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EVALUATION OF RHEOLOGICAL PROPERTIES OF ASPHALT BINDERS FOR PAVEMENT DESIGN APPLICATIONS

A DISSERTATION APPROVED FOR THE
SCHOOL OF CIVIL ENGINEERING AND ENVIRONMENTAL SCIENCE

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ABSTRACT

More that 90% of total paved roadways in the U.S. are asphalt concrete (AC) pavements. The annual expenditures for construction and maintenance of these pavements exceed $10 billion. To reduce these maintenance costs, it is crucial to design and construct pavements that perform well during the design life. Even though significant advances have been made in the analysis and design of hot mix asphalt (HMA) pavements during the last two decades, pavement communities are still challenged with evaluating the performance of HMA materials, in which the asphalt binder plays a significant role. Such challenges are getting augmented by the introduction of new paving technologies and construction materials such as warm mix asphalt (WMA) and asphalt recycling.

To attain a good and reliable design and performance evaluation of AC pavements, the Superpave® mix design procedures as well as the newly released mechanistic-empirical pavement design guide (MEPDG) analyses require estimations of rheological properties of asphalt binders. Basic rheological parameters of a performance grade (PG) binder are dynamic shear modulus (G*) and phase angle (δ) at warm temperatures, and flexural creep stiffness (S) and rate of stress relaxation (m-value) at cold temperatures. Furthermore, viscosity data are often needed to estimate the flow and performance properties of the mix (i.e., dynamic modulus, E*). The main objectives of this study were to evaluate local binders with and without different modifiers in the laboratory and predict the performance of the mixes.

To fulfill the objectives of this study, the Superpave® binder specification tests were conducted by using a rotational viscometer (RV), a dynamic shear rheometer

(DSR), and a bending beam rheometer (BBR). The short-term and long-term aging of asphalt binders were simulated in the laboratory by using a rotational thin film oven (RTFO) and a pressure aging vessel, respectively. Non-specification binder tests related to shear-hardening/thinning behavior, and strain and frequency dependencies were conducted by using a dynamic mechanical analyzer (DMA). To ensure the repeatability of test results, at least three replicate samples were tested at each testing temperature or testing condition. The DMA used in this study was found to be an effective and useful alternative device to the DSR, especially in evaluating asphalt binders at very low temperatures (12.7^{o}C or below). Because of the versatility of the DMA, it was found to be a valuable device for non-specification testing of asphalt binders.

One of the common distresses of hot mix asphalt (HMA) pavements is moisture-induced damage (i.e., stripping). To mitigate stripping in HMA pavements, liquid anti-stripping (AS) agents can be used. The current study evaluated the effects of different dosages of two commonly used liquid AS agents namely, AD-here® HP (0.25%, 0.50%, and 0.75%) Plus and Perma-Tac® Plus (0.50%, 0.75%, and 1.0%) on the rheological properties of a selected base (PG 64-22) binder. The maximum allowable dosage of either of these AS agents was found to be 0.5% (by the weight of the binder). The liquid AS agents were also found to reduce the rutting resistance of the base binder, but they did not adversely affect the PG grade of the binder.

Nationwide, approximately 4,000 asphalt plants produce over 500 million tons of asphalt paving materials annually; about 90 percent of these materials are HMA. However, the production of WMA has increased significantly in recent years due to its

beneficial effects in terms of energy saving and environmental stewardship. On the detrimental side, the low production temperature in WMA may lead to increased moisture induced damage. The current study evaluates the effects of different dosages two selected WMA additives, namely Advera® (4%, 6%, and 8%, by the weight of the binder) and Sasobit® (1%, 2%, and 3%) on the viscosity and temperature susceptibility of the modified binder both at high and low service temperatures.

Advera®, a water-bearing additive, was not found to be effective in reducing the mixing and compaction temperatures of the binder. Advera® was found to increase the high temperature stiffness and decrease the low temperature stiffness of the base binder. The optimum dosage of Advera® was found to be 6%. This dosage level of Advera® was not expected to change the PG grade of the base binder. However, the reduced RTFO-aging (at 135°C) of 6% Advera®-modified binder failed to meet the high PG temperature of the base binder. Thus, the poorer rut resistance of Advera® mixes is suspected to be due to the reduced production temperature rather than Advera® itself.

At production temperatures, Sasobit® was found to reduce the viscosity of the binder, and 3% Sasobit® was expected to reduce the mixing and compaction temperatures by 16°C and 19°C, respectively. Also, Sasobit® was found to increase the stiffness of the binder at both high and low service temperatures. Three percent Sasobit® was expected to alter the PG 64-22 binder to a PG 70-16 binder, which is expected to beneficial the rutting resistance but detrimental to the low temperature cracking. An alternative solution could be to use a softer binder (PG 58-28), which is expected to maintain the design grade of the Sasobit® binder as PG 64-22. The

optimum dosage of Sasobit® was found to be 1.5%. As expected, the reduced RTFO aging led to reduced oxidative age hardening of the Sasobit®-modified binder. The linear viscoelastic (LVE) limit was found to decrease with an increase in the dosage level of Sasobit®. A small amount (0.5%) of AD-here® HP Plus did not show any adverse impacts on the performance factors of the WMA additive--modified binder. Rather, it was expected to improve the fatigue fracture and low temperature resistance of the WMA additive-modified binders.

In regard to asphalt recycling, about 60 million tons of recycled asphalt pavement (RAP) materials were reused or recycled directly in the construction of new pavements in 2010. The usage of RAP was expected to be doubled by 2014 because of its beneficial effects in conserving raw materials and in realizing the goals of ongoing "green technology" initiatives. To this end, binders recovered from three RAP samples were evaluated. As expected, the recovered binders were found to be significantly stiffer than their virgin counterparts. Also, the prolonged use of the centrifuge and heat in the Abson recovery method was suspected to harden the binder. It was believed that the extra age-hardening, during the recovery process, caused the high PG and low PG temperatures of the recovered binder to increase up to 4°C and 3°C, respectively.

The current study also developed an inventory of the MEPDG input parameters for local binders by evaluating three PG binders (PG 64-22, PG 70-28, and PG 76-28) collected from three different refineries. Test results indicate that these binders met their PG grades. However, the viscosity and stiffness of these binders were found to vary significantly from one source to another.

While using the MEDPG-adopted Witczak model, it was observed that the DSR test data of the asphalt binder significantly underestimated the E* values of mixes. On the other hand, the RV test data somewhat overestimated the E* values. The other commonly used Hirsch model, based on the G* data of the asphalt binder from frequency sweep tests, was found to be a better-fit than the Witczak model. The estimated E* values of the WMA (Sasobit®)-modified mix was found to be significantly higher than the control mix. The liquid AS agent did show significant effects in the E* values of the WMA mix.

Major pavement distress factors of a typical pavement section in Oklahoma were estimated by using the MEPDG software. It was predicted that about 40% of the total rutting occurred in AC layers, of which 80% would occur in the surface course. It was also predicted that about 39% of rutting would occur within two years of construction. The AC layers' rut depths were found to vary significantly with the change of PG grade of the binder. It was observed that the AC layers' rutting was somewhat sensitive to the binder source. Fatigue fracture, thermal cracking, and international roughness index (IRI) were not found to be critical distresses in this study. Also, binder type and source did not seem to have significant influence on these distresses.

In conclusion, the current study evaluated rheological properties of asphalt binders modified with AS and WMA additives. It also evaluated binders recovered from RAP materials. Furthermore, an inventory of the MEDPG input parameters for local binders was developed. Finally, the evaluated rheological data were used to predict mixes' E* values, and estimate the sensitiveness of pavement distresses to

binder type and source. The findings of the current study are expected to provide pavement professionals a better understanding of the rheological evaluation of unmodified and modified binders for pavement design applications.

1 INTRODUCTION

1.1 BACKGROUND AND RESEARCH NEED

More than 90 percent of the 2.6 million miles of paved highways in the United Sates are asphalt concrete (AC) pavements. The annual expenditures for construction and maintenance of these roads exceed $10 billion (NECEPT, 2010). Nationwide, approximately 4,000 asphalt plants produce over 500 million tons of asphalt paving materials annually; about 90 percent of these materials are hot mix asphalt (HMA) (NAPA, 2009). It includes about 60 million tons of reclaimed materials, which are reused or recycled directly in the construction of new pavements. Significant gains can be achieved in addressing global issues such as climate change by accelerating research and deployment of new technologies that conserve energy and reduce emissions (NAPA, 2009). Among others, these technologies include warm mix asphalt (WMA) and recycled asphalt pavement (RAP). Furthermore, substantial benefits can be achieved by designing longer lasting AC pavements, which are less susceptible to common distresses such as rutting, fatigue fracture and thermal cracking.

AC pavement distresses can be predicted by using the rheological characteristics of the asphalt binder (Roberts et al., 1996). Basic rheological parameters of a performance grade (PG) binder are dynamic shear modulus (G*) and phase angle (δ) at warm temperatures, and flexural creep stiffness (S) and rate of stress relaxation (m-value) at cold temperatures. These parameters are also used to characterize recovered binders from RAP materials. Furthermore, G* and δ values of the asphalt binder at a desired loading frequency over a range of temperatures are used

as inputs in the new mechanistic-empirical pavement design guide (MEPDG) to predict the performance of the asphalt mix (NCHRP, 2004a).

Rheological parameters of asphalt binders at their high and intermediate temperatures are characterized as per AASHTO T 135 by using a dynamic shear rheometer (DSR) (AASHTO, 2008). However, the DSR has some inherent limitations with respect to test conditions (strain, frequency and temperature) and repeatability (AI, 2002; Carswell, 2001; Johnson et al., 2007). For example, the DSR estimates the PG grade of the asphalt binder based on a loading frequency of 1.59 Hz (10 rad/sec) rather than capturing its behavior for a larger frequency range. Furthermore, the use of the DSR to evaluate high grade (polymer-modified) asphalt binders at relatively low temperatures (i.e., 4.4^{o}C) while generating the MEPDG input data may be problematic or practically unsound. Some of these issues can be addressed by performing similar tests using a dynamic mechanical analyzer (DMA). Also, the DMA can be used an alternative tool to validate test results obtained from the DSR. However, no protocols are available for testing asphalt binders using the DMA. The current study aims at establishing such test protocols and using the DMA in selective cases when the DSR is not practical. For all other specification tests of asphalt binders, the DSR will be used in this study.

Another important concern arises from an ongoing practice where asphalt binders are often modified to enhance their performance by mixing additives. To enhance the performance of AC pavements against stripping (moisture-induced damage), amine-based liquid anti-stripping (AS) agents such as AD-here® HP Plus and Perma-Tac® Plus are often used in HMA mixes (Arr-Maz Chemicals, 2002; Akzo-

Nobel, 2008; Selvaratnam et al., 2007). These AS agents are expected reduce premature failures of bonds between asphalt binder and aggregates (Anderson, 1982; Malsch, 1986; Hurely and Prowell, 2005a-b, 2006). However, such modification can show detrimental effects on the other performance factors of the pavement, especially on the rut resistance.

In recent years, there has been an increasing trend in the use WMA technologies, in which asphalt binders are modified to reduce production temperatures of AC mixes. Since the first public demonstration in the United States in 2004, additive-based (e.g., wax, zeolite, chemical) WMA pavements have been constructed in all but ten states including Oklahoma. It is expected that WMA pavements will represent the majority of all the AC pavements produced nationwide by 2014 (NAPA, 2009). It should be noted that a few contractors in Oklahoma use the water-based WMA technology (e.g., Astec Double Barrel Green, Terex Warm Mix Asphalt) on limited basis (Gierhart, 2009). In this technology, water is injected in the mixing plant, which creates foaming effects and provides a better coating of aggregates. However, this technology requires significant adjustments of the asphalt plant. As of 2007, 10% of HMA plants in Oklahoma were equipped with the aforementioned WMA technology. The national asphalt pavement association (NAPA) reported that 40% (12 out of 30) of AC producers in Oklahoma are capable of producing some kind of WMA mixes (Gierhart, 2011). However, only about 2% of all Oklahoma Department of Transportation (ODOT) AC mix designs, associated with a project in the last 15 months, are WMA (Gierhart, 2011). Minimal cost savings and lack of specifications are believed to be the major setbacks for Oklahoma contractors for not adopting WMA

technologies to a greater extent (Gierhart, 2009). Generating laboratory data of WMA technologies are expected to be helpful for ODOT to formulate such specifications.

A recent collaborative study, conducted by researchers at the University of Oklahoma (OU), Oklahoma State University (OSU) and Langston University (LU), evaluated two selective WMA additives (Sasobit® and Aspha-Min®), and reported them as viable WMA technologies for Oklahoma mixes (Hossain et al., 2009). Rutting and fatigue properties, and surface free energy characteristics of these additives modified binders have been reported in the pertinent literature (Sneed, 2007; Wasiuddin et al., 2007; Buddhala, 2009). The current study is a natural extension of these studies, and it encompasses rheological properties of the modified binders at high, intermediate and low critical temperatures. Furthermore, the current study evaluates the effectiveness of a liquid AS in the WMA additives-modified binders.

One of the most important attributes of the WMA technology is to lower the viscosity of asphalt mixes at lower production temperatures without compromising their workability and performance (Hurley and Prowell, 2005a; Goh et al., 2007; Kantipong et al., 2007). The WMA technology significantly conserves production fuel energy, reduces emissions, and increases construction benefits including a longer paving season in cool climates (FHWA, 2010). However, the low production temperature in the WMA is blamed to exhibit increased moisture induced damage. This moisture induced damage can be reduced by using a liquid AS agent, which was reported to be effective in HMA mixes (Gore, 2003).

The liquid AS agents and WMA additives can be added with a binder at different stages: at refineries, at distribution centers, or at mixing plants as a batch or

continuous process (Akzo Nobel, 2008; Austerman et al., 2009). The preferred method to add these additives to an asphalt binder is to introduce it at the plant. It is a common practice to test binders for their performance grades prior to the addition of these additives (Gore, 2003; Bennert et al., 2010). Thus, rheological characteristics of a binder mixed with an additive remain unknown in most cases. The amount of theses additives is also expected to have influence in the changes to the rheological characteristics of the asphalt binder, and the current study seeks to evaluate such influences.

Furthermore, asphalt recycling technology has become an important topic among transportation professionals in recent years because of its enhanced use in the construction of new AC pavements. The increasing demand of RAP usage is mainly due to the increasing cost of asphalt binders and scarcity of high quality virgin aggregates, as well as increases in environmental awareness. Nationally, the use of RAP in new pavements is expected to be doubled by 2014 (NAPA, 2009). Presently, the ODOT field divisions and contractors use 15-20% RAP for shoulders, turnouts, and base courses, while none for surface courses (O'Rear et al., 2008; ODOT, 2008; ODOT, 2009b). Comparatively, a number of neighboring states including Arkansas, Louisiana and Texas allow 30% or more RAP in base courses and 10% or more in surface courses (Jones, 2008). In the asphalt recycling process, processed RAPs are blended with virgin materials and new mixes are prepared. So, the rheological characterization (i.e., evaluation viscosity and estimation of high, intermediate and low critical temperatures) of the recovered binders from the RAP along with the virgin asphalt binder is needed to attain proper blending charts (Kandhal and Foo, 1997;

McDaniel and Anderson, 2001; NCHRP, 2001; NCHRP, 2004a). One of the major reasons for using a lower percentage of RAP in Oklahoma is the lack of available rheological data of recovered binders from local RAPs (ODOT, 2009a; TRB, 2009a-2009b). Additionally, the recovery process of the binder may have some influence on the PG grading of the recovered binder. The current study aims at evaluating such influences, if any.

Departments of Transportation (DOT) in many states in the United States have already prepared or are in process of creating rheological databases for local calibrations of the new MEPDG (Clyne and Marasteanu, 2004; Flintsch et al., 2007). ODOT is also actively working towards implementing the new MEPDG for analyzing and designing flexible pavements for local conditions (ODOT, 2009a; Cross et al., 2009). As mentioned earlier, properties of asphalt binders are important input parameters in all reliability levels of design and analysis of the MEDPG. For *Level 3* analysis and design asphalt binders PG grades are used as input. On the other hand, rheological test data of asphalt binders are used for both *Level 2* and *Level 1* analyses and designs. For PG binders, these parameters consist of the G* and δ values over a range of temperatures (from 4.4^{o}C to 54.4^{o}C) at a loading frequency of 1.59 Hz or rotational viscosity data obtained from a Brookfield viscometer. The NCHRP Report 1-37A also recommends that these measurements be made after rotational thin film oven (RTFO)-aging of the asphalt binder to simulate the short-term plant aging condition (NCHRP, 2004b). However, such rheological data for local PG binders from different sources are not available to pavement professionals in Oklahoma (Cross et

al., 2007; ODOT, 2009a). This study aims to generate such rheological data and perform relative comparisons among binder types and sources.

Combined with binders' rheological data and volumetric properties of asphalt mixes, the MEPDG estimates the master curves of dynamic modulus (E*) of asphalt mixes. Previous studies (e.g., King et al., 2005; Shah et al., 2005) reported that the asphalt binder's source and PG grade, among others, had significant effects on E* value of a mix, which is highly correlated with pavement distresses such as rutting. Therefore, it would be worthwhile to evaluate the sensitiveness of asphalt binder's input parameters (PG grade, viscosity, and G*and δ values) on common distress functions (rutting, fatigue, and thermal cracking) of HMA pavements using the MEPDG.

1.2 OBJECTIVE OF THE RESEARCH

The major goals of this study are to evaluate the effects of AS agents and WMA additives on rheological properties of PG binders and characterize recovered binders from RAP. The specific objectives of this study are listed below:

(1) Develop dynamic shear test protocols for the DMA to evaluate asphalt binders.

(2) Characterize rheological properties (viscosity, stiffness and PG grade) of a commonly used PG binder (PG 64-22) modified with different dosages of liquid AS agents, namely, AD-here® HP Plus and Perma-Tac® Plus.

(3) Assess rheological properties (i.e., viscosity, stiffness) of the selected PG binder modified with two commonly used WMA additives, namely, Sasobit®, and Advera®.

(4) Evaluate the effects of a selected liquid AS agent (AD-here® HP Plus) on the rheological properties of the WMA additive-modified binders.

(5) Evaluate viscosity and PG grades of asphalt binders recovered from recycled asphalt pavements. Also, evaluate the effects of the commonly used recovery technique, the Abson method, on the viscosity and PG grading of the recovered binder.

(6) Develop an inventory of MEPDG input parameters for asphalt binders collected from different refineries in Oklahoma.

(7) Perform sensitivity analyses of the MEPDG input parameters of asphalt binder on distress factors (rut, fatigue fracture, and thermal cracking) of a typical HMA pavement section.

1.3 ORGANIZATION OF THE DISSERTATION

This dissertation is focused on evaluating the effects of various additives on the rheological properties of asphalt binders. Following the introduction presented in Chapter 1, the findings of this study are presented in this dissertation in the format of six journal publications (two published, three under review and one prepared for submission). Each chapter, from Chapters 2 to 7, contains a manuscript of one technical paper. Chapter 8 presents the overall summary of this dissertation and recommendations for future research.

Chapter 2 entitled "*Effects of Liquid Anti-Stripping Additives on Rheological Properties of Performance Grade Binders"* presents the effects of liquid AS agents on the rheological properties of asphalt binders for rutting and fatigue potentials. One commonly used performance grade asphalt binder, PG 64-22, and two amine-based

liquid anti-strip agents, namely, AD-here® HP Plus (ASA1) and Perma-Tac® Plus (ASA2), were evaluated at varying dosages. The amounts of ASA1 was 0.25%, 0.50%, and 0.75% (by the weight of the binder), and those of ASA2 was 0.50%, 0.75%, and 1.00% (by the weight of the binder). Asphalt binder specimens were prepared from unaged and RTFO-aged samples, and tested by following the Superpave® specifications. This chapter also presents the validation of results of newly developed binder testing protocols using the DMA that was used to conduct sweep tests (time, temperature, strain, and frequency). Furthermore, it presents developed correlations between the dynamic shear moduli of the unmodified and AS-modified binders with the dynamic moduli of corresponding mixes.

Chapter 3 entitled "*Effectiveness of Water-bearing and Anti-stripping Additives in Warm Mix Asphalt Technology*" evaluates the effects of varying dosages of a water-bearing WMA additive, Advera®, on the PG 64-22 binder. The effectiveness of 0.5% AD-here® HP Plus (ASA1) on the Advera®-modified binder was also studied. Furthermore, the effect of reduced oxidative aging on the Advera®-modified binder was investigated.

Chapter 4 entitled "*Effectiveness of Warm Mix and Liquid Anti-Stripping Additives on Performance Grade Binders*" examines the effectiveness of another WMA additive, Sasobit®, and the liquid AS agent, AD-here® HP Plus, on the PG 64-22 binder. A small amount (0.5%) of AD-here® HP Plus was maintained in the selected binder modified with the optimum dosage (1.5%) of Sasobit®. Effects on the mixing temperature and critical PG temperatures of the modified binders were evaluated following the Superpave® test methods.

Chapter 5 entitled "*Viscoelastic Characteristics of Warm Mix Additive Modified Binders and Prediction of Dynamic Modulus of Mixes"* illustrates the evaluation of viscoelastic properties of Sasobit®-modified binders at high service temperature and prediction of dynamic modulus (E*) value WMA mixes. Viscoelastic properties of the WMA-additive modified binders included their LVE limits, temperature susceptibilities and load frequency dependencies. Also, the effects of reduced RTFO-aging on the stiffness of a selected dosage of Sasobit®-modified were investigated. Furthermore, viscoelastic data of the unmodified and WMA-additive modified asphalt binders were used to estimate E* values of corresponding mixes through the deployment of Witczak and Hirsch models. The estimated E* values were then used to determine E* master curves by using time-temperature superposition principles (TTS). Finally, this chapter presents the effects of 0.5% AD-here® HP Plus on E* values of the WMA mix.

Chapter 6 entitled "*Influence of Recovery Processes on Properties of Binders and Aggregates Recovered from Recycled Asphalt Pavement*" presents the evaluation of recovered binders from field and simulated RAPs. Superpave® test results of virgin and recovered binders from one field RAP of known source and mix design, and two simulated RAPs (one with PG 76-28 binder and the other with a PG 64-22 binder) are discussed in this paper. It also presents possible influence of the commonly used "Abson" method on the PG grading of the recovered binder.

Chapter 7 entitled "*Sensitivity Analysis of Asphalt Pavements Using Performance Grade Binders for Oklahoma Conditions"* includes a parametric study of performance factors of typical flexible pavement sections using the new MEPDG.

Following different hierarchical design and analysis levels of the MEPDG, it evaluates the sensitivity of different input parameters on rutting and fatigue performance.

Chapter 8 summarizes the important conclusions from this study. It also presents a few recommendations for future studies.

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2 EFFECTS OF LIQUID ANTI-STRIPPING ADDITIVES ON RHEOLOGICAL PROPERTIES OF PERFORMANCE GRADE BINDERS[1]

2.1 ABSTRACT

This study presents a testing protocol for evaluating the viscoelastic properties of selected performance grade (PG) binders using a Dynamic Mechanical Analyzer (DMA). It also presents the effects of amine-based liquid anti-stripping additives on the binders' rheological properties. Out of total 183 samples tested, 51 samples for three PG binders (PG 64-22, PG 70-28, and PG 76-28) were tested to establish the DMA-based testing protocol. The remaining samples were tested to obtain rheological data of the PG 64-22 binder with different dosages of two anti-stripping (AS) additives. Test results of the DMA were validated by comparing with those obtained from a Dynamic Shear Rheometer (DSR). Test results show that the DMA can be used as an alternative tool for examining the viscoelastic behavior of binders. It was observed that the rutting factor ($G^*/\sin\delta$) of the binder decreased when the amount of AS additive was increased. The optimum dosage of either of these AS additives was found to be 0.50%. AS additives did not alter the mechanical workability and the linear viscoelastic limit of the binder. Also, a good correlation between the complex modulus of the PG 64-22 binder and the dynamic modulus of the corresponding mix was observed.

Keywords: DMA, complex modulus, anti-stripping additive, viscoelastic, sweep test

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2.2 INTRODUCTION

Major asphalt concrete (AC) pavement distresses such as rutting, cracking, and stripping can be assessed by using the rheological characteristics of asphalt binders (Soleymani et al., 2004). For example, stripping in AC pavements occurs when the bond between asphalt binder and aggregate is broken in presence of moisture. To address premature failures of bonds, amine-based liquid anti-stripping (AS) additives are often used in hot mix asphalt (HMA) pavements (Selvaratnam et al., 2007; ODOT, 2008).

Performance Grade (PG) binders are generally tested at high and intermediate temperatures using a Dynamic Shear Rheometer (DSR) as per AASHTO T 135 (AASHTO, 2008). Although DSRs are widely used in the PG grading of asphalt binders, it has some inherent limitations with respect to test conditions and reproducibility (AI, 2002; Carswell, 2001). For example, the DSR selects the PG grade of an asphalt binder based on a loading frequency of 1.59 Hz rather than capturing its behavior for a larger frequency range. The repeatability and the reproducibility issues can be challenging for polymer-modified binders. These issues can be resolved by following the ASTM D3244 guidelines to some extent (AI, 2002). A dynamic mechanical analyzer (DMA) can be a viable device to validate rheological characteristics obtained from a DSR. However, no guidelines or specifications are available for rheological characterizations of PG binders using a DMA.

As noted earlier, AS additives are added with binders to reduce premature bond failure in asphalt pavements. It is reported that out of 82% agencies that treat their asphalt mixes for premature bond failure, 56% use liquid amines, 15% use either

liquid amines or lime, and 29% use lime (Cheng et al., 2002). Several liquid AS additives are certified by Oklahoma Department of Transportation (ODOT) (ODOT, 2008). These AS additives can be added with binders at different stages: at refineries, at distribution centers, or at HMA plants as a batch or continuous process (Akzo Nobel, 2008). The preferred method for adding these additives to an asphalt binder is to introduce it at the HMA plant. On the other hand, it is a common practice to test binders for their performance grades prior to the addition of AS additives (Gore, 2003). Consequently, the rheological characteristics of a binder mixed with an AS additive remain unknown and the premature rut prediction in AC pavements may be underestimated in most cases. Moreover, the amount of AS additive can also change the rheological characteristics of a binder. Also, establishing a correlation between the complex modulus (G*) of AS additive-modified binder and the dynamic modulus (E*) of the corresponding mix would be useful for a better pavement design as currently such correlation does not exist.

The primary objective of this study is to generate rheological data of ODOT certified binders by using a DMA and to examine the influence of different dosages of AS additives. The optimum dosage of AS additive is then determined. This study also examines the mechanical workability, linear viscoelastic (LVE) limit, and temperature dependency of a binder mixed with different dosages of AS additives. Finally, it correlates the complex modulus (G*) value of AS additive-modified binder with the dynamic modulus (E*) value of a corresponding mix.

2.3 OVERVIEW OF PREVIOUS STUDIES

To examine the viscoelastic behavior of an asphalt binder, it is necessary to understand its stress-strain response under different environmental and loading conditions (Airey et al., 2002; Bahia et al., 2001). It is desirable that the rheological properties of an asphalt binder be time-independent, which means that the rheological properties should remain unchanged if the asphalt binder is not subjected to any loading or environmental changes (Iqbal et al., 2006). The Superpave® specifications did not consider the effect possible thixotropic behavior (a decrease in viscosity over time at a constant shear rate) of a binder on its G* and phase angle (δ) values (Bahia et al., 2001). The thixotropic network structure of binder can be destroyed or altered by repeated shearing due to addition of certain additives (Bahia and Perdomo, 1997). Furthermore, most of the modified asphalt binders exhibit a phenomenon known as pseudoplasticity, in which the binder displays decreasing viscosity with an increasing shear rate (Yildirim et al., 2000). The Superpave® specifications did not consider the pseudoplasticity (shear thinning) of modified binders.

Clyne and Marasteanu (2004) conducted strain controlled time sweep tests on long-term aged samples at intermediate temperatures to assess the fatigue behavior of nine PG binders certified in Minnesota. These researchers performed these tests with an oscillating stress of 500 kPa applied at 1.59 Hz by using a conventional DSR. Theses tests lasted from 15 minutes to 2.5 hours. Test data and model parameters were then populated in tables of a rheological database. Loh et al. (2000) examined the mechanical workability of two neat binders (AC 10 and AC 20) by performing time sweep tests at their high critical temperatures (Loh et al., 2000). It was reported that

significant reductions in the G* value were observed when the strain level decreased from 10% to 1%. However, none of these studies considered the influence of AS additives on the rheological properties of the binder.

As noted by several researchers (e.g., Soleymani et al., 2004; Airey et al., 2002; Zhai et al., 2006), it is important to perform specification-related dynamic testing of an asphalt binder within its LVE limit. The LVE limit of an asphalt binder is defined as the range of strain where the G* value is at least 95% of the zero strain modulus (Soleymani et al., 2004). Zhai et al. (2006) conducted strain sweep tests on selected emulsified asphalt binders and reported that some had limited LVE regions (as low as 1% strain). Clyne and Marasteanu (2004) also performed strain sweep tests on nine PG binders certified in Minnesota to obtain their LVE limits. These researchers observed that heavily polymer-modified binders showed sharper reduction in modulus with increasing strain. The sharp reduction of complex modulus with increase in strain indicates that under increased strain in the pavement, the materials may rut faster than binders that do not lose stiffness as quickly (Clyne and Marasteanu, 2004). These researchers did not consider AS additives in their respective studies.

Temperature sweep tests on binders can be used to approximate the temperature at which a binder will satisfy the Superpave® reflecting rutting resistance criteria. Selvaratnam et al. (2007) and Gore (2003) observed possible grade changes of selected PG binders due to the addition of two selected AS additives (AD-here® HP Plus and Perma-Tac® Plus). Henceforth, AD-here® HP Plus and Perma-Tac® Plus refer to ASA1 and ASA2, respectively. These researchers used DSRs to test binder

samples in accordance with AASHTO T-315. Selvaratnam et al. (2007) reported that the addition of ASA1 up to 0.75% and ASA2 up to 1.0% met the Superpave® criteria. Gore (2003) also reported that there was a slight change in the δ value (not more than 0.5^{o}) due to the addition of ASA1 and ASA2. However, these researchers did not evaluate mechanical workability, LVE limits, and frequency dependency of AS additive modified binders.

The stiffness of a HMA mix decreases as the loading time increases or the loading frequency decreases. The dynamic modulus (E*) value of a HMA mix can reduce as much as ten-fold when the loading frequency is reduced from 10 Hz to 0.01 Hz. The corresponding G* value of the binder exhibits a similar frequency dependency (Walker and Buncher, 1999). Accordingly, the traffic speed on newly constructed asphalt pavements can significantly influence its rutting potential. A pavement section experiencing slower traffic at early stage is expected to experience higher rutting damage. Frequency sweep tests can be conducted on asphalt binder to simulate this condition.

DMAs have been used by polymer and food processing industries for the last several years (TA Instruments, 2006). Lately, some researchers have used a DMA to examine the fatigue and healing characteristics of asphalt mastic and specially designed HMA mixtures (Kim et al., 2002). Hossain and Zaman (2008) analyzed a neat PG 64-22 binder using a DMA. That study, however, did not include any AS additive.

2.4 MATERIALS, EQUIPMENT AND TEST METHODOLOGY

2.4.1 Materials

An unmodified PG 64-22 binder and two styrene-butadien-styrene (SBS) modified (PG 70-28 and PG 76-28) binders, all collected from a local refinery in Oklahoma, were used to establish the DMA testing protocol. The PG 64-22 binder was then mixed with different dosages of ASA1 and ASA2, and its mechanical workability and rheological characteristics were examined by conducting time, temperature, strain, and frequency sweeps tests.

The atomic composition of the selected PG 64-22 binder has been reported to have 92% carbon, 6.7% hydrogen, 0.63% nitrogen, and 0.67% sulfur (Hossain et al., 2009). The viscosity of the PG 64-22 binder was found to be 134 mPa-s at ODOT's recommended mixing temperature (163°C). Selected amine based AS additives are organic compounds with a functional group containing a nitrogen (N) atom with a lone pair (valence electron) and at least one hydrogen (H) atom replaced with an alkyl or aryl group (hydrocarbons) (Figures 2.1a though 2.1c). These AS additives are surfactants with a lyophobic amine group which are highly surface active (Hossain et al., 2009). Surfactant molecules diffuse through the binder so that the "head" groups can adsorb onto the aggregate surface while the lyophilic hydrocarbon chain ("tail" group) still remains in the asphalt binder (Figure 2.1d). Thus, an AS additive acts as a bridge between the asphalt binder and the aggregate surface which resist the action of water. Depending on the asphalt grade and aggregate type, ASA1 is added to asphalt in the amount of 0.2 - 0.8% by the weight of the binder (Arr-Maz Chemicals, 2009). The recommended dosage of ASA2 is 0.5 to 1.0% (Akzo Nobel, 2008). Both of these

AS additives are commercial products. At 22°C, pH values of ASA1 and ASA2 were found to be 13 and 12.2, respectively. Some additional chemical and mechanical properties of these AS additives are presented in Table 2.1.

2.4.2 Equipment

The DSR used in this study is designed to permit testing of asphalt according to the AASHTO standards in both stress and strain controlled modes. Besides verifying the grading (pass or fail) of a binder sample at a pre-defined temperature, this DSR is also capable of determining PG grading, linearity, and temperature calibration of an asphalt binder. Once parameters for these tests are set, templates are created. On the other hand, the DMA is designed to perform sweep tests (time, strain, temperature, and frequency) of an asphalt binder. In addition to the testing parameters for a particular sweep test, the measuring head geometry and dimension, and material density are entered as inputs. Reusable oscillation procedure files containing the test specifications are then created.

A DMA can determine many rheological properties of an asphalt binder, including: storage modulus, viscous modulus (or loss modulus), complex modulus, damping, creep, stress relaxation, glass transition, and softening point (Kim et al., 2002). Tests relevant to these properties can be performed as a function of temperature, frequency or time in a constant (or step fashion), or under a fixed rate. It has also been reported that the DMA is the most sensitive of all thermal analytical techniques (Hossain et al., 2009). However, many of these features are out of scope of this paper.

The DMA used in this study is a fifth-generation commercial rheometer (TA Instruments, 2006). Figure 2.2a shows the DMA assembly, including an environmental testing chamber (ETC) capable of maintaining a temperature ranging from -150°C to 400°C (TA Instruments, 2006). The maximum heating/cooling rate in the ETC is 24°C/minute, and the accuracy of the temperature according to the manufacturer is ±0.1^{o}C. To attain rapid cooling and to maintain an equilibrium temperature for a binder sample, the ETC is connected to a liquid nitrogen supply which does not react with the asphalt binder being tested. Both unaged and rotational thin film oven (RTFO) aged binder samples were tested in accordance with AASHTO T 315 (AASHTO, 2008). In this test method, asphalt binder samples are sandwiched between two identical parallel plates. The bottom plate is fixed, and the top plate oscillates at a predefined stress or strain. While testing binder samples at high temperatures, 25 mm diameter plates are used. On the other hand, 8 mm diameter plates are used while testing binder samples at intermediate temperatures. During a test, the AASHTO T 315 specifications were maintained by inputting the specifications in the software of the DMA. The thermal equilibrium for a binder sample in the ETC chamber of the DMA was determined as per AASHTO T 315. The sample was tested at a constant speed of 1.59 Hz for 30 minutes. The G* value versus the testing time was plotted in Figure 2.2b, and the thermal equilibrium was found to be three minutes. To be on a conservative side, a five-minute thermal equilibrium was maintained at all testing temperatures.

The accuracy of the DSR and DMA measurements were validated by conducting dynamic shear test (AASHTO T 315) at different temperatures (64°C,

67°C, 70°C, and 76°C) on a standard fluid of known viscosity manufactured by Cannon. The dynamic viscosity (η) of the standard fluid was then calculated by dividing the measured G* value with the applied frequency (10 rad/sec). In case a measured viscosity value were found to be outside the specified tolerance limit (±1.5%) of its true viscosity, calibrations of measuring heads, geometry inertia and temperature would be performed. If the calibration efforts do not to resolve the validation issue, service(s) from the manufactures' certified technicians would be necessary.

Additionally, a rotational viscometer (RV) was used to measure viscosity of the binder as per AASHTO T 316 test method. Short-term aging of the binder was simulated in a RTFO as per AASHTO T 240. To simulate long-term aging of the binder, a pressure aging vessel (PAV) was used and AASHTO R 28 was followed. A bending beam rheometer (BBR) was used to evaluate the low temperature resistance of the same binder with 0.5% ASA1, and AASHTO T 313 was followed.

2.4.3 Mixing Additives

Three selected percentages of each AS additive (0.25%, 0.50% and 0.75% for ASA1, and 0.50%, 0.75% and 1.00% for ASA2) were mixed with the PG 64-22 binder. While mixing an AS additive with the PG 64-22 binder, ODOT's test specifications "OHD L-36: Method of Test for Retained Strength of Bituminous Paving Mixtures," were followed (ODOT, 2008). Before mixing, the binder was heated for two hours at 145°C. After pouring the AS additive in the heated binder, it was manually stirred for 30 seconds. The mix (AS additive and binder) was then kept

in a pre-heated oven at 145°C for an hour, and the sample was stirred for 30 seconds at ten minutes interval. The binder was then kept overnight for further testing.

2.4.4 Test Methodology in the DMA

The test matrix for the current study is shown in Table 2.2. Out of total 183 binder samples, 51 samples were tested for establishing the DMA protocol. The remainders of samples were tested to characterize rheological properties of PG 64-22 binder with different dosages of AS additives.

2.4.4.1 Rutting factor

The heated binder was poured into silicon rubber molds to prepare test samples (Figure 2.3a). Samples were then allowed to cool down for an hour. A Microsoft® window-based software "AR Instrument Control" networked with the testing unit was used to monitor and control the DMA. Immediately after attaching the measuring system, a standard three-minute rotational mapping of the equipment was carried out to obtain baseline data for the correction of torque. The ETC was then closed and the chamber temperature was raised to the sample loading temperature, which was 6°C below the testing temperature. Once the sample loading temperature maintained its equilibrium condition for two minutes, the upper plate was lowered to the zero-gap position. The ETC was opened, the upper plate was raised, and a sample was placed onto the lower plate. After placing the sample on the lower plate, the ETC was closed and the upper plate was lowered to the testing gap of 1.05 mm. The ETC was then opened and a trimmer was used to trim the bulged portion of the sample, as shown in Figure 2.3b. Depending upon the sample loading temperature and binder grade,

multiple rounds of trimming were necessary to obtain desired test samples (Figures 2.3c and 2.3d).

Following the trimming, the ETC was closed, the upper head was lowered to the geometry gap of 1 mm, and a five-minute thermal equilibrium was maintained at the testing temperature. The binder specimen was then pre-conditioned for one minute by pre-shearing with a loading frequency of 1.59 Hz and a strain level of 12%. The purpose of the pre-shearing was to remove any historical load associated with sample preparation, storage, handling, and loading. Following the pre-shearing, a three-minute time sweep test was conducted with a strain level of 12%, and a frequency level of 1.59 Hz. Data was collected every nine seconds. The last ten datasets were used to evaluate the $G^*/\sin\delta$ value. The post-test temperature was set to 100^oC as an aid to clean the plates and prepare the equipment for the next test.

2.4.4.2 Sweep Tests

Time Sweep — In a time sweep test, a binder sample was loaded at 58^oC. The temperature was raised to the testing temperature of 64^oC, and the thermal equilibrium was maintained for five minutes. At 64^oC, a 30-second pre-shearing was performed at a strain level of 5% and a frequency level of 1.59 Hz. The test was conducted at a constant strain (lateral strain at the outer perimeter of the upper side of the sample) of 12% and a constant frequency of 1.59 Hz, over a period of 15 minutes.

Strain Sweep — In a strain sweep test, a frequency of 1.59 Hz was kept constant while the oscillation amplitude was increased in some progression. The sample was loaded at 58^oC. After maintaining the thermal equilibrium for the testing

temperature, the sample was pre-sheared for one minute at a strain level of 0.1% and a frequency level of 1.59 Hz. During the testing phase, the sample was subjected to strains ranging from 0 to 51%.

Temperature Sweep — In a temperature sweep test, the frequency and the oscillation amplitude were kept constant, while the temperature was increased in some progression. The effect of high temperatures on the PG 64-22 binder was examined for a range of temperatures from 58°C to 73°C with increments of 3°C. Samples were loaded and trimmed at 58°C, followed by a 30-second pre-shearing at a strain level 1% and a frequency level of 1.59 Hz. During the temperature sweep test, the thermal equilibrium was maintained at each data collection point. Samples were tested using a progression going from low temperatures to high temperatures.

Frequency Sweep — It is known that several factors including aggregate type and characteristics, compaction effort, binder type, and binder content contribute to the E* value of a mix. For simplicity, frequency sweep tests on RTFO-aged binder samples (0% and 0.5% ASA2) were tested to correlate G* values of binders with E* values of corresponding mixes. In a frequency sweep test, the loading frequency ranged from 25 Hz to 0.1 Hz. Samples were pre-conditioned at a frequency of 25 Hz, and a temperature of 4.4^{o}C. Samples were then tested at 4.4^{o}C, 21.1^{o}C, 37.8^{o}C, and 54.4^{o}C. At each testing temperature, a five-minute thermal equilibrium was maintained.

2.5 RESULTS AND DISCUSSIONS

2.5.1 Validation of DMA Protocol

Test results obtained from the DMA are compared with those from the DSR (Figure 2.4). It is seen that all three unaged asphalt binders satisfy the Superpave® reflecting rutting factor of 1.0 kPa at their high critical temperatures. The value of δ is close to 90° for the PG 64-22 binder, whereas it is as low as 51° for polymer modified PG 76-28 binder. From Figures 2.4a and 2.4b it is observed that the test results ($G^*/\sin\delta$ and δ values) for PG 64-22 and PG 70-28 binders obtained from the DMA match well with those from the DSR. However, the DMA gives significantly higher $G^*/\sin\delta$ value for the polymer-modified PG 76-28 binder.

Student's t-tests (paired two-sample) were performed to compare the test results obtained from the DMA and the DSR to see if the differences had any statistical significance. It showed that at 95% confidence level ($p = 0.05$) the $G^*/\sin\delta$ and δ values for PG 64-22 and PG 70-28 binders from the DMA and the DSR did not differ significantly. In the case of the PG 76-28 binder, however, the t-test results ($p = 0.05$) suggested that there were significant differences in the mean $G^*/\sin\delta$ and δ values obtained from theses two pieces of equipment. The $G^*/\sin\delta$ values for the PG 76-28 binder obtained from the DSR and the DMA are 1.49 kPa, and 2.15 kPa, respectively. Possible reasons for the differences in the G^* and δ values are discussed below.

Some difficulties were encountered in conducting the DSR tests for the PG 76-28 binder. Naturally, PG 76-28 is a much stiffer and more viscid binder than the other

two. During the initial trial on the DSR, the δ value for the PG 76-28 binder was found to be relatively higher (63.7^{o} from the DSR tests compared to 51.2^{o} from the DMA tests) than expected, which led to lower G*/sinδ values. This could be due to the fact the parallel plates might not have been in smooth contact with the sample due to the presence of minute air bubbles. As shown in Figure 2.5, taking additional measures, namely extra trimmings and raising the sample loading temperature reduced the δ value by 15%, thus providing more reliable results from the DSR.

Figure 2.6 shows a comparison of the G*/sinδ and δ values obtained from the DSR and the DMA for the PG 64-22 binder at temperatures ranging from 58°C to 73°C at 3°C interval. Each data point in the chart represents the average value of three tests, and the vertical line at each point represents the error bar. It is evident that δ values obtained by using the DMA fit very well with those obtained from the DSR throughout the range of testing temperatures. The G*/sinδ values obtained from the DMA also match well with those from the DSR at temperatures of 61°C and higher. Although the mean G*/sinδ value from the DMA at 58°C differs from that obtained from the DSR, the Student's t-test results show that the difference does not have any statistical significance at 95% confidence.

2.5.2 Effect of Anti-stripping Additives

Both ASA1 and ASA2 reduced the G*/sinδ values of the PG 64-22 binder (Figure 2.7a). The higher the dosage level of an AS additive, the lower the G*/sinδ value, which means the lower rutting resistance. Selvaratnam et al. (2007) and Gore (2003) observed similar behavior for some selected PG binders tested in their studies.

ASA1 seems to have a higher influence in the reduction of G*/sinδ values than ASA2. This observation is supported by the viscosity data of ASA1 and AS2 which is 225 cps for the former and 350 cps for the latter at 25°C. Binder samples with 0.75% ASA1 and 1.0% ASA2 failed the Superpave® reflecting rutting criterion. An addition of either AS additive makes the binder a more liquid-like material, which is observed in their corresponding δ values (Figure 2.7b). When an AS additive is added, the primary amines present in these additives (aka surface active agents) react with the carboxylic acids present in the binder and form corresponding salts that also act as AS additives. As mentioned earlier, these AS additives are more viscous than the neat binder. The decrease in the viscosity of the modified binder increases the diffusivity of the amine groups in the AS additive through the binder to the surface. As viscosity of the binder decreases, its elastic modulus (G′) also decreases but δ value increases. Consequently, the complex modulus (G*) of asphalt binder decreases, and so does the rutting factor (G*/sinδ).

It is also important to note that any amount up to 0.50% ASA1 or 0.75% ASA2 can be blended with this PG 64-22 binder without jeopardizing the Superpave® acceptance criterion. However, a 4% and 6% reduction in rutting resistance is expected in the cases of 0.25% and 0.50% ASA1. On the other hand, about 1% and 6% reduction in rutting resistance is predicted in the cases of 0.5% and 0.75% ASA2. The reductions in rutting resistance in case of 0.75% ASA1 and 1.0% ASA2-modified binders are 22% and 9%, respectively.

2.5.3 Sweep Test Results

Mechanical workability: The mechanical working effects for the PG 64-22 binder with different dosages of AS additives were evaluated by performing 15-min time sweep tests at the high critical temperature. As expected, δ values for these binders remained constant throughout the testing period. However, a slight increase in G* values, representing marginal rheopectic or anti-thixotropic behavior, was observed. This behavior might be due to the fact that the hydrophilic suspended particles in asphalt binder form a lattice structure throughout the asphalt binder which causes an increase in viscosity and thus, hardening. Moffat (2003) also reported similar behavior for the Canadian Tar Sand bitumen in viscosity measurements.

It is expected that keeping the binder at elevated temperature for an extended period of time may affect the binder stiffness. At the same time, the storage time of the AS additive may influence the binder stiffness. To evaluate the effect of storage time (two years in an air-conditioned room at 22°C) of ASA1, PG 64-22 binder samples were tested at 64°C. The G*/sinδ value of the binder with 0.5% ASA1 was found to be 1.01 kPa, which was only 0.03 kPa lower than its fresh counterpart. Therefore, it is expected that the functionally of ASA1 will remain the same if it is stored for such short-period of time.

Linear Viscoelastic Limits: Figure 2.8 presents the effect of strain on the G* values for the PG 64-22 binder with different amounts of ASA1 and ASA2. It is evident that the G* value remained constant for all binder types up to a strain level of 51%. For this PG 64-22 binder, the LVE behavior was exhibited up to a strain level of 51% as 95% of $G^*_{\varepsilon=0}$ (G* corresponding to zero strain) was not achieved at that point.

This behavior is observed because the PG 64-22 binder behaves like a Newtonian fluid above 50°C (Edwards et al., 2006).

Temperature Dependency: The G*/sinδ and δ values for the ASA1-mixed binder are plotted against the testing temperatures in Figure 2.9a. As expected, the G*/sinδ value decreased and the δ value increased with increasing temperature. This is because at a low temperature, the asphaltenes (n-heptane insoluble material) in asphalt binder are able to form a compact structure, whereas at high end of the testing temperature they disperse as free particles (Wasiuddin, 2006; Wasiuddin et al. 2006). It is also observed that the higher the dosage of ASA1, the lower the G*/sinδ value and the higher the δ value. Similar behavior was observed for ASA2-mixed binder (Figure 2.9b). This could be due to the decrease in the number of polar molecules in the asphalt binders resulting in a decrease in the intermolecular forces.

The horizontal dotted lines in Figures 2.9a and 2.9b represent the Superpave® specified limiting value (1.0 kPa). Temperatures for each binder type that meets the Superpave® criterion for unaged condition are presented in Table 2.3. Similar observations were made by Selvaratnam et al. (2007), who reported G*/sinδ values RTFO-aged samples; the limiting high critical temperatures were found to be higher than unaged condition. Taking the minimum of unaged and RTFO-aged limiting temperatures, the high critical temperatures of the PG 64-22 binder with AS additives were calculated and are reported in Table 2.3. The true high critical temperature for the neat PG 64-22 binder was found to be 65°C. Correlating with limiting values of Superpave® specified rutting factors (1.0 kPa for unaged, and 2.2 kPa for RTFO-aged conditions), the optimum dosage of both ASA1 and ASA2 was found to be 0.50%.

Loading frequency dependency: To correlate the G* value of the binder with the E* value of a corresponding surface mix (ODOT Insoluble S4), frequency sweep tests were conducted on RTFO-aged PG 64-22 binder samples with and without 0.5% ASA2. Binder samples were tested at 4.4˚C, 21.1˚C, 37.8˚C and 54.4˚C at loading frequencies ranging from 25 Hz to 0.1 Hz at each temperature. Once the sample had reached the required test temperature, a strain controlled oscillating torque was applied and the sample was preconditioned for one-minute at 25 Hz. This set of frequency sweep tests were performed in a step fashion. The *first step* was designed to conduct the test in the frequency range of 25 Hz to 5 Hz, and test data was recorded at 5 Hz interval. A *second step* was added to continue the test for a loading frequency ranging from 1 Hz to 0.1 Hz, and test data was recorded for loading frequencies of 1 Hz, 0.5 Hz and 0.1 Hz. Samples were tested at temperatures going from coldest to warmest. Testing at a given temperature started with the highest frequency of loading and proceeded to the lowest.

The mix data used in this study was obtained from a related study (Hossain et al., 2009). The nominal maximum aggregate size of this mix was 19 mm with a binder content of 5.3%. The dynamic modulus (E*) value of the mix was evaluated in accordance with the AASHTO TP 62 test method. The mixing and compaction temperatures for the mix were 163°C and 149°C, respectively. Isotherms of G* and E* for the tested binder and mix samples are shown in Figures 10a and 10b. The test results indicate that ASA2 does not appear to have any significant effect on the E* value. As expected, testing temperature had significant influence on both G* and E* values. Also, the loading frequency exhibited significant influence on both the G*

value of the binder and the E* value of the mix. The G* value of the binder reduced as much as 18-fold when the loading frequency reduced from 10 Hz to 0.1 Hz, and the E* value of the corresponding mix reduced as much as five-fold. The E* value of the mix was found to be a power function ($E^*=A(G^*)^N$) of the G* value of the binder. Model parameters A and N for the established correlations are shown in Table 2.4. A good correlation (R^2 = 0.98 or higher) was observed for loading frequencies ranging from 0.5 Hz to 25 Hz.

2.5.4 Selection of Rheometer

It is recognized that although advanced DSRs might be capable of performing these sweep tests, the DSR used in the present study is not equipped with such features. As mentioned earlier, having the flexibility of adding multiple *steps* in one test provides some competitive advantages to the DSR in terms of efficiency. Enhanced efficiency was clearly observed for the frequency sweep tests (from 25 Hz to 0.1 Hz) in which the *first step* involved collection of data at 5 Hz interval, while the *second step* involved collection of data at 0.5 Hz interval. Temperature sweep tests demonstrated similar efficiencies. A DMA-based temperature sweep test (58°C to 70°C, at 3°C interval) is far more efficient than its DSR counterpart. As noted earlier, a 5-minute thermal equilibrium time was sufficient for the DMA, compared to the recommended 10 minutes equilibrium time for the DSR. Overall, a DSR is found to be more convenient for binder verification and grading, as templates for the associated AASHTO specifications can be readily used. It worth noting that the purpose of this comparison is to highlight the strengths and weaknesses of both pieces of equipment for a given testing situation, not to promote one over the other.

2.5.5 Optimum Dosage of Anti-stripping additive

The optimum dosage of an AS additive depends on both rheological characteristics of an asphalt binder and performance of the asphalt mix. In addition to rutting factor, other rheological characteristics such as fatigue factor and low temperature cracking resistance govern the acceptable dosage level. As noted earlier, to fulfill the Superpave® specified rutting criterion, the maximum dosage level of both ASA1 and ASA2 was found to be 0.5% by weight of the binder.

To examine the Superpave® specified fatigue and thermal cracking criteria, limited laboratory tests were conducted in this study. To evaluate the fatigue resistance, pressure aging vessel (PAV)-aged PG 64-22 binder samples with and without 0.5% ASA1 were tested at its intermediate temperature (25°C) using a DSR. The fatigue factor (G*sinδ) of neat PG 64-22 binder was found to be 2,855 kPa and that of the ASA1-modified binder was found to be 2,810 kPa. Both of these values are within the acceptable Superpave® specifications (less than or equal to 5,000 kPa), and ASA1 found to reduce the fatigue potential of a pavement. Similarly, bending beam rheometer (BBR) test results at -12°C on PAV-aged samples showed that 0.5% ASA1 decreased the stiffness, S(t), of neat PG 64-22 binder from 195 MPa to 184 MPa, which passed the Superpave® criterion (S(t) should be no more than 300 MPa). As expected, the m-value, denoting rate of stress relaxation, of ASA1-modified binder increased from 0.316 to 0.320, which satisfied the Superpave® criterion (m-value should be at least 0.300).

The bond strength of the same PG 64-22 and AS additives was studied as part of a related study and reported in Hossain et al. (2009). In that study, an increase in

bond strength was reported for the same PG 64-22 binder when either of these AS additives was added. The bond strength was estimated by evaluating the tensile strength ratio (TSR) for asphalt mixes with AS2 and surface free energy (SFE) for binder with ASA1. The tensile strength ratio (TSR) increased from 0.56 to 0.89 when 0.5% ASA2 was added. Because of the nature of that project, locally available high moisture susceptible granite aggregates were used in the control mix. The TSR was determined as per AASHTO T 283, except for the sample curing procedure which was same as the corresponding mix design procedure (AASHTO R 30). The ODOT follows this modified curing procedure and uses a TSR value of 0.8 or higher for acceptable mixes (Hossain et al., 2009; Wasiuddin, 2009). While evaluating the SFE of the same PG 64-22 binder, it was reported that the corresponding values of the free energy of adhesion of the control binder and the same binder with 0.5% ASA1 are 11.2 ergs/cm^2 and 15.4 ergs/cm^2, respectively (Hossain et al., 2003). This represents a 37% increase from the control binder. An increase in SFE indicates increased resistance against moisture damage in pavement (Moffat, 2003; Buddhala et al., 2009). Thus, the optimum dosage for both AS additives was selected as 0.5% (by weight of the binder)

2.6 CONCLUSIONS AND RECOMMENDATIONS

This study presents rheological data of three locally available PG binders in Oklahoma using a Dynamic Mechanical Analyzer. The mechanical workability, linear viscoelastic limit, and temperature and frequency dependency on a PG 64-22 binder mixed with two anti-stripping (AS) additives namely, ASA1 and ASA2, were

examined. Based on the analyses of laboratory test data, the following conclusions can be drawn:

- The DMA was found to be an effective and efficient tool for examining the viscoelastic properties of unmodified PG 64-22 and low polymer modified binder such as PG 70-28.
- A thermal equilibrium time of five minutes was sufficient for the DMA, when testing PG binders at high temperatures. In comparison, the recommended thermal equilibrium time for the DSR was much longer (ten minutes).
- Based on the Superpave® reflecting rutting factor of unaged and RTFO-aged PG 64-22 binder, the maximum allowable dosage of ASA1 and ASA2 was found to be 0.5% for the tested PG 64-22 binder.
- Anti-stripping additives did not have any influence on the mechanical workability of the PG 64-22 binder
- Neither ASA1 nor ASA2 altered the linear viscoelastic limit of the PG 64-22 binder. No noticeable drop of G* was observed up to a strain level of 51%.
- Anti-stripping additives stored (up to two years at 22°C) in an air-conditioned facility did not degrade the functionality of anti-stripping additives.
- The high critical temperature for the PG 64-22 binder found to be 65°C, but it degraded when a liquid ant-striping additive was mixed with the binder. The corresponding reduction of the high PG temperature was 2.3^{o}C, and 1.8^{o}C in case of 0.75% ASA1 and 1.0% ASA2-mixed binder, respectively.
- Frequency sweep tests data at various testing temperatures showed loading frequency had significant influence on the G* values of a binder. The G* value

reduced as much as 18 folds when the loading frequency was reduced from 10 Hz to 0.1 Hz. Similarly, the E* value of the corresponding mix reduced as much as five folds.

- Good correlations between the G* values of the binder and the E* values of the mix were established. The dynamic modulus of the mix was found to be a power function of the complex modulus of the binder ($E^*=A(G^*)^N$). The anti-stripping additive did not seem to show any significant influence on the E* value of the mix.

Based on the findings and limited scope of the current study, additional laboratory testing is needed to validate the testing protocol for a high polymer modified asphalt binder such as PG 76-28, subjected to short-term and long-term aging. Also, additional frequency sweep tests on binders mixed with other AS additives (i.e., hydrated lime) could be conducted at various temperatures and time-temperature superposition could be used to obtain their master curves. Furthermore, dynamic mechanical analyses of asphalt mastic and mixture samples with and without ant-stripping additives could be conducted to predict the fatigue life of a pavement

2.7 ACKNOWLEDGEMENT

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(WMA) project for their valuable assistance and support.

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Table 2.1 Properties of Anti-stripping Additives used in this Study (Akzo Nobel, 2008; Arr-Maz Chemicals, 2009)

Name of Additive	ASA1	ASA2
Dosage	0.2% – 0.8%	0.5% – 1.0%
Physical State	Brown to Dark Brown Liquid	Brown to Dark Brown Liquid
Viscosity	225 cps at 25°C	350 cps at 25°C
Flash Point	> 149°C	> 200°C
Boiling/Condensation	> 150°C	> 150°C
Melting/freezing	< 0°C	< 0°C
Density	0.95 g/cm^{3} at 25°C	1.03 g/cm^{3} at 25°C
Solubility	Partially soluble in cold water	Partially soluble in cold water
Storage Temperature	32°C to 38°C	Below 48.9^{o}C
P^{H}	13 at 22°C	12.2 at 22°C
Specific Gravity	0.99 to 1.03	1.02
Solubility in Water	Slight	Partially soluble in cold water
Composition	Bis-hexamethylenetriamine >30%, Aminoethylethanolamine (AEEA) >1%, and the rest is unknown, if any.	Alcohol ethoxylate (33%), Fatty amine derivative (25%), Distillate residues (19%), Polyamines (18%), and Fatty acid (5%)

Table 2.2 Binder Test Matrix

a) DMA Testing Protocol Establishment and Validation					
Procedural and Validation Purpose	Binder Type	Sample Loading Temp (^oC)	Testing Temp (^oC)	No. of DMA Tests	No. of DSR Tests
Thermal equilibrium	PG 64-22	58	64	3	-
Rutting factor	PG 64-22	58	64	3	3
Rutting factor	PG 70-28	64	70	3	3
Rutting factor	PG 76-22	70	76	3	3
Temperature Dependency	PG 64-22	DMA: 52; DSR: 52 to 67 @ 3^oC interval	58 to 73 @ 3^oC	3	18
DSR Fine Tuning	PG 76-28	70, 76, 85 (for DSR)	76	-	9
Total Number of Tests:				15	36

b) Superpave Rutting Factor and Phase Angle Determination					
Purpose	Binder Type	AS Agent	Dosage (by Weight)	Testing Temp (^oC)	No. of DMA tests
Rutting Factor	PG 64-22	ASA1	0.25%, 0.50%, 0.75%	64	9
Rutting Factor	PG 64-22	ASA2	0.50%, 0.75%, 1.0%	64	9
Total Number of Tests:					18

c) Sweep Tests						
Binder Type	AS Agent	Dosage (by Wight)	Sweep Type and Number of Tests on DMA			
			Time[a]	*Strain*[b]	*Temperatue*[c]	*Frequency*[d]
PG 64-22	ASA1	0.00%	3	3	3	3
		0.25%	3	3	3	3
		0.50%	3+6[f]	3	3	3
		0.75%	3	3	3	3
	ASA2	0.00%	-	-	-	-
		0.50%	3	3	3	3+12[g]
		0.75%	3	3	3	3
		1.00%	3	3	3	3
Total Number of Tests:						102

d) Fatigue and Thermal Cracking				
Binder Type	AS Agent	Dosage (by Wight)	No. of DSR Tests	No. of BBR Test
PG 64-22	ASA1	0.00%	3	3
		0.50%	3	3
Total Number of Tests:				12

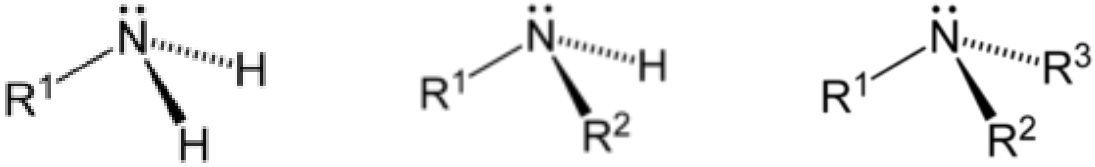
[a] Testing temperature = 64^oC; strain = 12%, and frequency =10 rad/sec, time = 15 mins; [b] Testing temperature = 64^oC; strain = 0 to 51%, and frequency =10 rad/sec; [c] Testing temperature = 58° to 73° @ 3^oC; strain = 12%, and frequency =10 rad/sec; [d] Testing temperature = 64^oC; strain = 12%, and frequency =0.15 rad/sec to 15.15 rad/sec; [f] 6 samples were tested to determine the influence of storage time; and [g] 12 samples were tested at 4.4^oC, 21.1. 37.8^oC, and 54.4^oC to compare G* binder with E* of mixes.

Table 2.3 True High PG Grade for Each Binder Type

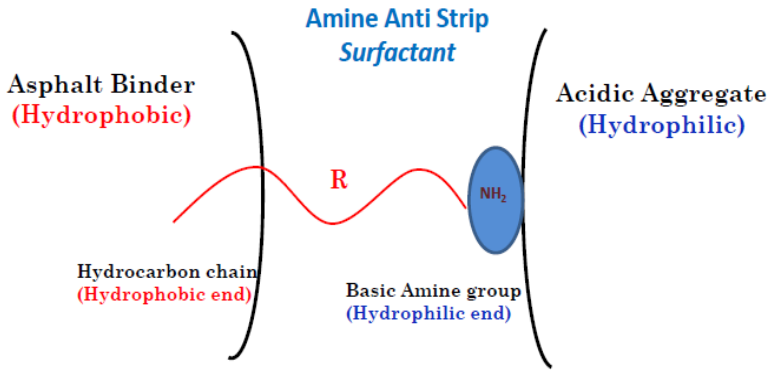
Binder Type	Unaged Condition Limiting Temperature	RTFO-aged Condition Limiting Temperature	True high PG Temperature	Pass/Fail?
Neat PG 64-22	65.0	66.0	65.0	Pass
PG 64-22 + 0.25% ASA1	64.3	65.5	64.3	Pass
PG 64-22 + 0.50% ASA1	64.1	64.7	64.1	Pass
PG 64-22 + 0.75% ASA1	62.7	64.5	62.7	Fail
PG 64-22 + 0.50% ASA2	64.8	65.1	64.8	Pass
PG 64-22 + 0.75% ASA2	63.4	64.7	63.4	Fail
PG 64-22 + 1.0% ASA2	63.2	64.2	63.2	Fail

Table 2.4 Model Parameters for Correlation between Binder's Complex Modulus (G*) and Mix's dynamic Modulus (E*)

Binder Type	Frequency (Hz)	Power Model, $E^*=A(G^*)^N$		
		A	N	R^2
PG 64-22	25	9813.9	0.7033	0.975
	10	18661	0.6441	0.995
	5	22150	0.6312	0.987
	1	13289	0.7063	0.984
	0.5	18617	0.6774	0.975
	0.1	14927	0.5556	0.711
PG 64-22+0.5% ASA2	25	13015	0.6927	0.998
	10	12484	0.6924	0.998
	5	11944	0.7029	0.997
	1	14071	0.7076	0.967
	0.5	18221	0.6843	0.964
	0.1	40641	0.6116	0.949

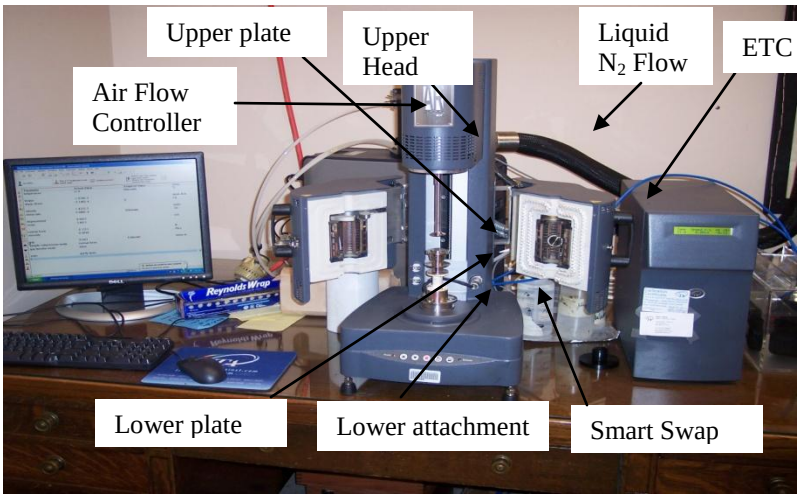


a) primary amine b) Secondary amine c) Tertiary amine

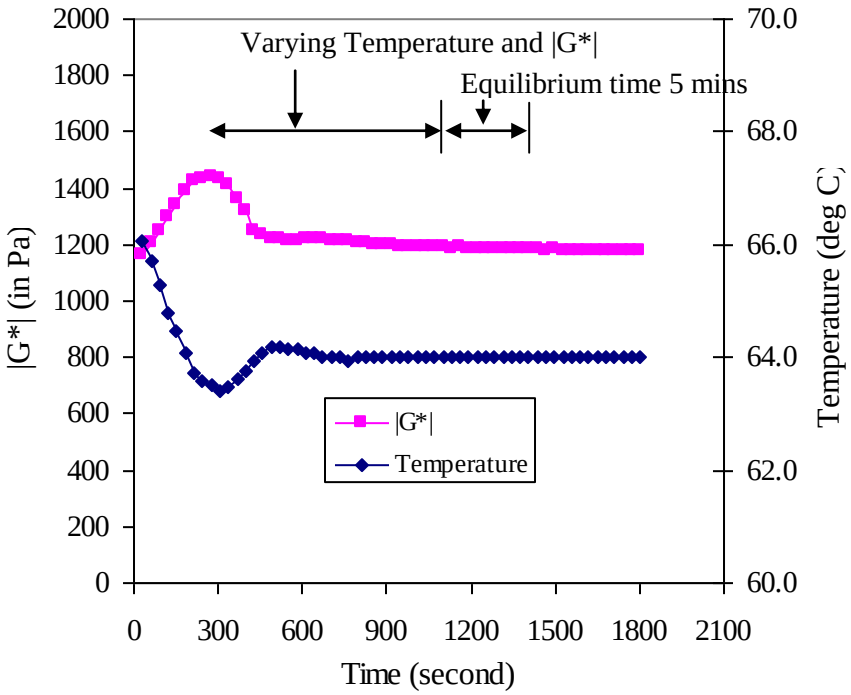


d) Surface Modification of Acidic Binder with Amine Anti-Stripping Agent

Figure 2.1 Functional Group of Asphalt Binder and Amine-based Liquid Anti-stripping and Surface Modification (Harnish, 2009; Hossain et al., 2009).



a) DMA



b) Thermal equilibrium of the DMA

Figure 2.2 (a) AR2000ex Rheometer (DMA) Used in the Current Study, and (b) Thermal Equilibrium Data.

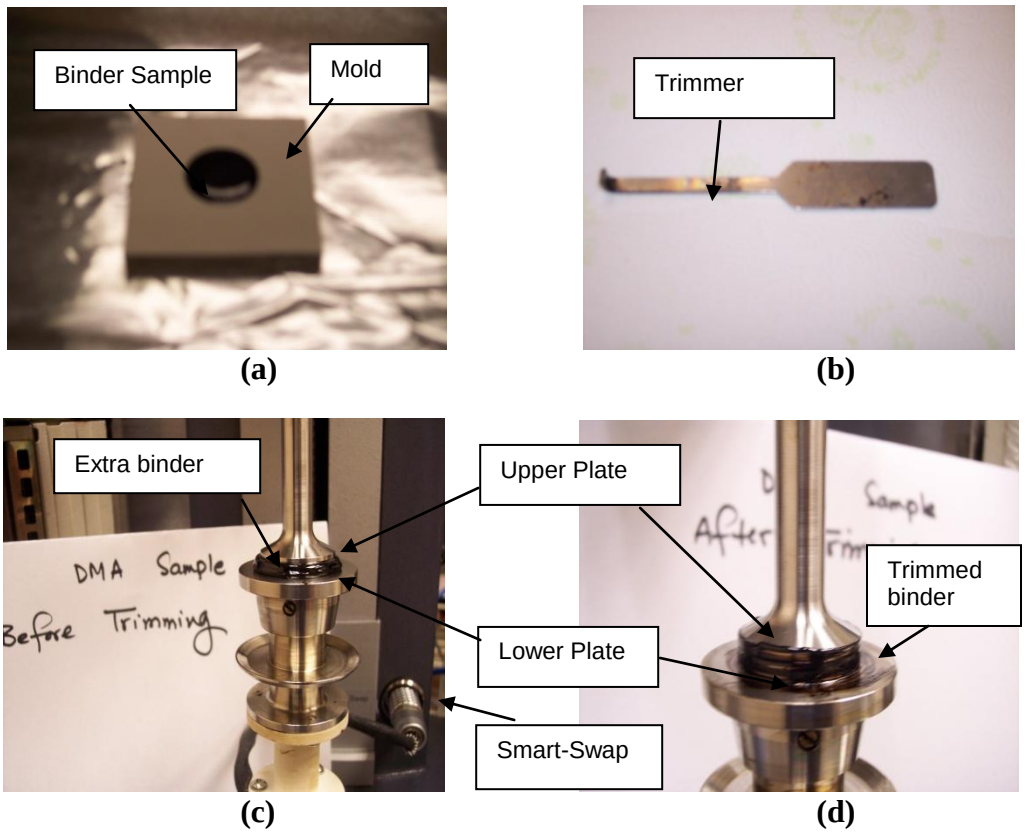


Figure 2.3 (a) Binder Sample on Silicon Rubber Mold (b) Trimmer for DMA (c) Sample in DMA Before Trimming, and (d) Sample in DMA After Trimming.

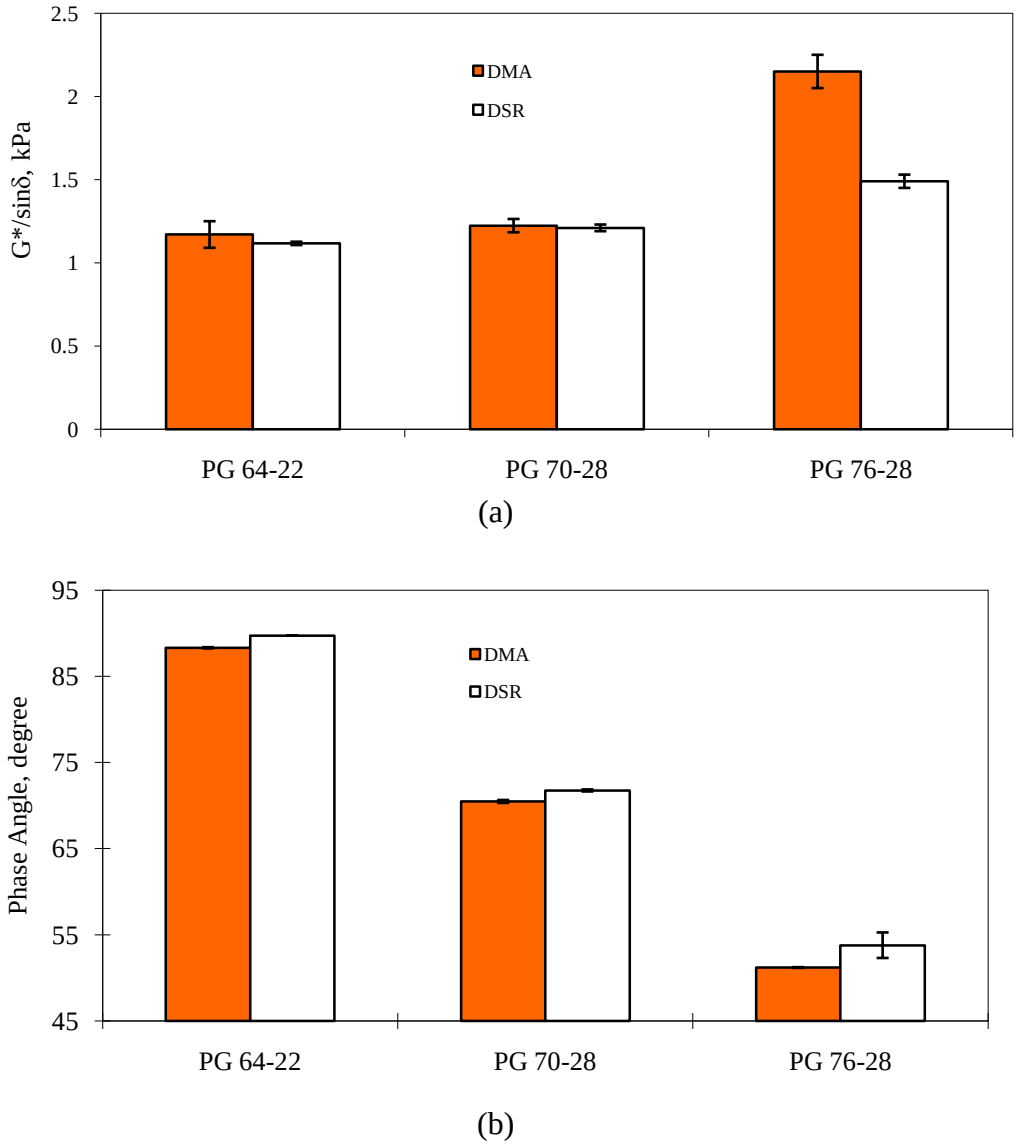


Figure 2.4 Test Results of Selected PG Binders at High Grade Temperatures Using DMA and DSR: a) G*/sinδ, and (b) δ values. (Note: Each Data Point is the Average of Three Replications and Vertical Bars Denote ± One Standard Deviation of the Population):

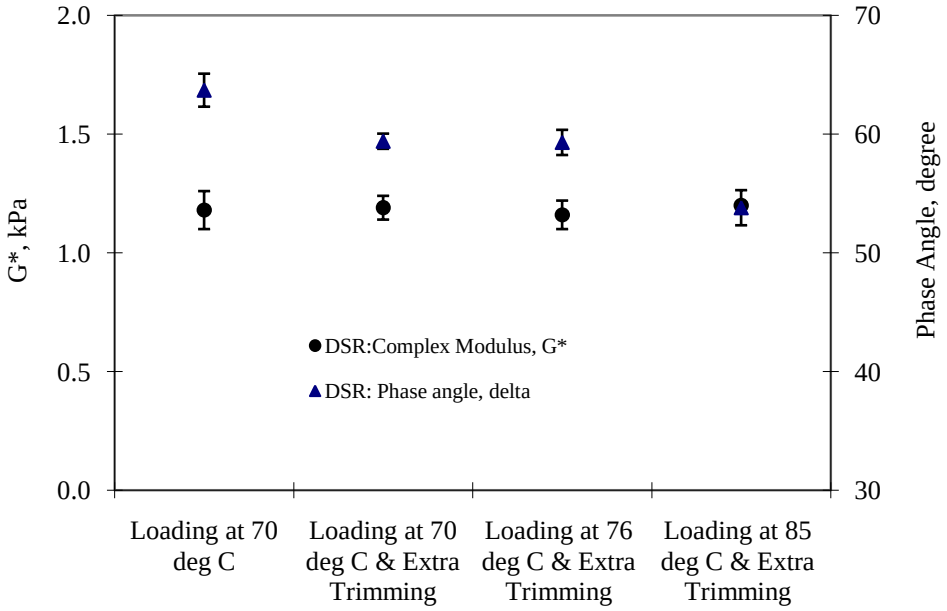


Figure 2.5 Variation G* and δ Values of PG 76-28 Binder with Higher Sample Loading Temperature and Extra Trimming Efforts While Using DSR (Note: Vertical Lines Represent Standard Deviation).

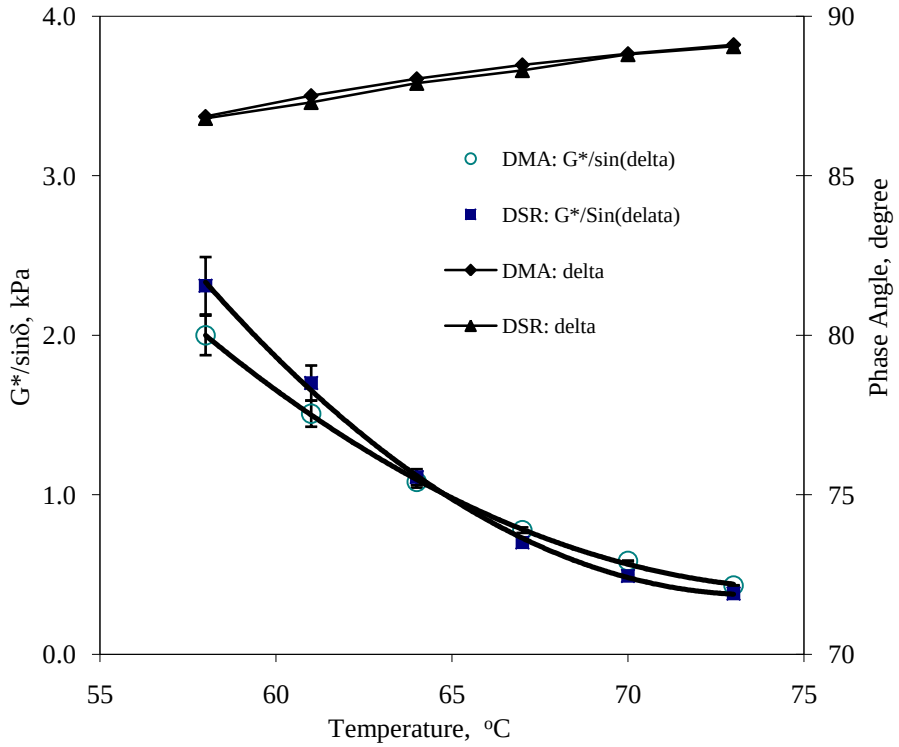
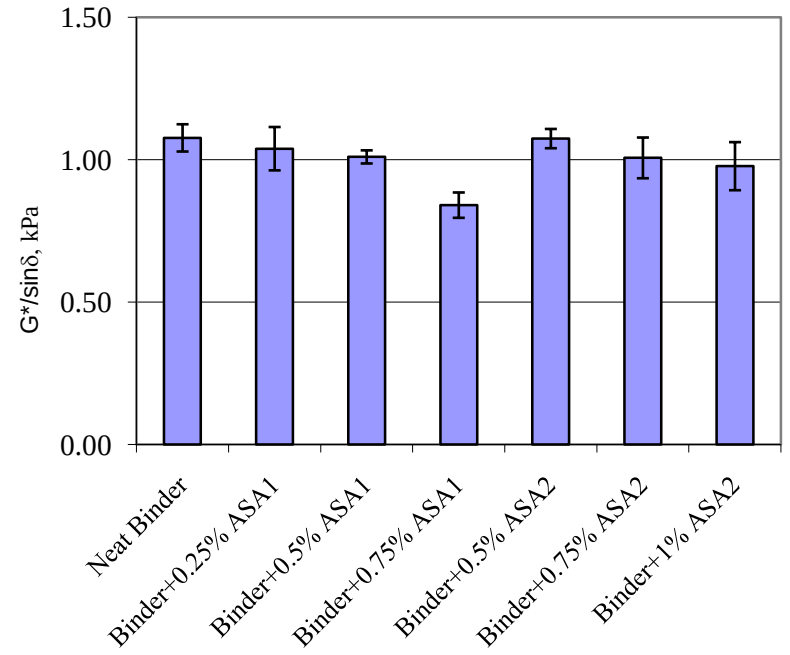
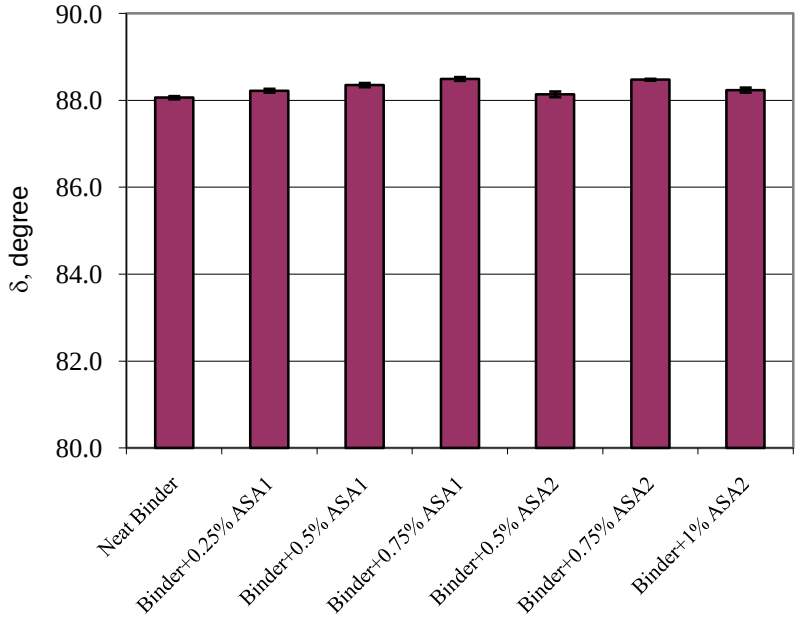


Figure 2.6 Validation of DMA Results for a Wide Range (Near high PG) of Temperature.

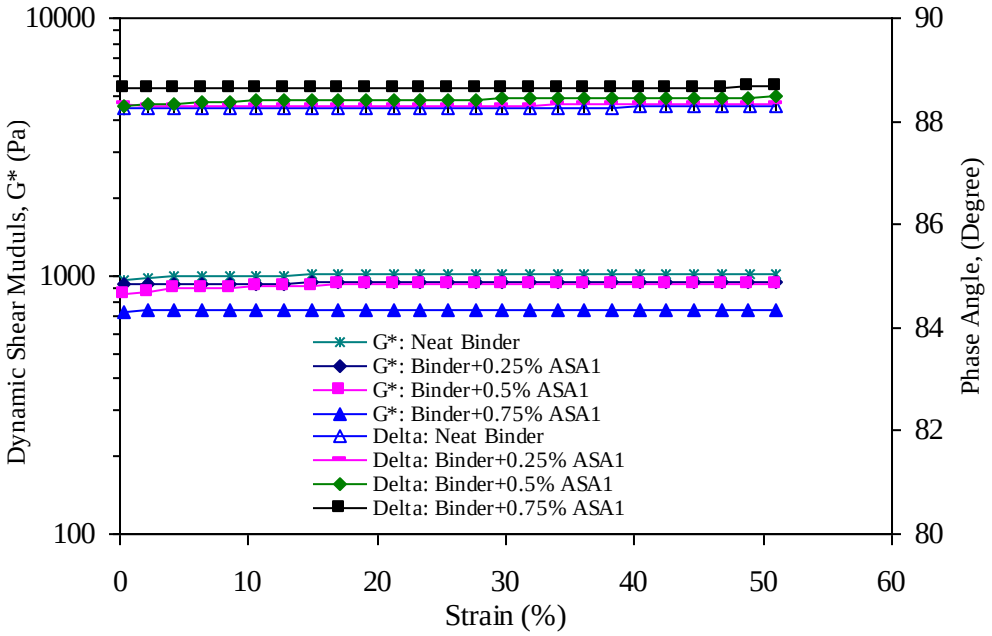


(a)

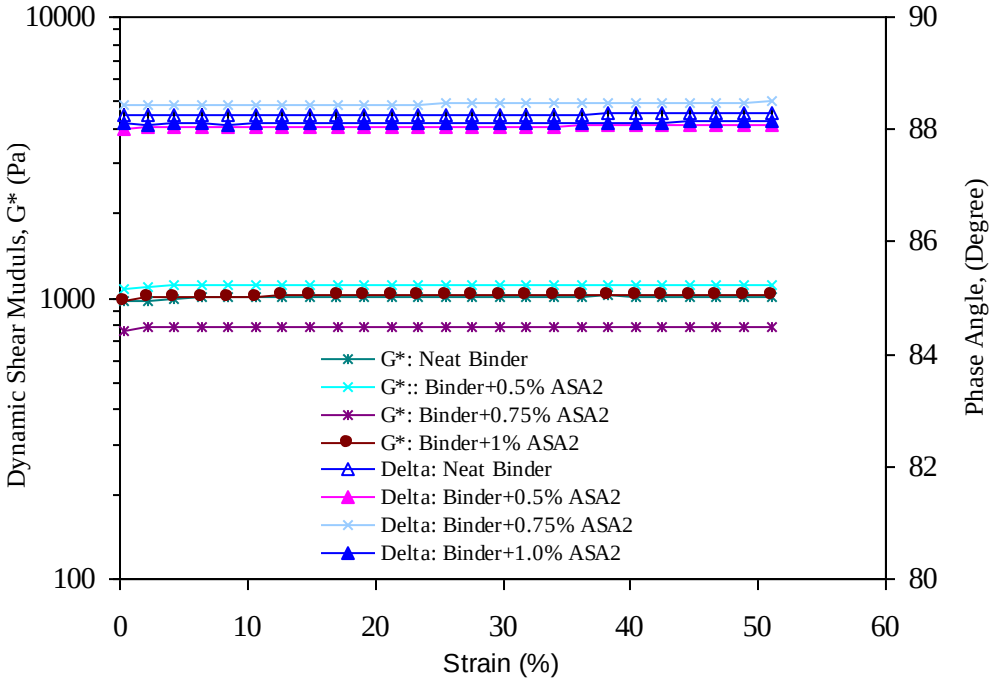


(b)

Figure 2.7 Effect of Anti-stripping Agents on the Base Binder: (a) G*/sinδ, and b) Phase Angle, δ

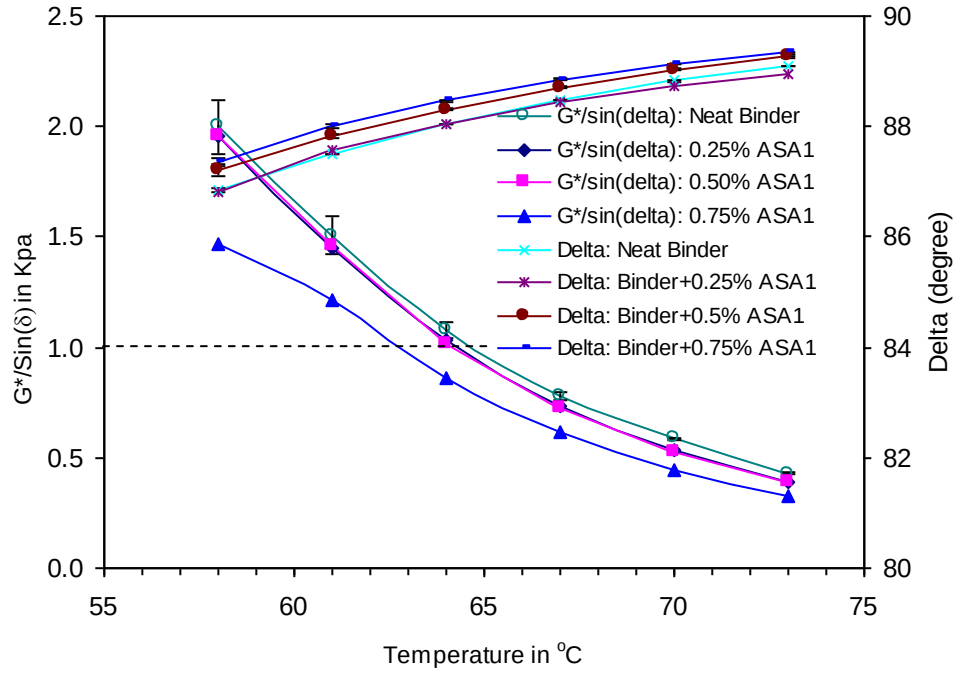


(a)

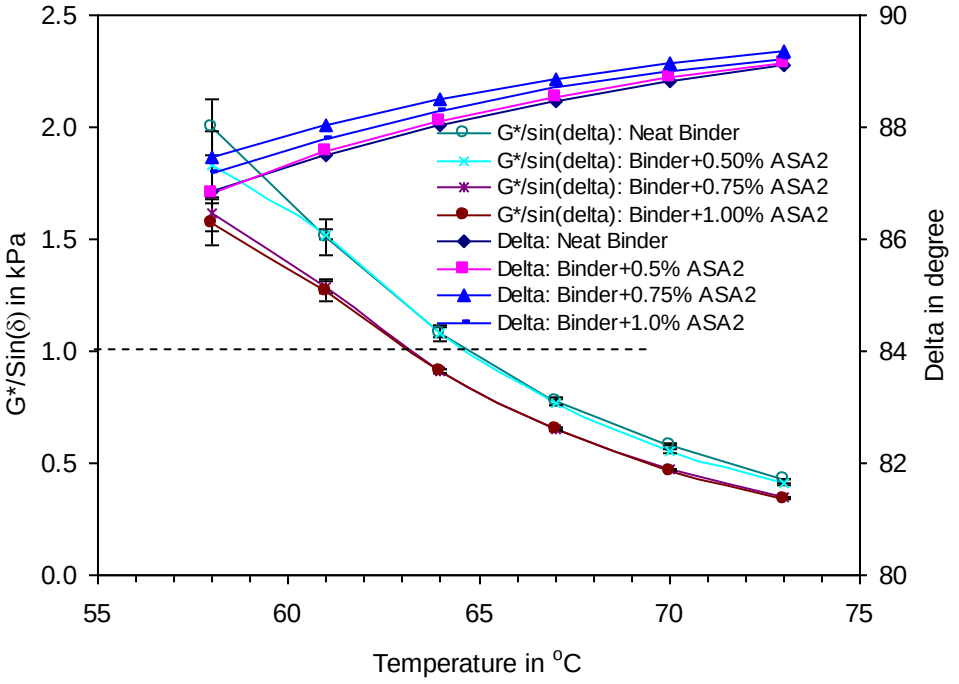


(b)

Figure 2.8 Strain Sweep Test Data off PG 64-22 Binder at 64°C: (a) ASA1, and (b) ASA2.

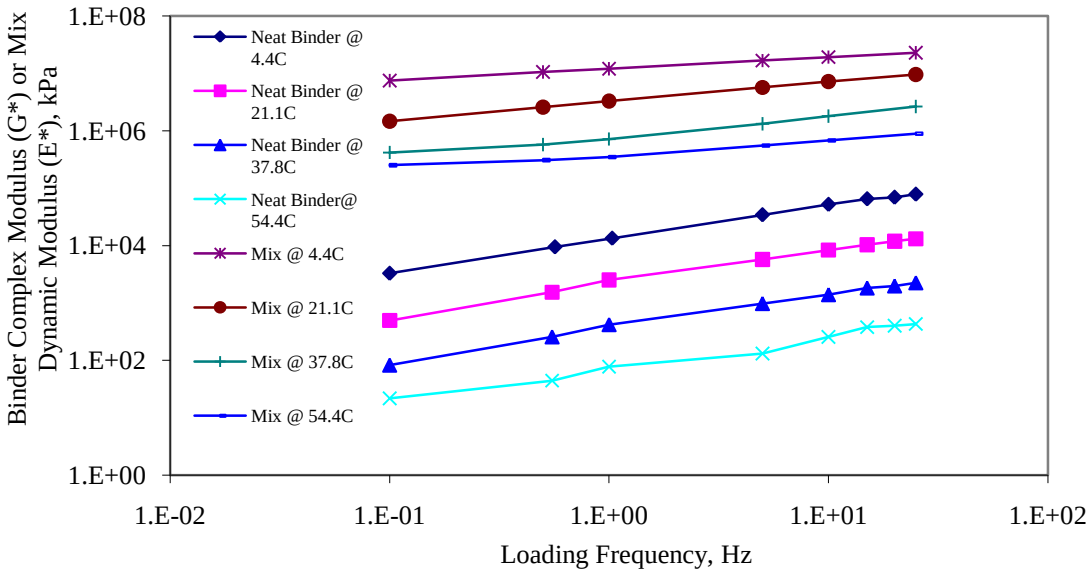


(a)

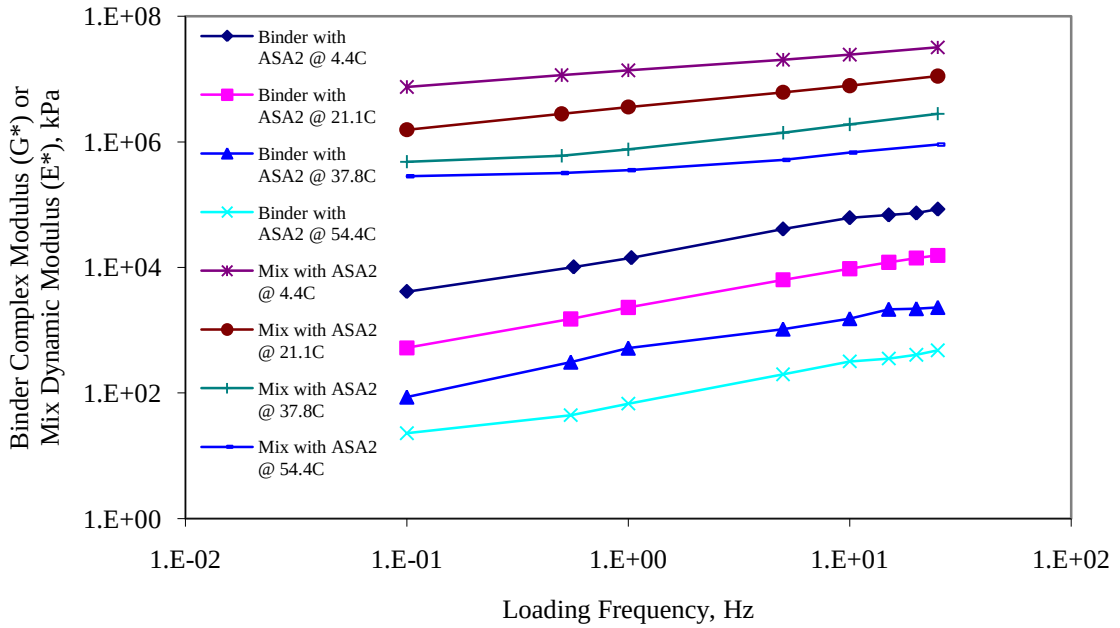


(b)

Figure 2.9 Temperature Effects on Anti-stripping-modified PG 64-22 Binder: a) ASA1; b) ASA2.



(a)



(b)

Figure 2.10 Binder's Complex Modulus Versus Mix's Dynamic Modulus Versus Over a Range of Temperatures: a) With no Anti-stripping Agent; b) with 0.5% ASA2 (Note: Dynamic Modulus data is obtained from Hossain et al., 2009).

3 LABORATORY EVALUATION OF WATER-BEARING ADDITIVE (ADVERA®) FOR WARM MIX ASPHALT[2]

3.1 ABSTRACT

Benefits of warm mix asphalt (WMA) technologies in terms of energy savings and air quality improvements are highly promising. However, further investigation is needed to validate their performance as laboratory and field data are significantly lacking for conditions in Oklahoma. To this end, effects of varying dosages (4, 6 and 8% by the weight of the binder) of a water-bearing WMA additive, Advera®, on the performance grade (PG) of a local binder, PG 64-22, were evaluated. The effectiveness of a commonly used amine-based liquid anti-stripping (AS) agent in Oklahoma, AD-here® HP Plus, on the WMA-modified binder was also studied. Furthermore, the effect of reduced oxidative aging on Advera®-modified binder was investigated. The optimum dosage of Advera® was found to be 6% (by the weight of the binder), which did not alter the PG grade of the base binder. A fairly small amount (0.5% by the weight of the binder) of the AS agent was found to be effective in increasing the fatigue fracture and low temperature resistances of the WMA-modified binder. A slight reduction of the high PG temperature was observed when a reduced oven temperature was maintained during short-term aging of the Advera®-modified binder. Selective performance test data of a corresponding WMA mix from a closely related study was found to consistent with the rheological properties of the modified binder. The findings of the current study are expected to enhance the inventory of

[2] This chapter or portions thereof has been published previously in the Compendium of Papers of the 90th Transportation Research Board Annual Meeting, Washington, D.C., January 23-27, 2011. The current version has been formatted for this dissertation.

rheological database for local materials and help in implementing WMA mixes in Oklahoma.

3.2 INTRODUCTION

WMA technologies are relatively new processes and products. These technologies use various mechanical and chemical means to reduce the shear resistance of the mix at relatively low production temperatures, while reportedly maintaining or improving the pavement performance. These technologies can reduce the production temperature of the hot mix asphalt (HMA) by 16°C to over 55°C (Newcomb, 2010). The lower production temperatures lead to reduced green house gas emissions (i.e., volatile hydrocarbons and CO_2), dusts and production costs (FHWA, 2010; Goh et al., 2007; Kristjansdottir, 2007). It also extends the paving season in certain locations where the HMA construction is restricted to warmer months. However, test data are significantly lacking in terms of rheological properties of modified binders as well as strength and performance-related properties of the mix.

During the mixing and compaction of WMA, the viscosity of asphalt binder is reduced, allowing the binder to sufficiently coat aggregates at lower temperatures (Weilinski et al., 2009). The hardness of asphalt binder can be reduced by using an additive in the asphalt binder or by foaming the asphalt binder in the mix. Some of these additives include waxes, chemicals, or zeolite, which reduce the viscosity of the asphalt binder at production temperatures. The reduction of viscosity of the asphalt binder at comparatively low production temperatures is a result of the expansion of the asphalt binder.

A commonly used water-bearing WMA additive named Advera® was selected for evaluation in this study. Unlike foamed asphalt or free-water systems (e.g., Terex and Astec Double Barrel Green), Advera® is a finely powdered synthetic zeolite (sodium aluminum silicate hydrate) that is hydro-thermally crystallized, and it holds from 18 to 22% (by mass) of water (PQ Corporation, 2010). Theoretically, the zeolite releases water which creates foam that reduces the viscosity and increases the workability. It facilitates better coatings of the asphalt binder on aggregates. Once the binder is cooled off, the water condenses and, ideally, is reabsorbed by the zeolite, thus presumably leaving no significant effect on the rheology thereafter (PQ Corporation, 2010). The recommended dosage of Advera® is 0.25% (by the weight of the mix), which can be introduced into the plant using a feeder, with minor modification to the plant.

In practice, many agencies simply allow the addition of a WMA additive to an approved PG binder without accounting for possible grade change to the base binder (Austerman et al., 2009). As the modified binder needs to meet the Superpave® specifications, it is important to examine the impacts of the additive on the PG grading of the binder. Previous WMA studies (e.g., Austerman et al., 2009; Hurley and Prowell, 2005; Sneed, 2007; Carter et al., 2010) demonstrated significant changes in both the high PG and the low PG temperatures of base binders. The extent of changes in PG temperatures depends on the amount of additive being added to the binder. Therefore, performance factors (rutting, fatigue fracture and thermal cracking) of the Advera®-modified binder need to be evaluated. For instance, the reduced aging of asphalt binder can lead to excessive rutting during the early age of the pavement.

Another common concern for the WMA stems from the argument that the low production temperature would fail to eliminate moisture as effectively, leaving behind more moisture within the mix during the construction process. Additionally, if the moisture contained in Advera® does not completely evaporate during the production process or become reabsorbed, it may further worsen the situation. The moisture damage potential of a mix is generally evaluated through the determination of tensile strength ratio (TSR), in accordance with AASHTO T 283 (Hurley and Prowell, 2005; Cross et al., 2000). It can also be determined through the observation of the point of inflection in Hamburg-wheel tracking (HWT) tests (Hurley and Prowell, 2005).

The working mechanism of Advera® is similar to that of another water-bearing WMA additive named Aspha-Min® zeolite. Hurley and Prowell (2005) studied the viability of Aspha-Min® at different production temperatures ranging from 149°C down to 88°C. These researchers evaluated two binders (PG 58-28 and PG 64-22) and reported that the zeolite (0.3% by the weight of the mix) did not contribute to the increased Asphalt Pavement Analyzer (APA) rut depth. Rather, the increased rut depth was associated with the reduced mixing and compaction temperatures. These researchers also suspected significant increase in moisture susceptibility of the zeolite-modified mix, which seemed to be mitigated with hydrated lime.

The Montana Department of Transportation (MDT) studied the rutting potential of loose mix samples collected from an Advera®-modified WMA section in Yellowstone National Park (YNP) (Perkins, 2009). The production temperature for the WMA section was 121°C, and the dosage level of Advera® was maintained as 0.25% (by the weight of the mix). HWT data from laboratory prepared slabs showed the

Advera® mix rutted much faster than the control mix to the point that the former did not pass the MDT specifications (maximum 13 mm rut depth after 20,000 cycles). Based on the field experience with the YNP project, the MDT conducted a laboratory study on several WMA technologies including Advera®. It was reported that Advera® was found be one of the worst WMA technologies in terms of rut resistance.

Schiebel (2009) presented tests results from a case study performed on I-70 pavement sections in Colorado that incorporated three WMA technologies including Advera®. The production temperature for the WMA mix was 121°C, while that for the control HMA with a PG 58-28 binder was 138°C. Data showed Advera® did not change the PG grade of the control binder. Test results showed that the Advera® mix performed the worst in terms of rutting and stripping potentials. Advera® mix showed 9.5 mm rut depth after 5,100 cycles compared to the control mix's 9 mm rut depth after 9,700 cycles while the reported stripping inflection cycles were 3,300 and 7,800 for the Advera® and control mixes, respectively.

Tao and Mallick (2009) investigated the feasibility of using 100% recycled asphalt pavement (RAP) as a base course with varying dosages (0.3%, 0.5%, and 0.7% by the weight of the mix) of Advera® at low compaction temperatures. It was reported that workability of the RAP improved with the addition of Advera® at temperatures as low as 110°C. At temperatures less than 80°C, the addition of Advera® was found to stiffen the mix which was also reflected in increased seismic moduli and indirect tensile strength (ITS) data. The largest ITS value was obtained when 0.3% Advera® was added to the mix. The effect of added amounts of Advera® on bulk specific gravity did not show any particular trend. It was also suspected that the interaction of

the RAP binder with Advera® played a significant role in compaction by preventing the asphalt binder from fully foaming.

Moisture susceptibility was also a concern for an Advera®-modified mix that used the same base binder (PG 64-22 from the same refinery) and additives (6% Advera®, and 6% Advera® plus 0.5% AD-here® HP Plus) as the current study (Hossain et al., 2009). TSR tests of asphalt mixes were conducted as per AASHTO T 283 except for the sample curing procedure. Asphalt mixes were cured as per AASHTO R 30, a two hour oven curing at the compaction temperature. Repeatability of these test results were ensured by testing three replicate samples for each curing condition, and the average value was reported. As shown in Table 3.1, TSR values of the control and Advera® mixes compacted at 149°C were found to be 0.56 and 0.48, respectively. At a lower compaction temperature (121°C), the TSR value of the Advera®-modified mix was found to be significantly reduced (i.e., TSR = 0.48). These mixes did not meet the TSR requirement (≥ 0.80) of the Oklahoma Department of Transportation (ODOT). It is important to note that the indirect tensile strength (ITS) values of Advera®-modified mixes at 121°C were also significantly lower than those at 149°C. The ITS value of the control mix at 149°C also dropped significantly when AS agent (0.5%) was added in the mix. This was possibly due to the fact that the low viscosity AS agent constituted a relatively softer binder. A relevant study (Selvaratnam et al., 2007) reported 0.7°C reduction of the high PG temperature and -1.3°C reduction of the low PG temperature of a PG 64-22 binder with an AS agent. On the other hand, the TSR value of the Advera®-modified mix increased to 0.77 when the AS agent was used. Similar observations were made from the HWT stripping

inflection point tests. There was a significant increase in the stripping inflection point when the AS additive was added into the mix. However, rheological properties (i.e., PG grade) of the modified binders remained unknown. Furthermore, the AS additive needs to be evaluated if there is any change in asphalt binder supplier, crude source or aggregate source (Harnish, 2009). To this end, the viability of these additives was evaluated as per Superpave® test methods. Specific objectives of this study were as follows: (i) evaluate the effects of dosage levels of Advera® on the PG grade of the base binder, (ii) investigate the effects of AD-here® HP Plus on the WMA-modified binder, and (iii) evaluate the effects of reduced operating temperature of the rolling thin film oven (RTFO) on the WMA-modified binder.

3.3 TEST MATERIALS

Advera® was obtained from PQ Corporation in Malvern, Pennsylvania. Selective dosages of Advera® were 4%, 6%, and 8% (by the weight of the binder). These dosages are around the recommended amount for a typical Advera®-modified mix (0.3% by the weight of the mix). The base binder (PG 64-22, from lot number TK 118) was obtained from Valero refinery in Ardmore, Oklahoma. The liquid AS agent, AD-here® HP Plus, was obtained from Arr-Maz Custom Chemicals, Florida. The dosage level (0.5% by the weight of the binder) of the AS agent was maintained constant as recommended in Hossain et al. (2010).

In terms of mixing Advera® with the asphalt binder, no standard procedures are available. However, a variation of the sample preparation used by Edwards et al. (2005) was conducted in this study. Small tin canisters filled with the selected binder were heated for two hours at 135°C. Advera® was then added and stirred by hand with

a spatula for approximately one minute. Leaving the spatula in the canister, the top of the canister was covered with aluminum foil, and the covered canister was placed back into the oven for 10 minutes. The foil was then removed and the mixture was hand stirred for approximately 30 seconds. It was again covered with a foil and placed back into the oven. To help ensure that it was mixed thoroughly, the sample was stirred every ten minutes for an hour. Binder mixed with the additive was then left overnight for further testing.

3.4 EFFECT OF ADVERA® ON PERFORMANCE GRADE

Like other WMA additives, Advera® showed the potential for altering the base binder's PG grade. A summary of dynamic shear rheometer (DSR) test (AASHTO T 315) results for un-aged and RTFO-aged (at 163°C) binder with Advera® at different temperatures is presented in Figure 3.1. The horizontal dotted lines in this figure represent the Superpave® criteria for rutting factor ($G^*/\sin\delta$) at different aging conditions. Each data point in the chart is the average of at least three replicates, and an error bar represents ±one standard deviation from the mean value. In general, any dosages of Advera® in the base binder increased the $G^*/\sin\delta$ value for both un-aged and RTFO-aged conditions. Apparently there was no significant difference in $G^*/\sin\delta$ values between 4% and 6% Advera®-modified binders. Tao and Mallick (2009) observed similar behaviour for Advera® (0.5% and 0.7% by the weight of the mix) mixes; seismic moduli were also found to be higher than that of the control mix, but the modulus values seemed independent of the amount of Advera®.

From DSR test results, it was also observed that 8% Advera® increased the high PG temperature by 3°C (from 64.8°C to 67.8°C), whereas 4% and 6% Advera®

did not show any increase in the high PG temperature. Advera® tends to enhance the elasticity of the binder, but it is barely noticeable. The slight increase in the complex modulus (G*) could be from the added zeolite which acted as a "reinforcement" in the binder and increased its strength. However, this is a minor contribution as it has no significant effect on the high PG temperature. On the contrary, the Advera®-modified mixes compacted at 149°C were comparatively more susceptible (about 8%) to rutting than the control mixes. This could be related to comparatively lower voids in total mix (VTM) values (about 4%) for the Advera®-modified mixes than the control mixes.

The aforementioned findings should be applied with caution since the lower production temperatures of the WMA may further reduce the G*/sinδ value, resulting in reduced rut resistance of the mix. Previous studies (Perkins, 2009; Schiebel, 2009) made such observations for Advera® mixes where a significant reduction of rut resistance was reported. The HWT data listed in Table 3.1 also showed significance reduction in rut resistance for mixes with Advera®, at a lower curing and compaction temperature (121°C), compared to the control HMA mix. At a curing and compaction temperature of 149°C, the number of cycles for 12.5 mm rut for the control mix was found to be 9,950. Comparatively, the number of cycles to the same depth of rut for the Advera® mix compacted at 121°C was reported as 2,250. A possible explanation of the reduced rut resistance for the Advera® mix could be due to the reduced oxidation hardening of the binder at a low production temperature. To validate this hypothesis, rutting factors for binder samples aged at a reduced RTFO oven temperature were evaluated and discussed later in this paper.

DSR testing of pressure aging vessel (PAV)-aged binder samples was carried out for different dosages of Advera®-modified binder at intermediate service temperatures. All binder samples satisfied the Superpave® specified fatigue factor criterion (≤ 5000 kPa) at an intermediate temperature (IT) of 25°C except for 8% Advera®-modified samples (see Figure 3.2). It was observed that the fatigue factor of the binder increased with the addition of Advera®. The critical IT for the base binder was found to be 22.5°C. On the other hand, the critical IT for 4%, 6%, and 8% Advera® samples was found to be 24°C, 25°C, and 25.8°C, respectively. Thus, Advera® mixes were expected to exhibit more fatigue fracture during their service life than the control mix.

Bending beam rheometer (BBR) test (AASHTO T 313) results (see Figure 3.3) revealed that the stiffness (S) values for different dosages of Advera® were found to be well within the Superpave® limit (≤ 300 MPa $_{\text{at t= 60 sec}}$) at -12°C. On the other hand, 8% Advera® failed to meet the Superpave® limit for the m-value (≥ 0.300 $_{\text{at t = 60 sec}}$). The corresponding m-value for 6% Advera® did not meet the m-value criterion either. However, Student's t-test results showed that the difference between the mean m-value for 6% Advera® samples and 0.300 was insignificant at 95% confidence (p = 0.05) level. Therefore, 6% Advera® was considered the optimum dosage in the current study. As discussed herein, this dosage level (6% by the weight of the binder) Advera® did not notably change the PG grade of the base binder (Table 3.2). The corresponding continuous PG grades of the base and 6% Advera®-modified binders were found to be PG 64.8-24.8, PG 67.2-21.9, respectively. Similar observations were made by

Schiebel (2009) for the tested PG 58-28 binder for which Advera® had significance influence on its PG grade.

Rotational viscometer (RV) test results on unaged Advera®-modified binders at 135°C and 150°C are shown in Table 3.3. It is observed that the viscosity of the binder increases with any amount of Advera®. This phenomenon is possibly due to the fact the water entrapped in crystalline structures of Advera® has already expelled out at high testing temperatures. Similar observations were made in a relevant study by Wasiuddin et al. (2007), which reported that the addition of a zeolite had no reduction of the viscosity of the base binder. Thus, the laboratory-based viscosity measurement of the Advera®-modified binder is not expected to provide any insightful findings in terms of the reduction of the mixing temperature. As explained earlier, the zeolite releases water during mixing in the production plant which creates foam, increases fluidity and increases the workability of the mix.

3.5 EFFECTIVENESS OF ANTI-STRIPPING AGENT

Liquid AS additives are surfactants or surface active materials which concentrate at the interface between asphalt binder and the aggregate surface. While the “head” groups on the surface active agents can bind strongly to the aggregate surface, the hydrocarbon "tails" of the molecules are compatible with the asphalt binder (see Figure 3.4). When added to an asphalt binder, an AS additive acts as a bridge between the asphalt binder and the surface which resists the action of water, displaces the moisture on the surface of the aggregate, and thus promotes the adhesion of the binder to the aggregate surface (Gore, 2003). Liquid AS additives can also coat large amounts of fine aggregates or dusts. These AS additives are also reported to be

effective in promoting adhesion between highly acidic asphalt binders to acidic or siliceous aggregates such as granites and quartzite. Like many other state departments of transportation (DOTs), the ODOT requires contractors to use AS additives to increase tensile strength ratios of a mix under the AASHTO T-283 test criteria, when the ratios cannot be met simply by using a neat or polymer-modified binder (ODOT, 2010). AD-here® HP Plus is one of the liquid AS additives for HMA certified by the ODOT (ODOT, 2010). The most active components in AD-here® HP Plus are polyamine hydrocarbons based on bis-hexamethylene triamine (BHMT) with a very high boiling point (>150°C), which is expected to be effective in WMA mixing and compaction temperatures.

DSR test results of WMA samples with and without the AS are compared in Figure 3.5. As explained earlier, the rutting factor of WMA-modified binder samples was expected to be higher than the base binder. With further addition of the AS (0.5% AD-here® HP Plus), a slight reduction of rutting factors was noticed for both un-aged and RTFO-aged conditions, but it was not enough to lower the high PG temperature of the WMA additive-modified binder .

To facilitate the prediction of the rutting potential of modified binders, aging index was used here. The aging index is the ratio of the G*/sinδ values of aged and un-aged conditions (Edwards et al., 2007). The aging indices for modified binders are summarized in Table 3.4. Advera® (6%) was found to reduce the aging index with an increase in testing temperature, but AD-here® HP Plus did not show any particular trend. At a particular testing temperature (e.g., 64°C), the aging index of the Advera®-modified binder was found to be significantly reduced from that of the base binder. On

the other hand, AD-here® HP Plus was found to increase the aging index of the Advera®-modified binder. These findings are in agreement with the HWT data of the mixes presented in Table 3.1. At a compaction temperature of 149°C, the reported number of cycles to 12.5 mm rut depth for the WMA- and AS-modified (combined) mix was 10,300, and that for the mix with WMA alone was 9,100.

The viscosity of the Advera®-modified binder around the mixing temperature was found to decrease with the addition of AS additive which was expected to be a positive effect for the WMA mix. For instance, the RV test results at 135°C also showed an 8% reduction in the viscosity while using the AS in the WMA. At this temperature, the viscosity of the WMA binder samples was found to be 519 mPa-s, and that of the WMA- and AS-modified samples was found to be 475 mPa-s. Thus, the AS was expected to facilitate a better coating of aggregates with the asphalt binder modified by Advera®.

The effects of the AS agent on the fatigue factor at intermediate service temperatures are also shown in Figure 3.5. It was observed that all samples passed the Superpave® specified fatigue criterion at 25°C. As discussed earlier, the fatigue potential of the WMA was expected to be higher than the base binder. However, the fatigue potential of the WMA-modified binder was reduced when the liquid AS was used. For instance, the fatigue factor ($G^* \cdot \sin\delta$) of WMA samples increased by 14% from that of the base binder, but with further addition of the AS, it was almost the same as that of the base binder. This was due to the fact that the AD-here® HP Plus helped to slow down the age-hardening of the binder. Thus, the AS-modified WMA

was expected to lead to reduced fatigue cracking during the service life of the pavement.

BBR test results of the asphalt binder samples with the WMA and AS additives are shown in Figure 3.6. It was observed that all tested samples passed the Superpave® criteria ($S_{t=60\ sec} \leq 300$ MPa, and $m_{t=60\ sec} \geq 0.300$) at -12°C. While the liquid AS additive was expected to reduce the m-value, the extent of the change was not sufficient to alter the PG grade of the WMA-modified binder. This observation is in agreement with the findings from a previous study (Selvaratnam et al., 2007), where the AS alone reduced the low PG temperature of a PG 64-22 binder up to 1.3°C.

3.6 EFFECT OF REDUCED RTFO TEMPERATURE ON RUTTING FACTOR

The reduced production temperature of WMA is believed to be the leading cause of reduced oxidation hardening of the binder. This reduced oxidation hardening can exhibit an increased rutting during the service life of the pavement. As mentioned earlier, the AASHTO T 240 method specifies the oven temperature for short-term aging of asphalt binder to be 163°C. To obtain a better understanding of the effects of reduced oxidative hardening of WMA additive-modified binder, it is recommended to age (short-term) the asphalt binder at a significantly low temperature (e.g., 150°C, 135°C). Such low temperature controlled short-term aging is expected to mimic the field curing and compaction condition of WMA. Besides the reduced RTFO operating temperature, a reduced aging time rather than the standard duration (85 minutes) can better simulate the field conditions for WMA.

To test this hypothesis, binder samples with and without the WMA was RTFO-aged at a reduced oven temperature of 150°C, a 13°C reduction from AASHTO T 240 specified temperature (163°C). The simulated reduced oxidative hardened binder samples were then tested using the DSR. It was observed that by dropping the operating temperature by 13°C, the rutting factor was reduced by 21% and 15% for the base binder, and the Advera® samples, respectively (see Figure 3.7). These findings are in agreement with rut data of mixes observed from HWT tests reported in Hossain et al. (2009). However, the reduced RTFO temperature of 150°C did not alter the high PG temperature of the 6% Advera®-modified asphalt binder.

To further investigate the aforementioned hypothesis, additional RTFO-aged samples of 6% Advera®-modified binder were produced by maintaining a further reduced operating temperature of 135°C. In addition to the standard duration (85 min), a reduced duration (75 min) was also employed during the RTFO-aging. Subsequent DSR test results of these samples discovered that the rutting factors for these samples reduced significantly (Figure 3.7). Consequently, the high PG temperature for 6% Advera®-modified samples RTFO-aged at 135°C for a duration of either 85 min or 75 min resulted 62.7^{o}C, which failed to meet the PG grade of the base binder. It was also observed both the reduced temperature and the reduced time contributed to the reduced rutting factor of the Advera®-modified binder. However, the reduction of rutting factor of the Advera®-modified binder RTFO-aged at 135°C for 85 min was higher than that of the same binder RTFO-aged at 150°C for 75 min. Thus, the low production temperature and reduced curing time of Advera®-modified mixes is expected to exhibit increased rutting. Previous studies (e.g., Tarefder et al., 2003) also

indicated the PG grade of the binder in HMA mixes as one of the most influencing factors for rutting.

3.7 CONCLUDING REMARKS

This paper presents the effects of a water-bearing WMA additive (Advera®) on rheological properties of a selected PG 64-22 binder. In particular, it demonstrates the influence of Advera® on the PG grading of the base binder. It also evaluates the effectiveness of an amine-based liquid anti-stripping agent (AD-here® HP Plus). Furthermore, it presents the effects of reduced RTFO aging on the stiffness on the Advera®-modified binder. Specifically, the following conclusions can be drawn from the results presented in this paper:

- The optimum dosage of Advera® was found to be 6% (by the weight of the asphalt binder), and this dosage level was not expected to change the PG grade of the base binder.
- Addition of 0.5% AD-here® HP Plus did not show any adverse impacts on the performance factors of the binder. Rather, it improved the fatigue fracture resistance of the Advera®-modified binder.
- RTFO aging at a reduced operating temperature (150°C) reduced the rutting resistance of the Advera®-modified binder. The poorer rut resistance was suspected to be due to the reduced production temperature rather than Advera®.

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Table 3.1 Results of Moisture Sensitivity and Rut Tests of Mixture Samples[a]

Sample	AS (Yes/No)	Compaction Temperature ($^oF[^oC]$)	ITS's (Wet / Dry)	ITS's Saturation (%)	ITS (psi)	TSR	VTM (%)	HWT cycles to 12.5 mm rut	HWT Stripping Inflection
Control	No	300[149]	Dry	NA	124.9	0.56	7.4	9,950	7,300
Control	No	300[149]	Wet	74.7	70.1				
Control	Yes	300[149]	Dry	NA	98.3	0.89	7.5	10,300	8,200
Control	Yes	300[149]	Wet	76.6	87.5				
Advera®	No	300[149]	Dry	NA	131.6	0.48	7.1	9,100	5,400
Advera®	No	300[149]	Wet	76.7	62.7				
Advera®	Yes	300[149]	Dry	NA	96.7	0.77	7.3	10,300	6,350
Advera®	Yes	300[149]	Wet	75.9	74.6				
Advera®	No	250[121]	Dry	NA	78.1	0.48	6.8	2,250	None
Advera®	No	250[121]	Wet	76.1	18.7				
Advera®	Yes	250[121]	Dry	NA	73.5	0.77	6.9	3,300	None
Advera®	Yes	250[121]	Wet	78.6	41.6				

[a]Asphalt mixes were tested by Dr. Cross and his team at Oklahoma State University laboratory (Hossain et a. 2009); AS = anti-stripping agent, $^oF = (9/5)^oC + 32$, 1 inch = 25.4 mm, 1 psi= 6.89 kPa, ITS = Indirect tensile strength, TSR = tensile strength ratio, VTM = void in total mix, HWT = Hamburg wheel-tracking test, NA = not applicable, None = sample rutted to 12.5 mm before showing any point of inflection.

Table 3.2 Continuous PG Grade of PG 64-22 with Different Dosages of Advera®

Amount of additive (%)	High critical temperature under unaged (^oC)	High critical temperature under RTFO-aged (^oC)	Low critical temperature from Stiffness (^oC)	Low critical temperature from m-value (^oC)	Continuous PG grade
0	64.8	66.0	<-24.8	-24.8	PG 64.8-24.8
4	67.0	66.1	<-22.0	-22.0	PG 67.0-22.0
6	67.2	66.2	<-21.9	-21.9	PG 67.2-21.9
8	67.8	68.5	<-20.9	-20.9	PG 67.8-20.9

Note: $^oF = (9/5)^oC + 32$; Number of replicates = 3

Table 3.3 Rotational Viscometer Results for Advera®

Temperature (°C)	Binder's viscosity with 0% Advera® (mPa-s)	Binder's viscosity with 4% Advera® (mPa-s)	Binder's viscosity with 6% Advera® (mPa-s)	Binder's viscosity with 8% Advera® (mPa-s)
135	438	490	519	556
150	221	258	271	296

Note: shear rate = 6.8 sec^{-1}. $^{o}F = (9/5)^{o}C + 32$; Number of replicates = 3.

Table 3.4 Aging Index of PG 64-22 with WMA and AS Additives

Testing temperature	Base binder	6% Advera®-modified binder	6% Advera® + 0.5% AD-here® HP Plus-modified binder
61°C	2.53	2.10	2.17
64°C	2.43	2.04	2.17
67°C	2.36	2.00	2.23
70°C	2.29	1.95	2.17

Note: $^{o}F = (9/5)^{o}C + 32$; Number of replicates = 3.

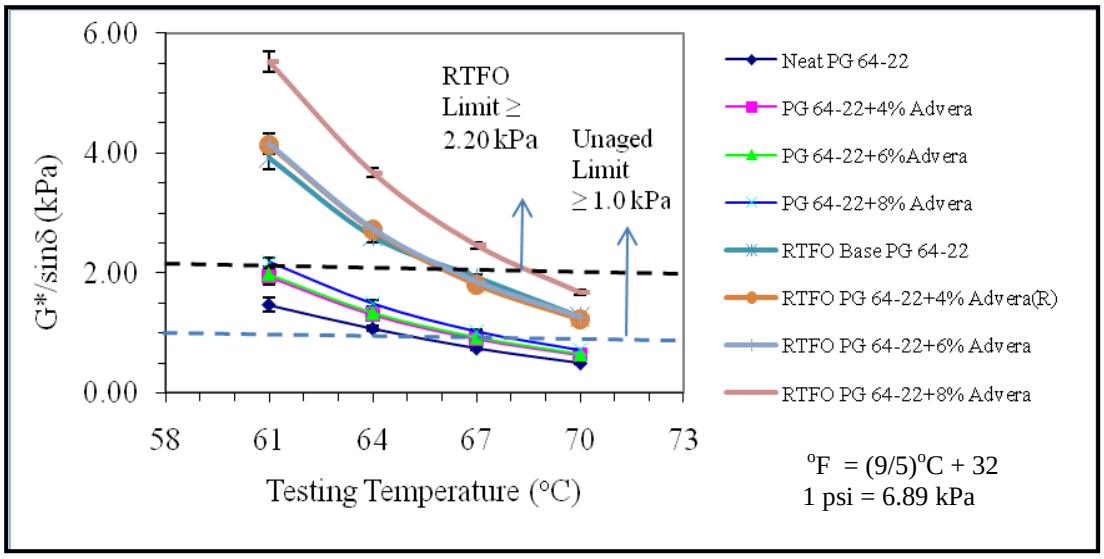


Figure 3.1 DSR Test Data of Advera® Samples.

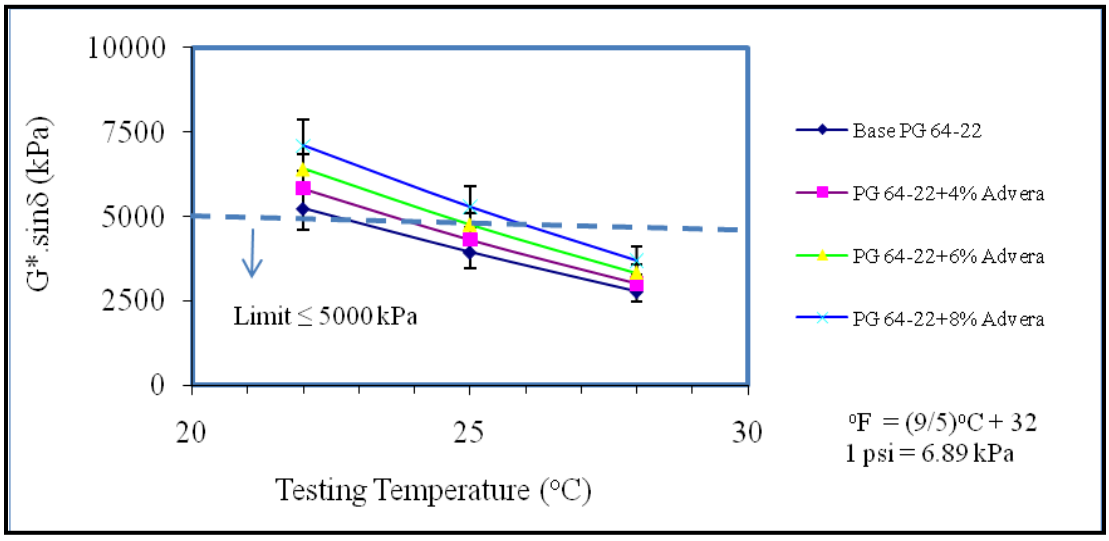


Figure 3.2 DSR Test Results for PAV-aged Advera® Samples.

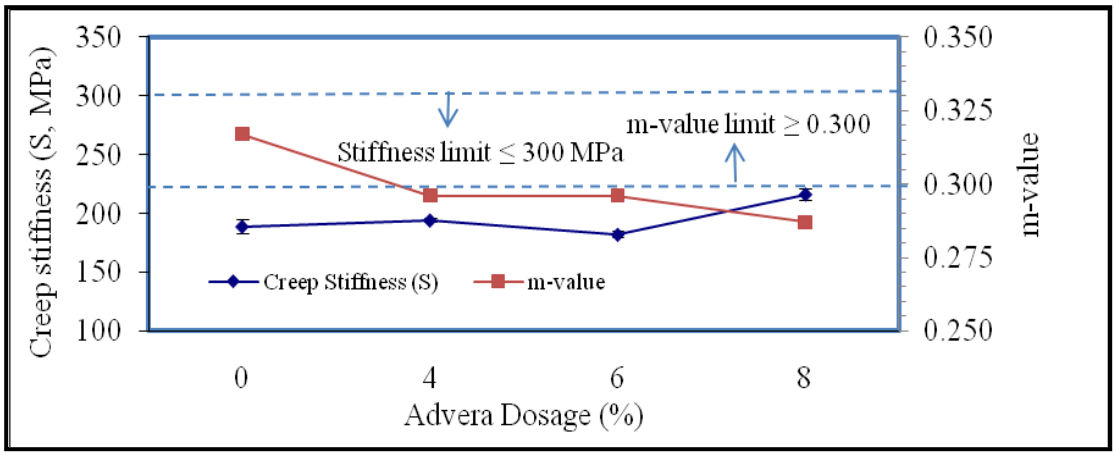


Figure 3.3 BBR Test Results with Advera® Samples.

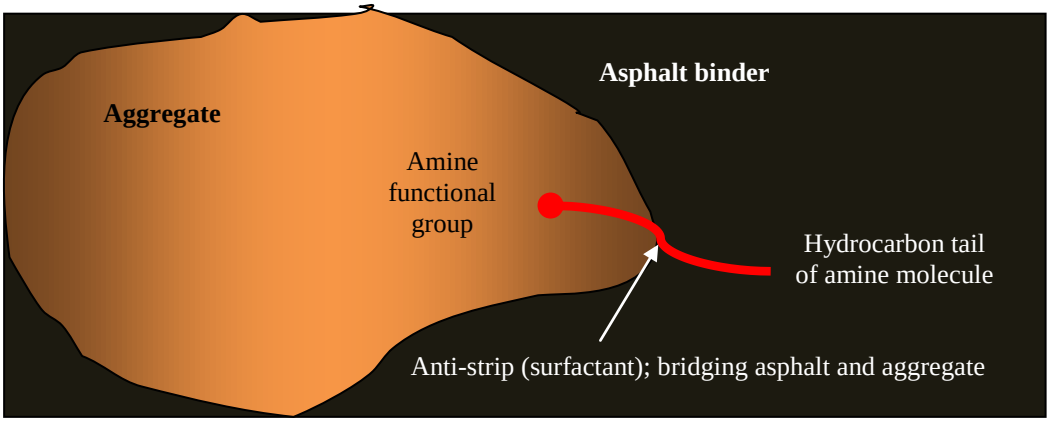


Figure 3.4 Amine-Based Liquid Anti-Stripping Additive and Surface Modification.

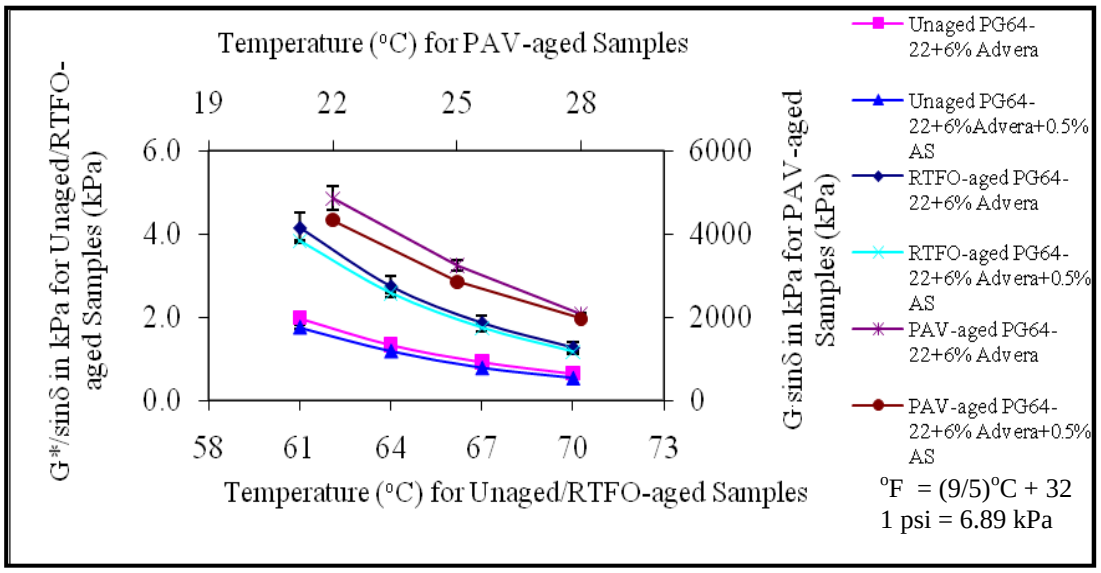
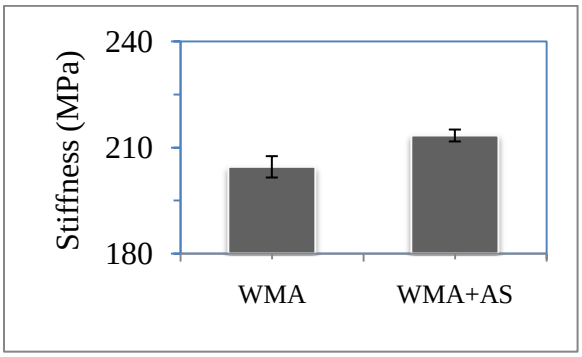
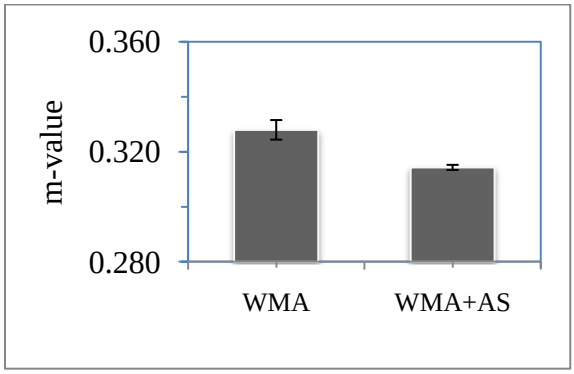


Figure 3.5 DSR Data of WMA And Anti-Stripping Additives Modified Binder.



(a)



(b)

Figure 3.6 BBR data of WMA and AS-modified Binders: a) Stiffness; b) m-value.

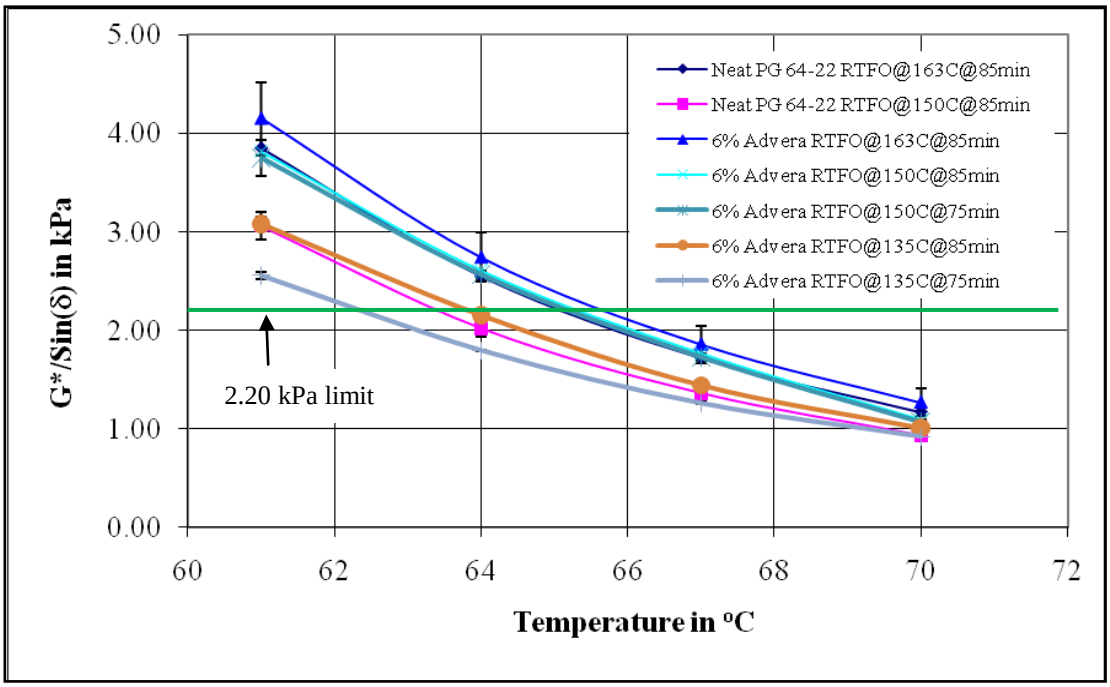


Figure 3.7 DSR Test Data of Binder Aged at Reduced Temperatures.

4 EFFECTIVENESS OF WARM MIX AND LIQUID ANTI-STRIPPING ADDITIVES ON PERFORMANCE GRADE BINDERS[3]

4.1 ABSTRACT

The effectiveness of a warm mix asphalt (WMA) additive, Sasobit®, and a liquid anti-stripping agent, AD-here® HP Plus, on a performance grade binder, PG 64-22, was evaluated in this study. Also, the effects of reduced rolling thin film oven (RTFO) temperature on the modified binder were investigated. The optimum dosage of Sasobit® was found to be 1.5% (by the weight of the binder), which was expected to reduce the mixing temperature by 9°C. It was also likely to increase the high PG temperature of the base binder by 4.5°C. The RTFO-aging at 150°C on the Sasobit®-modified binder predicted a significant reduction in the rut resistance. While a fairly small amount of AD-here® HP Plus was found to be effective in reducing the stripping potential, it was expected to increase the low temperature resistance of the binder. The current study is expected to enhance rheological database and help in implementing WMA mixes in Oklahoma.

KEYWORDS: WMA, Sasobit®, AD-here® HP Plus, RTFO, Performance Grade.

4.2 INTRODUCTION

The benefits of warm mix asphalt (WMA) technologies in the U.S. in terms of energy savings and air quality improvements are promising. WMA technologies can reduce the production temperature of the hot mix asphalt (HMA) by 16°C to over 55°C (Newcomb, 2010). The reduction in production temperatures leads to reduced

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emissions (i.e., volatile hydrocarbons and CO_2), dusts and production costs (de Groot et al., 2001; FHWA, 2010). WMA technologies can also extend the paving season in certain locations where the construction of the HMA is restricted to warmer months (Kristjánsdttir et al., 2007). However, laboratory and field data are significantly lacking in terms of rheological properties of modified binders as well as performance-related properties of WMA mixes for conditions in Oklahoma.

Lowering the viscosity of the asphalt mix is one of the most important attributes of WMA technologies (Hurley and Prowell, 2005; Goh et al., 2007; Kantipong et al., 2007). Among several available WMA technologies in the U.S., an organic additive named Sasobit® was evaluated in this study. Sasobit® is a mixture of long-chain hydrocarbon alkanes with chain lengths of 45 to 100 carbon atoms. It is produced from natural gas (methane), using the Fischer-Tropsch (FT) process (Sasolwax, 2008). It is identical to paraffin waxes that are found in crude oil, except that it has a higher molecular weight. For maximum effectiveness, the recommended dosage of Sasobit® is 0.8% to 3.0% (by the weight of the binder) (Sasolwax, 2008).

In practice, many agencies simply allow the addition of a WMA additive to an approved PG binder without accounting for possible grade changes to the base binder (Austerman et al., 2009). Previous studies (e.g., Hurley and Prowell, 2005; Butz, 2005; Austerman et al., 2009; Carter et al., 2010) demonstrated significant changes in both the high PG and the low PG temperatures of WMA additive-modified binders. The extent of changes in PG temperatures depends on the amount of additive being used. Another major concern for WMA stems from the argument that the lower production temperature would leave behind some superfluous moisture within the mix.

Among the detrimental influences of possible excessive moisture-induced damages, stripping (i.e., loss of strength through the weakening of the bond between the asphalt binder and the aggregate) is fairly common. The moisture damage potential of a mix is generally evaluated through the determination of tensile strength ratio (TSR), in accordance with AASHTO T 283 (Cross et al., 2000; Hurley and Prowell, 2005). The moisture damage potential can also be determined through the observation of point of inflection in Hamburg-wheel tracking (HWT) tests (Hurley and Prowell, 2005).

Hurley and Prowell (2005) at the National Center for Asphalt Technology (NCAT) studied the effects of Sasobit® on selected binders. In the NCAT study, the addition of 2.5% Sasobit® to a PG 58-28 produced a PG 64-22 binder. Rheological properties of the modified PG 64-22 binder were then compared with those of an unmodified PG 64-22 binder. From viscosity data, it was found that the compaction temperature for the Sasobit®-modified binder was approximately 18°C lower than that for the unmodified PG 64-22 binder. This study also observed low TSR values and visual stripping for both the control and Sasobit® mixes produced at warm temperatures (mixed at 135°C and compacted at 121°C). Compared to the control mix, Sasobit® was found to increase the TSR value from 0.65 to 0.91 for a limestone mix, whereas it decreased the TSR value from 0.76 to 0.71 for a granite mix. An anti-stripping (AS) agent named Kling Beta 2912 was used to improve the TSR value of the granite mix. Kling Beta 2912 (0.4% by the weight of the binder) was found to improve the TSR value of the Sasobit® mix to an acceptable performance level (i.e., from 0.71 to 0.94). The stripping inflection points from HWT test results for mixes with Sasobit® and Kling Beta 2912 were in agreement with the TSR values. It was

concluded that a lower production temperature in the WMA led to incomplete drying of aggregates and increased moisture damage.

Kanitpong et al. (2007) examined the effects of Sasobit® on the viscosity of selected binders. It was reported that 3% Sasobit® significantly reduced the viscosity of a polymer-modified asphalt binder. However, Sasobit® was found to be ineffective for an AC 60/70 binder (a penetration grade binder). These researchers also reported increased moisture induced damage in Sasobit® mixes and suspected that the reduced mixing temperature rather than Sasobit® caused the detrimental effects to the moisture induced damage.

In a related study, Wasiuddin et al. (2008) examined the effect of varying amounts of Sasobit® on the viscosity of two selected binders (PG 64-22 and PG 70-28). The viscosity of the PG 64-22 binder decreased with the addition of up to 2% Sasobit®, but it did remain unchanged with further increase of Sasobit® up to 4%. Thus, any amount Sasobit® from 2% to 4% led to a 16°C drop in the mixing temperature for the PG 64-22 binder. Comparatively, the amount of Sasobit® showed significant influence on the viscosity of the PG 70-28 binder. The mixing temperature of the PG 70-28 binder was expected to reduce by 10°C, 12°C, and 13°C, respectively, for 2%, 3%, and 4% Sasobit®. This study recommended additional testing to evaluate the allowable dosage of Sasobit®. Also, the effects of the AS agent on the viscosity and PG grade of the WMA-modified binder was out of scope of this study.

Austerman et al. (2009) observed changes in both PG temperatures of a Sasobit®-modified (1.5%) PG 64-28 binder by 6°C, thus the PG 64-28 binder resulted

in a PG 70-22 binder. On the other hand, an addition of 3% Sasobit®, the high PG temperature of the same binder increased by 6°C and the low PG temperature increased by 12°C, resulting in a PG 70-16 binder. Similar observations were made by Carter et al. (2010) while evaluating three selected binders (PG 58-28, PG 64-34, and PG 64-34 with crumb rubber) with 2% Sasobit®. Among these binders, the PG 64-34 binder was found to be most susceptible to viscosity reduction by 2% Sasobit®. Even though the high PG temperature of the PG 64-34 with 2% Sasobit® increased to 70.8°C, the low PG temperature of the binder decreased to -19.9°C. Because of the low PG temperature of the modified binder being too high, it was not acceptable based on the local climate condition and guidelines.

Bennert et al. (2010) evaluated the workability of a PG 76-22 binder modified with varying amounts of Sasobit® and reported unrealistic mixing and compaction temperatures for warm mix applications. The mixing and compaction temperatures of the PG 76-22 binder with 1.5% Sasobit® ranged from 163°C to 169°C, and from 150°C to 156°C, respectively. These mixing temperatures were significantly higher than those (the mixing temperatures ranged from 156°C to 161°C and the compaction temperature ranged from 145°C to 149°C) of the unmodified binder. These researchers also reported a slight increase in the high PG temperature with the addition of Sasobit®.

As explained earlier, moisture-induced damage in a WMA mix is a major concern. The degree of the moisture susceptibility problem can be mitigated by adding liquid AS agents (Hossain et al., 2009, 2011). AD-here® HP Plus is one of the liquid AS agents commonly used by the Oklahoma Department of Transportation (ODOT)

(ODOT, 2008). It can be added with binders at different stages: at refineries, at distribution centers, or at WMA plants as a batch or continuous process. Previous studies (Gore, 2003; Selvaratnam et al., 2007; Hossain et al., 2010) observed possible grade changes of selected PG binders due to the addition of AD-here® HP Plus in HMA. Therefore, it is important to evaluate the performance characteristics of a binder mixed with both Sasobit® and AD-here® HP Plus because such data do not exist in the public domain. To this end, in the present laboratory study, different dosages of Sasobit® in combination with AD-here® HP Plus were evaluated in terms of their contributions to the rheological properties of a selected PG binder. The specific objectives of this study are as follows:

- Evaluate the effect of Sasobit® on viscosity and performance characteristics of a selected PG binder.
- Investigate the effects of AD-here® HP Plus on the viscosity and performance characteristics of the Sasobit®-modified binder.
- Examine the effect of short-term aging on the rutting factor of the binder at a reduced rolling thin film oven (RTFO) operating temperature.

4.3 TEST MATERIALS AND EXPERIMENTAL DESIGN

Sasobit® in this study was obtained from the Sasol Wax plant in Richmond, California in the form of prills. Selective dosages (0%, 1%, 2%, 2.5%, and 3%) of Sasobit® were added to a PG 64-22 binder (lot number TK 118) obtained from Valero refinery in Ardmore, Oklahoma. The liquid anti-stripping additive, AD-here® HP Plus,

was obtained from Arr-Maz Custom Chemicals, Florida. The dosage level of AD-here® HP Plus was maintained constant (0.5% by the weight of the binder).

To achieve the objectives of this study, an experimental plan (Figure 4.1) was developed. Viscosity testing was conducted using a rotational viscometer (RV) in accordance with the AASHTO T316 test method. Viscosity measurements were taken in 15°C increments from 135°C to 165°C, and three replicates were tested for each combination. To determine the PG grade of the base binder with varying amounts of additives, the AASHTO R29 method was followed. The short-term aging of binder was simulated by using a rotational thin-film oven (RTFO) in accordance with AASHTO T 240, and the long-term aging was simulated using a pressure aging vessel (PAV) in accordance with AASHTO R 28. High PG temperatures of unmodified and modified binders were determined by performing dynamic shear rheometer (DSR) tests of unaged-, RTFO-, and PAV-aged binder samples in accordance with the AASHTO T 315 test method. Low PG temperatures of binder samples were validated by testing PAV-aged samples in a bending beam rheometer (BBR) in accordance with the AASHTO T 313 test method.

4.4 EVALUATION OF SASOBIT®

4.4.1 Viscosity

Viscosity data for the unaged binder with different amounts of Sasobit® is presented in Figure 4.2. The desired viscosity for proper mixing of an asphalt mix is 170 ± 20 mPa-s, measured in accordance with AASHTO T 316 (AASHTO, 2008). In the case of the base binder, the mixing temperature corresponding to a viscosity of 170

m mPa-s is found to be 158°C. The corresponding mixing temperature to achieve the same level of viscosity is found to be 153°C, 152°C, 150°C, and 147°C with 1%, 2%, 2.5%, and 3% Sasobit®, respectively.

The recommended mixing temperature for HMA mixes in Oklahoma is 163°C (ODOT, 2008). This mixing temperature was considered as a baseline to estimate the reduction in mixing temperature for the corresponding WMA mixes. At 163°C, the viscosity of the base binder was found to be 138 mPa-s. The corresponding temperatures for 1%, 2%, 2.5%, and 3% dosages of Sasobit® to achieve the same level of viscosity were 158°C, 154°C, 153.5°C, and 153°C, respectively. Thus, the expected reductions in the mixing temperature of the binder were 5°C, 9°C, 9.5°C, and 10°C with Sasobit® contents of 1%, 2%, 2.5%, and 3%, respectively. These results are similar to those reported in previous studies (e.g., Hurley and Prowell, 2005; Austerman et al., 2009).

4.4.2 Rut Resistance

The DSR test results for the Sasobit®-modified binder samples are presented in Figure 4.3. It is observed that the rutting factor ($G^*/\sin\delta$) increases significantly with an increase in the amount of Sasobit®, for both unaged and RTFO-aged conditions, indicating that the WMA mixes are expected to exhibit more rut resistance. These findings are consistent with the rutting behavior observed in the Hamburg wheel tracking (HWT) tests conducted on limited mixes with the same binder and WMA additive (1.5% Sasobit®). At a compaction temperature of 149°C, the number of cycles to 12.5 mm rut for the control and WMA mixes was reported as 9,950, and 10,500,

respectively. Prowell et al. (2007) also reported that field test trials of WMA mixes showed excellent rutting resistance.

Based on the Superpave® rutting factor criteria for unaged- ($G^*/\sin\delta \geq 1.00$ kPa) and RTFO-aged ($G^*/\sin\delta \geq 2.20$ kPa) conditions, high PG temperatures for 0%, 1%, 2%, 2.5%, and 3% Sasobit®-modified binders were found to be 64.5°C, 67°C, 70°C, 70.2°C, and 70.5°C, respectively. Similar observations were made by others researchers in their corresponding studies (Hurley and Prowell, 2005; Edwards et al., 2007; Austerman et al., 2009). However, these findings should be applied with a caution since the lower production temperatures of the WMA may further reduce the $G^*/\sin\delta$ value, resulting in reduced rut resistance of the mix. To test this hypothesis, additional tests have been conducted on binder samples aged (short-term) at a reduced RTFO operating temperature and discussed later (Section 5) in this paper.

The increase in the $G^*/\sin\delta$ values of Sasobit®-modified binders is related to chemical structure and compositions of the binder which are altered due to the addition of Sasobit®, as demonstrated by Loeber et al. (1998). These researchers observed that Sasobit® increased the concentration of asphaltenes in the binder which led the binder to exhibit more elastic behavior with a higher complex modulus (G*). In a separate study, it was indicated that the asphalt binder system was governed by a colloidal law, expressed in terms of an instability or colloidal index (Loeber et al., 1998; Lesueur, 2009). Lesueur (2009) defined the colloidal index as the ratio of the sum of the weight contents of the asphaltenes and flocculants (the part of the maltene generating asphaltenes flocculation) to the weight content of "peptidizing agents" (i.e.,

the molecules acting as a surfactant for asphaltenes dispersion). The "flocculants" would be a mix of saturates and aromatics, while the surfactants would be a mix of aromatics and resins (Lesueur, 2009). Sasobit® is expected to increase the asphaltene content, indicating associated increases in the colloidal index and complex modulus.

It is also observed that rutting factors of both modified and unmodified asphalt binders increase with short-term aging, irrespective of testing temperatures (Figures 4.3(a) and 4.2(b)). This is due to the fact that the RTFO aging changes the molecular size distribution of the asphalt binder (Amirkhanian and Kim, 2005). The molecular size of asphaltenes in an asphalt binder increases while resins and oils decrease when the binder is heated in the RTFO oven. Furthermore, the resins become asphaltenes when oxidized, creating a small increase in molecular weight, thus increasing the G*/sinδ value. Large increases in the G*/sinδ value from aging are also associated with binder hardening and pavement cracking. To test this hypothesis, the aging index has been created and is presented in Table 4.1. The aging index is the ratio of the G*/sinδ value of the binder at RTFO-aged condition to that at unaged condition (Edwards et al., 2007). Sasobit® was found to reduce the aging index of the binder, indicating a reduced hardening of the binder and an increased life of the pavement.

4.4.3 Fatigue Performance

From Figure 4.3(c) it is seen that the fatigue factor (G*.sinδ) of PAV-aged binder samples of the base PG 64-22 binder passes at 22°C, which is lower than its intermediate temperature (25°C). Samples with 1.0% and 2.0% Sasobit® passed at 25°C, while those with 2.5% and 3% Sasobit® passed at 28°C. Thus, it is evident that the fatigue fracture potential of the binder increases with an increase in the amount of

Sasobit®. However, the fatigue factors of base and Sasobit®-modified binders are within the Superpave® specified acceptable limit ($G^*.\sin\delta \leq 5000$ kPa).

4.4.4 Low Temperature Performance

BBR test results (Figure 4.4) at the low PG temperature (-12°C) of the binder show that the stiffness (S(t)) value increases significantly when the amount of Sasobit® is increased, specifically with 2% or higher. As the S(t) value increases, thermal stresses developed in the pavements increase and thermal cracking becomes more likely to occur. However, the Superpave® specified limiting stiffness of 300 MPa did not reach for the selected dosages of Sasobit® (up to 3%). From Figure 4.4 it is also evident that the rate of stress relaxation (m-value at t = 60 sec) decreases with the addition of Sasobit®. For 1% Sasobit®-modified binder samples, the m-value is found to be within the Superpave® specified limit (greater than or equal to 0.300). For 2% Sasobit®, the m-value is found to be 0.282, which is significantly lower than the acceptable limit. Thus, the m-value is found to control the optimum dosage of Sasobit® for this study.

Based on m-values, the low PG temperature of the Sasobit®-modified binder is expected to be higher (greater than -22°C) than that of the base binder. These findings are in agreement with in a recent study on a selected PG 64-28 binder with 1.5% and 3% Sasobit®, conducted by Austerman et al. (2009). Hurley and Prowell (2005) also reported a similar trend of the m-value for a PG 58-28 binder samples with 2.5% Sasobit®. This is due to the fact that at lower temperatures, non-polar molecules begin to re-organize into a structured form. Combined with the already structured polar

molecules, Sasobit® makes an asphalt binder more rigid which is likely to fracture rather than deform elastically under stresses. Thus, Sasobit® makes the binder more susceptible to low temperature cracking, as seen from the BBR test results.

Based on RV and DSR data discussed earlier (Sections 3.1 through 3.3), 1.5% of Sasobit® was expected to be effective in reducing viscosity and satisfactory against rutting and fatigue fracture re factors. To evaluate this dosage level of Sasobit® against the low temperature cracking performance, additional BBR tests were conducted for binder samples with 1.5% Sasobit®. The average m-value for these binder samples was found to be 0.296, which was slightly lower than the Superpave® criterion. However, Student's t-test (one-pair) at a confidence level of 95% showed the average m-value (0.296) did not differ significantly from 0.300. Therefore, 1.5% Sasobit® was recommended to be the optimum dosage for the selected binder, and further tests were carried out to evaluate the effects of the liquid AS agent.

4.5 EFFECT OF AD-HERE® HP PLUS

AD-here® HP Plus is an organic compound with a functional group containing a nitrogen (N) atom with a lone pair (valence electron) and at least one hydrogen (H) atom replaced with an alkyl or aryl group (hydrocarbons). It is a surfactant with a lyophobic amine group which is highly surface active. The "head" groups of this surfactant tend to diffuse through the lyophillic surface of the binder, while the lyophillic hydrocarbon chain ("tail" group) still remains in the asphalt binder (Figure 4.5). Thus, AD-here® HP Plus acts as a bridge between the asphalt binder and the aggregate surface which resist the action of water. The recommended dosage of AD-

here® HP Plus is 0.2 - 0.8% by the weight of the binder (Sasolwax, 2008). The amount (0.5%) of AD-here® HP Plus was used in the current study was based on the recommendations by ODOT and related studies (Selvaratnam et al., 2007; Hossain et al., 2010).

Moisture susceptibility was also a concern for control (HMA) and WMA mixes (Oklahoma S4 Insoluble) that used the same base binder (PG 64-22) and WMA additive (1.5% Sasobit®). As shown in Table 4.2, none of the mixes met the ODOT's minimum TSR requirement of 0.80 without the use of the AS agent. Adding 0.5% AS agent increased the TSR value of the WMA mix to 0.83. The TSR values of the control and WMA mixes cured and compacted at 149°C were found to be 0.56 and 0.73, respectively. At a lower curing and compaction temperature of 121°C, the TSR value of the WMA mix was also found to be 0.73. It is important to note that the indirect tensile strength (ITS) values at 121°C were also significantly lower than those at 149°C. The AS agent lowered the ITS value for the dry samples and increased the ITS value slightly for the wet samples, thus increasing the TSR value at 121°C.

From the HWT test results a significant increase in the stripping inflection point was observed when AS agent was added into the control mix. It was also observed that the rutting resistance of the WMA mix improved from that of the control mix when mixes were cured and compacted at 149°C (Table 4.2). However, the WMA samples cured and compacted at 121°C required considerably less cycles to 12.5 mm inch rut depth than those cured and compacted at 149°C. This could be related to the weakness in aggregate structure, inadequate binder stiffness, or moisture damage at low compaction temperature (Hossain et al., 2009). In regard to stripping point of

inflection, no well defined inflection point was observed for WMA mixes, indicating that the that the rutting observed was not due to moisture-induced damage but was due to weakness in the aggregate structure and inadequate binder stiffness.

Viscosity data (Figure 4.6) of the binder modified with 1.5% Sasobit® show that the mixing temperature can be reduced to 154°C, a 9°C reduction from the ODOT mixing temperature (163°C). From Figure 4.6, it is also seen that AD-here® HP Plus does not alter the beneficial effects (i.e., workability) of Sasobit®-modified binder at a low testing temperature (150°C or lower), which is realistic for the WMA technology. At a relatively higher testing temperature (150°C or higher), however, AD-here® HP Plus leads to a slight increase (statistically insignificant at 95% confidence level) of the viscosity of the base binder. Furthermore, it is observed that the viscosity of RTFO-aged PG 64-22 binder with 1.5% Sasobit® and 0.5% AD-here® is lower than the unaged counterpart at a testing temperature higher than 163°C. However, the viscosity of these RTFO-aged binder samples is higher than the unaged counterpart at a testing temperature of lower than 163°C. This behavior is expected because during the RTFO aging process the binder undergoes age hardening process at an operating temperature of 163°C. Thus, the RTFO-aged binder becomes more viscous than unaged samples at temperatures below 163°C.

The DSR test results of WMA along with AS-modified binders are presented in Figure 4.7. It is evident that the AS agent decreases the G*/sinδ values of the WMA-modified binder. For instance, at 64°C, AD-here® HP Plus decreases the G*/sinδ value of the WMA-modified binder (unaged condition) by 13% (Figure 4.6

and Table 4.3(a)). This is due to the fact that the AS agent is a low viscous (225 cps at 25°C) and low density (0.95 g/cc) liquid which makes the WMA-modified binder softer. When the AS is added, its primary amines (aka surface active agents) react with the carboxylic acids present in the binder and form corresponding salts that also act as an AS additive. The reduction in the viscosity of the modified binder increases the diffusivity of the amine molecules in the AS additive through the binder to the surface. As viscosity of the binder decreases, the complex modulus (G*) of the asphalt binder decreases, and so does the rutting factor. Correlating with the Superpave® limiting rutting factors for both unaged and RTFO-aged conditions, the high PG temperature for the WMA-modified binder is found to be 68.5^{o}C (minimum of 70°C and 68.5^{o}C) (Figure 4.7 and Table 4.3(b)). On the other hand, the high PG temperature for the same binder modified with the WMA along with the AS agent is 67°C (minimum of 67.5^{o}C and 67°C).

The effects of the AS agent on the fatigue factor (G*•sinδ) values of WMA-modified are also shown in Figure 4.7. It is observed that all the tested samples passed the Superpave® specified fatigue acceptance criterion at the intermediate temperature of 25°C. As presented in Table 4.3(c), the fatigue fracture potential of a mix with the WMA-modified binder is expected to be 15% higher than that of the base binder. When 0.5% AS agent is added with the WMA-modified binder, the fatigue fracture potential of the mix is expected to reduce by 6%.

As explained earlier, the stiffness (S(t)) of the binder increases and the rate of stress relaxation (m-value) decreases with an increase in the amount of Sasobit®. However, AD-here® HP Plus shows positive effects on the Sasobit®-modified binder

in terms of S(t) and m-values (Figure 4.8). When 0.5% AD-here® HP Plus is added with 1.5% Sasobit®-modified binder, the S(t) value decreased from 217 MPa to 209 MPa, and the m-value was found to increase from 0.296 to 0.301, meeting the Superpave® criteria. Thus, the low temperature resistance of a WMA mix is expected to increase when AD-here® HP Plus is added into a Sasobit®-modified binder.

4.6 EFFECT OF REDUCED RTFO OPERATING TEMPERATURE

High temperature and exposure to air are considered the leading factors for oxidation and hardening of an asphalt binder. However, the binder goes through less oxidation and hardening process in WMA mixes. This phenomenon can be simulated by reducing the RTFO operating temperature. Thus, a 13°C reduction in the RTFO operating temperature (150°C) was adopted while conducting the short-term aging on the binder. Subsequently DSR tests were conducted on PG 64-22 binder samples with and without Sasobit®. Findings of these DSR tests results are presented in Figure 4.9.

It is observed that the base PG 64-22 binder did not pass at 64°C when the RTFO aging was conducted at 150°C (Figure 4.9). The high PG temperature for the base binder (actual high PG temperature, 65°C) was found to be 61°C, a 4°C reduction in the high PG temperature. On the other hand, 3% Sasobit®-modified samples (actual high PG temperature 70°C) were able to maintain a high PG temperature of 67°C (i.e., a 3°C reduction from the actual PG temperature), when subjected to RTFO-aging at 150°C. It is also observed that by dropping the RTFO operating temperature by 13°C, the G*/sinδ value of the binder at 64°C is reduced by 21%, and 26% for the base PG 64-22, and 3% Sasobit®-modified binders, respectively. These findings are in

agreement with HWT test data for mixes explained earlier. The number of loading cycles required to reach 12.5 mm rut depth for the Sasobit® mix (mixed at 135°C and compacted at 121°C) and the control mix (mixed at 163°C and compacted at 150°C) were 3,850, and 10,500, respectively. As mentioned earlier, the base PG 64-22 binder failed its high PG temperature when RTFO-aged at 150°C. Thus, it was believed that the reduced production temperatures of Sasobit® mixes led to a reduced oxidation of the binder, thereby a reduced rut resistance. Sasobit® did increase the stiffness of the binder, but it was not enough to overcome the reduced oxidative hardening at reduced RTFO oven temperature.

4.7 CONCLUSIONS

The effects of varying dosages of a WMA additive (Sasobit®) on a selected PG binder (PG 64-22) were evaluated as per Superpave® specifications. Also, the effectiveness of a commonly used liquid anti-stripping agent (AD-here® HP Plus) on the Sasobit®-modified binder was investigated for local conditions in Oklahoma. Furthermore, the influences of a reduced RTFO operating temperature on stiffness of unmodified and modified binders were studied. Based on the results and analyses presented in the preceding sections, the following conclusions can be drawn from the results presented in the preceding sections.

- For any dosages of Sasobit® 2.0% and greater, the high PG temperature of the asphalt binder was increased from 64°C to 70°C, one grade higher than the high PG grade of the base binder. However, the rate of stress relaxation (m-value) at the

low PG temperature (-22°C) of the base binder was found to be inadequate to meet the Superpave® criterion.

- The optimum dosage of Sasobit® for the selected binder found to be 1.5% (by the weight of the binder). This dosage of Sasobit® was expected to reduce the mixing temperature by 9°C. The continuous PG grading of 1.5% Sasobit®-modified binder was found to be PG 68.5-22.

- A fairly small amount (0.5%) of AD-here® HP Plus did not show any adverse effects on the viscosity of the Sasobit®-modified binder. It was also expected to improve the fatigue fracture (reduced fatigue factor) and low temperature cracking potentials (decreased stiffness and increased m-value) without decreasing the rutting resistance significantly.

- A reduced RTFO operating temperature (150°C) on the Sasobit®-modified binder resulted in a reduced rut resistance, which indicated that the poorer rut resistance of the WMA mix was due to the reduced production temperature (i.e., lower aging) rather than Sasobit® itself.

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Table 4.1 Aging Indices of Sasobit®-Modified PG 64-22 Binder

Temperature	Aging Index				
	Base Binder	1% Sasobit®	2% Sasobit®	2.5% Sasobit®	3% Sasobit®
61°C	2.53	2.15	2.20	2.13	2.13
64°C	2.43	2.09	2.07	2.08	2.06
67°C	2.36	2.01	1.95	2.01	1.96
70°C	2.29	1.91	1.85	1.94	1.82

Table 4.2 Moisture Sensitivity Test Data of Mixes (Hossain et al., 2009a)

a) Indirect Tensile Strength					
Sample	AS	Compaction temperature (°C)	Dry/Wet (saturation)	ITS (psi)	TSR
Control	No	149	Dry	124.9	0.56
Control	No	149	Wet (74.7%)	70.1	
Control	Yes	149	Dry	98.3	0.89
Control	Yes	149	Wet (76.6%)	87.5	
Sasobit®	No	149	Dry	106.4	0.73
Sasobit®	No	149	Wet (74.0%)	78.1	
Sasobit®	Yes	149	Dry	67.7	0.83
Sasobit®	Yes	149	Wet (77.5%)	56.2	
Sasobit®	No	121	Dry	79.3	0.73
Sasobit®	No	121	Wet (73.3%)	59.2	
Sasobit®	Yes	121	Dry	74.8	0.83
Sasobit®	Yes	121	Wet (79.0%)	63.7	
b) Hamburg Wheel-Tracking Test					
Sample	AS	Compaction temperature (°C)	Voids in total mix (%)	Cycles to 12.5 mm rut depth	Stripping inflection point
Control	No	149	7.4	9950	7300
Control	Yes	149	7.5	10300	8200
Sasobit®	No	149	7.0	10500	None
Sasobit®	Yes	149	6.9	11600	None
Sasobit®	No	121	6.8	3850	None
Sasobit®	Yes	121	6.9	4650	None

[a]Mixes were tested by Dr. Cross's team at Oklahoma State University laboratory

Table 4.3 Summary of DSR test Results of WMA- and AS-Modified Binder

a) Changes in Rutting Factor (G*/sinδ) from Base PG 64-22		
Testing Temperature	WMA (Unaged)	WMA+AS (Unaged)
61°C	+17%	+6%
64°C	+23%	+10%
67°C	+29%	+14%
70°C	+21%	+14%
b) High PG temperature		
Aging Condition	WMA	WMA+AS
Unaged	70.0°C	67.5°C
RTFO	68.5°C	67.0°C
Uanged and RTFO	68.5°C	67.0°C
c) Changes in Fatigue Factor (G*•sinδ) from Base PG 64-22		
Testing Temperature	WMA	WMA+AS
22°C	+15%	+9%
25°C	+25%	+19%
28°C	+37%	+29%

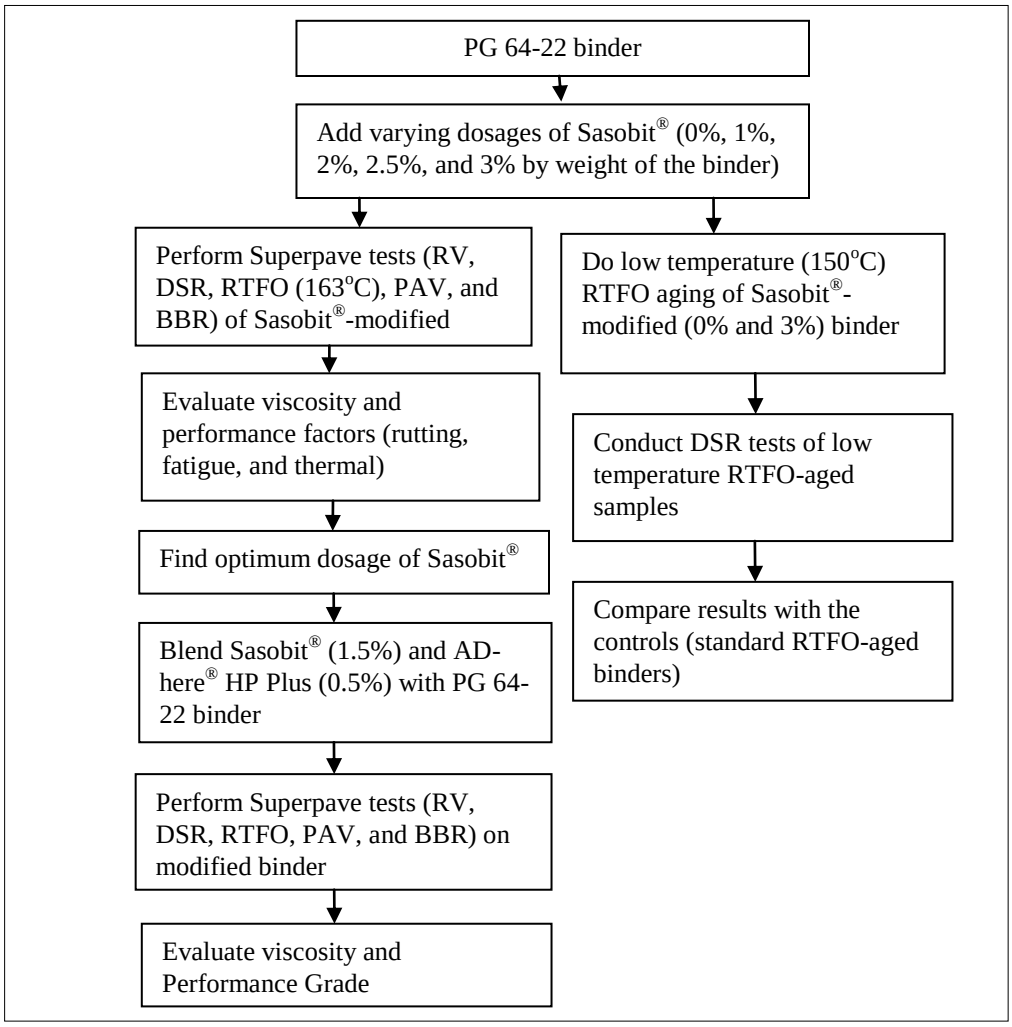


Figure 4.1 Experimental Design.

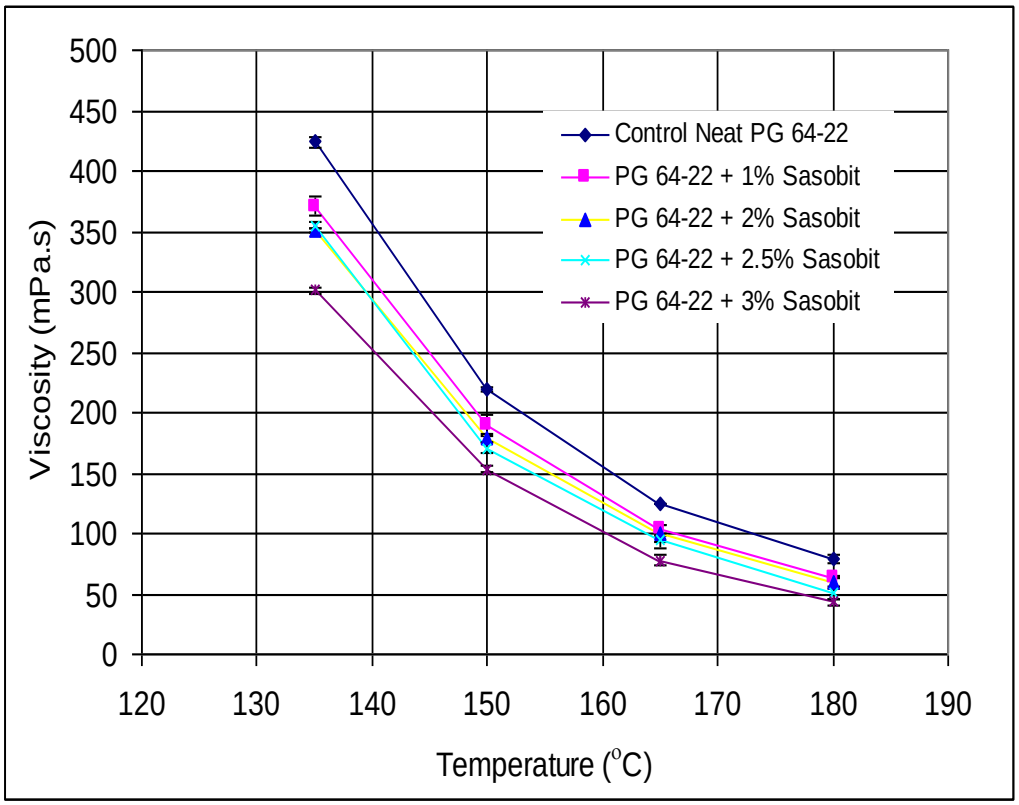
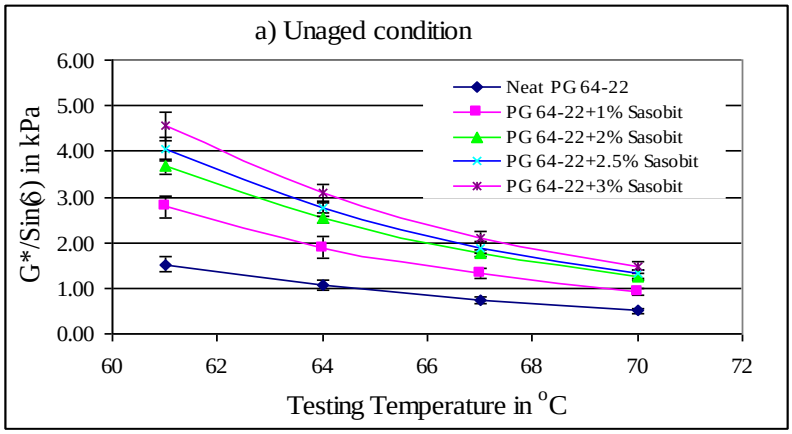
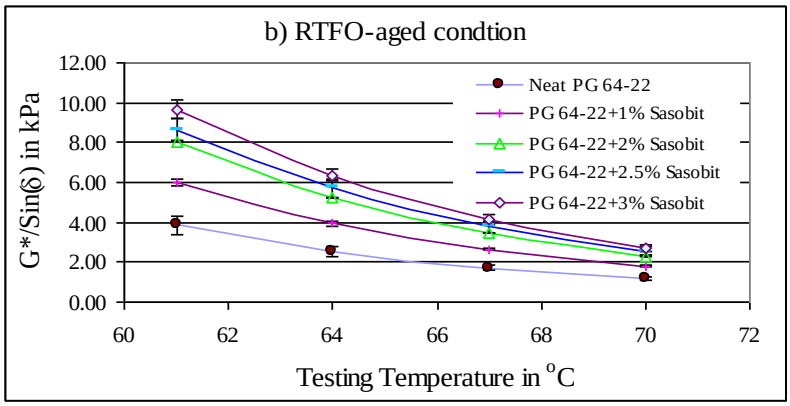


Figure 4.2 Rotational Viscosity of Sasobit®-modified Binder.





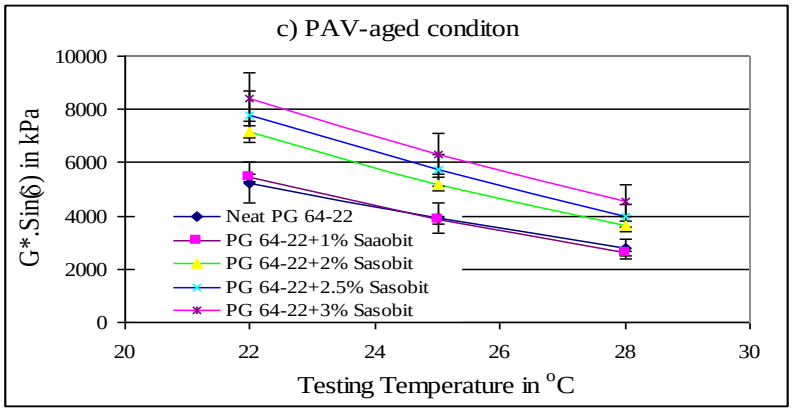


Figure 4.3 DSR Test Data of Sasobit®-Modified Binder Samples.

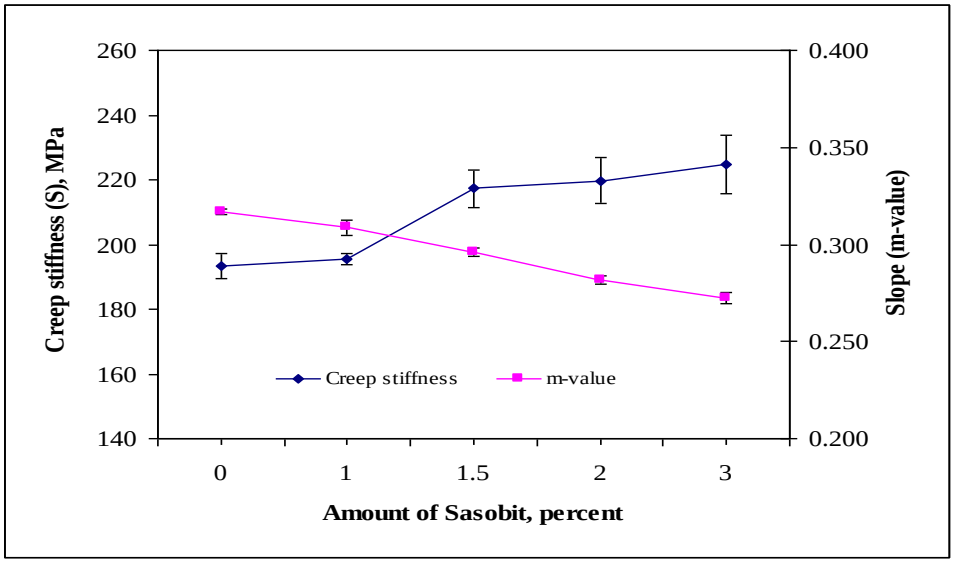
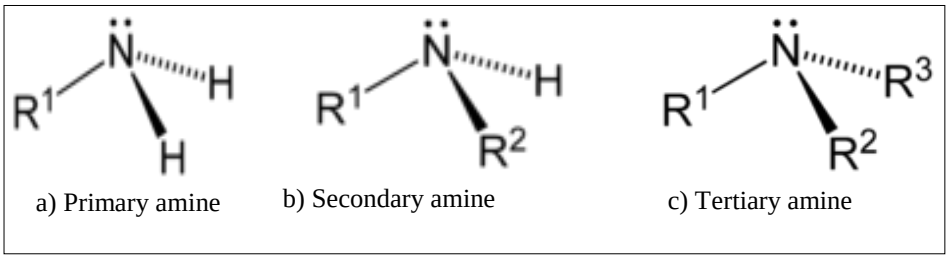
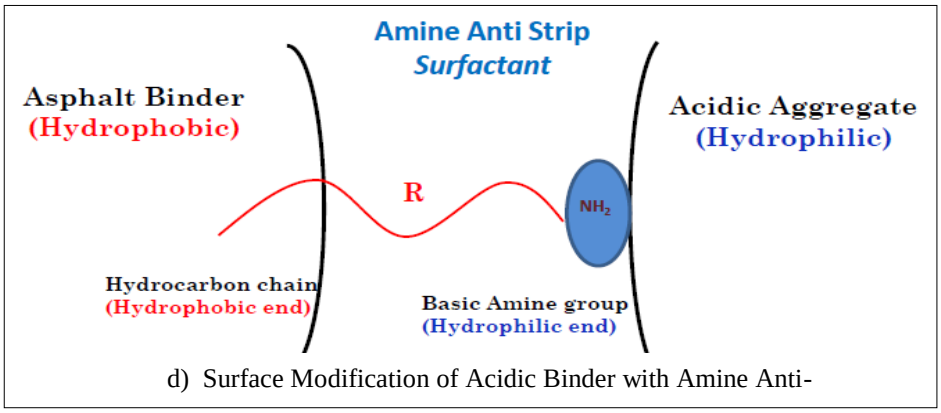


Figure 4.4 BBR Test Results of PG 64-22 Binder with Different Dosages of Sasobit®.



a) Primary amine b) Secondary amine c) Tertiary amine



d) Surface Modification of Acidic Binder with Amine Anti-

Figure 4.5 Functional Group of Amine-Based Liquid AS Agent and Surface Modification.

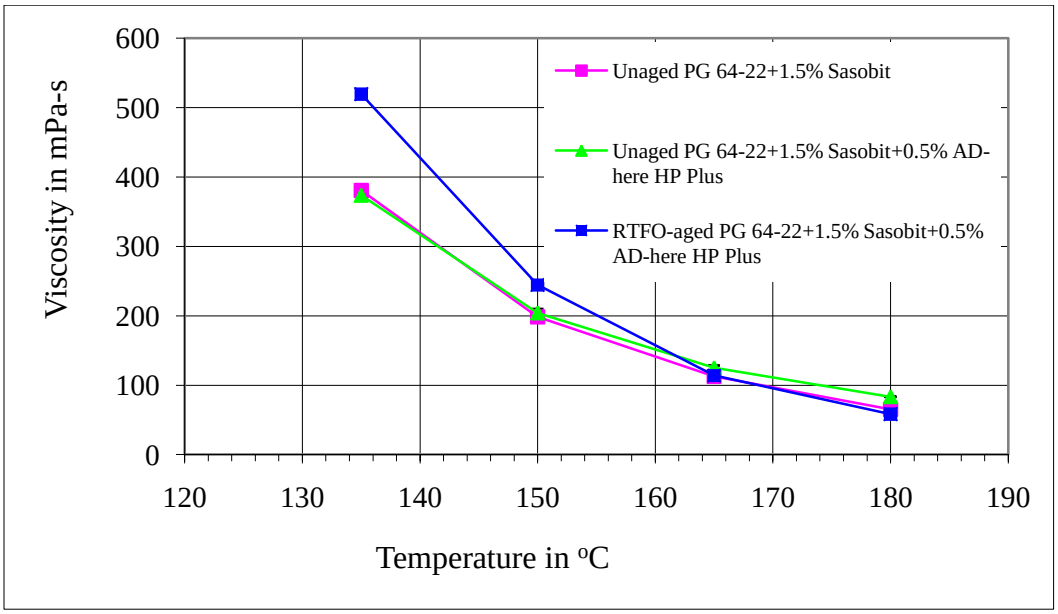


Figure 4.6 Effect of AD-here® HP Plus on Viscosity of Sasobit®-Modified Binder.

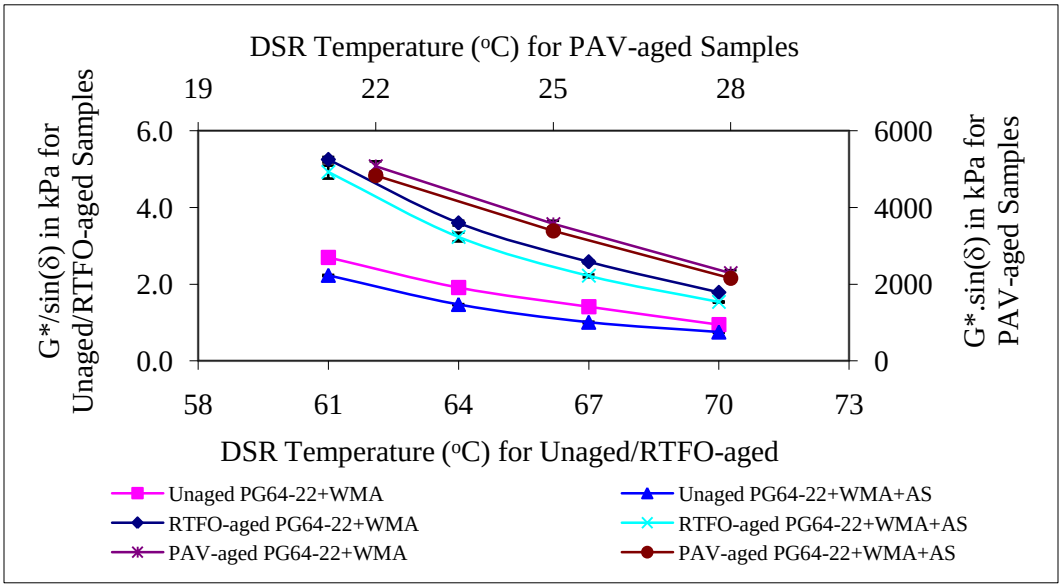
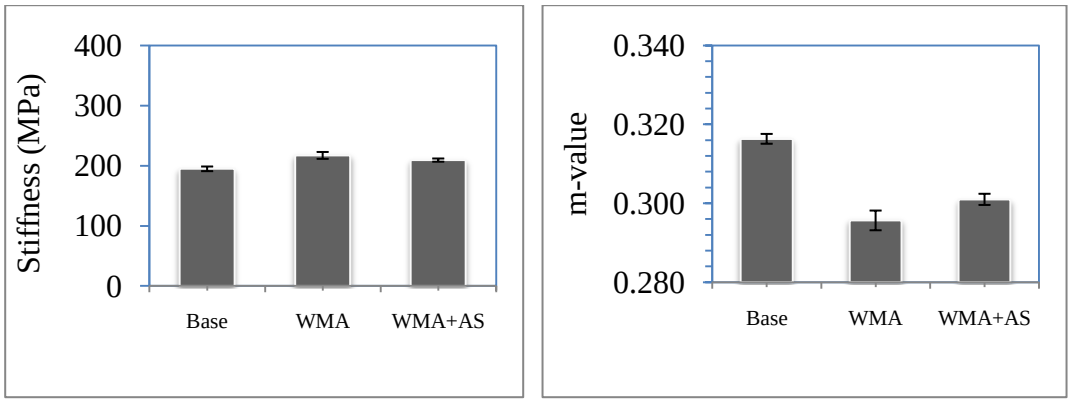


Figure 4.7 Effect of AS agent on Rutting and Fatigue Factors of WMA®-Modified Binder.



(a) (b)

Figure 4.8 BBR Test Results of WMA- and AS-Modified Binder at -12°C: (a) Stiffness, and (b) m-value.

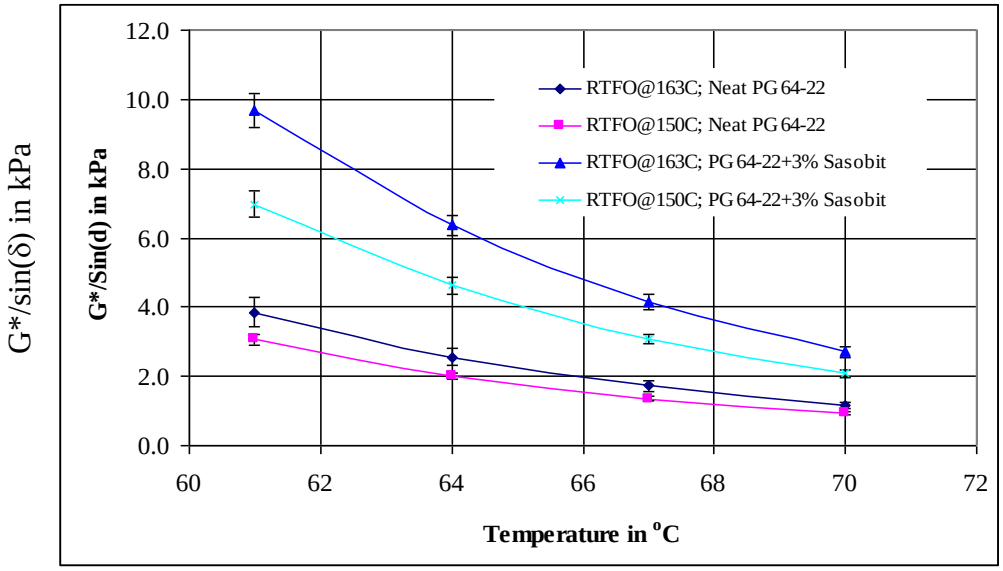


Figure 4.9 Effect of low RTFO Oven Temperature on G*/sinδ Values.

5 EVALUATION OF HIGH TEMPERATURE VISCOELASTIC CHARACTERISTICS OF WARM MIX ADDITIVE MODIFIED BINDERS AND PREDICTION OF DYNAMIC MODULUS OF MIXES [4]

5.1 ABSTRACT

This study was pursued to evaluate high temperature viscoelastic properties of a selected performance grade (PG) binder modified with different dosages of Sasobit®. The evaluated viscoelastic properties included viscosity, linear viscoelastic (LVE) limits, temperature susceptibility, and loading frequency dependency. The effects of reduced rolling thin film oven (RTFO) aging on the stiffness of the Sasobit®-modified binder were also evaluated. The binders' viscoelastic data were used to estimate dynamic modulus (E*) values of the warm mix asphalt (WMA) mixes using Witczak and Hirsch models. E* master curves of these mixes were then determined by following time temperature superposition (TTS) principles. Furthermore, the effects of AD-here® HP Plus, an anti-stripping agent (ASA), on the viscoeslatic properties of the modified binder were investigated. It was observed that LVE limits of Sasobit®-modified binders reduced with an increasing dosage of Sosobit®. The LVE limit of 3% Sasobit®-modified binder samples, under unaged condition, was found to be in compliance with the requirement. However, it was slightly lower than the required limit under the RTFO-aged condition. An amount of 3% Sasobit® was found to increase the high PG temperature of the PG 64-22 binder to 69°C. Reduced RTFO aging of the Sasobit®-modified binder was found to have significant effects on the binder stiffness. The high PG temperature of 1.5% Sasobit®-modified binder, RTFO-

[4] This chapter has been prepared for possible publication as a technical paper in ASCE Journal of Materials in Civil Engineering. The current version has been formatted for this dissertation.

aged at 120°C, was found to be about 4°C lower than that of the same binder RTFO-aged at 163°C. While predicting E* values of mixes, the Hirsch model, based on binder's frequency sweep test data, was found to provide better approximations of the E* master curves than the Witczak model. The Witczak model, based on dynamic shear rheometer (DSR) data, was found to significantly underestimate the E* values. While ASA did not reduce the beneficial effects of Sasobit®, it was found to increase the E* values of the WMA mix to some extent. The findings of this study are expected to provide transportation professionals a better understanding in evaluating WMA additive modified binders and mixes.

Keywords: warm mix asphalt, dynamic modulus, linear viscoelastic limit, anti-stripping agent, time temperature superposition.

5.2 INTRODUCTION

Warm mix asphalt (WMA) technologies allow a reduction in the temperatures at which asphalt mixes are produced and paved. The benefits of these technologies to the U.S. in terms of energy savings and air quality improvements are promising, but these technologies need further investigation and research in order to validate their expected performance and added value (FHWA, 2010). WMA technologies can reduce the production temperature of the hot mix asphalt (HMA) by 16°C to over 55°C (Newcomb, 2010). The reduction in production temperatures leads to reduced emissions, dusts, and production costs (FHWA, 2010). WMA technologies can also extend the paving season in certain locations where the construction of the HMA is restricted to warmer months (Kristjánsdttir et al., 2007). However, there is a lack of

information about the viscoelastic properties of modified binders as well as WMA mixes for conditions in Oklahoma.

Lowering the viscosity of the asphalt mix is one of the most important attributes of WMA technologies (Hurley and Prowell, 2005; Kantipong et al., 2007). The reduced viscosity allows the aggregate to be adequately coated at a lower temperature than what is traditionally required in HMA production. Among several available WMA technologies in the U.S., an organic additive named Sasobit®, a mixture of long-chain hydrocarbon alkanes with chain lengths of 45 to 100 carbon atoms, was evaluated in this study. It is produced from coal gasification using the Fischer-Tropsch (FT) process and is otherwise known as an FT paraffin wax (Sasolwax, 2008). Sasobit® is often described as a flow modifier or "asphalt flow improver" in WMA technology. For maximum effectiveness, the recommended dosage of Sasobit® is 0.8% to 3.0% (by the weight of the binder) (Sasolwax, 2008). The dosage level of Sasobit® should not exceed 4% due to the possible impact on the binder's low temperature properties (FHWA, 2010). A major concern about the detrimental effects of Sasobit®-modified WMA stems from the argument that the lower production temperature would leave behind some unwanted moisture within the mix, which might facilitate excessive stripping. A recent study by Hossain et al. (2009) reported a liquid ASA to be effective in increasing tensile strength ratio (TSR) of WMA mixes. On the other hand, Xiao et al. (2010) reported a liquid ASA exhibited weak moisture resistance compared to hydrated lime in the cases of WMA mixes.

Several researchers (e.g., Airey et al., 2002; Soleymani et al., 2004) noted that it is important to perform specification-related dynamic testing of an asphalt binder

within its LVE limit. The LVE limit of an asphalt binder is defined as the range of strain where the dynamic shear modulus (G*) value is at least 95% of the zero strain modulus. Zhai et al. (2006) observed reported that the LVE limits of selected emulsified asphalt binders were as low as 1% (strain). Clyne and Marasteanu (2004) also observed that heavily polymer-modified PG binders in Minnesota showed sharper reduction in the G* value with increasing strain. The sharp reduction of the G* value with increase in strain indicates that under increased strain in the pavement, the pavement may rut faster than a binder that does not lose stiffness as quickly. This may be the case for Sasobit®-modified binder, whose viscoelastic properties are expected to differ from the base binder. A recent study by Hossain and Zaman (2010) reported that ASA did not seem to reduce the LVE limit of a base PG 64-22 binder. These researchers, however, did not consider WMA additives in their respective studies.

In practice, many agencies simply allow the addition of a WMA additive to an approved PG binder without accounting for possible viscoelastic characteristics' changes to the base binder. Previous studies (e.g., Hurley and Prowell, 2005; Butz, 2005; Kanitpong et al., 2007; Austerman et al., 2009; Wasiuddin et al., 2008; Bennert et al., 2010; Carter et al., 2010) demonstrated significant changes in PG temperatures of WMA additive-modified binders. The extent of changes in PG temperatures depends on the amount of additive being used. Temperature sweep tests on asphalt binders can be used to approximate the temperature at which the asphalt material will satisfy the Superpave® specified rutting factor criterion. These data can also be used as input parameters in the new Mechanistic-Empirical Pavement Design Guide

(MEPDG). Edwards et al. (2005) performed temperature sweep tests on selected asphalt binders to evaluate the aging properties of wax-mix asphalts.

It can also be noted that the stiffness of an asphalt mix decreases as the loading time increases or the loading frequency decreases. The mix's dynamic modulus (E*) value can reduce as much as a factor of ten when the loading frequency is reduced from 10 Hz to 0.01 Hz. The corresponding G* value of the binder will exhibit a similar frequency dependency (Walker and Buncher, 1999). Thus, G* values from frequency sweep tests can be used to predict E* values of the mix (Christensen et al., 2003).

As mentioned earlier, asphalt binder's rheological data are input parameters in the new MEPDG, which is believed to be less empirical than the widely used 1993 AASHTO Design Guide for Pavement Structures. At the lowest (*Level 3*) design reliability, an asphalt binder's PG grade is used as input, which estimates the E* master curve for the design life of pavement. In the estimation process of the E* master curve, the MEPDG uses typical ASTM A and VTS (Viscosity Temperature Susceptibility) parameters. To obtain intermediate level (*Level 2*) reliability, the asphalt binder's viscoelastic properties are used as input. These viscoelastic properties include G* and phase angle (δ) values of RTFO-aged asphalt binder, determined by using a dynamic shear rheometer (DSR), in accordance with AASHTO T 315. Alternatively, rotational viscometer (RV) data (in accordance with AASHTO T 316 with a shear rate of 6.8 sec^{-1}) with some other conventional binder test data (flash point, and absolute and kinematic viscosities) can be used as input. These binder test data are used with simple volumetric properties of the asphalt mix to estimate E*

master curves. Expensive and time consuming laboratory E* test data are not needed at either Level 3 or Level 2. The commonly used processes for the estimation of E* values are explained next.

The Witczak's predictive equation (Equation 5.1), adopted in the MEPDG, is used to estimate the E* values of a mix. In Equation 5.1, the asphalt binder viscosity (η) is the measurements of RV test of the RTFO-aged binder. The η value can also be determined by using Equation 5.2 if the binder G* and δ values are known (Bari and Witczak, 2006). Once the η value is determined, the ASTM viscosity temperature susceptibility (VTS) parameters, shown in Equation 5.3, can be found by a linear regression analysis after a log-log transformation of the viscosity and log transformation of the temperature data (Bari and Witczak, 2006). In case neither the required DSR nor RV test data of asphalt binder is available, the predictive equation (Equation 5.1) can be used to estimate E* value of the mix by using typical ASTM A and VTS parameters based on the binder's PG grade.

$$\log E^* = 1.249937 + 0.249937 + 0.02932P_{200} - 0.001767(P_4)^2 - 0.002841P_4 - 0.058097V_a - 0.802208\left(\frac{V_{beff}}{V_{eff}+V_a}\right) + \frac{3.871977 - 0.0021P_4 + 0.003958P_{38} - 0.000017(P_{38})^2 + 0.00547P_{34}}{1+e^{(-0.603313-0.313351\log(f)-0.393532\log(\eta))}} \quad (5.1)$$

where,

E* = dynamic modulus (10^5 psi),

η = asphalt binder (RTFO-aged) viscosity at temperature of interest (10^6 Poise),

f = loading frequency (Hz),

V_a = air void content (%),

V_{beff} = effective asphalt content (% by volume),

P_{34} = cumulative % retained on 3/4 in (19 mm) sieve,

P_{38} = cumulative % retained on 3/8 in (9.5 mm) sieve,

P_4 = cumulative % retained on #4 (4.76 mm) sieve, and

P_{200} = % passing #200 (0.075 mm) sieve.

$$\mu = \frac{G^*}{10}\left(\frac{1}{\sin\delta}\right) \tag{5.2}$$

where,

η = asphalt viscosity (cP),

G^* = binder complex shear modulus (Pa), and

δ = binder phase angle (^{o}).

$$\log\log\mu = A + VTS\log T_R \tag{5.3}$$

where,

η = asphalt viscosity (cP),

A, VTS = regression parameters, and

T_R = temperature (°Rankine).

Another simplistic micromechanical model, introduced by T. J. Hirsch in the 1960s, estimates the HMA modulus based on a law of mixtures for composite materials (Christensen et al., 2003; Al-Qadi et al., 2009). In applying the Hirsch model to HMA, Christensen et al. (2003) developed a model (Equation 5.4) to predict the E* values from the G* values of the binder and easily measured volumetric properties of

the mix. The G* values from frequency sweep tests at different temperatures are used to predict E* values at corresponding frequency levels and temperatures.

$$|E^*|_{mix} = P_c\left[4{,}200{,}000\left(1-\frac{VMA}{100}\right)\right]+3\times|G^*|_{binder}\left(\frac{VFA\times VMA}{10{,}000}\right)+ \left(1-P_c\right)\left[\frac{1-\frac{VMA}{100}}{4{,}200{,}000}+\frac{VMA}{VFA\times 3\times|G^*|_{binder}}\right]^{-1} \quad (5.4)$$

where,

$|E^*|_{mix}$ = compressive dynamic modulus of mix (psi),

$|G^*|_{binder}$ = dynamic shear modulus of binder (psi),

VMA = voids in mineral aggregate (%),

VFA = voids in aggregate filled with mastic (%), and

P_c = Aggregate contact volume, as shown in Equation 5.5.

$$P_c = \left[\frac{\left(20+\frac{VMA\times 3\times|G^*|_{binder}}{VFA}\right)^{0.58}}{650+\left(\frac{VFA\times 3\times|G^*|_b}{VMA}\right)^{0.58}}\right] \quad (5.5)$$

The E* values at different temperatures and frequencies are used to develop master curves at a reference temperature, which is generally 70°F (21.1^{o}C) (Bari and Witczak, 2006). Master curves are constructed using the time-temperature superposition (TTS) principles. The E* values at various temperatures are shifted with respect to time until the curves merge into a single smooth sigmoidal function, as given in Equation 5.6.

$$\log[E^*] = \delta + \frac{\alpha}{1 + e^{\beta + \gamma(\log t_r)}} \quad (5.6)$$

where,

E* = mix's dynamic modulus (psi),

t_r = reduced time of loading at reference temperature (sec),

δ = minimum value of E*,

$\delta + \alpha$ = maximum value of E*, and

β, γ = parameters describing the shape of the sigmoidal function.

The shift factor can be shown in the form as shown in Equation 5.7.

$$a(T) = \frac{t}{t_r} \quad (5.7)$$

where,

a(T) = shift factor as a function of temperature,

t = time of loading at desired temperature,

t_r = reduced time of loading at reference temperature, and

T = temperature of interest.

For precision, a second order polynomial relationship between the logarithm of the shift factor and the temperature in degrees Fahrenheit as shown in Equation 5.8, is used (Bari and Witczak, 2006).

$$\log a(T_i) = aT_i^2 + bT_i + c \quad (5.8)$$

where,

$a(T_i)$ = shift factor as a function of temperature T_i,

T_i = temperature of interest (°F), and

a, b and c = coefficients of the second order polynomial.

As mentioned earlier, moisture-induced damage is a major concern for a WMA mix. The extent of the moisture susceptibility problem can be mitigated by adding a liquid ASA (Hossain et al., 2009, 2011). AD-here® HP Plus is a common liquid ASA, used by the Oklahoma Department of Transportation (ODOT) (ODOT, 2008). It can be added with binders at different stages: at refineries, at distribution centers, or at plants as a batch or continuous process. Previous studies (Gore, 2003; Selvaratnam et al., 2007; Hossain et al., 2010) observed possible grade changes of selected PG binders due to the addition of AD-here® HP Plus in HMA. However, none of these studies attempted to evaluate the possible change in LVE limits. Also, there exists only a limited research that has estimated and predicted E* value of WMA mixes modified with a liquid ASA.

5.3 OBJECTIVES

Major objectives of this study were:

- Evaluate the effects of Sasobit® on LVE limits and performance characteristics at high PG temperature of a selected PG binder.
- Evaluate the effects of reduced RTFO operating temperature on the stiffness of the Sasobit®-modified binder.
- Estimate the E* values of WMA mixes from viscoelastic properties of Sasobit®-modified asphalt binders.
- Assess the effects of AD-here® HP Plus on the E* values of WMA mixes.

5.4 MATERIALS AND METHODOLOGY

5.4.1 Materials

Selective dosages (0%, 1%, 2%, and 3%) of a commonly used additive called Sasobit® were added with a PG 64-22 binder, which was obtained from Valero refinery in Ardmore, Oklahoma. Sasobit® used in this study was obtained from the Sasol Wax plant in Richmond, California in the form of prills. The liquid anti-stripping additive, AD-here® HP Plus, was obtained from Arr-Maz Custom Chemicals, Florida. The dosage level of AD-here® HP Plus was maintained constant (0.5% by the weight of the binder).

5.4.2 Mixing Additives

While mixing different dosages of Sasobit® with the PG 64-22 binder, ODOT's test specifications "OHD L-36: Method of Test for Retained Strength of Bituminous Paving Mixtures," were followed [ODOT, 2010]. Before mixing, the base binder was heated for two hours at 135°C. Sasobit® prills were then poured in the heated binder, and the blend was stirred manually for 30 seconds. The blend was then kept in the pre-heated oven at 135°C for an hour, and it was stirred for 30 seconds at ten minutes intervals. In the case of both Sasobit® and ASA modification of the binder, both additives were blended with the base binder at the same time to minimize the age hardening and duplicate the plant mixing process. The blended binder was then kept overnight at room temperature for further testing.

5.4.3 Test Methods

5.4.3.1 Superpave® Tests

To achieve the objectives of this study, several binder test protocols were followed. Dynamic sweep tests (strain, temperature and frequency) were conducted by using a dynamic mechanical analyzer (DMA). To determine the PG grade of the base binder with varying amounts of additives, the AASHTO R29 method was followed. The short-term aging of binder was simulated by using a RTFO in accordance with AASHTO T 240. High PG temperatures of unmodified and modified binders were determined by performing DSR tests of unaged, and RTFO-aged binder samples in accordance with the AASHTO T 315 test method. At least three replicate samples were tested at each testing temperature to ensure the repeatability of test results.

5.4.3.2 Dynamic Modulus of Mix

Volumetric properties and E* test (AASTHO TP 62) data of asphalt mixes with and without the additives, presented in Table 5.1, were conducted by a collaborative research team at Oklahoma State University and reported in Hossain et al. (2009). The E* test specimens were prepared from cylindrical samples, which were mixed at 163°C and compacted at 149°C. Aggregates used in the mix design were predominately granite from Snyder quarry in south-west Oklahoma. Both the HMA (the control) and WMA mixes were designed as a surface mix (Oklahoma S4 mix) with a nominal maximum size (NMS) of 3/8 inch (9.5 mm) and a binder content of 5.3%.

5.4.3.3 Dynamic Sweep Tests

Strain Sweep -In a strain sweep test, a frequency level of 1.59 Hz was kept constant while the oscillation amplitude followed an increasing progression. The sample was loaded at 58°C. After maintaining 5-min thermal equilibrium for the testing temperature, the sample was pre-sheared for one minute at a strain level of 0.1% and a frequency level of 1.59 Hz. During the testing phase, the sample was subjected to strains ranging from 0 to 51%.

Temperature Sweep - In a temperature sweep test, the frequency and oscillation amplitude were kept constant, while the temperature followed an increasing progression. The effect of high temperatures on the unmodified and modified binder samples was examined for a range of temperature from 58°C to 73°C with increments of 3°C. Samples were loaded and trimmed at 58°C, followed by a 30-second pre-shearing at a strain level 1% and a frequency level of 1.59 Hz. During the temperature sweep test, a 5-min thermal equilibrium was maintained at each data collection point. Samples were tested using the progression going from low temperatures to high temperatures.

Frequency Sweep -It is known that several factors including aggregate type and characteristics, compaction effort, binder type, and binder content contribute to the E* value of a mix. For simplicity, frequency sweep tests on RTFO-aged binder samples were performed to correlate G* values of binders with E* values of corresponding mixes. In a frequency sweep test, the loading frequency ranged from 25 Hz to 0.1 Hz. Samples were pre-conditioned at a frequency of 25 Hz, and a

temperature of -6.7°C. Samples were then tested at -6.7°C, 4.4°C, 21.1°C, 37.8°C, and 54.4°C. At each testing temperature, a 5-min thermal equilibrium was maintained.

5.5 ASPHALT BINDER TEST RESULTS

As mentioned earlier, the main goal of WMA technology is to mix and compact the asphalt mix at relatively lower temperature than traditional HMA mixes without compromising the durability or performance of the pavement throughout its service life. The Sasobit® technology achieves this goal by lowering the viscosity of the binder and allowing a better coating of aggregates during mixing at low temperatures. The following subsection explains viscoelastic properties of Sasobit®-modified binders in terms of their viscosity measurements, LVE limits, temperature susceptibility and frequency dependency.

5.5.1 Viscosity

It should be noted that the Superpave® mixture design requires that gyratory specimens be mixed and compacted at equiviscous binder temperatures corresponding to viscosities of 0.170 and 0.280 Pa.s, as recommended in ASTM D4293 (ASTM, 2009). Using these viscosity values as baselines, the mixing and compaction temperatures for the base binder were found to be 168°C and 154°C, respectively (Figure 5.1a). The equiviscous mixing temperatures for 1%, 2%, and 3% Sasobit®-modified binders were found to be 161°C, 156°C, and 152°C, respectively. Thus, 1%, 2%, and 3% Sasobit®-modified binders were expected to reduce the mixing temperature by 7°C, 12°C, and 16°C. On the other hand, the equiviscous compaction temperatures for 1%, 2%, and 3% Sasobit®-modified binders were found to be 147°C,

140°C, and 135°C, respectively. So, the 1%, 2%, and 3% Sasobit®-modified binders were expected to reduce the compaction temperatures by 7°C, 14°C, and 19°C, respectively. It should be noted that ODOT recommends the mixing and compaction temperatures for the PG 64-22 be 163°C and 149°C, respectively. Considering the ODOT recommended mixing temperature as the baseline, relevant studies (Sneed, 2007; Hossain et al., 2009) reported that 1%, 2%, and 3% Sasobit®-modified binders would reduce the mixing temperature by 4°C, 5°C, and 10°C, respectively. On the other hand, 1%, 2%, and 3% Sasobit®-modified binders were expected to reduce the ODOT recommended compaction temperature by 5°C, 8°C, and 13°C, respectively.

It is also worthwhile to emphasize that ASTM D2493 was established for unmodified asphalt binders, which are Newtonian fluids at high temperatures. The viscosity of a polymer-modified binder generally changes with a change in shear rate. To verify that the Sasobit®-modified binders followed the characteristics of a Newtonian fluid, their viscosities were measured for a shear rate ranging from 5 sec^{-1} to 500 sec-^{1} using the DMA. It is clear from Figure 5.1b that the viscosity values of these binders, at 135°C, did not change notably with an increase in shear rate. A slight decrease in viscosity with an increase in shear rate was possibly caused by the shear thinning behavior of theses binders.

5.5.2 Strain Sweep

The applied strain rates versus G* values of different dosages of Sasobit®-modified binders under unaged and RTFO-aging conditions are shown in Figures 5.2a and 5.2b, respectively. The base binder, under unaged condition, exhibited the LVE behavior (i.e., strain corresponding to the 95% of the initial G*) up to the tested strain

level of 51%. This behavior is observed because the PG 64-22 binder behaves like a Newtonian fluid above 50°C (Airey, 2002). However, Sasobit® changes the LVE limit of the base binder as the G* value reduces with an increase in the strain. The LVE limits for 1%, 2%, and 3% Sasobit®-modified binders, under the unaged condition, were found to be 48%, 25%, and 16%, respectively. These LVE limits are well above the Superpave® specified AASHTO T 315 strain limit of 12% for unaged binders. On the other hand, the LVE limits for 0%, 1%, 2%, and 3% Sasobit®-modified binders, under RTFO aging condition, were found to be 44%, 29%, 13%, and 8%, respectively. The low LVE limit of the 3% Sasobit®-modified binder, under RTFO-aged condition, is somewhat lower than the AASHTO T 315 strain rate of 10%. Thus, in the case of a high dosage (3% or above) of Sasobit®-modified binder, under the RTFO-aging condition, it is recommended to perform specification tests at a strain rate that does not exceed the actual LVE limit of the modified binder.

5.5.3 Temperature Sweep

The G*/sinδ (rutting factor) values for the Sasobit®-modified binders under unaged and RTFO-aged conditions are plotted against the testing temperatures in Figures 5.3a and 5.3b, respectively. It is evident that the G*/sinδ value decreases with increasing temperature, as expected. At a low temperature, the asphaltenes in asphalt binder are able to form a compact structure, whereas at high end of the testing temperature they disperse as free particles. With the addition of Sasobit®, the G*/sinδ value increased compared to the base binder, and the curves shifted upward. The higher the dosage level of Sasobit®, the larger the G*/sinδ value. It is also observed

that DSR test data under the RTFO-aging condition governed the high PG temperatures of the base binder, whereas those under the unaged condition dictated the high PG temperatures of Sasobit®-modified binders. The high PG temperature for the base binder was found to be 64.7°C. As shown in Table 5.2, the corresponding high PG temperatures for 1%, 2% and 3% Sasobit®-modified binders were found to be 66.5°C, 67.5°C and 69.0°C, respectively. Thus, at the highest tested dosage (3%) of Sasobit®, the high critical temperature was found to be 5°C higher than the high PG temperature of the base binder. The increase in stiffness of the base binder is due to the fact that Sasobit® is a synthetic wax that forms crystal structures in the binder. The shapes of these structures in the modified binders, based on morphology analyses using a Differential Scanning Calorimetry (DSC), vary from tiny "needles" to elongated "needles," flakes and even crescent shaped structures, as observed by Lu et al. (2005). Similar morphological observations were made by Loeber et al. (1995) in their corresponding study, which observed nano-scale "bee" structured surface roughness on the surface of the asphalt binders using Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM). From aforementioned findings it appears that the high PG temperatures of Sasobit®-modified binders are expected to increase. However, these findings should be applied with caution as the actual plant mixing temperature of WMA is significantly lower than standard RTFO temperature. At a reduced RTFO temperature, the high PG temperature of the Sasobit®-modified binders is expected to experience less age-hardening processes, which can result in a reduced high PG temperature.

Viscosity-temperature susceptibility (VTS) properties of the base and Sasobit®-modified binders are presented as the double logarithm of viscosity versus logarithm of temperature in degree Rankine, as shown in Figure 5.3c. DSR test data were used to calculate viscosities of these binders by using Equation 5.2. As seen in Figure 5.3c, VTS (slope) value of the base binder is slightly larger than the Sasobit®-modified binder, indicating the latter is less susceptible to temperature than the former. Such characteristic is expected as the Sasobit®-modified binders are comparatively stiffer that the base binder at high service temperatures of the pavement. To further compare the aging, the aging indices of Sasobit®-modified binders were computed, as shown in Table 5.3. The aging index is defined as the ratio of the rutting factor of RTFO-aged binder to that of the unaged binder. The general trend is that the aging index increases with an increase in the amount of Sasobit®. It is also seen that the aging index decreases with an increase in temperature. It should be noted that the optimum dosage of Sasobit® for this binder was controlled by the low temperature characteristics. As reported by Hossain et al. (2009), an amount of 1.5% Sasobit® was found to be optimum and this dosage level was used while evaluating the effects of ASA.

5.5.4 Effects of ASA

To fulfill the objective of this study, an amount of 0.5% (by weight of the binder) ASA was used, as recommended in a related study (Hossain et al., 2010). The effect of ASA on the viscosity of unaged asphalt binders already modified by a WMA additive (1.5% Sasobit®) is shown in Figure 5.4a. The mixing and compaction temperatures with 1.5% Sasobit®-modified binder are expected to be reduced by 9°C,

and 10°C, respectively. ASA was found to somewhat decrease the viscosity of the WMA-modified binder. This is due to the fact that AD-here® HP Plus is less viscous than the binder itself. Figure 5.4b shows viscosity measurements of RTFO-aged WMA- and ASA-modified binder samples. As seen, the viscosity of the Sasobit®-modified binder, RTFO-aged at a reduced temperature, is significantly lower than that of the same binder RTFO-aged at 163°C. This behavior will be discussed later in this paper.

DSR test data of WMA- and ASA-modified binder samples under unaged and RTFO-aged conditions are shown in Figures 5.5a and 5.5b, respectively. The continuous PG grades of the modified binders are presented in Table 5.4. The base binder passes the Superpave® rutting factor criteria up to a temperature of 64.7°C, and with 1.5% Sasobit® the corresponding temperature is 68.5°C, and with 1.5% Sasobit® and 0.5% AD-Here® HP Plus it is 66.9°C. As mentioned earlier, Sasobit® enhances the elasticity of the binder. On the other hand, AD-here® HP Plus makes a binder softer, and it reduces its G* value. With only 0.5% ASA, the high PG temperature of the binder was found to be 64.2°C, which is 0.5°C lower than that of the base binder.

5.5.5 Low Temperature RTFO Aging

Viscosity measurements of RTFO-aged samples at reduced aging temperatures, presented in Figure 5.4b, showed significant reductions of viscosities due to less oxidative hardening. The viscosity values of RTFO-aged Sasobit®-modified binder samples are used to estimate E* values of corresponding mixes, which are presented later in this paper. The G*/sinδ values for the Sasobit®-modified binder samples under reduced RTFO aging conditions are plotted against the DSR test

temperatures in Figure 5.5b. It is evident that the G*/sinδ value decreases with a decrease in the RTFO aging temperature. This is expected as the binder goes through less oxidative hardening processes due to low RTFO oven temperature. From Table 5.5 it is clear that the aging index decreases with a decrease in RTFO operating temperature. Consequently, the high PG temperature of 1.5% Sasobit®-modified binder samples is reduced (Table 5.5). The high PG temperature of 1.5% Sasobit®-modified samples, RTFO-aged at 121°C, was found to be 3.8°C lower than that of the same binder RTFO-aged at 163°C.

5.6 PREDICTION OF E* VALUES

The E* values of the HMA (control) and WMA mixes were predicted by using Equations 5.1 through 5.4. The E* master curves for these mixes were then constructed using the TTS principle at a reference temperature of 21.1°C (70°F), as recommended by the MEPDG. As shown in Figure 5.6(a), the E* data at all temperatures other than the reference temperature were shifted with respect to time until the E* curves merged into a single smooth sigmoidal function, representing the master curve. The master curve was constructed by using a second order polynomial relationship (Equation 5.6) between the logarithm of the shift factors (loga(Ti)) and the temperature. It should be noted that the shift factor at the reference temperature (a(T(70)) is zero. The TTS was performed by simultaneously solving for the four coefficients of the sigmoidal function (δ, α, β, and γ) as described in Equation 5.6 and the four shift factors (a(T20), a(T40), a(T100), and a(T130)). A Microsoft™ Excel Solver program was used to conduct the nonlinear optimization to fit the sigmoidal function of a master curve. An example of shift factor versus reduced log time is

shown in Figure 5.6(b). The logarithm of shift factor is presented as a second order polynomial function of temperature with an R^2 value of 0.997.

E* master curves of the S4 mix with the base and Sasobit®-modified binders from the measured and predicted E* values are presented in Figures 5.7a through 5.7c. The predicted E* values from Witczak and Hirsch models are shown in these figures. It is seen that that the predicted E* values from the typical ASTM A and VTS parameters are somewhat close to the measured E* values except for those corresponding to large log reduced time values (6 to 10 sec). It is also seen that the DSR test data significantly underestimates E* values. On the other hand, RV test data overestimated E* values in most cases except for large log reduced time values (6 to 10 sec). Similar observations were made by Birgisson et al. (2005) for HMA mixes in Florida. That study reported that the predicted E* values from DSR data were significantly lower than the measured E* values, and the predicted E* values from RV test data were significantly higher than the measured E* values. These researchers also found a bias in the results and recommended a multiplier to correlate Witczak-based predicted E* values with the measured E* values. On the other hand, Tran and Hall (2005) reported no significant difference between the measured and predicted E* values for Arkansas mixes, indicating that the Witczak predictive equation could be used to estimate E* values. The current study, however, shows that the predicted E* values from the Hirsch model fits reasonably well with the measured E* values.

The *goodness-of-fit* statistics of the aforementioned sigmoidal master curves were assessed by calculating their R^2 (correlation coefficient) and S_e/S_y (standard error of estimate/standard deviation) values. The correlation coefficient, R^2, is a measure of

accuracy of the model. The closer the R^2 value is to 1.00, the better the prediction. The ratio S_e/S_y is a measure of the accuracy of the prediction that indicates how well the variations of the predicted E* values are explainable by the predictive equations. The lower the S_e/S_y value (close to zero) the better the accuracy of the prediction. Table 5.6 presents the *goodness-of-fit* statistics for all master curves. Overall, master curves for all mixes had "excellent" ($R^2 > 0.9$ and $S_e/S_y < 0.35$) correlations, based on the criteria given in NCHRP Report 465 (Witczak et al., 2002).

Figures 5.8a through 5.8c show the measured and predicted E* values for the mix for the base, 1.5% Sasobit®-, and 1.5% Sasobit® and 0.5% ASA- modified binders, respectively. If the data points distribute themselves around the "Equality Line," as shown in these figures, then there exists a good correlation. In order to evaluate the quality of predictions, linear regressions with an intercept of zero were performed. The slope of a regression line is a measure of the quality of fit; the closer the slope to unity, the less of a bias in the prediction. If the slope is greater than unity, then the predicted E* values are less than the measured values. If the slope is less than unity, then the predicted E* values are higher than the measured values. The goodness-of-fit statistics parameters of the measured versus predicted E* values are also presented in Table 5.6. In general, "excellent" correlations are observed for the predicted E* values from the typical ASTM A and V parameters and frequency sweep test data of both modified and unmodified binders. However, the major shortcomings of using the typical ASTM A and VTS parameters in predicting E* of modified binders is that these parameters are based on their Superpave® PG grades. Since the Superpave® PG of the modified binder does not vary from the base binder, the

predicted E* values for the WMA mixes based on the ASTM A and VTS parameters are the same as those of the unmodified binder.

Based on R^2 and S_e/S_y values, the correlation between the measured and predicted E* values from DSR test data is found to be "good" for both unmodified and modified binders. The "Equality Line" is also notably above the distribution of the measured and predicted E* values from DSR test data. Based on the R^2 value, the correlations between the measured and predicted E* values from RV test results for the base binder is good. However, an excellent correlation is found between the measured and predicted E* values, predicted from RV test data, in the case of the WMA mix. These observations reiterate that the Witczak model based on DSR test data significantly underestimates the E* value of the mix even though this model is being used by the MEPDG in Level 1 analysis. Overall, the Hirsch model, based on frequency sweep test data of binders, is found to be the best model, which is followed by the Witczak model, based on RV test data.

The effects of additives on E* master curves at a reference temperature of 21.1^{o}C, based on the measured and predicted E* values from the Hirsch model, are shown in Figures 5.9a and 5.9b. From Figure 5.9a, it is seen that the Sasobit® changes the viscoelastic properties of the mix. In the case of measured E* master curves, the WMA mix shows notably higher E* values than the control mix (HMA for a log reduced time from -10 to 3 sec. For a higher log reduced time (3 to 10 sec) the WMA mix shows lower E* values than the control mix. The estimated E* master curves, obtained from the Hirsch model, are in reasonable agreement with the measured E* master curves except that the estimated E* values are significantly lower at a very low

log reduced time. This could be related to possible intrinsic limitations of the Hirsch model, which makes an assumption for the calculation of the compressive modulus from shear modulus using linear elastic theory and a constant Poisson's ratio (Al-Qadi, 2009). The Hirsch model assumes a Poisson's ratio of 0.5, which is applicable to elastic materials and may not be true for viscoelastic asphalt binder.

A recent study by Zhang (2010) reported that no significant changes in E* values were observed due to 1.5% Sasobit®. Shrum (2010), however, reported significant reduction of E* values due to the addition of Sasobit® on a WMA mix with a PG 52-28 binder at a mixing temperature of 135°C. From Figures 5.9a and 5.9b, it is also evident that the predictive E* master curves of WMA mixes show the same similar trend as the E* master curves obtained from laboratory test data. The current study also reveals that the ASA does not have any significant effects on E* values of the WMA, and the measured and predicted E* master curves shows a similar trend. This signifies the fact that modified binder's frequency sweep test data can predict the E* master curves of WMA mixes reasonably well.

5.7 SUMMARY

This study evaluates viscoelastic properties of a selected PG 64-22 binder modified by different dosages (1%, 2%, and 3%) of a warm mix asphalt (WMA) additive, Sasobit®, at high service temperatures. The viscoelastic properties included viscosity, linear viscoelastic limits, temperature susceptibility and frequency dependency. This study also evaluates the effect of reduced RTFO aging temperatures on the stiffness of a selected dosage of Sasobit®-modified binder. The evaluated viscoelastic properties have been used to predict dynamic modulus (E*) of surface

mixes with unmodified and Sasobit®-modified binders using Witczak and Hirsch models. This study also presents the effects of a liquid anti-stripping agent (ASA), AD-Here® HP Plus, on the viscoelastic properties of a Sasobit®-modified binder and E* values of the WMA mix. Based on the findings of the study, the following conclusions can be drawn:

- A significant reduction of the production temperature can be achieved by using Sasobit® technology. With 3 % Sasobit®, the mixing and compaction temperatures are expected to be reduced by 16°C, and 19°C, respectively.
- At 135°C, the viscosity values of Sasobit®-modified binders remain unchanged with a varying range of shear rate (5 to 500 sec^{-1}), indicating that these binders follow the characteristics of a Newtonian fluid.
- The linear viscoelastic (LVE) limit reduces with an increase in the dosage level of Sasobit®. In the case of 3% Sasobit®-modified binder the corresponding LVE limits under unaged and RTFO-aged conditions are found to be 16% and 8%, respectively.
- The LVE limit of the Sasobit®-modified binder is not expected to change due to the addition of ASA. ASA is expected to reduce the stiffness of the Sasobit®-modified binder. With 1.5% Sasobit®, and 1.5% Sasobit® and 0.5% ASA the corresponding high PG temperature of the modified binders are found to be 68.5°C and 66.9°C, respectively.
- As expected, reduced RTFO aging leads to reduced oxidative age hardening of the Sasobit®-modified binder. The high PG temperature of 1.5% Sasobit®-

modified binder RTFO-aged at 121°C is expected to be 3.8^{o}C lower than that of the same binder RTFO-aged at 163°C.

- While using the Witczak model, the DSR test data, which is used in the MEPDG *Level 1* analysis for mixes with PG binders, significantly underestimates E* values of both the control (HMA) and WMA mixes. On the other hand, rotational viscosity (RV) test data overestimate E* values of these mixes.
- The Hirsch model, based on the dynamic shear modulus (G*) data from frequency sweep tests, is found to be a reasonably better than the Witczak model, based on RV test data, for predicting E* master curves of mixes. The correlation coefficient (R^2) values of the Hirsch model for the control, WMA, and WMA with ASA mixes are found to be 0.95, 0.91 and 0.92, respectively. Conversely, the R^2 values of the Witczak model, based on DSR test data, for the same mixes were found to be 0.84, 0.83, and 0.81, respectively.
- For a medium range of log reduced time (-3 to 3 sec), the measured and estimated E* values of the Sasobit®-modified mix were found to be significantly higher than the control (HMA) mix. The changes of E* values for these mixes due to the addition of ASA are not statistically significant.

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Table 5.1 Summary of Aggregate and Mix Properties (Hossain et al., 2009[1])

Properties	Parameter	Results	ODOT Specifications
Aggregate	Aggregate Type	Granite	
	L. A Abrasion (%)	20	40 max.
	Durability Index (%)	81	40 min.
	Insoluble Residue (%)	99.4	40 min.
	Micro-Deval (%)	5.5	25.5 max.
	Sand Equivalent (%)	80	40 min.
Mix volumetric	Mix Type	S4 (Oklahoma)	
	Binder Grade	PG 64-22	
	V_a (%)	4.5 ± 1	
	Vb_{eff} (%)	7.1 ± 1	
	Asphalt binder content, P_b (%)	5.3	4.6 min.
	Effective binder content, P_{be} (%)	4.5	
	Specific gravity of binder, G_b	1.026	
	Specific gravity of mix, G_{mm}	2.430	
	Specific gravity of aggregate, G_{sb}	2.579	
	Void in total mix, VTM[2] (%)	4.0	
	Voids in mineral aggregate, VMA (%)	14.1	
	Void filled with asphalt, VFA (%)	71.9	
	Dust proportion, DP	1.1	
	% Retained ¾″	0	0
	% Retained ½”	5	0 to 10
	% Retained 3/8″	15	>10
	% Retained # 4	40	
	Percent passing #200, P_{200} (%)	5	2 to 10

[1] Mixture tests were conducted by Dr. Steve Cross' team at Oklahoma State University.
[2] After 100 revolution in the Superpave® gyratory compactor (SGC), the average VTM values of the control, and Sosobit®-modified mixes was reported to be 4.4%, and 3.1%, respectively; V_{beff} = effective asphalt content by volume, V_a = air void content.

Table 5.2 Continuous PG grade of PG 64-22 with different dosages of Additives

Binder Type	High critical temperature under unaged (^oC)	High critical temperature under RTFO-aged (^oC)	Low critical Temperature	Continuous PG grade
Base PG 64-22	64.9	64.8	<-22.0	PG 64.8-XX
PG 64-22+1% Sasobit®	66.0	68.2	<-22.0	PG 66.0-XX
PG 64-22+2% Sasobit®	68.7	70.0	>-22.0	PG 68.7-XX
PG 64-22+3% Sasobit®	69.0	71.0	>-22	PG 69.0-XX

Note: $^oF = (9/5)^oC + 32$; Number of replicates = 3

Table 5.3 Aging Index of Sasobit®-modified Binder RTFO aged at 325°F (163°C)

DSR Testing Temperature	Base Binder	1% Sasobit®	2% Sasobit®	3% Sasobit®
58	2.70	3.41	3.51	3.62
61	2.58	3.39	3.41	3.61
64	2.32	3.25	3.36	3.59
67	2.17	3.06	3.16	3.51
70	2.13	2.77	3.12	3.24
73	2.10	2.71	3.07	3.20

Table 5.4 Continuous PG grade of PG 64-22 with different dosages of Additives

Binder Type	High critical temperature under unaged (^oC)	High critical temperature under RTFO-aged (^oC)	Low critical temperature from Stiffness (^oC)	Low critical temperature from m-value (^oC)	Continuous PG grade
Base PG 64-22	64.7	66.0	<-24.8	-24.8	PG 64.7-24.8
PG 64-22+0.5% AD-here® HP Plus	64.2	64.7	<-23.3	-23.3	PG 64.2-23.3
PG 64-22+1.5% Sasobit®	69.9	68.5	<-22.0	-22.0	PG 68.5-22.0
PG 64-22+1.5% Sasobit®+0.5% ASA1	67.0	66.9	<-22.0	-22.0	PG 66.9-22.0

Note: $^oF = (9/5)^oC + 32$; Number of replicates = 3

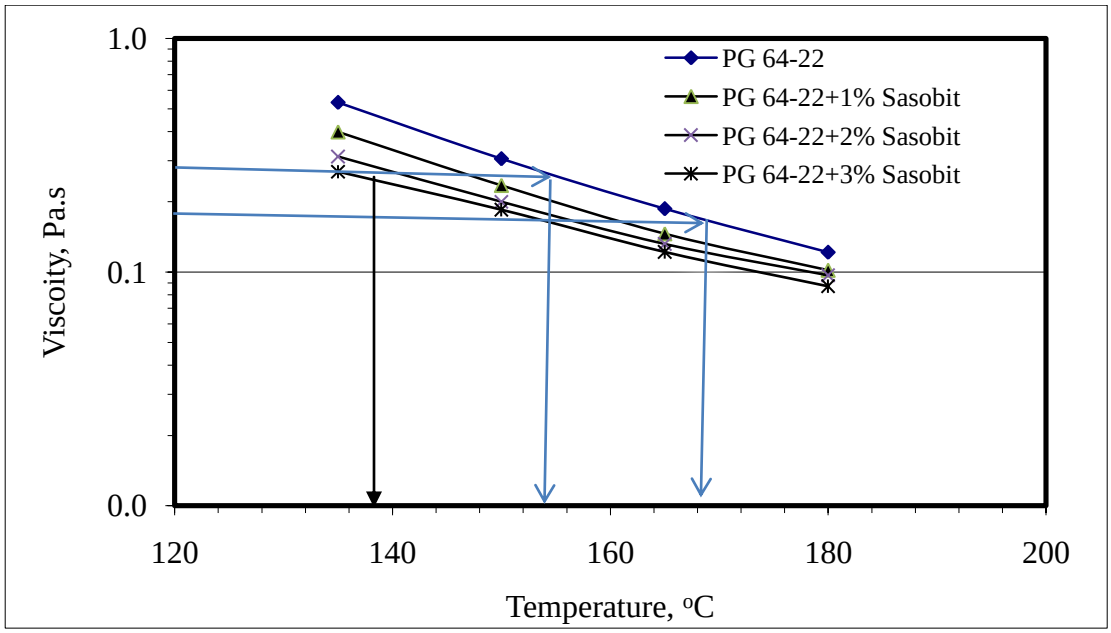
Table 5.5 Variation of Aging Index and high PG Temperatures with Respect to Reduce RTFO-Aging

Aging Index						
DSR Test Temperature (^oC)	RTFO 163C: PG 64-22	RTFO 163C: PG 64-22 with 1.5% Sasobit®	RTFO 149C: PG 64-22+ 1.5% Sasobit®	RTFO 135C: PG 64-22+ 1.5% Sasobit®	RTFO 120C: PG 64-22+ 1.5% Sasobit®	RTFO 163C: PG 64-22+ 1.5% Sasobit® +.5% AD-HP
61	2.60	2.19	1.94	1.78	1.46	2.21
64	2.41	2.11	2.00	1.65	1.37	2.20
67	2.43	2.00	1.74	1.55	1.38	2.21
70	2.53	1.89	1.71	1.50	1.49	2.05
High PG Temperature						
Aging Condition	RTFO 163C:PG 64-22	RTFO 163C:PG 64-22 with 1.5% Sasobit®	RTFO 149C:PG 64-22+ 1.5% Sasobit®	RTFO 135C:PG 64-22+ 1.5% Sasobit®	RTFO 120C:PG 64-22+ 1.5% Sasobit®	RTFO 163C:PG 64-22+ 1.5% Sasobit® +.5% AD-HP
Unaged	64.7	69.9	69.9	69.9	69.9	67.0
RTFO	66.0	68.5	67.5	66.5	64.7	66.9
High PG	64.7	68.5	67.5	66.5	64.7	66.9

Table 5.6 Goodness-of-fit Statistics for the Witczak Model Predictions

	ODOT S4 Mix Binder Type	Model Name	Measured/Predicted E* Values From	S_e/S_y		R^2	
				Value[1]	Evaluation	Value[2]	Evaluation
Master Curve	PG 64-22	N/A	Direct Estimation	0.07	Excellent	0.99	Excellent
		Witczak	Typical ASTM A VTS	0.00	Excellent	1.00	Excellent
		Witczak	RV Test Data	0.00	Excellent	1.00	Excellent
		Witczak	DSR Test Data	0.00	Excellent	1.00	Excellent
		Hirsch	Freq. Sweep Test Data	0.04	Excellent	0.99	Excellent
	PG 64-22 + WMA	N/A	Direct Estimation	0.10	Excellent	0.99	Excellent
		Witczak	Typical ASTM A VTS	0.00	Excellent	1.00	Excellent
		Witczak	RV Test Data	0.00	Excellent	1.00	Excellent
		Witczak	DSR Test Data	0.00	Excellent	1.00	Excellent
		Hirsch	Freq. Sweep Test Data	0.06	Excellent	0.99	Excellent
	PG 64-22 + WMA + ASA	N/A	Direct Estimation	0.10	Excellent	0.99	Excellent
		Witczak	Typical ASTM A VTS	0.00	Excellent	1.000	Excellent
		Witczak	RV Test Data	0.00	Excellent	1.000	Excellent
		Witczak	DSR Test Data	0.00	Excellent	1.000	Excellent
		Hirsch	Freq. Sweep Test Data	0.06	Excellent	0.999	Excellent
Measured Vs. Predicted	PG 64-22	Witczak	Typical ASTM A VTS	0.22	Excellent	0.92	Excellent
		Witczak	RV Test Data	0.31	Excellent	0.89	Good
		Witczak	DSR Test Data	0.40	Good	0.84	Good
		Hirsch	Freq. Sweep Test Data	0.06	Excellent	0.95	Excellent
	PG 64-22 + WMA	Witczak	Typical ASTM A VTS	0.18	Excellent	0.93	Excellent
		Witczak	RV Test Data	0.13	Excellent	0.94	Excellent
		Witczak	DSR Test Data	0.42	Good	0.83	Good
		Hirsch	Freq. Sweep Test Data	0.27	Excellent	0.91	Excellent
	PG 64-22 + WMA+ ASA	Witczak	Typical ASTM A VTS	0.18	Excellent	0.93	Excellent
		Witczak	RV Test Data	0.17	Excellent	0.94	Excellent
		Witczak	DSR Test Data	0.46	Good	0.81	Good
		Hirsch	Freq. Sweep Test Data	0.26	Excellent	0.92	Excellent

[1] $R^2 > 0.90$ means Excellent, $R^2 = 0.70$ to 0.89 means Good, $R^2 = 0.40$ to 0.69 means Fair, $R^2 = 0.20$ to 0.39 means Poor, and $R^2 < 0.19$ means Very Poor. [2] $S_e/S_y < 0.35$ means Excellent, $S_e/S_y = 0.36$-0.55 means Good, $S_e/S_y = 0.56$ to 0.75 means Fair, $S_e/S_y = 0.76$ to 0.89 means Poor, and $S_e/S_y > 9.0$ means Very Poor.



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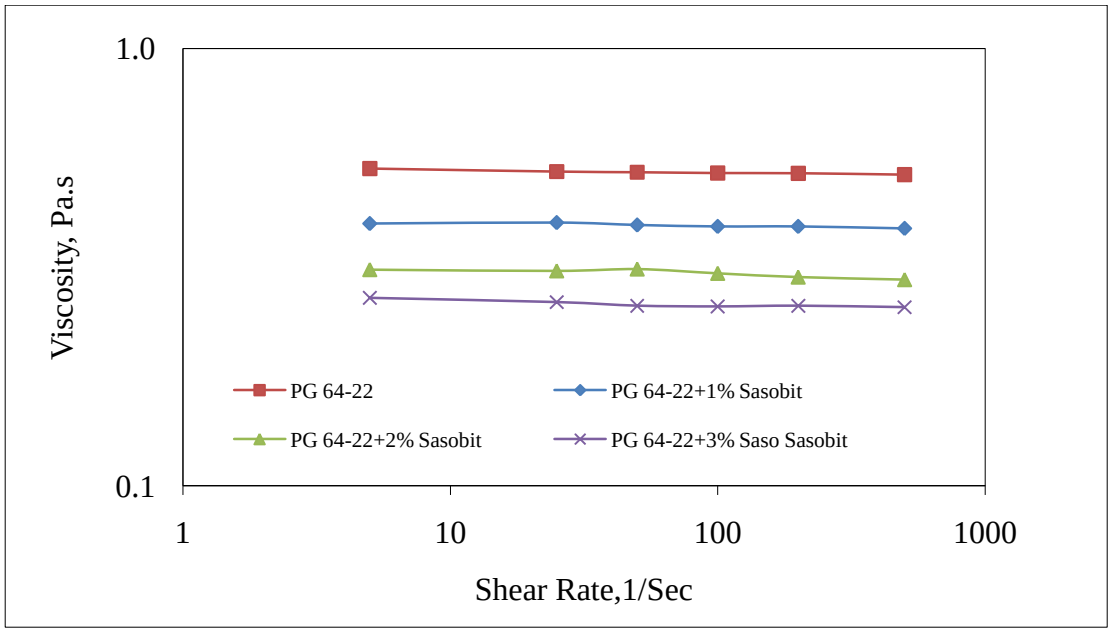
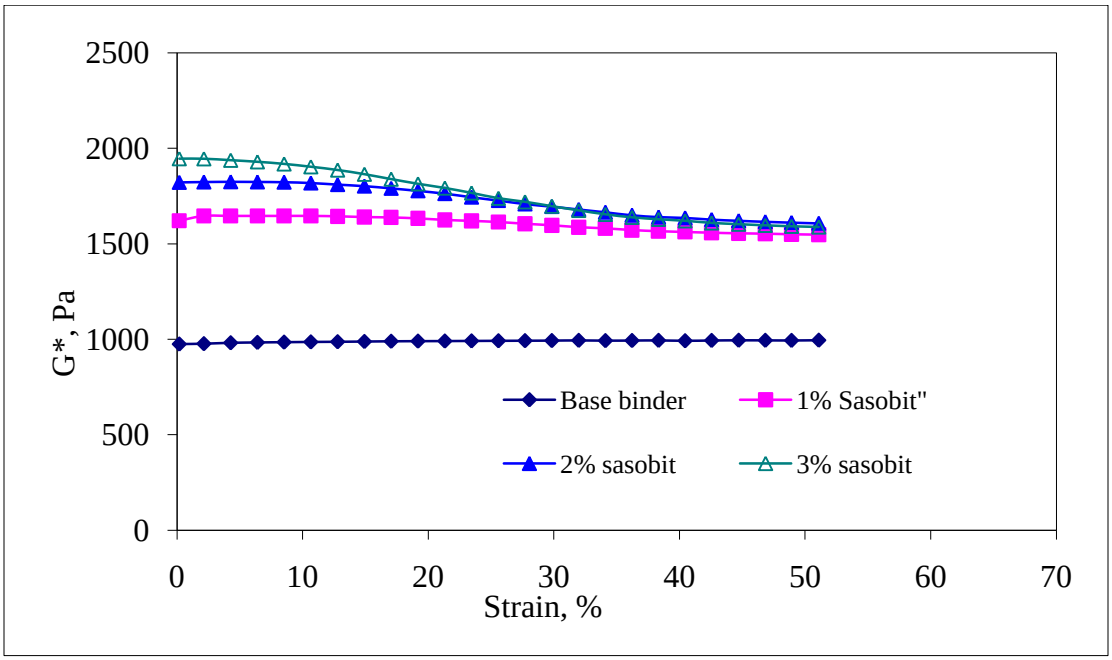


(b)

Figure 5.1 (a) Viscosity versus Temperature for PG 64-22 binder Modified with Sasobit®, and (b) Viscosity versus shear rate at 135°C for PG 64-22 binder Modified with Sasobit®.



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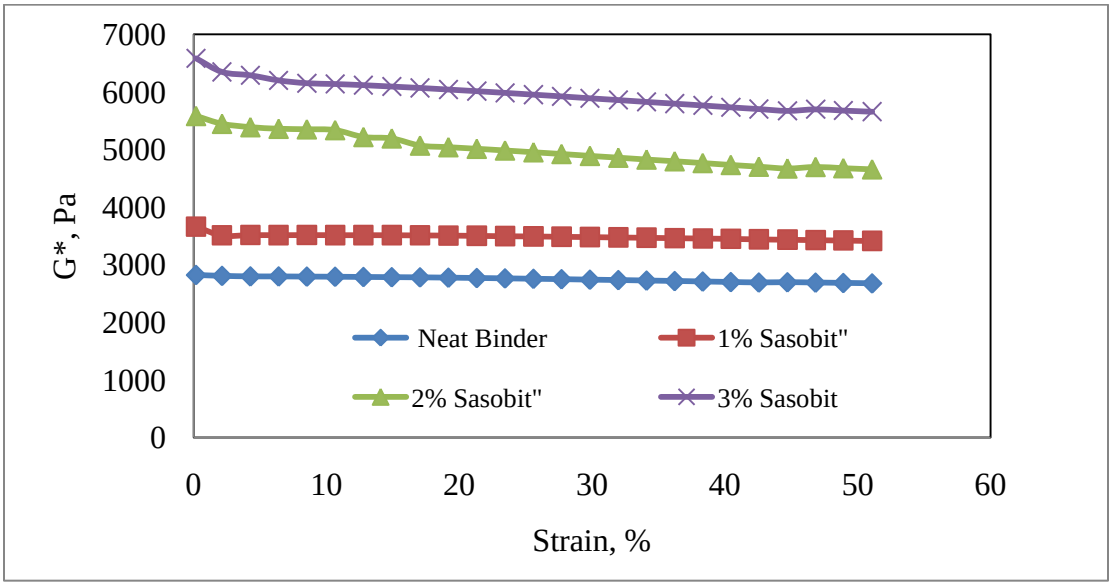
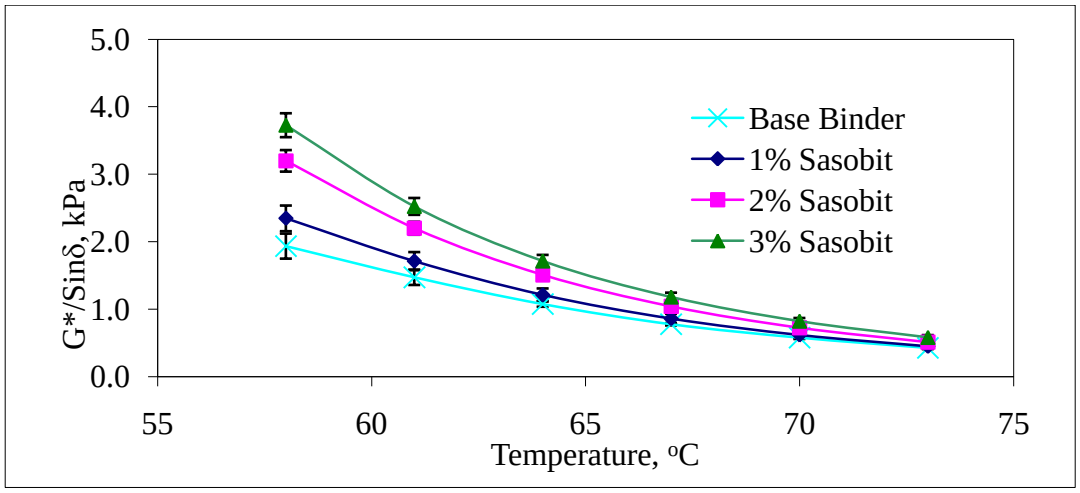


(b)

Figure 5.2 Strain Sweep Test Results of Sasobit®-modified Binder at 64°C: (a) Unaged, and (b) RTFO-aged.



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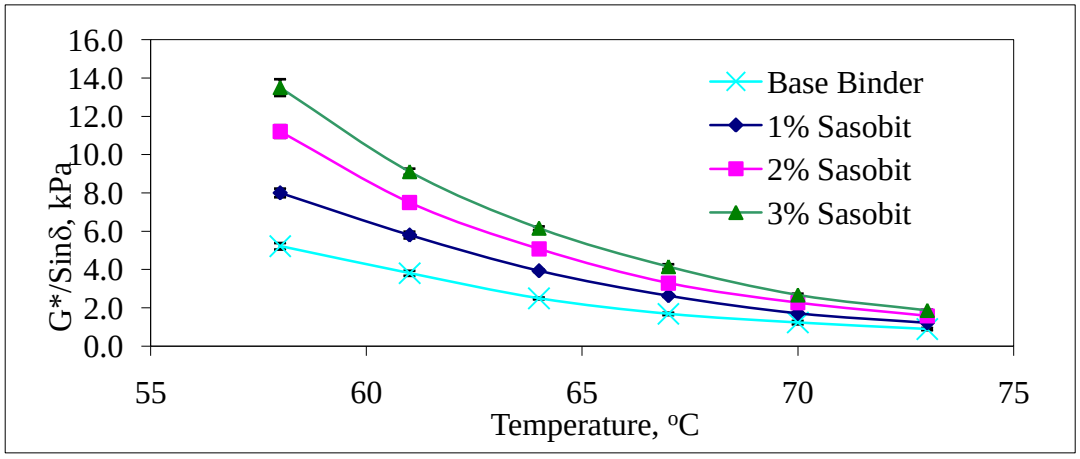

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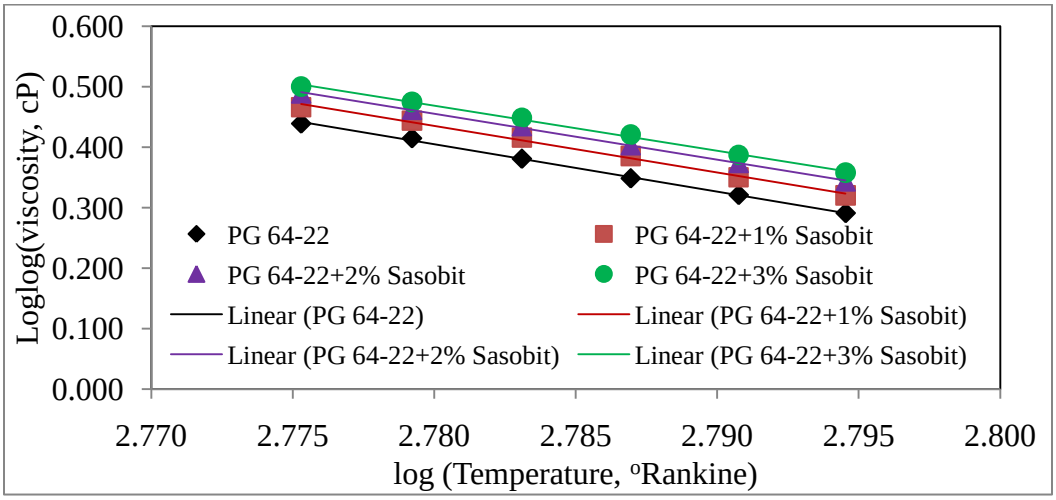
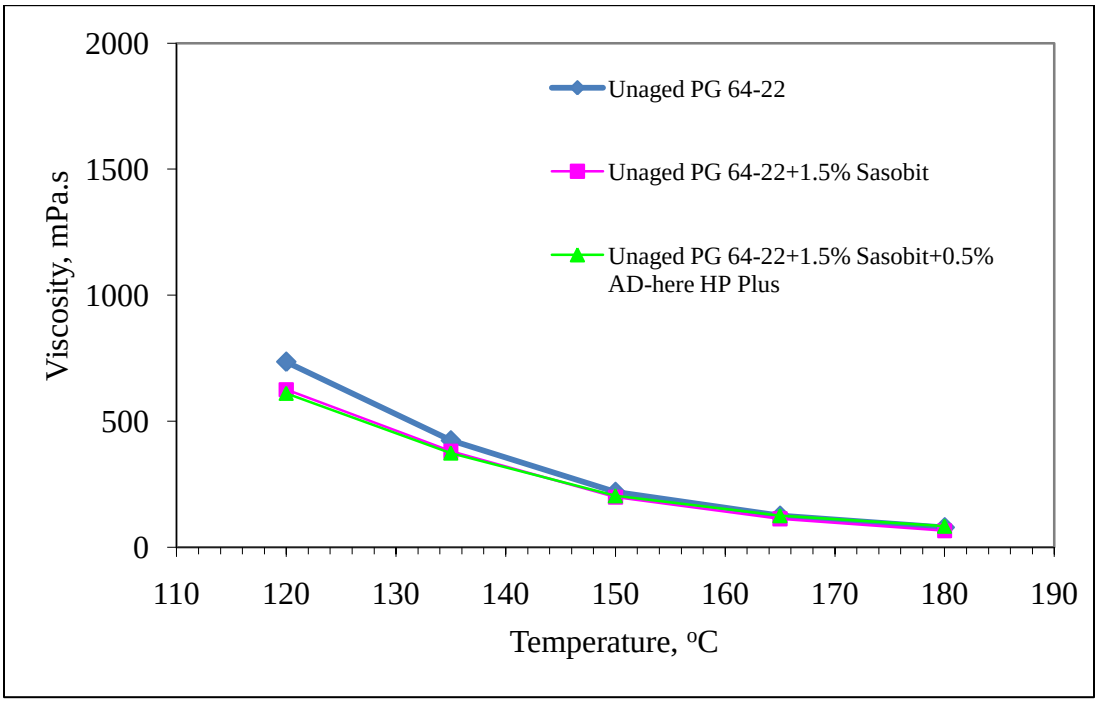


(c)

Figure 5.3 Temperature Susceptibility of Sasobit®-modified Binders Samples: (a) Unaged, (b) RTFO-aged, and (c) VTS of RTFO-aged.



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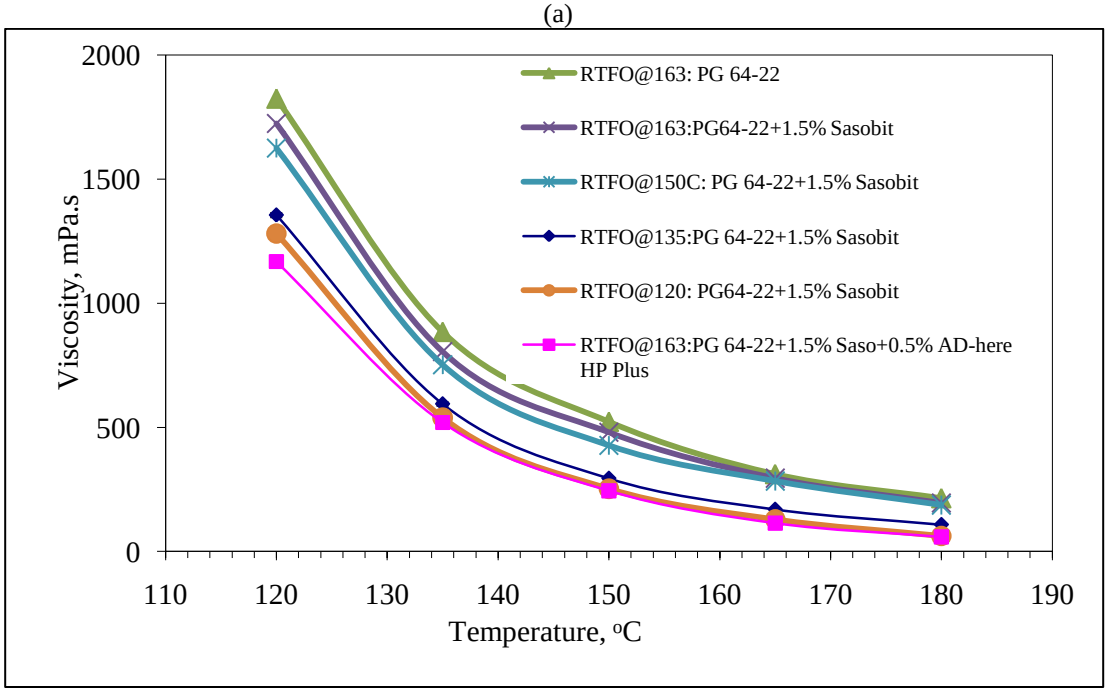

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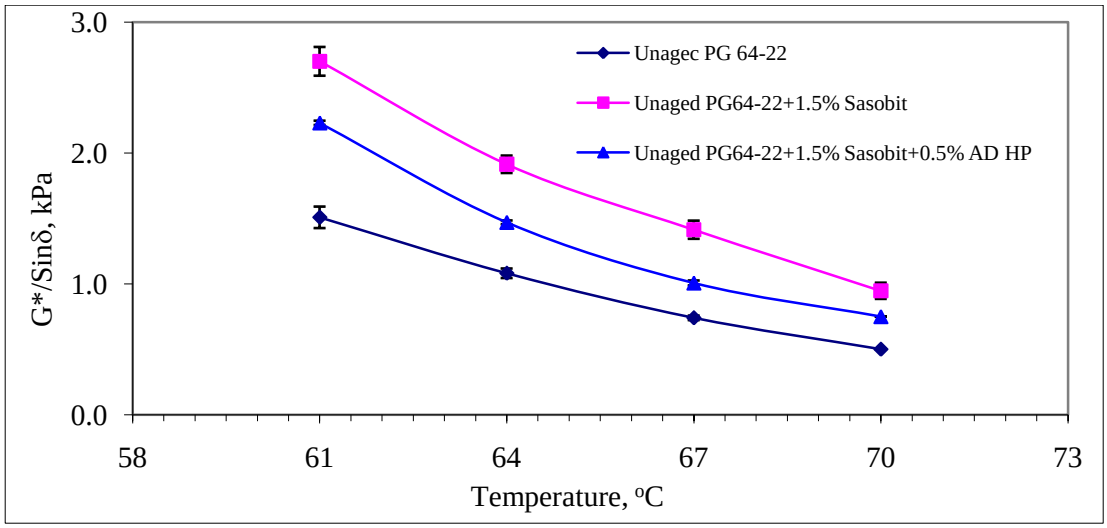
Figure 5.4 Viscosity Test Results of WMA- and ASA-modified Binders: (a) Unaged, and (b) RTFO-aged.



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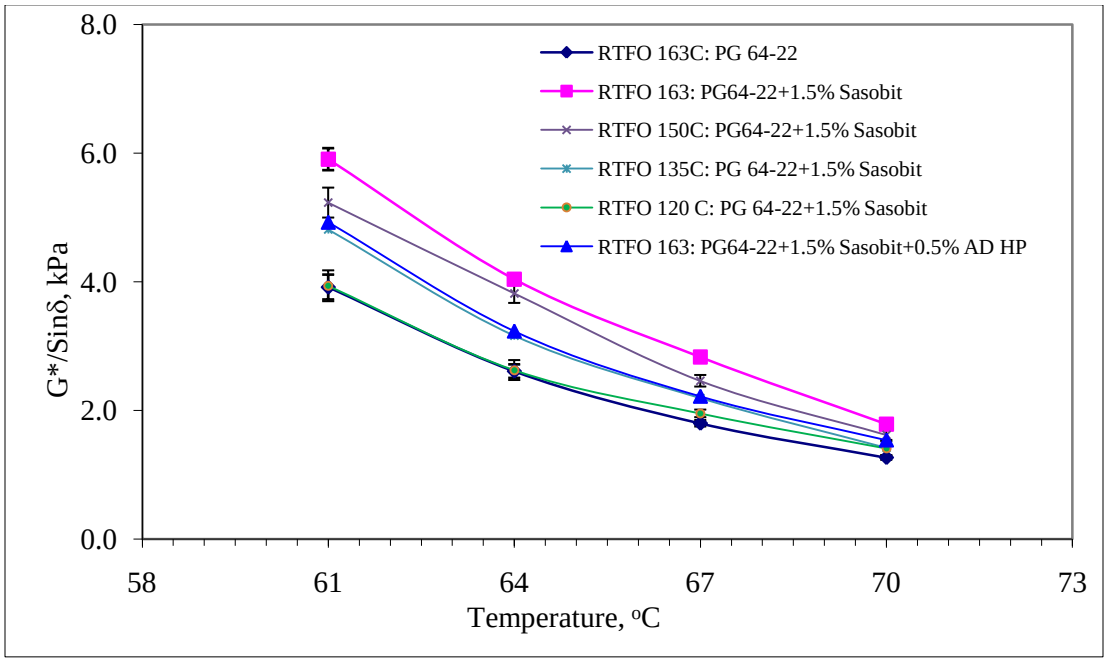
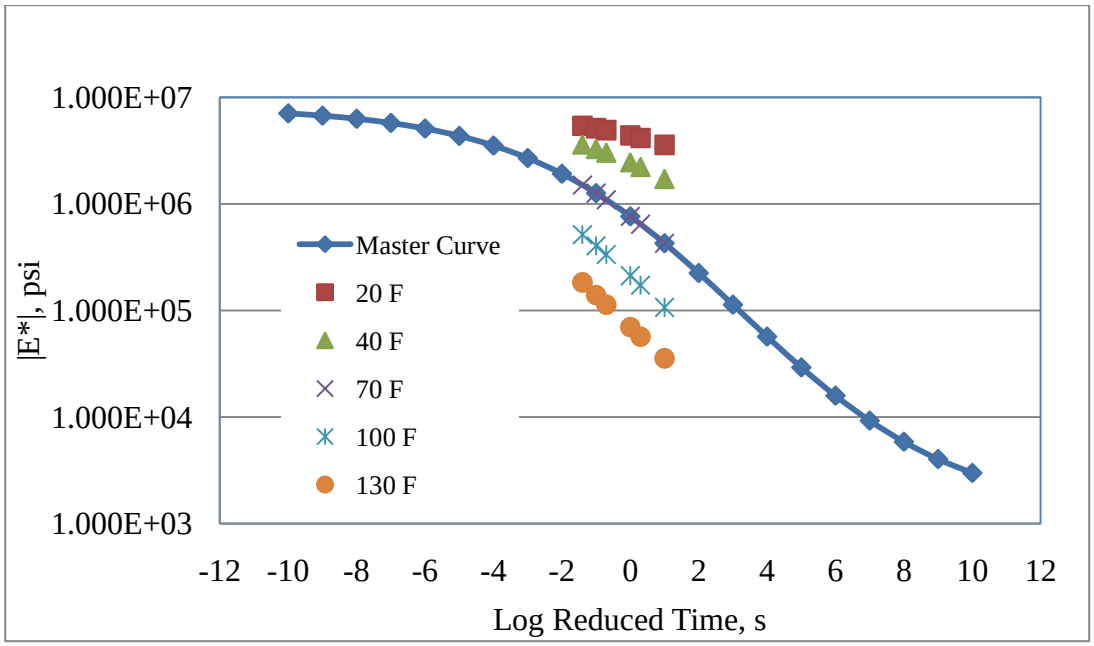


Figure 5.5 Temperature Sweep Test Data of Sasobit® and ASA-modified binders: (a) Unaged, and (b) RTFO-aged.



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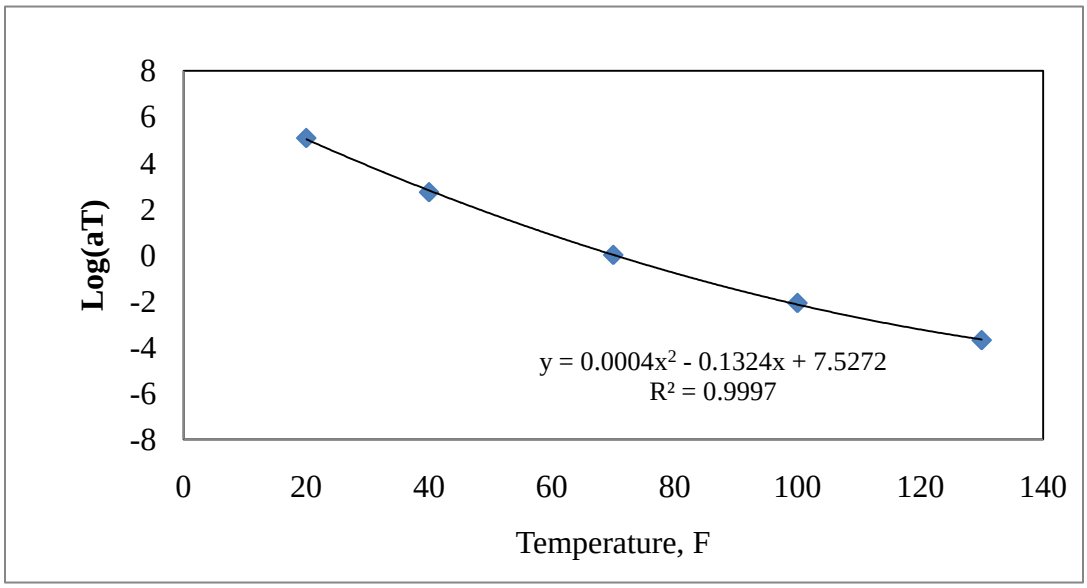
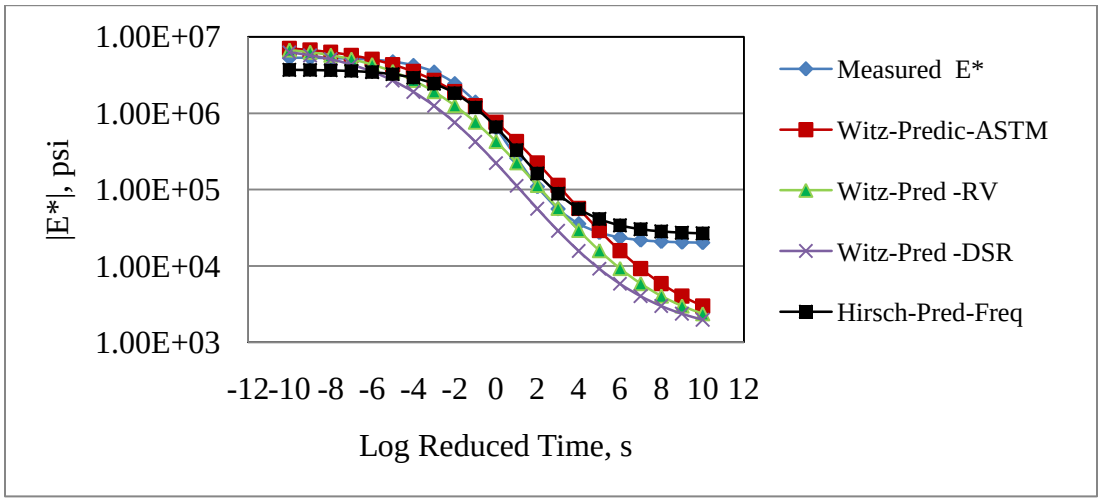


(b)

Figure 5.6 (a) Master Curve for S4 Mix Based on Typical ASTM A VTS Parameters of PG 64-22 Binder; and (b) Time-temperature Shift factor for E* Master Curve for a S4 Mix at Reference Temperature of 21.1°C.



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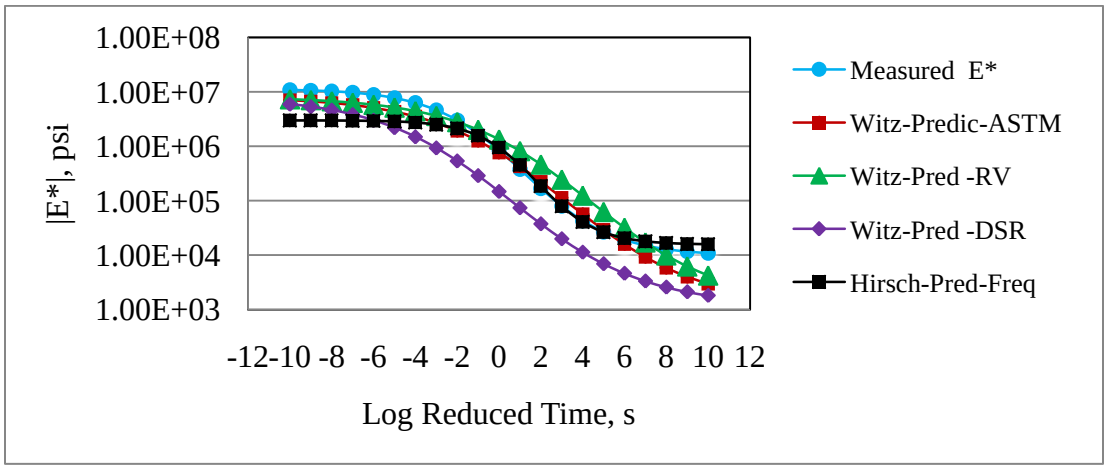

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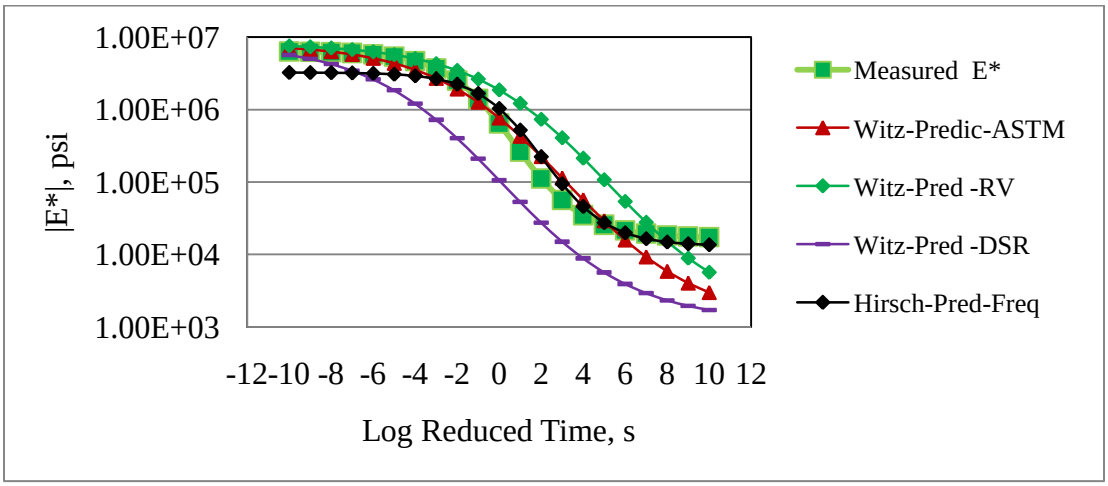
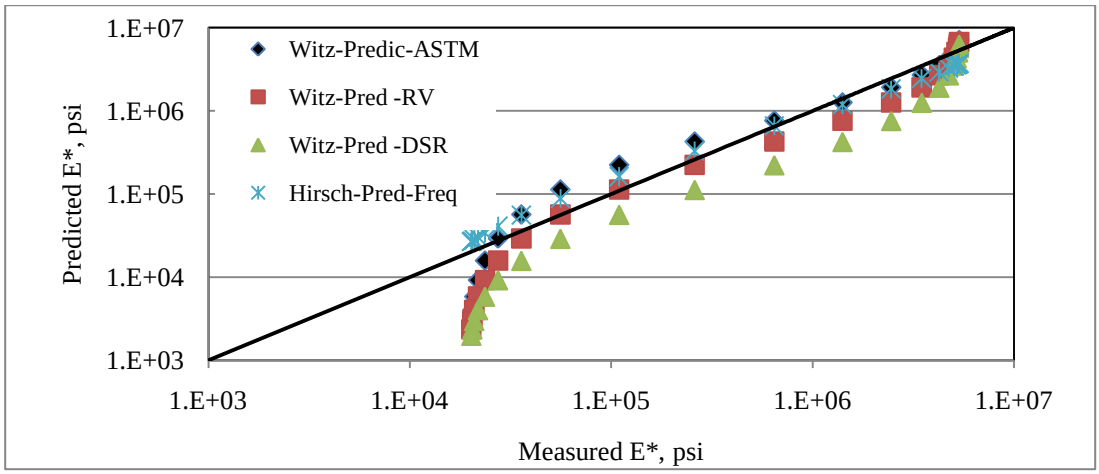
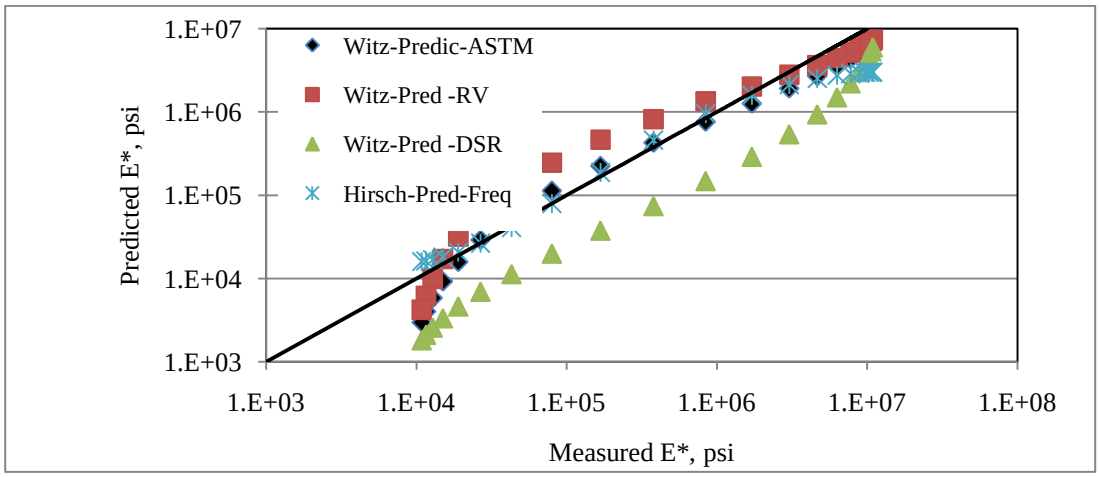


(c)

Figure 5.7 E* Master Curves for S4 Mix at a Reference Temperature of 21.1°C: (a) PG 64-22, (b) PG 64-22+WMA, and (c) PG 64-22+WMA+ASA.



(a)



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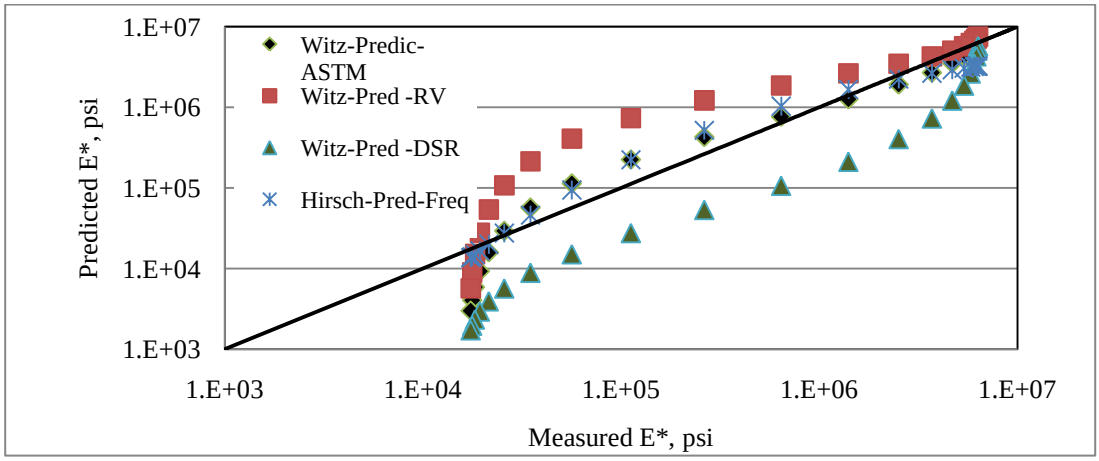
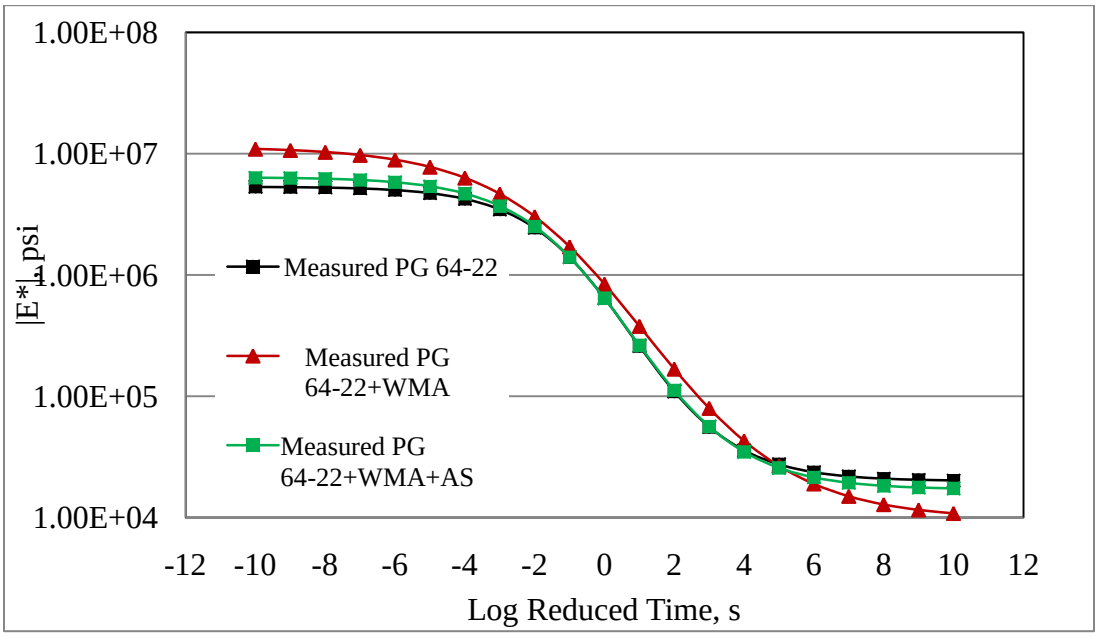
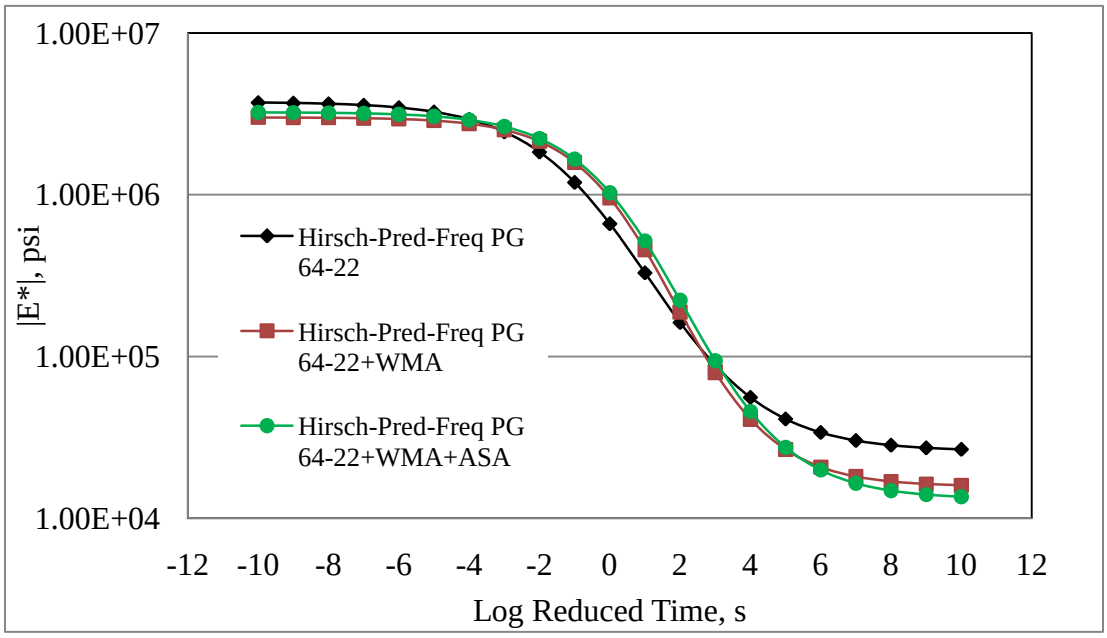


(c)

Figure 5.8 Measured Versus Predicted E* Master Curves at a Reference Temperature of 21.1°C: (a) PG 64-22, (b) PG 64-22+1.5% Sasobit®, and (c) PG 64-22+1.5% Sasobit®+0.5% ASA.



(a)



(b)

Figure 5.9 Effects of Additives on E* Master Curves at a Reference Temperature of 21.1°C: (a) Measured, (b) Predicted From Frequency Sweep Tests.

6 INFLUENCE OF RECOVERY PROCESSES ON PROPERTIES OF BINDERS AND AGGREGATES RECOVERED FROM RECYCLED ASPHALT PAVEMENT[5]

6.1 ABSTRACT

Usage of recycled asphalt pavement (RAP) in the construction of new pavements has increased in recent years due to the movement to conserve energy and raw materials, and reuse waste materials. To assess the effectiveness of RAP materials in new asphalt mixes, it is important to evaluate the properties of the recovered binders and aggregates. The widely used "Abson" method is employed in this study to recover asphalt binder from RAP. Also, the frequently used "NCAT Ignition" method is used to extract aggregates. A laboratory study comprising of two field RAP materials, two simulated RAP materials and corresponding virgin materials, was undertaken to assess possible influences of the aforementioned recovery processes. Performance grade (PG) of the recovered binders, and gradation, durability (LA Abrasion and Micro-Deval), specific gravity, sand equivalent, and insoluble residue of the extracted aggregates were evaluated as per the AASHTO and Oklahoma Department of Transportation (ODOT) standards. The test results showed that the Abson method notably influenced the critical PG temperatures of the recovered binder. It was also observed that some mechanical properties (durability and sand equivalent) of RAP aggregates were inconsistent with their virgin counterparts. Furthermore, field RAP aggregates showed significant variations in LA Abrasion loss and insoluble residue

[5] This chapter or portion thereof has been submitted for possible publication as a technical paper in the Journal of ASTM International (JAI). The manuscript is currently being reviewed by peers. The current version has been formatted for this dissertation.

test results. The findings of this study are expected to be helpful to the evaluation of RAP for reuse in asphalt paving.

Keywords: RAP, Abson, PG grading, NCAT ignition oven, aggregate extraction, durability

6.2 INTRODUCTION

Asphalt recycling has become an important topic in recent years because of its enhanced use in the construction of new asphalt concrete (AC) pavements. The increasing demand of recycled asphalt pavement (RAP) is mainly due to the increasing cost of asphalt binders and scarcity of quality virgin aggregates, as well as due to increasing environmental awareness. RAP has already become one the most widely used recycled materials in the United States. Nationally, the use of RAP in new pavements is expected to be doubled by 2014 (NAPA, 2009). In the asphalt recycling process, the processed RAP is blended with virgin materials, and new mixes are prepared. Therefore, the characterization of the recovered binders and aggregates from RAP is essential to attain proper blending in the mix design.

Among existing recovery techniques, the "Abson" method (AASHTO T 170) is widely used by the transportation industry. In this method, the asphalt binder is recovered by distilling previously solvent-extracted asphalt residues in a centrifuge, as per AASHTO T 164 (AASHTO, 2008). This method involves boiling the solvent (i.e., trichloroethylene [TCE]) off and leaving the asphalt binder behind. The solvent is then condensed back into a liquid. Sometimes the solvent removal may be incomplete. It is also possible that the asphalt binder is overheated during the recovery process. Several

studies (e.g., Loh and Olek, 1999; Anderson, 2001; McDaniel and Anderson, 2001) have raised some concerns on the variability of test results when recovering binder in accordance with the Abson method. On the other hand, in the commonly used aggregate extraction technology, the National Center for Asphalt Technology (NCAT) ignition method (AASHTO T 308), aggregates are extracted by burning off the asphalt binder at a very high temperature (538°C). Therefore, it is important to examine the influences, if any, of these recovery techniques on the recovered materials. The current study was undertaken to achieve the following objectives: (i) evaluate the effects of the Abson method on the PG grading of the recovered binder, and (ii) evaluate the influence of the NCAT ignition oven on the engineering properties (gradation, durability, specific gravity, sand equivalent, and insoluble residue) of the extracted aggregates.

6.3 REVIEW OF PREVIOUS STUDIES

Findings of some previous studies (McKeen, 1997; Burr et al., 1999; Stroup-Gardiner and Nelson, 2001; Ahmad et al. 2004; Tao et al., 2010; Daniel et al., 2010; Doh et al., 2010) pertinent to the current study are summarized in Table 6.1. Even though the Abson method is used frequently to recover asphalt binder from RAP, several studies (McKeen, 1997; Burr et al., 1999; Stroup-Gardiner and Nelson, 2001; Ahmad et al. 2004) have warned that it may cause excessive hardening of the binder. This excessive oxidative hardening of the recovered binder is partly due to chemical and physical hardening processes which the asphalt binder experiences during the removal process of the solvent. Likewise, the high operating temperature of the NCAT ignition oven may alter some engineering properties (i.e., LA Abrasion loss) of the

extracted aggregates (Tao et al., 2010; Daniel et al., 2010; Doh et al., 2010). Such effects may be more prominent in some aggregates (i.e., dolomite, limestone) as the chemical structures of these aggregates may change due to their exposure to high heat in the NCAT ignition oven.

6.4 MATERIALS AND METHODOLOGY

6.4.1 Materials

Two asphalt milling sites were selected for laboratory evaluation in this study. About 600 kg bulk RAP was collected from each site. These collected field RAP materials are referred as FRAP1 and FRAP2. The source of FRAP1 is a pavement section, located at Shields Blvd. in Moore, Oklahoma. The original pavement of this RAP was a Type B Insoluble (Oklahoma) mix with a PG 76-28 binder, constructed in May, 2003. Relevant properties of aggregates and the mix are shown in Table 6.2. FRAP1 was collected from the contractor's plant site where it was separated from other stockpiles. The asphalt binder and aggregates corresponding to FRAP1 were collected from the same physical location. The PG 76-28 binder (Canadian crude) was collected from Ergon Asphalts and Emulsion, Inc. located at Muskogee, Oklahoma. Virgin aggregates were collected from four different quarries: 16 mm (5/8 inch) chips (limestone) from Cyril, coarse screenings (limestone) from Richard Spur, stone sand from Davis, and asphalt sand Meridian Pit, all from Oklahoma.

The location of FRAP2 was a city street named North May Avenue, constructed in 1995. This pavement section of FRAP2 was a Type B Recycled (Oklahoma) mix, which included 25% recycled asphalt from an unknown source

(Table 6.3). Bulk FRAP2 sample was collected from the contractor's plant site where it was kept in a separate stockpile. Based on the mix design sheet for the original pavement section, virgin materials were collected from the same geographical locations. Thus, a PG 64-22 binder (Boscan crude) was collected from Valero refinery at Ardmore, Oklahoma, and virgin aggregates were collected from different sources: 19 mm (3/4 inch) rock (limestone) at Davis, screenings (limestone) at Davis, stone sand at Davis, and natural sand at Yukon, all from Oklahoma.

Two new loose hot mix asphalt (HMA) mixes, HMAMix1 and HMAMix2, were prepared as per the mix design data of the original pavement sections of FRAP1 and FRAP2. These HMA mixes were made by using the corresponding virgin aggregates (AGR1 and AGR2), and asphalt binders (PG 76-28 and PG 64-22), as noted earlier. These mixes were then long-term conditioned in the laboratory as per the AASHTO T 30 method to obtain simulated RAP materials. Major steps involving the laboratory simulation of RAP are discussed later on. Henceforth, the simulated RAP materials prepared from HMAMix1 and HMAMix2 are called as SRAP1 and SRAP2, respectively.

About 400 gm of binder was recovered from a representative sample of each RAP in accordance with the Abson method (AASHTO T 170). Since the Abson method can recover only a small amount of asphalt binder at a time, the recovered binder samples from several trials of each RAP were blended for homogeneity. The blended recovered binder was then tested to determine its PG grade. Also, collected virgin binders were long-term aged by using a pressure aging vessel (PAV) as per AASHTO R 28, which exposes the asphalt binder to heat and pressure to simulate in-

service aging over a 7- to 10-year period. PG grades of the recovered and PAV-aged binders were then compared. On the other hand, the aggregates were extracted from representative samples of RAP by burning the asphalt binder off in an NCAT ignition oven as per the AASHTO T 308 method. The extracted aggregates were then blended for homogeneity and tested to determine their engineering properties. The engineering properties of burned off aggregates were then compared with those of their virgin counterparts.

To verify repeatability of test results, replicate samples (at least three) were tested at each test temperature or test condition. Average values and error bars (± one standard deviation; shown as vertical error bars) were reported in appropriate figures. Furthermore, Student's t-tests were conducted with a confidence level of 95% ($p = 0.05$) to verify if the average of one set of test results statistically differed from the average value of another set. Microsoft® Excel software was used for presenting test results and performing statistical analyses.

6.4.2 Laboratory Simulation of RAP

Both short-term and long-term-conditioning of HMA mixes can easily be accomplished by following the AASHTO R 30 method (AASHTO, 2008). The short-term conditioning of HMA mixes simulates the pre-compaction phase of the construction process. To accomplish this, loose mixes were placed in a force-drift conditioning oven for 4 hours ± 5 minutes at a temperature of 135 ± 3°C. The long-term-conditioning of HMA mixes simulates the aging that occurs over the service life. The short-term-conditioned loose mixes were cooled at room temperature for 16 ± 1

hours. The specimen was then placed in the conditioning oven for 120 ± 0.5 hours at a temperature of 85 ± 3°C. Some recent studies (e.g., Hamzah et al., 2006; NCHRP, 2001) have used similar methods to prepare simulated RAP in the laboratory. Even though this method does not take into account the effects of HMA mix properties and environmental factors, the long-term conditioning is designed to simulate the aging the mix undergoes during seven to ten years of service. Thus, it is stipulated that the age hardening of the asphalt binder experiences in this method is similar to that the asphalt binder undergoes in the PAV-aging process (AASHTO R 28).

Since the aim of this study was to assess the influence of the aforementioned test methods rather than the performance of the RAP itself, the evaluation of the simulated RAP is expected to be a better approach than that of the field RAP. This was because the simulated RAP had a fewer unknowns and assumptions than the field RAP. For example, the mix of the original pavement section of FRAP2 had 25% RAP from an unknown source. Thus, it was not practical to reproduce a new mix with the same type of RAP in the laboratory. Because of such anomalies in FRAP2, it was not evaluated further in this study. For the same reason, SRAP2 (simulated RAP from HMAMix2) was prepared only with virgin aggregates and asphalt binder from the same geographical locations of FRAP2 except that 25% RAP was substituted by other aggregates to maintain the overall gradation within the specification limits.

6.4.3 Abson Recovery

Prior to the recovery of the asphalt binder, it was extracted from RAP by using TCE (reagent grade) as a solvent in accordance with the AASHTO T 164 method

(AASHTO, 2008). In the Abson method (AASHTO, 2008), the solution from the previous extraction was centrifuged for a minimum of 30 minutes at 770 g in 250-mL to 500-mL wide-mouth bottles. The residue was then transferred from the primary distillation flask, using several washes of solvent to rinse the residue into the distillation flask. Afterwards, carbon dioxide (CO_2) gas was introduced at a low rate (~100 mL/min). The distillation process was continued until the temperature reached 157ºC to 160ºC. The flow rate of CO_2 was then increased to approximately ~ 900 mL/min. This flow rate and a temperature of 160 to 166ºC were maintained for 10 minutes to complete the process.

6.4.4 The NCAT Ignition Oven

The amount of material for each batch of the extraction process was determined based on the nominal maximum size (NMAS). For instance, 2 kg of FRAP1 sample was used during each extraction as its NMAS was 19 mm. The NCAT oven was preheated at 538°C, and an automated ignition process was set by inputting a calibration based correction factor, a set point temperature of 538°C and the initial mass of the specimen. Each test was concluded in approximately 45 minutes.

6.4.5 Other Test Methods

Test methods involving the determination of PG grades of asphalt binders and engineering properties of aggregates are listed in Table 6.4. While determining the high PG temperature of the recovered binder, DSR tests were conducted on binder specimens as if the asphalt binder were unaged. The remainder of the binder was subjected to RTFO aging, and additional DSR tests were conducted at high

temperatures. Even though the recovered binder went through long-term aging in the field, this RTFO-aging was done to comply with linear blending equations for recovered and virgin binders as per recommendation of the National Cooperative Highway Research Program (NCHRP) Report No. 452 (McDaniel and Anderson, 2001). The RTFO-aged recovered binder was also subjected to BBR tests for evaluating its low PG temperature as if the binder were PAV-aged (McDaniel and Anderson, 2001). Furthermore, elemental analysis of selected binders was conducted using a CE 440 Elemental Analyzer. All test protocols for evaluating engineering properties of aggregates followed in this study were AASHTO specifications (AASHTO, 2008) except for an ODOT standard (OHD L-25). The OHD L-25 method determines the acid insolubility of coarse aggregates with concentrated HCL, an indicator for skid resistance in high volume traffic road (ODOT, 2010).

6.5 EFFECT OF THE ABSON METHOD

6.5.1 Performance Grade

DSR test results of the recovered binders from FRAP1 and SRAP2, and their counterparts are shown in Figures 6.1a-6.1d and 6.2a-6.2c. High critical temperatures of RAP binders of under "As Is" and RTFO-aged conditions were calculated by extrapolating DSR test data, as recommended by McDaniel and Anderson (McDaniel and Anderson, 2001). It should be noted that the rutting factor ($G^*/\sin\delta$; where, G^* = complex modulus, and δ = phase angle) under RTFO-aging condition governed the high PG temperature of the tested binders. Low critical temperatures (Table 6.5) of RAP binders with respect to the stiffness (S) and the rate of stress relaxation (m-value)

were calculated by extrapolating the BBR test data, as suggested by McDaniel and Anderson (McDaniel and Anderson, 2001). It was observed that the m-value rather than the S-value governed the low PG temperature of the tested binders. For example, the low critical temperature corresponding to the m-value of FRAP1 binder was found to be -19.8^{o}C, whereas it was found to be -26.6^{o}C with respect to its S value. Therefore, the low critical temperature of the recovered binder from FRAP1 was reported as -19.8^{o}C. Similar observations were made for the binder recovered from SRAP2 and its counterparts.

Based on the DSR and BBR test results, the continuous PG grades of the virgin PG 76-28 and PG 64-22 binders were found to be PG 79.8-33.7, and PG 64.8-24.0, respectively (Figures 6.3a and 6.3b). The continuous PG grades of the PAV-aged PG 76-28, FRAP1, and SRAP1 binders were found to be PG 94.8-30.6, PG 81.1-19.8, and PG 98.9-27.7, respectively. It is also important to note that the high PG temperature of FRAP1 binder was only 5.1^{o}C (i.e., 81.1^{o}C minus76^{o}C) higher than that of the virgin binder (PG 76-28) used in the old pavement assuming that the continuous PG grade of the latter was same as its standard grade. However, the high PG temperature of FRAP1 binder was expected to be significantly (at least two PG grades or so) higher than 76°C as the binder must have hardened over its service life. Since the continuous PG grade, crude source, and the modifier (polymer) of the binder used in the original mix of the pavement of FRAP1 is unknown, it would not be worthwhile to compare its PG grade with the tested virgin binder (Ergon 76-28). Furthermore, the collection of the virgin aggregates used in the original pavement section of FRAP1 was a challenging issue, which is discussed later on.

As seen in Figure 6.3a, the PG grade of SRAP1 binder shifted upward from that of the PAV-aged PG 76-28 binder. The high PG temperature and the low PG temperature of SRAP1 binder (PG 98.9-27.7) increased by 4.1^{o}C and 2.9^{o}C, respectively, from those of the corresponding PAV-aged PG 76-28 binder. In case of the PG 64-22 binder, the continuous PG grades of the PAV-aged binder, and SRAP2 binder were found to be PG 82.5-18.8, and PG 86.2-18.6, respectively (Figure 6.3b); the high PG temperature for the former was found to be 3.7^{o}C higher than that of the latter. On the other hand, a slight increase in the low PG temperature was observed for SRAP2 binder compared to the PAV-aged PG 64-22 binder. Such differences were possibly due to the fact that the recovered binder went through excessive oxidative hardening (chemical and physical) in the centrifuge. The purge gas (CO_2) used in the recovery method may have accelerated the aforementioned age hardening. Furthermore, it is possible that very fine particles escaped through the filter which increased the complex modulus of the binder. It is believed that even small traces of the TCE solvent make the asphalt binder softer (Houston et al., 2001; Daniel et al. 2010). However, the combined effect of prolong oxidative hardening and inadequate filtering may have offset the softening effect of the TCE; thus it increased the overall stiffness of the recovered binder. To verify the aforementioned findings, elemental analysis of a selected binder was conducted to determine its composition under different aging conditions and the results are discussed next.

6.5.2 Elemental Analysis

Elemental analysis of the PG 64-22 binder under unaged and PAV-aged conditions, and SRAP2 binder is shown in Table 6.6. The hydrocarbon (carbon and

oxygen) content of the binder under unaged condition was found about 94.5%, which is within the typical range of asphalt binders refined from Boscan crude source (Lewandowski, 1994). The amount of hydrocarbon was found to decrease with physical and chemical hardening that the binder experienced during the aging process. As expected, the content of oxygen in PAV-aged PG 64-22 binder was found to be 43% higher than that of the unaged binder. In case of SRAP2 binder, the amount of oxygen was found to be 241% higher than that of the unaged binder. The significant increase in oxygen content in SRAP2 binder enlightens the increased oxidative hardening (i.e., carboxyl functional group) that the binder experienced during the Abson recovery process. This observation supports the PG grades of the tested binders presented earlier; both high and low PG temperatures of the recovered (Abson) binder shifted upward compared to the PAV-aged binder.

6.6 EFFECTS OF THE NCAT IGNITION OVEN

6.6.1 Binder content

While extracting aggregates from RAP, binder contents were determined by using the NCAT ignition oven in accordance with the AASHTO T 308 method. Three representative samples from each asphalt mix were burnt off, and the average binder content was found to be reasonably in agreement with the true binder content. For example, the binder content in FRAP1 was found to be 4.8%, whereas the true binder content reported in the mix design sheet of the original pavement section was 4.7±0.4%. Similar observations were made in a recent study (ARC, 2010) conducted by researchers at Asphalt Research Consortium (ARC), which evaluated the binder

contents in simulated RAP materials by using three extraction methods including the NCAT ignition oven method. It was reported that the NCAT ignition oven method gave the best approximation of the true binder content, and it was followed by the reflux method. The centrifuge extraction method provided the worst approximation of the true binder content. In that study, for RAP samples with soft limestone and hard limestone aggregates, the NCAT ignition oven method estimated the binder contents as 5.1% and 5.8%, where the true binder contents were 5.3% and 6.0%, respectively (ARC, 2010).

6.6.2 Gradation

Gradations (average of three trials) of FRAP1 and SRAP1 aggregates were found to be in agreement with that of AGR1 (Figure 6.4a). Similar observations were made for SRAP2 and AGR2 aggregates (Figure 6.4b). It was also observed that gradations of these aggregates (extracted and virgin) are well within the minimum and maximum limits of the corresponding job mix formula (JMF). This implies that even though the NCAT ignition oven may change the chemical composition of aggregates, it does not affect the volume. Consequently, the overall gradation of burned off aggregates remained unchanged. However, some excessive fine particles (passing No. 200 sieve) were observed in the case of aggregates extracted from FRAP1. One of the reasons for the excessive fines in FRAP1 aggregates could be due to the weathering action, traffic load, and processes involved with millings and handling which the old pavement experienced throughout its life cycle. These factors could break down the asperities of aggregates in old pavement (Krukoswki, 2005). However, the ARC study (ARC, 2010) did not find any particular trend in the gradation chart for RAP

aggregates (extracted via NCAT ignition oven) particles passing No. 200 sieve; it over-estimated in 50% of time and under-estimated in the other 50% of time.

6.6.3 Specific Gravity

Specific gravity values of both coarse and fine aggregates of FRAP1, SRAP1, and AGR1 are listed in Table 6.7, and they were found very comparable. Specific gravity values of SRAP2 aggregates were also comparable with those of their virgin counterparts (AGR2) (Table 6.7). Furthermore, these specific gravity values are in agreement with the ODOT materials division database (ODOT, 2010), where the agency stores quality control (QC) data for engineering properties (gradation, specific gravity, LA Abrasion, etc.) of local aggregates. As noted earlier, the volume of burned off aggregates does not seem to change. Therefore, the specific gravity of the burned off aggregates is also expected to remain the same.

6.6.4 Durability

Durability of aggregates was evaluated by conducting LA Abrasion and Micro-Deval tests. As shown in Figure 6.5a, LA Abrasion loss values of aggregates in FRAP1, SRAP1, and AGR1 were very comparable. However, the LA Abrasion loss value of SRAP2 aggregates was significantly (about 30%) higher than that of AGR2 (Figure 6.5b). The increased LA Abrasion loss for the SRAP2 aggregates could be related to the breakdown of asperities of the aggregate (limestone) due to excessive heat in the NCAT ignition oven, resulting excessive wearing in the LA Abrasion process. In fact, limestone aggregate has a dissociation temperature of 700°C (Krukoswki, 2005), which is close not far from the operation temperature of the

NCAT ignition oven. It can be presumed that partial dissociations have occurred in some burned off aggregates and some of these aggregates disintegrated during the LA Abrasion process.

It is also observed that the LA Abrasion loss values for all tested aggregates are within the limits specified by the ODOT, and they are in agreement with the ODOT's QC database (ODOT, 2010). The increased LA Abrasion value of FRAP1 aggregates compared to that of SRAP1 aggregates could be partly due to the fact that FRAP1 aggregates lost a reasonable degree of hardness during the course of their life in the pavement. These findings are in agreement with test results reported in recent studies (Ahmed et al., 2004; ARC, 2010). Ahmad et al. (2005) reported reduced aggregate crushing and aggregate impact values for RAP aggregates. The ARC study (2010) reported that the estimated measured the LA Abrasion loss values for different aggregates extracted via the NCAT ignition oven method was comparatively higher than those for virgin aggregates. Such over-estimation of LA Abrasion loss values was observed in 75% of time (trials), and it can be concluded that using these values would lead to a conservative design (ARC, 2010). A quite different observation was made for aggregates extracted via other extraction methods (centrifuge and reflux); the measured LA Abrasion loss values were close to the actual values in 75% of time for both cases. The LA Abrasion loss values were under-estimated in 25% of time in case of the centrifuge method, and they were over-estimated in 25% time in case the reflux method.

It was also observed that the variation of the LA Abrasion loss of FRAP1 aggregates was comparatively higher than the others (Figure 6.5a). Recent studies

(O'Rear et al., 2008; ODOT, 2009) have reported significant variations in the quality of RAP aggregates in Oklahoma because of inadequate supervision and QC guidelines. On the contrary, Watson et al. (2008) reported very little variation (within 3%) of the LA Abrasion loss among a few RAP materials.

Micro-Deval test (i.e., durability under wet condition) data of these aggregates are shown in Figures 6.5c and 6.5d, and they show a similar trend to those of the LA Abrasion loss values. The wet durability of SRAP2 aggregates was significantly lower than that of their virgin counterparts. As noted earlier, partial dissociations may have occurred in some burned off aggregates and the associations become weaker due to the absorption of water in the Micro-Deval testing process. Similar to the LA Abrasion test results, the variation of Micro-Deval loss value of FRAP1 aggregates was comparatively higher than that of other aggregates. It should be noted that the original mix design did not contain the Micro-Deval loss value, and it was not practical to produce such data for aggregates used in the mix 15 years ago. Thus, there is no data for aggregate used in the original mix design of the pavement section of FRAP2.

6.6.5 Sand Equivalent

The sand equivalent test results, indicating clay-like particles in fine aggregates, of tested materials are presented in Figures 6.6a and 6.6b. It was observed that SRAP1 aggregates showed a slight increase in the sand equivalent value compared to AGR1 (89 for the former and 85 for the later) (Figure 6.6a). The sand equivalent value of SRAP2 aggregates was significantly higher (about 55%) than that that of AGR2 (Figure 6.6b). Such over-estimate of sand equivalent test data for burned off aggregates imply that a "correction factor" is needed to consider the influence of

the NCAT ignition oven. Prowell and Carter (2000) reported similar findings; the sand equivalent values of the burnt samples (8 out of 10 cases) were considerably higher than that of the virgin samples. The ARC study (2010) also reported that the sand equivalent values of aggregates extracted via the NCAT ignition oven over-estimated 50% of time, indicating un-conservative designs. It should also be noted that the FHWA Mixture Expert Task Group recommended treating RAP as an aggregate stockpile and did not recommend measurement of sand equivalent criterion outlined by Superpave® (Bukowski, 1997).

6.6.6 Insoluble Residue

The insoluble residue test results for tested aggregates are presented in Figures 6.7a and 6.7b. As stated earlier, the original mix of FRAP1 was an Insoluble Type B Mix, and the aggregates had a high insoluble residue value of 40.2% (Figure 6.7a). FRAP1 aggregates do not meet the ODOT solubility requirement anymore. Possible reasons for the loss in percent insoluble residue could be degradation of particles under heavy traffic (3M+) and weathering action. Also, it is clear from Figure 6.6a that the insoluble residue value for SRAP1 aggregates and AGR1 were extremely low, but they were very comparable to each other. Similar observations were made for insoluble test results of SRAP2 aggregates and AGR2 (Figure 6.7b). Insoluble residue data for the same virgin aggregates (AGR1 and AGR2) available in ODOT's QC database (ODOT, 2010) are in agreement with the current study. So, the NCAT ignition oven did not seem to have any noticeable influence on the insoluble residue of extracted aggregates. The reason for virgin aggregates having extremely low insoluble residue was because of changes in source of aggregates. Currently aggregates in the

quarry appear to be limestone, whereas they were mostly sandstone several years ago when the original pavement sections were constructed. Therefore, concentrated HCL acid used in the insoluble residue test was more reactive with carbonates (M_xCO_3) in the limestone aggregates compared to sandstones. Thus, these limestone aggregates do not meet the ODOT insolubility requirements for high traffic volume road anymore.

6.7 CONCLUDING REMARKS

This study evaluates the influences of the Abson method on the PG grading of recovered binder from RAP. It also evaluates the effects of the NCAT Ignition method on the engineering properties of extracted aggregates from RAP. To this end, two bulk RAP materials, along with their virgin materials used in the original pavement sections, were collected. Virgin materials were used to prepare to two HMA mixes and they were long-term conditioned (i.e., simulated RAP) in the laboratory. Properties of extracted aggregates and recovered binders from RAP materials were compared with those of their virgin counterparts. Specifically, the following conclusions can be drawn from the results presented in this paper:

- Laboratory test results of RAP samples with selected binders (PG 76-28 and PG 64-22) reveal that the prolonged use of the centrifuge and heat in the Abson recovery method is expected to make the binder stiffer. The expected increase in the high PG temperature of the recovered binder is up to 4.1^{o}C and that of the low PG temperature is about 3°C. The polymer-modified binder (PG 76-28) was found to be more sensitive to the recovery process than the unmodified binder (PG 64-22).
- Elemental analysis of a selected RAP binder (recovered via Abson) showed a significant increase in the oxygen content compared to the PAV-aged binder,

indicating increased oxidative hardening of the binder during the recovery process.

- The LA Abrasion loss value for the aggregate extracted (NCAT) from RAP was found to be about 30% higher than its virgin counterpart. Similar observations were made in the Micro-Deval test results. Thus, the use of the LA Abrasion and Micro-Deval results of the NCAT ignition oven extracted aggregate is expected to lead to a conservation design.
- The excessive heat in the NCAT ignition oven is expected to increase the sand equivalent value of extracted fine aggregates approximately by 53% compared to their virgin counterparts, which are expected to result in un-conservative designs.
- The NCAT ignition oven does not appear to have significant influence on some other tested engineering properties of extracted aggregates which include specific gravity, gradation, and insoluble residue.
- Variations of some engineering properties (gradation, LA Abrasion loss, Micro-Deval loss, and sand equivalent) of aggregates extracted from the field RAP were found to be comparatively higher than their virgin counterparts, indicating inadequate supervision and quality control guidelines for RAP.

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Table 6.1 Relevant Existing Literature Related to Binder Recovery

Reference	Materials and methodology	Findings and Recommendations
Stroup-Gardiner and Nelson (*6*)	Evaluated TCE and four normal propyl bromide (nPB) solvents (Lenium, Leksol, Hypersolv, and EnSolv) in extraction and recovery of binders.	• TCE produced stiffer recovered binder than its virgin counterpart; Hypersolv was found to be incompatible with polymer-modified binder. • Recommended to use nPB solvents as direct replacements for the TCE solvent.
Tao et al. (*7*)	Recovered binders from RAP using Abson and suspected that some chemicals remain as residuals	• Proposed a new testing procedure to estimate the low-temperature properties of the RAP binder without extraction and/or any chemical treatments. • Tested RAP mortar (mix of fresh binder and RAP materials, using a bending beam rheometer (BBR) with minor modifications.
Burr et al. (*8*).	Investigated chemical interactions of solvent and binder in the Abson method	• Suspected chemical reactions during the process which are very likely to produce a very narrow range of products, and the reaction rates vary considerably with asphalt binder source, solvent and solution conditions
Daniel et al. (*9*)	Evaluated PG grading of recovered binders and their critical temperatures.	• Used an additional step to remove the last traces of TCE, if any, from the recovered binder. • Observed that the high PG temperature increased up to one grade for the various percentages of RAP and the low PG temperature increased up to one grade from the virgin mix.
Doh et al. (*10*)	Studied two PG binders and an aged binder (recovered from RAP).	• Reported significant aging of the recovered binder in terms of kinematic viscosity and penetration, among others.
McKeen (*11*)	Conducted a round-robin study to obtain data to determine the precision of the NCAT ignition method.	• Reported, "test results indicated the precision is equal to that reported for reflux extractions and nuclear asphalt gages. Aggregate gradations were not changed by the ignition test based on a comparison of before and after gradation data." • Recommended adopting the NCAT oven for aggregate extraction in AASHTO specifications.
Ahmad et al. (*12*)	Studied the Abrasion of RAP aggregates after extracting from RAP using an ignition oven.	• Reported that extracted aggregates were finer (gradation) than the virgin counterparts; Aggregate crushing value (ACV) and aggregate impact value (ACV) of recovered aggregates was lower than virgin aggregates. However, all three parameters (gradation, ACV and AIV) of RAP aggregates were within their corresponding acceptable ranges.
Watson et al. (*13*)	Studied LA Abrasion loss of blended aggregates composed of different percentages of recycled SMA mixes and four virgin aggregates, all were granite materials.	• Reported that the variation of LA Abrasion loss among RAP materials found to be very little (within 3% difference). • Observed that RAP consisted of aggregate that had many of its rough edges broken off during original production, through the milling process, and through additional crushing.

Table 6.2 Gradation and Mix Design Properties of Original Pavement of FRAP1

Source and proportion of materials							
Material			Source				% Used
5/8-inch chips			The Dolese Co. @ Cyril, Oklahoma				51
Coarse Screenings			The Dolese Co. @ Richard Spur, Oklahoma				13
Stone Sand			The Dolese Co. @ Davis, Oklahoma				25
Asphalt Sand			GMI (Meridian Pit) @ Oklahoma City, Oklahoma				11
Asphalt Binder PG 76-28			Koch Materials[1] (Ergon) @ Muskogee, Oklahoma				
Percent Passing from different sieve sizes							
Aggregate size	5/8 inch chips	Coarse Screenings	Stone Sand	Asphalt Sand	Combined Aggregate	Job Formula	JMF Tolerance
¾-inch	100	100	100	100	100	100	±0
½-inch	90	100	100	100	95	95	±7
3/8-inch	66	100	100	100	83	83	±7
No. 4	11	92	98	98	53	53	±7
No. 10	4	47	68	97	33	36	±4
No. 40	4	17	10	85	16	16	±4
No. 80	4	12	4	27	8	8	±4
No. 200	2.5	9.8	4.7	2.0	3.9	3.9	±2
% Asphalt Binder PG 76-28						4.7	±0.4
Mixing Temperature						163	±11
Optimum Roadway Compaction Temperature						152	
Aggregate Test Data							
Properties			Measured Value			ODOT Requirement	
LA Abrasion loss			27.0			< 40	
Sand Equivalent			81			> 45	
Insoluble Residue			40.2			> 40	
Ignition Oven Correction			0.48				
Mixture Test Data							
Asphalt Binder (%)	Sp. Gr. of mix	Maximum Theoretical Sp. Gr.	Density % of maximum Theoretical	Density % required of maximum Theoretical	Void in Mineral Aggregate (%)	Void in Mineral Aggregate (Minimum)	Hveen Stability
4.3	2.354	2.506	93.9		16.0		50
4.8	2.374	2.487	95.5	94-96	15.7	15	51
5.3	2.406	2.469	97.4		15.0		49

[1]Currently owned by Ergon

Table 6.3 Gradation and Mix Design Properties of Original Pavement of FRAP2

Source and proportion of materials			
Material		Source	% Used
3/4-inch rocks		The Dolese Co. @ Davis, Oklahoma	17
Screenings		Western Rock @ Davis, Oklahoma	36
Stone Sand		The Dolese Co. @ Davis, Oklahoma	12
RAP		Plant site	25
Sand		The Dolese Co. @ Yukon, Oklahoma	10
Asphalt Binder PG 76-28		Total Petroleum[1] (Valero) @ Ardmore, Oklahoma	

Percent Passing from different sieve sizes								
Aggregate size	3/4-inch rocks	Screenings	Stone Sand	RAP	Sand	Combined Aggregate	Job Formula	JMF Tolerance
¾-inch	100	100	100	100	100	100	100	±0
½-inch	41	100	100	98	100	100	90	±7
3/8-inch	8	100	100	95	100	100	83	±7
No. 4	4	81	99	74	99	99	70	±7
No. 10	3	43	64	54	97	97	47	±4
No. 40	2	18	14	32	74	74	24	±4
No. 80	2	12	4	16	20	20	11	±4
No. 200	0.6	8.2	2.0	9.2	20	2.0	5.8	±2
% Asphalt Binder PG 64-22							5.6	±0.4
Mixing Temperature, ^{o}C							152	±11
Optimum Roadway Compaction Temperature, ^{o}C							143	

Aggregate Test Data			
Properties		Measured Value	ODOT Requirement
LA Abrasion loss		23.5	< 40
Sand Equivalent		63	> 45
Ignition Oven Correction		0.32	

Mixture Test Data								
Asphalt Binder (%)	Sp. Gr. of mix	Maximum Theoretical Sp. Gr.	Density % of maximum Theoretical	Density % required of maximum Theoretical		Void in Mineral Aggregate (%)	Void in Mineral Aggregate (Minimum)	Hveen Stability
4.7	2.355	2.477	95.1			15.7		45
5.2	2.373	2.458	96.7	95-97		15.5	15	43
5.7	2.391	2.440	98.0			15.3		41

[1]Currently owned by Valero

Table 6.4 List of Tests and Their Designations

Material	Test name and designation	Field RAP	Simulated RAP	Virgin Materials
Binder	PG grade: AASHTO M 320	Yes	Yes	Yes
	DSR: AASHTO T 315	Yes	Yes	Yes
	RTFO: AASHTO T 240	Yes	Yes	Yes
	PAV: AASHTO R 28	Yes	Yes	Yes
	BBR: AASHTO T 313	Yes	Yes	Yes
Aggregate	Gradation: AASHTO T 30, T 27	Yes	Yes	Yes
	LA Abrasion: AASHTO T 96	Yes	Yes	Yes
	Micro-Deval: AASHTO T 327	Yes	Yes	Yes
	Sp. Gr.: AASHTO T 84, T 85	Yes	Yes	Yes
	Sand equivalent: AASHTO T 176	Yes	Yes	Yes
	Insoluble residue: OHD L-25	Yes	Yes	Yes

Note: RV = Rotational viscosity, DSR = Dynamic shear rheometer, RTFO = Rotational thin film oven, PAV = Pressure aging vessel, and BBR = Bending beam rheometer.

Table 6.5 BBR Test Results for FRAP1 Binder and its Counterparts

Binder Type	Aging Condition	Testing Temp	Stiffness (MPa)	m-value	Low PG Temp (^{o}C)		
					From Stiffness	From m-value	Overall low PG Temp
PG 76-28	PAV	-18	156.34	0.337	-38.0	-33.7	-33.7
		-21	210.50	0.317			
		-24	262.29	0.298			
PAV-aged PG76-28	RTFO	-12	46.14	0.380	-31.1	-30.6	-30.6
		-15	83.85	0.352			
Recovered from FRAP1	RTFO	-9	124.74	0.307	-26.6	-19.8	-19.8
		-12	176.07	0.285			
Recovered from SRAP1	RTFO	-9	56.53	0.382	-32.5	-22.7	-22.7
		-12	82.02	0.361			
PG 64-22	PAV	-9	108.90	0.330	-33.7	-24.0	-24.0
		-12	145.70	0.316			
		-15	185.40	0.292			
PAV-aged PG 64-22	RTFO	-6	76.53	0.317	-35.1	-18.8	-18.8
		-9	111.65	0.299			
Recovered from SRAP2	RTFO	-6	86.45	0.326	-32.1	-18.6	-18.6
		-9	126.34	0.296			

Table 6.6 Elemental Analysis of Virgin and Recovered Binders

Binder Type	Composition[1] (%)			
	Carbon	Hydrogen	Nitrogen	Oxygen
Unaged PG 64-22	85.06	10.43	0.69	0.81
PAV-aged PG 64-22	84.69	10.44	0.72	1.16
Recovered from SRAP2	77.12	9.14	0.62	2.76

[1] Sulfur content was not determined

Table 6.7 Specific Gravity of Aggregates

Aggregate Type	Coarse/Fine	Specific Gravity
Extracted From FRAP1	Coarse	2.646
	Fine	2.627
Extracted from SRAP1	Coarse	2.559
	Fine	2.637
Virgin Aggregates used in SRAP1	Coarse	2.656
	Fine	2.635
Extracted from SRAP2	Coarse	2.570
	Fine	2.445
Virgin Aggregates used in SRAP2	Coarse	2.607
	Fine	2.502

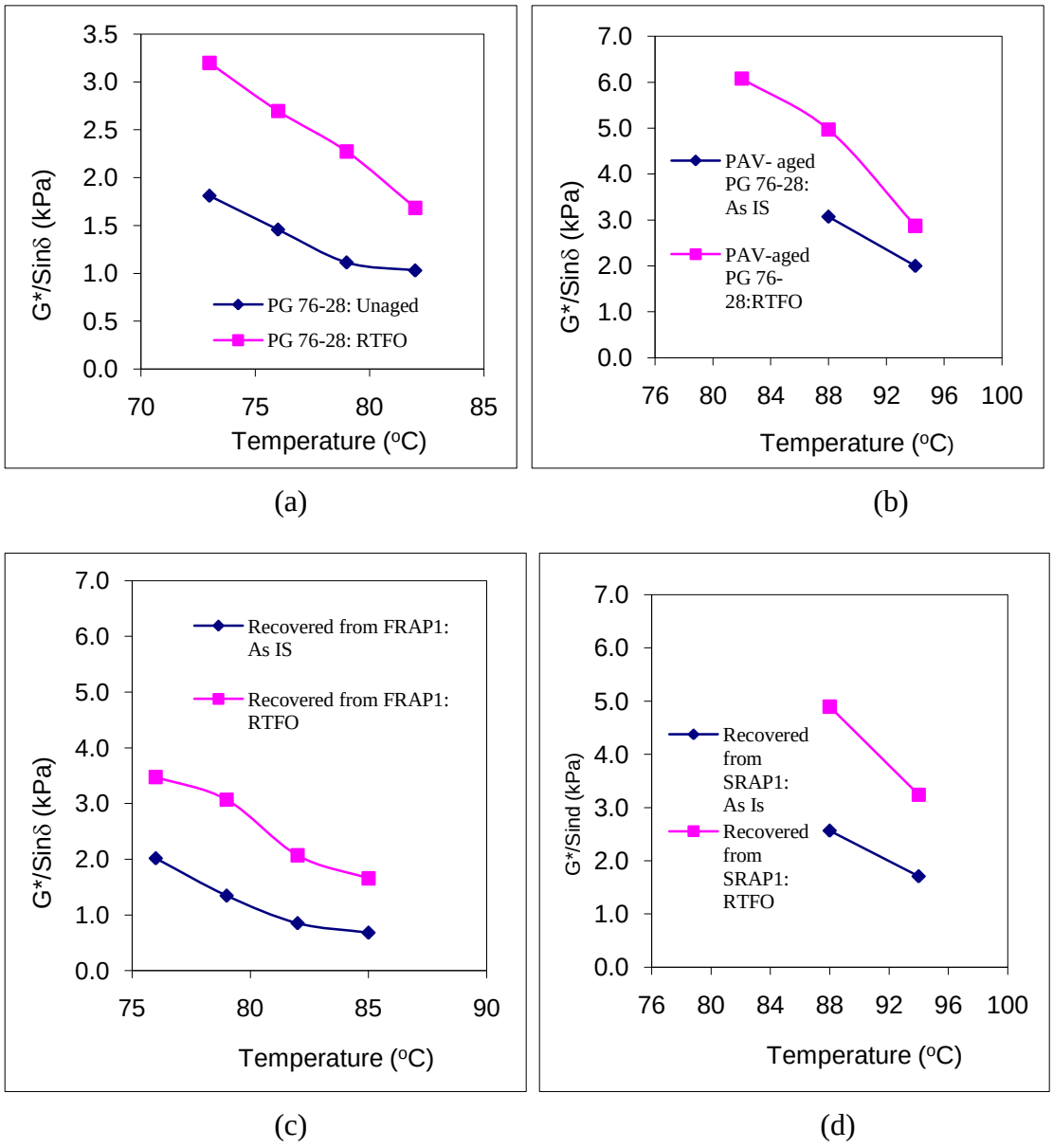
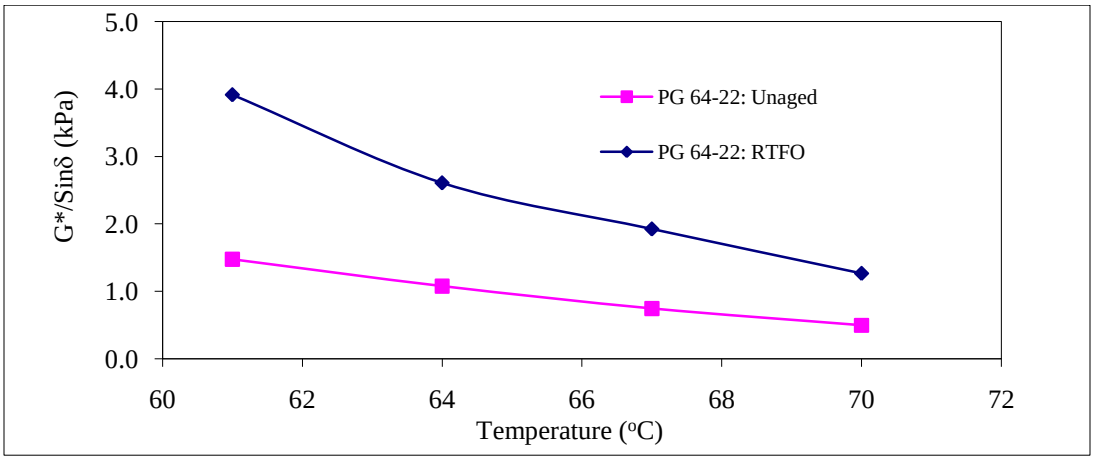
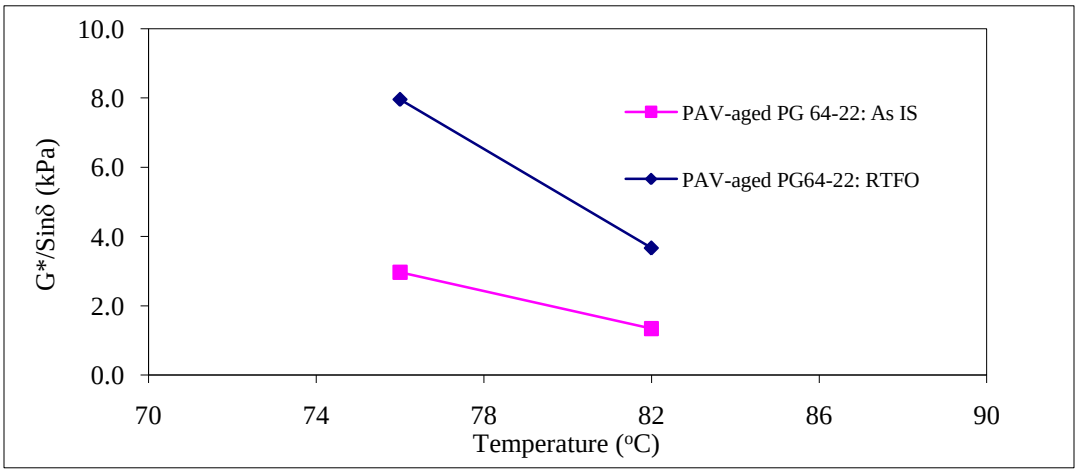


Figure 6.1 DSR Test Results: (a) Virgin PG 76-28, (b) PAV-aged PG 76-28, (c) Recovered Binder from FRAP1, and (d) Recovered Binder from SRAP1.



(a)



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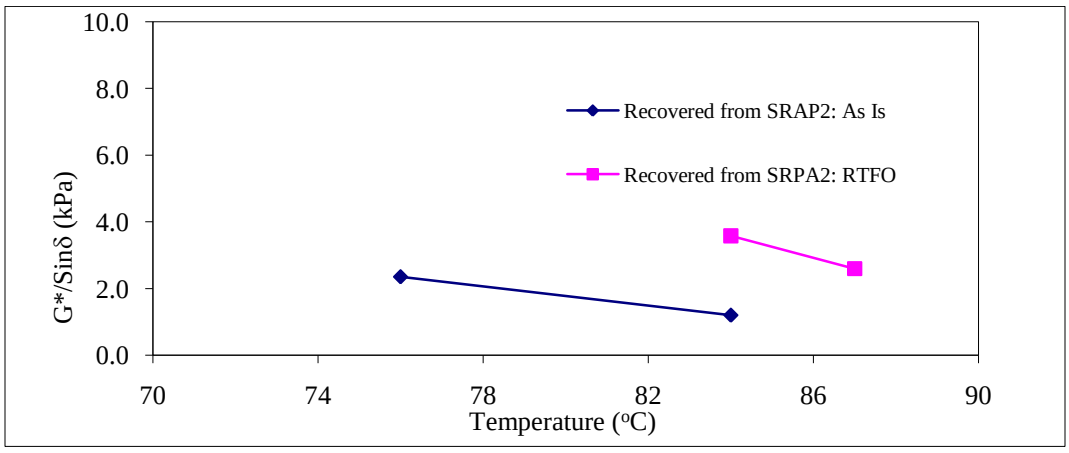
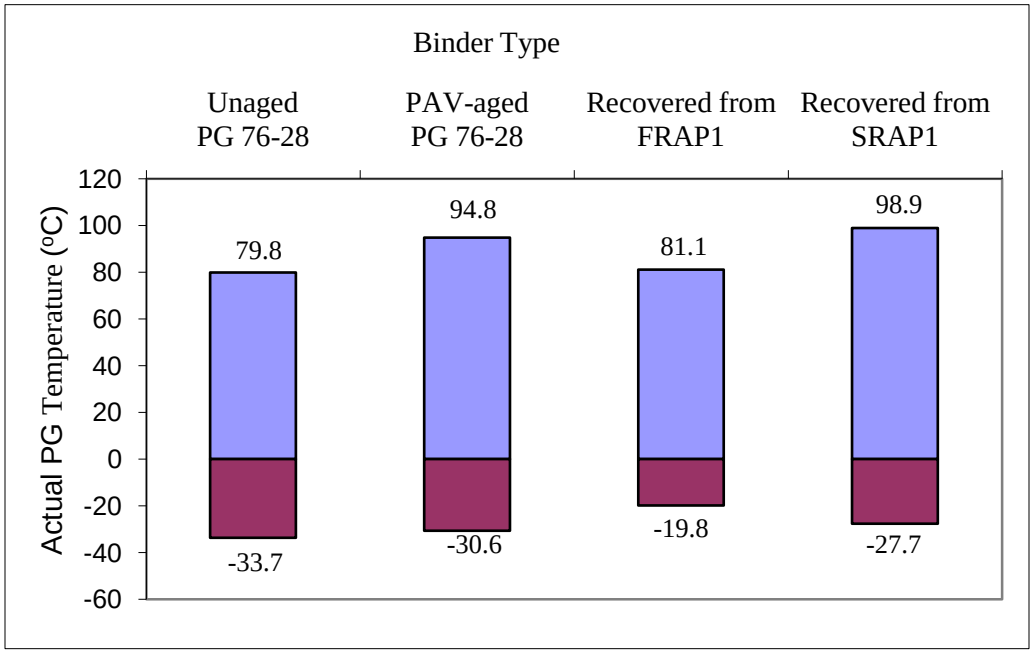
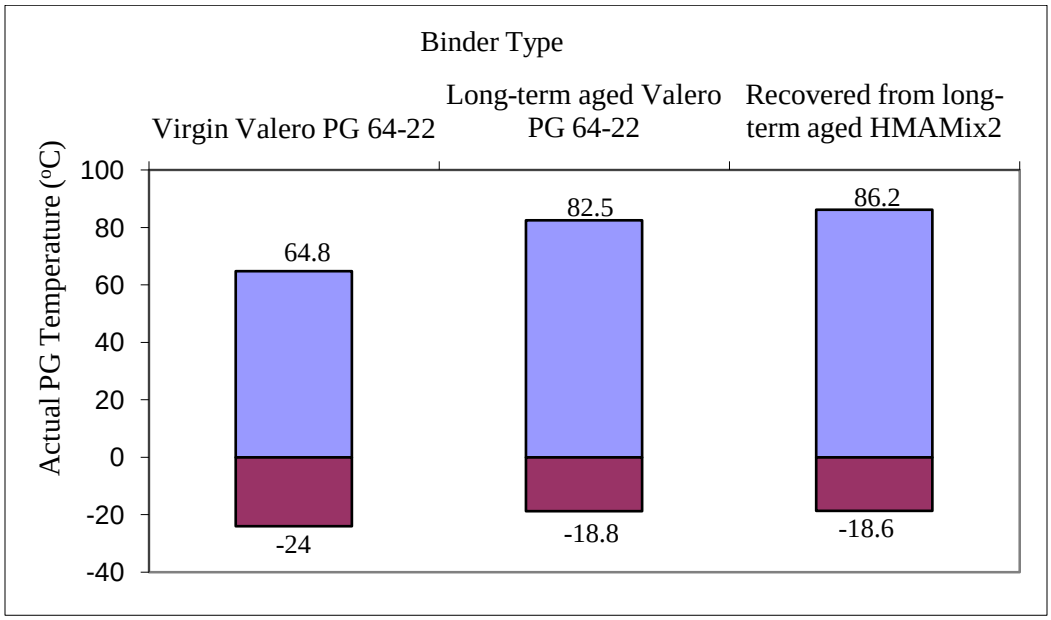


(c)

Figure 6.2 DSR Test Results: (a) Virgin PG 64-22, (b) PAV-aged PG 64-22, and (c) Recovered Binder from SRAP2.

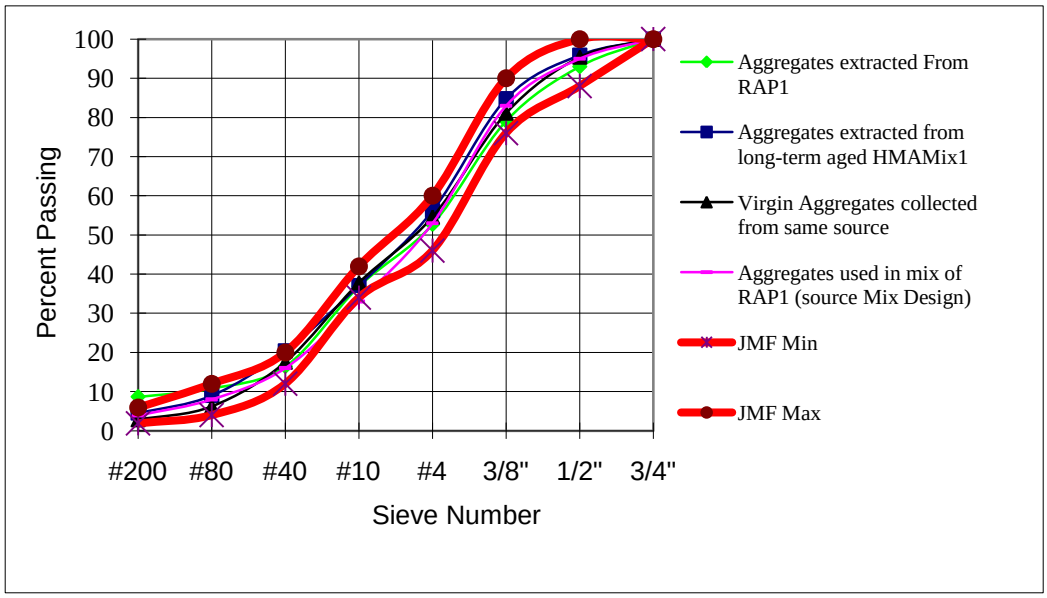


(a)

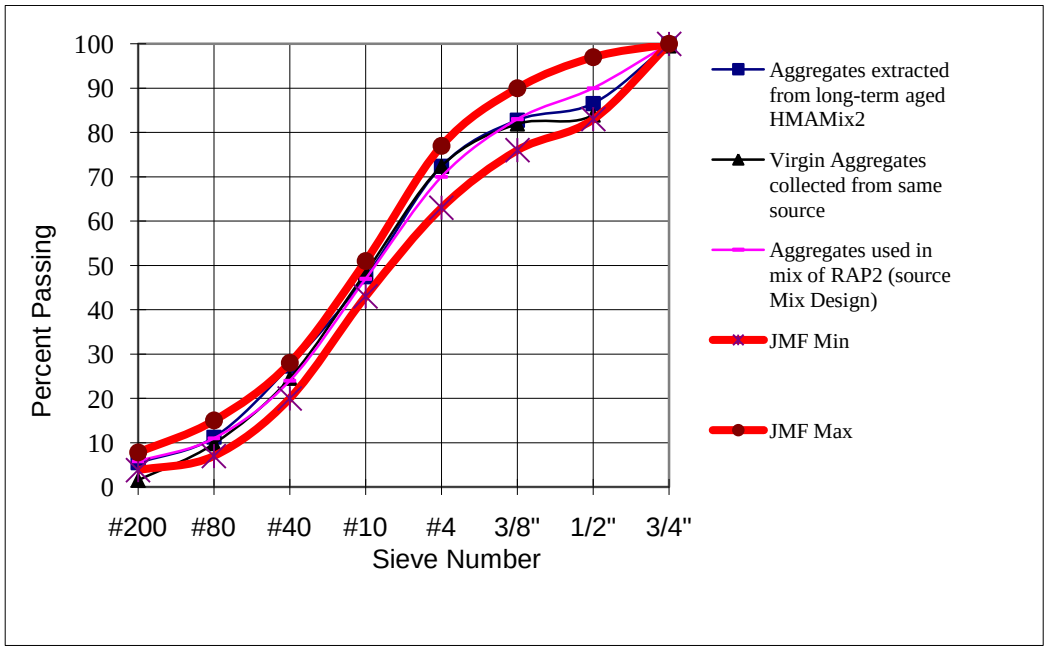


(b)

Figure 6.3 Grades of Virgin, Laboratory-Conditioned and Recovered Binders: (a) Ergon PG 76-28, and (b) Valero PG 64-22.

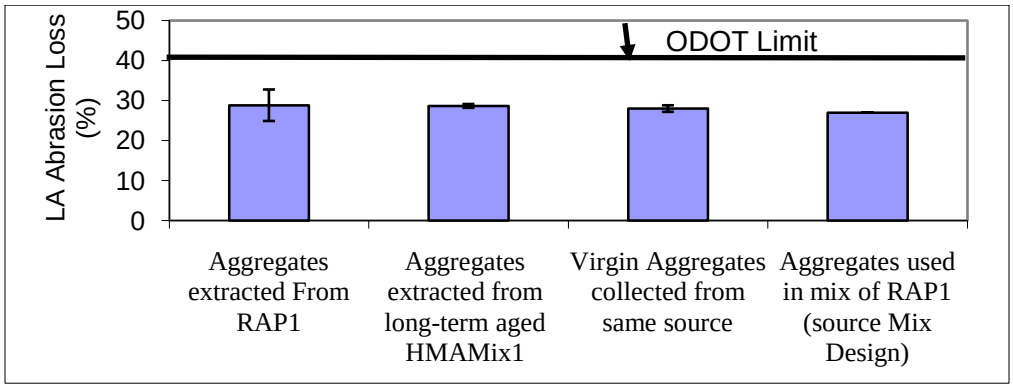


(a)

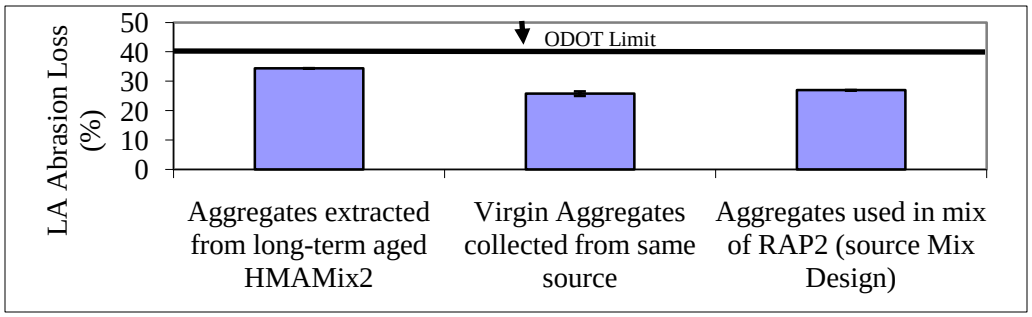


(b)

Figure 6.4 Gradations charts and Sp. Gr. of Aggregates: (a) Extracted from FRAP1 and its Counterparts, and (b) Extracted from SRAP2 and its Counterpart.



(a)



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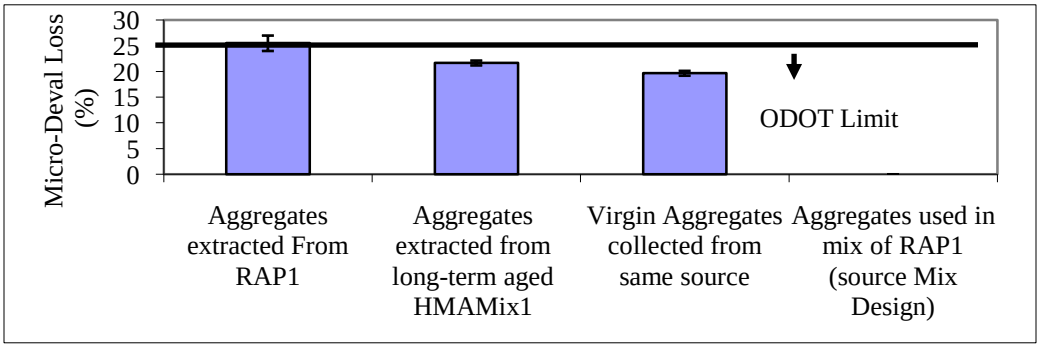
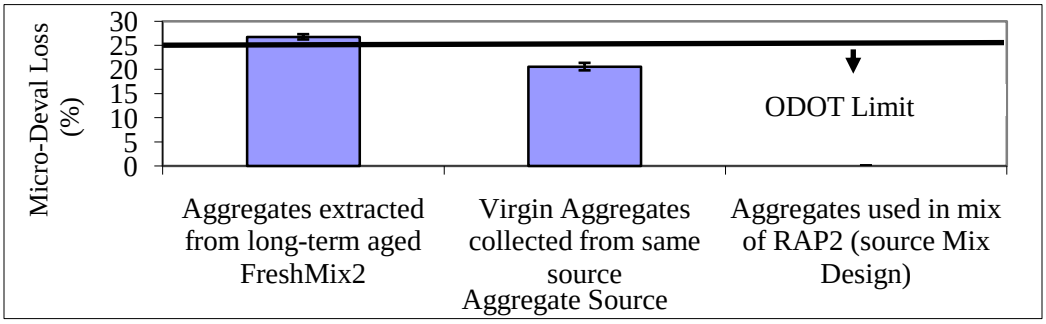
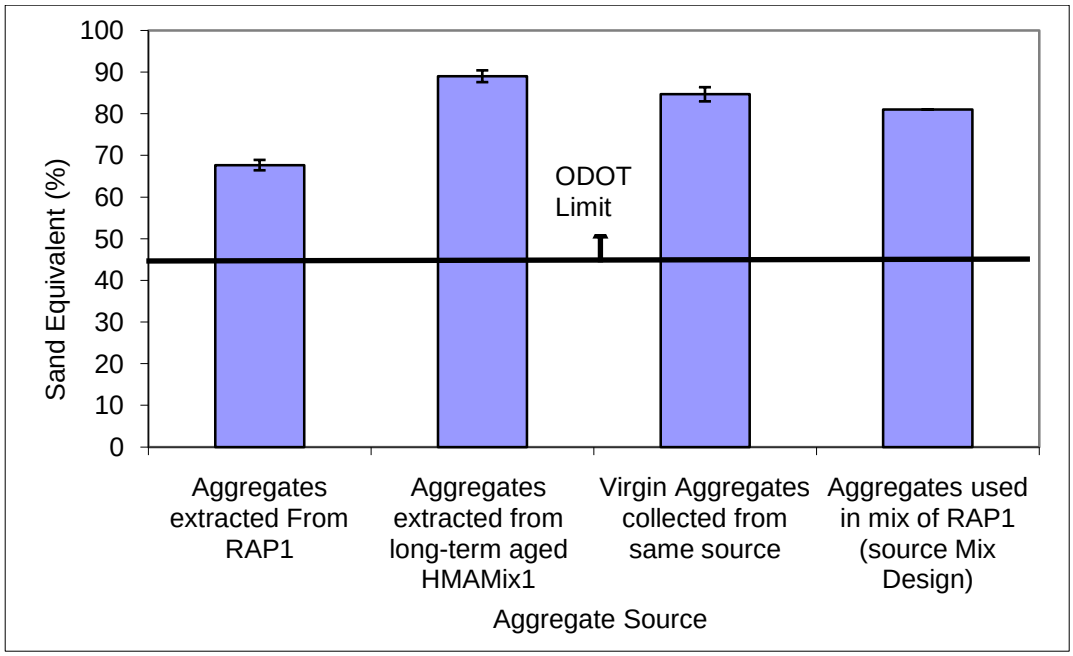


(c)

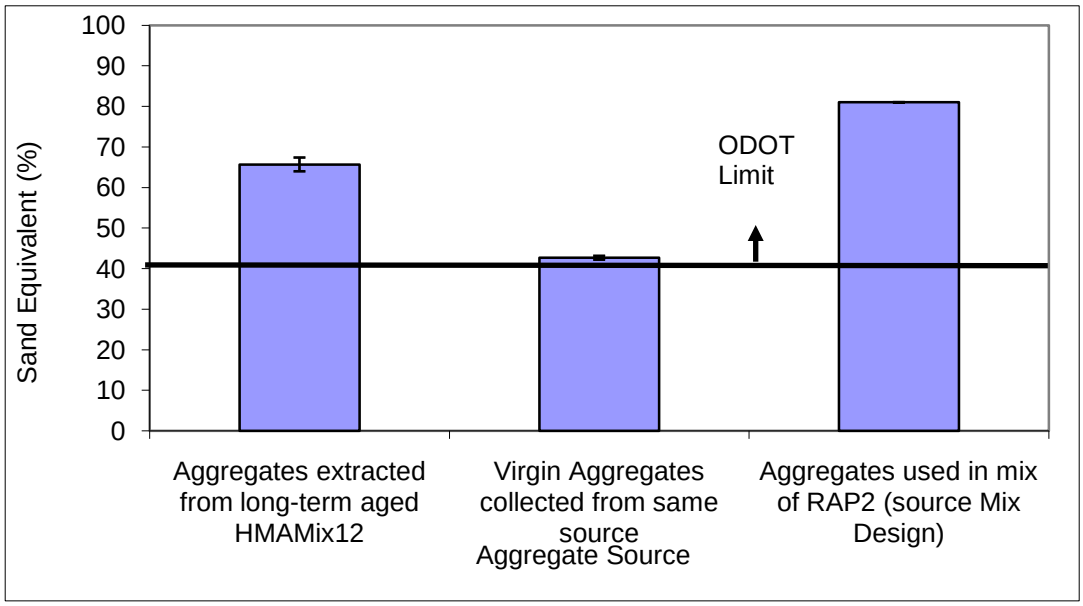


(d)

Figure 6.5 Durability Test Results: (a)-(b) LA Abrasion, and (c)-(d) Micro-Deval.

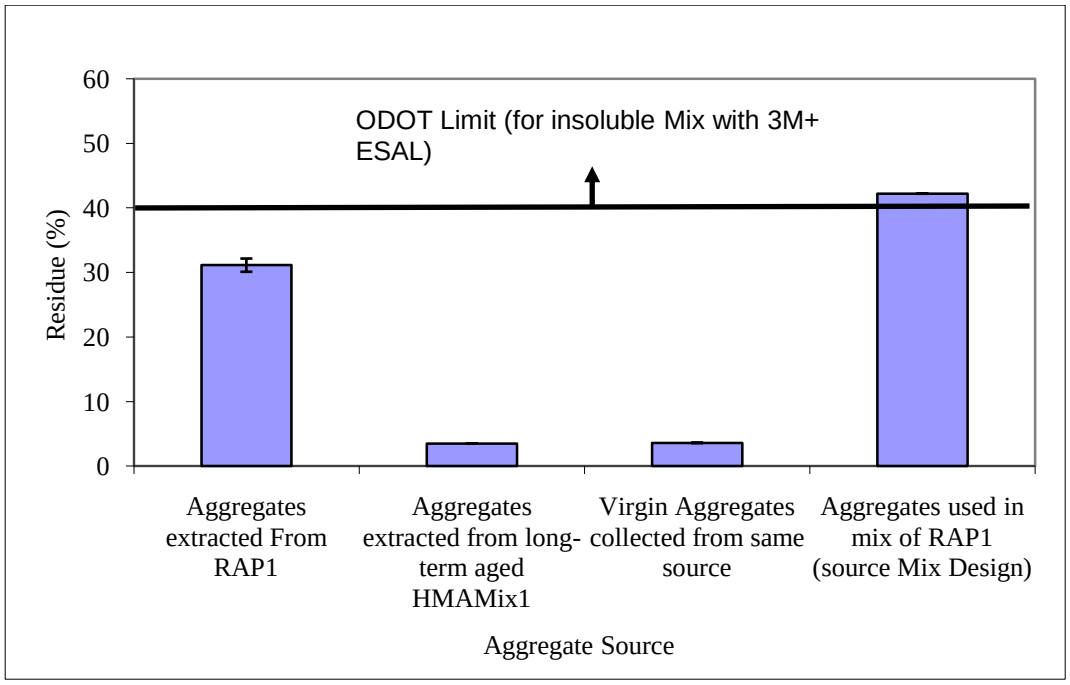


(a)

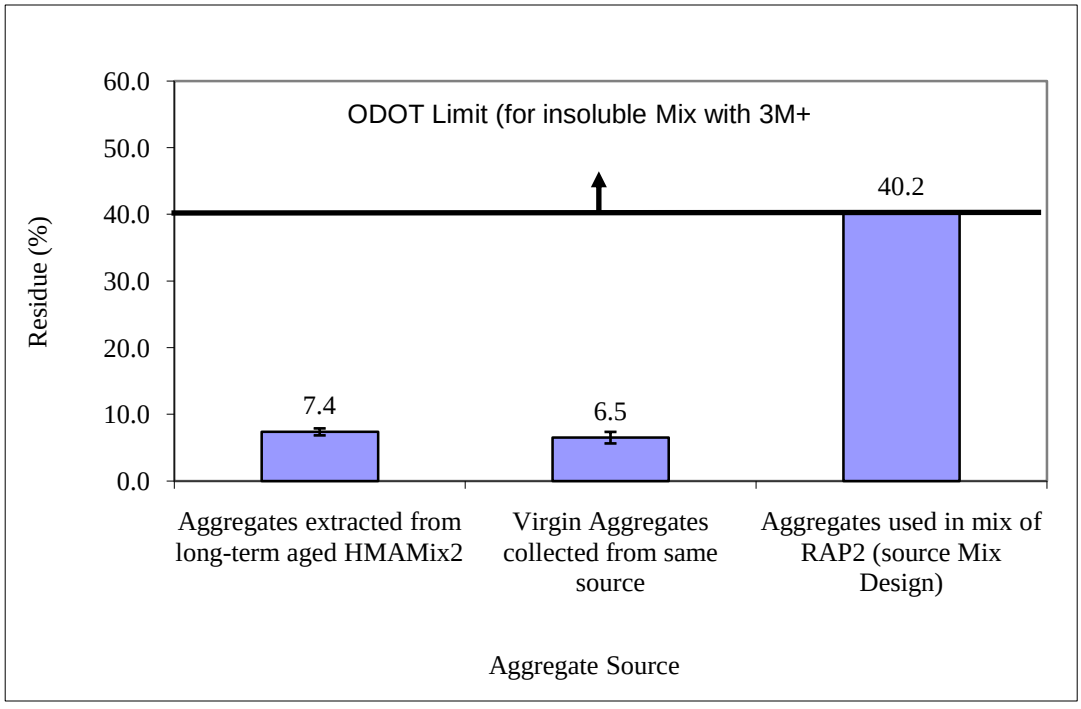


(b)

Figure 6.6 Sand Equivalent Test Results of Extracted and Virgin Aggregates: (a) FRAP1 and its Counterparts, and (b) SRAP2 and its Counterparts.



(a)



(b)

Figure 6.7 Acid Insoluble Residue Test Results of Different Aggregates: (a) FRAP1 and its Counterparts, and (b) SRAP2 and its Counterparts.

7 SENSITIVITY ANALYSIS OF DISTRESS FACTORS USING LOCAL PERFORMANCE GRADE BINDERS[6]

7.1 ABSTRACT

For designing pavements many state agencies, including Oklahoma, use the 1993 AASHTO Guide for Design of Pavement Structures, which is empirical in nature. The new 2002 Mechanistic-Empirical Pavement Design Guide (MEPDG) predicts pavement distresses in a more mechanistic approach, based on material properties, local traffic and climate conditions. Among material properties in the MEPDG, the dynamic modulus (E*) of the asphalt mix is one of the key parameters to achieve the highest level of design reliability. The present study was conducted to estimate E* values of two commonly used hot mix asphalt (HMA) mixes (S3 and S4) in Oklahoma. Different design reliability levels, based on rheological properties of three performance grade (PG) binders, were considered. These asphalt binders were collected from three different sources in Oklahoma. Furthermore, the sensitivity of four major pavement distresses (rutting, fatigue cracking, thermal cracking, and roughness) to the design reliability, and asphalt binder type and source was evaluated by using the MEPDG software version 1.100. It was observed that rotational viscometer (RV) test data overestimate the E* values in the case of stiff binders. Conversely, dynamic shear rheometer (DSR) test data significantly underestimate the E* values of asphalt mixes with all PG binders. Among distress factors, the rutting in HMA layers' was found to be highly sensitive to the design reliability and asphalt binder's PG grade. The asphalt binder source was also found to be somewhat sensitive

[6] This chapter or portion thereof has been submitted for possible publication as a technical paper in the Journal of Materials in Civil Engineering. The current version has been formatted for this dissertation.

to rutting. Other distresses were not significantly influenced by the design reliability, asphalt binder PG grade, and source. The findings of this study are expected to provide transportation professionals with a better understanding of material input parameters that influence E* values of asphalt mixes and pavement distresses, and assist in implementing the MEPDG for local conditions.

Keywords: Mechanistic-empirical pavement design guide, sensitivity analysis, rutting, hot mix asphalt, asphalt binder, dynamic shear modulus.

7.2 INTRODUCTION

The new Mechanistic-Empirical Pavement Design Guide (MEPDG), introduced under the National Cooperative Highway Research Program (NCHRP) project 1-37A, replaces the widely used 1993 AASHTO Design Guide, which is more empirical in nature. The implementation of the MEPDG with enhanced design reliability requires mechanistic characterization of pavement materials and calibration of performance prediction models for local conditions (Khazanovich, 2010; Quintus, 2010). Among several material properties in the analysis and design of hot mix asphalt (HMA) pavements, mechanical and rheological properties of mixes and asphalt binders are required input parameters.

At the highest possible design reliability level (*Level 1*) in the hierarchical approach of the MEPDG, the dynamic modulus (E*) of the mix and Superpave® test data of the asphalt binder are used as input. Superpave® binder test data consist of dynamic shear modulus (G*) and phase angle (δ) values of rotational thin film oven (RTFO)-aged asphalt binder, determined by using a dynamic shear rheometer (DSR) in accordance with AASHTO T 315 (NCHRP, 2004). As an alternative to the

Superpave® test data, conventional binder test data (flash point, absolute and kinematic viscosities, and penetration or Brookfield viscosity) can be used as input. The MEPDG uses the E* values of the mix and the asphalt binder data to estimate the E* master curve.

Level 2, intermediate reliability level, does not require laboratory testing of E*. The Witczak's predictive equation (Equation 7.1) is used to calculate E* of the mix by using the asphalt binder test data, which is same as in *Level 1*. In Equation 7.1, the asphalt binder viscosity (η) can be determined by using Equation 7.2, if the binder's G* and δ values are known (Bari and Witczak, 2006). Once the η value is determined, the ASTM viscosity temperature susceptibility (VTS) parameters, shown in Equation 7.3, are found by linear regression analysis after log-log transformation of the viscosity and log transformation of the temperature data (Bari and Witczak, 2006).

Level 3, the lowest reliability level, does not require any laboratory E* testing of the mix. Superpave® binder properties include its standard (6°C interval) PG grading. Predictive equation (Equation 7.1) is then used to calculate E* of the mix by using typical ASTM A and VTS parameters based on the binder's PG grade.

$$\log E^* = 1.249937 + 0.249937 + 0.02932P_{200} - 0.001767(P_4)^2 - 0.002841P_4 - 0.058097V_a - 0.802208\left(\frac{V_{beff}}{V_{eff} + V_a}\right) + \frac{3.871977 - 0.0021P_4 + 0.003958P_{38} - 0.000017(P_{38})^2 + 0.00547P_{34}}{1 + e^{(-0.603313 - 0.313351\log(f) - 0.393532\log(\eta))}} \quad (7.1)$$

where,

E* = dynamic modulus (10^5 psi),

η = asphalt binder (RTFO-aged) viscosity at temperature of interest (10^6 Poise),

f = loading frequency (Hz),

V_a = air void content (%),

V_{beff} = effective asphalt content (% by volume),

P_{34} = cumulative % retained on 3/4 in (19 mm) sieve,

P_{38} = cumulative % retained on 3/8 in (9.5 mm) sieve,

P_4 = cumulative % retained on #4 (4.76 mm) sieve, and

P_{200} = % passing #200 (0.075 mm) sieve.

$$\mu = \frac{G^*}{10}\left(\frac{1}{\sin\delta}\right) \tag{7.2}$$

where,

η = asphalt viscosity (cP),

G^* = binder complex shear modulus (Pa), and

δ = binder phase angle (^{o}).

$$\log\log\mu = A + VTS\log T_R \tag{7.3}$$

where,

η = asphalt viscosity (cP),

A, VTS = regression parameters, and

T_R = temperature (°Rankine).

To perform *Level 1* analysis, the MEPDG recommends using E* data of five test temperatures and four frequencies (NCHRP, 2004). The MEPDG, however, allows the use of E* data up to eight test temperatures and six frequencies. The E*

values at different temperatures and frequencies are used to develop master curves at a reference temperature, which is generally 70°F (21.1^{o}C) (Bari and Witczak, 2006). Master curves are constructed using the principle of time-temperature superposition (TTS) principles. The E* data at various temperatures are shifted with respect to time until the curves merge into a single smooth sigmoidal function, as given in Equation 7.4.

$$\log[E^*] + \delta + \frac{\alpha}{1 + e^{\beta + \gamma(\log t_r)}} \tag{7.4}$$

where,

E* = dynamic modulus of mix (psi),

t_r = reduced time of loading at reference temperature (sec),

δ = minimum value of E*,

$\delta + \alpha$ = maximum value of E*, and

β, γ = parameters describing the shape of the sigmoidal function.

The shift factor can be shown in the form as shown in Equation 7.5.

$$a(T) = \frac{t}{t_r} \tag{7.5}$$

where,

a(T) = shift factor as a function of temperature,

t = time of loading at desired temperature,

t_r = reduced time of loading at reference temperature, and

T = temperature of interest.

For precision, a second order polynomial relationship between the logarithm of the shift factor and the temperature in degrees Fahrenheit, as shown in Equation 7.6, is used (Bari and Witczak, 2006).

$$\log a(T_i) = aT_i^2 + bT_i + c \quad (7.6)$$

where,

$a(T_i)$ = shift factor as a function of temperature T_i,

T_i = temperature of interest (°F), and

a, b and c = coefficients of the second order polynomial.

Previous studies (e.g., Shah et al., 2005; King et al., 2005; El-Badawy et al., 2009) reported binder stiffness as one of the most influential factors of the E* value of the mix. Shah et al. (2005) reported that E* values of mixes made with PG 70-28 binders were found to be significantly higher than those of mixes containing PG 58-28 binders. According to El-Badawy et al. (2009), binder stiffness had significant impact on the development of the fatigue damage. It was recommended that this parameter (binder stiffness) be considered as a variable in the final fatigue damage model. These and other studies indicate that rheological characterization of asphalt binders is getting more attention from transportation communities, especially while using the MEPDG. Realizing the need and urgency for the implementation of the MEPDG, many state agencies have already developed or are in the process of developing inventories of rheological data of their asphalt binders. Some of these initiatives are mentioned next.

Clyne and Marasteanu (2004) tested nine certified asphalt binders used in Minnesota from six refineries around the state and created an inventory of rheological properties by conducting Superpave® binder tests. Kim et al. (2005) evaluated the

relative sensitivity of several distress factors of two flexible pavement sections to asphalt concrete (AC) properties, traffic, and climatic conditions in Iowa. PG grade of the asphalt binder was found to be one of the key parameters that generally influenced most of these distresses. It was reported that the predicted transverse cracking was very sensitive to material properties and climate. Furthermore, the surface course rutting dominated the total rutting in relatively thick pavements and it was very sensitive to binder's PG grade. Alligator cracking was not found to be a critical distress in the selected thick pavement section. Longitudinal cracking was influenced by most input parameters, whereas roughness was not sensitive to the asphalt binder's PG grade. Another study by Li et al. (2009), reported similar findings for conditions in Washington. These researchers reported longitudinal cracking to be highly sensitive, whereas transverse cracking, alligator cracking and rutting were only moderately sensitive to the asphalt binder's PG grade.

Flintsch et al. (2007) conducted laboratory testing on 11 plant mixes with a PG 64-22 binder toward implementing the MEPDG in Virginia. These researchers used *Level 1* input for these mixes. However, they used *Level 3* input for the asphalt binder, which is a major limitation of that study. Bahia et al. (2009) suggested that actual values of G* and δ be used as inputs into the MEPDG rather than an asphalt binder's PG grade for a more reliable estimation of performance. Another study by Birgisson et al. (2005), however, reported that the DSR data significantly underestimated the E* value of the mix, meaning that it overestimated pavement distresses.

Like many other state departments of transportation, the Oklahoma Department of Transportation (ODOT) is actively working toward implementing the

MEPDG for flexible pavements (Hossain et al., 2011). A successful implementation of the MEPDG will require a comprehensive database for local asphalt materials and an assessment of the database through calibrations of local materials. The findings of the present study are expected to generate necessary data for calibrating the MEPDG software for conditions prevailing in Oklahoma. The outcome of this study is also expected to provide a better understanding of how to evaluate and incorporate new materials into the MEPDG.

7.3 OBJECTIVES

Major objectives of this study are to: (a) develop an inventory of rheological data of some common PG binders in Oklahoma, (b) estimate the MEPDG material input parameters of these binders at different design reliability levels, (c) predict E* values of selected HMA mixes by using asphalt binders' properties, and (d) perform sensitiveness of major MEPDG distress factors of a typical pavement section in Oklahoma to binder input parameters.

7.4 MATERIALS AND METHODOLOGY

Three commonly used PG binders (PG 64-22, PG 70-28 and PG 76-28) certified by ODOT were collected from three major refineries (sources) in Oklahoma. Henceforth, these sources are called SRC1, SRC2, and SRC3. The collected asphalt binders were tested in the laboratory to determine their MEPDG input parameters, continuous PG grades, and rotational viscosity values. Superpave® binder test methods included measurement of viscosity using a Brookfield viscometer (AASHTO T 316), evaluation of G* and δ using a DSR (AASHTO T 315), and determination of low

temperature stiffness (S) and rate of stress relaxation (m-value) using a bending beam rheometer (BBR) (AASHTO T 313). Short-term and long-term aging of binders were accomplished by using a RTFO (AASHTO T 240) and a pressure aging vessel (PAV) (AASHTO R 28), respectively. At least three replicate samples were tested at each temperature to ensure the repeatability of test results.

Volumetric properties and E* data of two asphalt mixes (one surface course (S4) mix and one base course (S3) mix), each with three selected PG binders from SRC2, as shown in Table 7.1, were reported in a related study (Cross et al., 2007). Henceforth, the mix designs of the S4 and S3 mixes are called MixDesign#1 and MixDesign#2, respectively. Aggregates used in MixDesign#1 and MixDesign#2 were predominately limestone and rhyolites, respectively.

7.5 TEST RESULTS AND DISCUSSIONS

7.5.1 Viscosity

As suggested by the Superpave®, for proper pumping and flow behavior, the rotational viscosity of the unaged binder at 135°C must be less than 3 Pa.s (Roberts et al., 1996). Figure 7.1(a) shows that the tested binders met the Superpave® specified viscosity requirement. In general, within the same PG grade level, an asphalt binder from SRC3 was found to be more viscous than that from SRC1 or SRC2. In particular, the PG 76-28 binder from SRC3 was found to be the most viscous binder, which barely passed the Superpave® viscosity requirement. It is also observed that viscosity values of the asphalt binders from SRC1 and SRC2 are very comparable. Viscosity data of the RTFO-aged binders, shown in Figure 7.1(b), was used to calculate ASTM

A and VTS parameters using Equation 7.3, which were then used to estimate E* values of the mixes using Equation 7.1.

7.5.2 Performance Grade

It can be noted that, for *Level 3* analysis, the MEPDG allows users to input the Superpave® PG grade (6°C interval) of the asphalt binder. However, the continuous PG grades, as shown in Table 7.2, indicates the binder's actual high and low critical temperatures. All tested binders met the manufacturer-specified PG grades. A few of the tested asphalt binders' actual high PG temperatures were found to be significantly higher than their Superpave® PG grades. For example, the actual high critical temperature of the PG 76-28 binder from SRC2 was found to be 81.8°C. Therefore, the manufacturer-certified Superpave® PG grade is expected to be a conservative design. However, the MEPDG does not allow the user to enter the actual PG grade of the asphalt binder. Other available input options in *Level 3* analysis are viscosity and penetration grading. It should be noted that all tested binders' viscosity grades were stiffer than AC-40, which is the stiffest binder option available in the MEPDG software. Therefore, the viscosity grading of all tested binders would be the same. On the other hand, the penetration grading itself is an empirical method, which can be deceptive to performance at higher and lower service temperatures (Roberts et al., 1996). Therefore, *Level 3* analysis is not expected to provide reliable pavement performance prediction. On the other hand, asphalt binder's inputs at *Level 2* are the same as those at *Level 1*. Since *Level 3* is meant not to require any laboratory test data, it is recommended that the MEPDG software allow the users to use the continuous PG

grade of the binder at *Level 2* analysis in a future release, which is acknowledged to be an interesting concept by the MEPDG team (Gibson, 2011).

7.5.3 G* and δ values

MEPDG *Level 1* input parameters for the tested asphalt binders are also presented in Table 7.2. These input parameters are essentially G* and δ values of RTFO-aged samples over a range of testing temperatures, as recommended by the MEDPG. As expected, the G* value increases with a decrease in testing temperature. For example, the G* values for SRC1 PG 64-22 binder at 54.4^{o}C and 4.4^{o}C were found to be 9.28 kPa and 18300.00 kPa, respectively. Among three selected sources, at a majority of test temperatures, binders from SRC3 were found to be comparatively stiffer (i.e., higher G* and lower δ) than those from the other two sources. A similar trend of stiffness was observed in the case of viscosity measurements and continuous PG grades.

7.5.4 ASTM A and VTS Parameters

The ASTM A and VTS parameters of the tested binders, based on RV and DSR test data, are presented in Table 7.3. It is seen that the estimated A and VTS values significantly vary from the MEPDG suggested ASTM A and VTS parameters, which are based on the nationally calibrated model. The estimated A values of the tested binders are significantly higher than the corresponding values suggested by the MEPDG. Comparatively, the estimated VTS values of these binders are significantly lower than the typical values. Furthermore, in the case of the nationally calibrated model, the A values decrease and the VTS values increase with an increase in PG

grade. A similar trend holds only for the SRC1 binders used in the current study. Asphalt binders from the other two sources do not follow any particular trend.

7.5.5 Dynamic Modulus

The E* values of HMA mixes with the three selected binders were predicted using Equations 7.1, 7.2, and 7.3. The E* master curves for these mixes were then constructed using the time-temperature superposition (TTS) principle at a reference temperature of 70°F (21.1^{o}C), as recommended by the MEPDG. As shown in Figure 7.2(a), the E* data at all temperatures other than the reference temperature were shifted with respect to time until the E* curves merged into a single smooth sigmoidal function, representing the master curve. The master curve was constructed by using a second order polynomial relationship (Equation 7.6) between the logarithm of the shift factors (loga(T_i)) and the temperature. It should be noted that the shift factor at the reference temperature (a(T70)) is zero. The TTS was performed by simultaneously solving for the four coefficients of the sigmoidal function (δ, α, β, and γ), as described in Equation 7.4, and the four shift factors (a(T20), a(T40), a(T100), and a(T130)). A Microsoft^{TM} Excel Solver program was used to conduct the nonlinear optimization to fit the sigmoidal function of the master curves. An example of shift factor versus reduced log time is shown in Figure 7.2(b). The logarithm of the shift factor is presented as a second order polynomial function of temperature with a R^2 value of 1.00.

E* master curves of MixDesign#1 and MixDesign#2 with different binders are presented in Figures 7.3 and 7.4, respectively. As expected, in both mixes, the E* value of a stiff binder (e.g., PG 76-28) is notably higher than that of the soft binder

(PG 64-22). Shah et al. (2005) reported similar findings for a PG 70-28 binder, which showed significantly higher E* values than a PG 58-28 binder. It is clear from these master curves that typical ASTM A and VTS parameters, as well as RV test data, can predict E* values of the mixes with the tested binders reasonably well, except for a few cases. In the case of MixDesign#1, at a higher log reduced time from 8 to 10 seconds, typical ASTM A and VTS values and RV data of PG 70-28 and PG 76-28 binders underestimated the E* values. Similar observations were made for PG 64-22 and PG 76-28 binders in the case MixDesign#2. The RV test data overestimated the E* values in the case of PG 76-28 binder from both mixes. In general, for mixes with a stiffer binder, the trend is that RV test data tended to overestimate E* values. Birgisson et al. (2005) also reported that the predicted E* values from RV test data were significantly higher than the measured E* values for HMA mixes in Florida. On the other hand, Tran and Hall (2005) reported no significant difference between the measured and predicted E* values for Arkansas mixes, indicating that the Witzack predictive equation could be used to estimate E* values.

From Figures 7.3 and 7.4, it is also evident that the predicted E* values, based on DSR test data, significantly underestimated the measured E* values for both mixes with all three binders. The study performed by Birgisson et al. (2005) reported similar findings; the predicted E* values from DSR data were also found to be lower than the measured E* values. These researchers also found bias in the results and recommended that a multiplier be used to correlate predicted E* values with the measured E* values.

The *goodness-of-fit* statistics of the aforementioned sigmoidal master curves was assessed by calculating their R^2 (correlation coefficient) and S_e/S_y (standard error of estimate divided by standard deviation) values. The correlation coefficient, R^2, is a measure of the accuracy of the model. The higher the R^2 value (close to 1.00), the better the prediction is. The ratio S_e/S_y is a measure of the accuracy of the prediction and indicates how well the variations of the predicted E* values are explainable by the predictive equations. The lower the S_e/S_y value, the better the accuracy of the prediction. Table 7.4 presents the *goodness-of-fit* statistics for all master curves. Overall, master curves for all mixes had "excellent" ($R^2 > 0.9$ and $S_e/S_y < 0.35$) correlations, based on the criteria given in NCHRP Report 465 (Witczak et al., 2002).

Figures 7.5 and 7.6 show the measured and predicted E* values, respectively, for the mixes with all tested binders. If the data points distribute themselves around the "Equality Line," as shown in these figures, then there exists a good correlation. In order to evaluate the quality of predictions, linear regressions with zero intercept were performed. The slope of a regression line is a measure of the quality of fit; the closer the slope to unity, the less bias of a prediction. If the slope is greater than unity, then the predicted E* values are less than the measured values. If the slope is less than unity, the predicted E* values are higher than the measured values. Similarly, the R^2 value of the correlation also indicates a measure of the *goodness-of-fit* of the regression line. The *goodness-of-fit* statistics parameters of the measured versus predicted E* values are also presented in Table 7.4. In general, "excellent" correlations are observed form predicted E* values from typical ASTM A and V parameters and RV test results. Even though the R^2 values of correlations between the

predicted E* values from DSR test results and the measured E* values indicate "good" fits, the high S_e/S_y values (greater than 0.9) indicate that the variations of E* values cannot be explained well and the accuracies of established correlations are "very poor."

7.5.6 MEPDG Analysis

The MEPDG software allows the user to change over 150 variables that impact the performance of the pavement. These variables are grouped by category including: climate, traffic, pavement layers and their material properties. Among material properties of different layers of a typical pavement section, as shown in Figure 7.7(a), this study focused on the parametric study of asphalt binders. The surface course and base course represent MixDesign#1 and MixDesign#2, respectively. The 10-inch sub-base layer was considered to be crushed gravel (AASHTO Classification A-2-4) from Marshall County with a resilient modulus (M_r) of 14,218 psi (98.04 MPa), as reported by Hossain et al. (2011). The semi-infinite subgrade layer consisted of soil type A-7-6 with a M_r value of 10,852 psi (74.82 MPa).

The pavement section was analyzed for a design life of 20 years. Traffic data of *Interstate 35* near Grand Avenue and SE 36th Street in Oklahoma City was considered in this study. The annual average daily traffic (AADT) of this location was found to be 13,350, of which 5.92% were trucks with 3 or more axles (FHWA Classes 6-13) (ODOT, 2011). A default growth rate of 1.5% was assumed, as per recommendation of the MEPDG. The depth of ground water table for this location was found to be 141 ft, as reported by Ley et al. (2009). The existing weather files of Will

Rogers World Airport at Oklahoma City and Willey Post Airport at Bethany (both in Oklahoma), were used to model the climate condition of the pavement site.

Four major distress parameters (rutting, fatigue cracking, thermal cracking and international roughness index (IRI)) were evaluated in the current study using the MEPDG software version 1.100. As recommended by the MEPDG, the acceptable design values of these distresses are 0.25 inch for AC layers' rutting, 0.75 inch for the total rutting, 25% for AC layers' bottom up (alligator) cracking, 1000 ft/mi for AC layer thermal (transverse) cracking, and 172 inch/mi for IRI, all with a target reliability of 90% or higher. The naming convention of a *project* in the MEPDG software was as follows: LNSRCNPGXX-YYPGZZ-AA, where LN = design reliability level (L1, L2, or L3), SRCN = binder source (SRC1, SRC2, or SRC3), PGXX-YY= PG grade (e.g., PG76-22) of the binder used in the surface course, and PGZZ-AA = PG grade (e.g., PG64-22) of the binder used in the base course. For example, a *project* named as L1SRC2PG76-28PG64-22 indicates the *project* was run at a design reliability of *Level 1*, and the surface and base mixes was constructed with PG 78-28 and PG 64-22 binders, respectively, from SRC2. For simplicity, only MixDesign#2 with the PG 64-22 binder was considered in the base course, which would be a realistic approach as a softer binder is usually used in base courses in Oklahoma. Meanwhile, MixDesign#1 with all three binders from all three sources were considered in the surface course. Also, asphalt binders from only one source were considered in each *project*. It should be noted that *Level 3* analysis does not consider the effect of binder source. However, the effects of PG grade of the binders on selected distress parameters can be evaluated from *Level 3* analysis. *Level 1*

analysis was limited to all three binders from SRC2 as only E* data of HMA mixes for these binders were available. *Level 2* analysis, however, was conducted for mixes with binders from all three sources.

7.5.6.1 Rut Depth

The MEPDG estimates the total rutting by summing the rutting in the surface, base, sub-base, and subgrade layers. The contribution of all pavement layers to the total rutting from a typical *project* is shown in Figure 7.7(b). The predicted total rutting and AC layers' rutting did not exceed their corresponding design rut depths of 0.75 inch and 0.25 inch, respectively. About 40% of the total rutting occurs in the AC layers, of which 80% was in the surface course. The subgrade layer contributed about 50% of the total rutting, and the sub-base course contributed the remaining 10% rutting. It is also seen that significant rutting (about 40%) occurs within the first two years of construction, which simulates reduced production aging of the binder and reduced compaction of the mix. Furthermore, it is observed that a majority of rutting occurs during the warmer months (April to September) compared to the colder months (October to March), which is expected. A recent study (Hoegh et al., 2010) at Minnesota DOT (MnDOT) also reported similar findings for pavement sections constructed with PG 64-22 and PG 58-28 binders. However, the predicted total rutting in that study was significantly higher than the measured (field) rutting for an analysis period of 10 years. On the other hand, the predicted AC layers' rutting was fairly in agreement with the measured rutting. Since the measured granular base and subgrade layer rutting was highly overestimated in that study, these researchers recommended modification of the rutting models for these layers in the MEPDG.

As seen in Figure 7.8(a), the AC layers' rutting varies significantly with the change in PG grade of the binder, which is expected. The general trend is that the higher the binder grade, the lower the AC layer rutting. At *Level 3* analysis, compared to the PG 76-28 binder, in cases of surface course mixes with PG 70-28 and PG 64-22 binders, the corresponding increases in the predicted rutting were found to be 14% and 21%, respectively. Similar observations were made at *Level 1* analysis, but the variations of the rut depths are lower than *Level 3* analysis. These observations are in agreement with the binders' DSR and mixes' E* test data, where the stiffer grade binders showed reasonably higher G*/sin(δ) values and the corresponding mixes exhibited higher E* values compared to the softer grade binder. Kim et al. (2005) reported similar findings in their corresponding study, where the predicted rut depths for mixes with PG 52-XX binders predicted significantly higher rut depths than those of PG 58-XX binders.

In regard to the design reliability, significantly lower rutting was predicted at *Level 1* compared to that at *Level 3*, for all three PG binders, as shown in Figure 7.8(b). The reduction in the predicted rutting from *Level 3* to *Level 1* input was as much as 36% in the case of the PG 70-28 binder. These findings are in agreement with E* master curves where the predicted E* values at *Level 1* are significantly lower than those at *Level 3*. The MnDOT study reported similar findings while comparing rut depths between *Level 2* and *Level 3* analyses (Hoegh et al., 2010). In that study, the predicted rut depth from *Level 2* input was about 74% lower than that from *Level 3* input. These researchers also reported inconsistent rutting prediction form *Level 2*

input due to a logical bug in the MEPDG software (Hoegh et al., 2010). The current study, however, did not experience such issues.

Sensitivity of the AC layers' rutting to the binder source was evaluated by performing *Level 2* analysis, which uses DSR data of binders and volumetric properties of the asphalt mixes. For the comparison purpose, binders from SRC2 were selected as the controls. The AC layers' rut depths of the pavement section with SRC1 and SRC3 binders were somewhat different from those with SRC2 binders. In regard to the binder's PG grade, as shown in Figure 7.8(c), these variations are relatively higher in PG 64-22 binders than in PG 70-28 or PG 76-28 binders. For instance, the predicted AC layers' rut depths in cases of PG 64-22 and PG 76-28 binders from SRC3 were about 9% and 7% higher than those of their counterparts from SRC2. On the other hand, the pavement section with the PG 70-28 binder from SRC1 predicted about 7% reduced rutting compared to that with the same PG binder from SRC2. These findings are in agreement with predicted E* master curves obtained from DSR test data explained earlier.

7.5.6.2 Thermal Cracking

The evaluation of thermal cracking of this study was limited to *Level 3* analysis. *Level 2* and *Level 1* analyses could not be done as required laboratory creep compliance data of the mixes were not available. In the cases of PG 70-28 and PG 76-28 binders in the surface course, no thermal cracking was predicted for the design life of the pavement. In the case of PG 64-22 binder, negligible thermal depth (in order of 10^{-5} inch) was predicted to start after 4.5 years of construction. In this case, the predicted thermal crack length (ft/mi) was still zero. Velasquez et al. (2009), however,

reported significant thermal cracking for pavement sections in Minnesota at *Level 1* analysis. These researchers also reported that the MEPDG underestimated thermal cracking compared to field measurements. Therefore, for Minnesota conditions, a recalibration factor of 1.85 was suggested based on linear regression between the measured and predicted thermal cracking (transverse) measurements (Velasquez et al., 2009). Li et al. (2009) also reported longitudinal cracking to be highly sensitive to asphalt binder's PG at *Level 2* analysis for conditions in Washington.

7.5.6.3 Fatigue Cracking

Alligator cracking, a series of interconnected cracks, is a form of fatigue load-related cracking. The MEPDG assumes that alligator cracking initiates at the bottom of the AC layers and propagates to the pavement surface with continued truck traffic. At *Level 2* analysis, the predicted alligator cracking was found insignificant (up to 0.48%) compared to its design limit of 25%, as shown in Figure 7.9(a). A general trend is that alligator cracking decreases with an increase in the intermediate PG temperature of the binder. However, the variation of alligator was not significant among mixes with the tested binder types. It should be noted that the intermediate PG temperatures of PG 64-22 and PG 70-28 are the same, which is 25°C. Therefore, no variation in fatigue cracking is expected between mixes with these two binders, and this is the case for the current study (Figure 7.8a). Alternatively, the intermediate temperature of the PG 76-28 binder is 28°C. Therefore, reduced alligator cracking is expected for the mix with a PG 76-28 binder compared to the other two binders. In regard to binder source, SRC1 binders showed the highest alligator cracking, followed by SRC2 binders, which was followed by SRC3 binders. The largest difference (about

13%) was observed between PG 64-22 binders from SRC3 and SRC1. Similar observations were made by Kim et al. (2005) for conditions in Iowa. These researchers reported a very small bottom up alligator cracking and found that the binder type to be insensitive to this distress. Li et al. (2009) also observed significantly less alligator cracking in the MEPDG analysis than field measurements.

7.5.6.4 International Roughness Index

International roughness index (IRI) is estimated based on site factors (pavement age, annual rainfall, soil properties, etc.), rut depth, fatigue and thermal cracking of the pavement section. As seen in Figure 7.9(b), the predicted IRI of the pavement section in its design life is found to be as high as 118 in/mi, which is comfortably below the MEPDG recommended target design value of 172 in/mi. It is also observed that the binder's PG grade and source are insensitive to IRI. This could be due to the fact that the IRI model considers alligator cracking and transverse cracking, which are not significantly sensitive to binder type and source. Because of the suspected limitation of the MEPDG to predict thermal cracking, the MnDOT study could not calibrate the IRI model (Velasquez et al., 2009). Li et al. (2009) also reported the asphalt binder's PG grade to be of little or no sensitivity to IRI. Kim et al. (2005), however, reported positive correlation between binder PG grade and IRI.

7.6 SUMMARY AND CONCLUSIONS

An inventory of rheological data including rotational viscosity and continuous PG grades of three certified PG binders (PG 64-22, PG 70-28, and PG 76-28) from three different sources (SRC1, SRC2 and SRC3) in Oklahoma were evaluated by conducting Superpave® tests. Material input parameters of these binders were also evaluated as per the MEPDG recommendations. The generated asphalt binder input

parameters and volumetric properties of six HMA mixes were used to estimate dynamic modulus (E*) of mixes. E* master curves obtained from estimated and laboratory measured E* values were then compared. Finally, relative sensitiveness of the asphalt binder's PG grade and source on four major pavement distresses were evaluated by using the MEPDG version 1.100 software. Based on the findings of the study, the following conclusions can be drawn:

- All tested asphalt binders met the Superpave® specified viscosity requirement (≤ 3 Pa.s). The general trend is that asphalt binders from SRC3 appear to be more viscous than those from the other two sources. In particular, the PG 76-28 binder from SRC3 barely passed the Superpave® criterion for rotational viscosity. There is no particular trend in the viscosity measurements of asphalt binders from SRC2 and SRC1.
- All three tested binders from all three sources met the manufacturers' specified PG grades. The actual PG grades of the majority of the tested binders were found to be significantly higher than their standard (6^oC interval) PG grades. Thus, using standard PG grades of these binders at *Level 3* analysis is expected to be a conservative design.
- Estimated ASTM A and VTS parameters from RV and DSR test data of the tested asphalt binders vary significantly from the MEPDG suggested typical values.
- The MEPDG suggested typical A and VTS parameters were found to predict E* values of mixes reasonably well except for a log reduced time from 8 to 10 seconds. Theses parameters, estimated from the RV data, can also predict E*

values of mixes with a softer binder fairly well. In the case of stiffer binders (i.e., PG 70-28), however, the RV test data tend to overestimate E* values. The extent of overestimation of E* values increased with the increase of stiffness of the binder. On the other hand, these parameters, obtained from the DSR data, significantly underestimated E* values of the mixes.

- The MEPDG software predicted that about 40% of the total rutting occurred in AC layers, of which 80% would occur in the surface course. It also estimated that about 39% of rutting would occur within two years of construction.
- The AC layers' rut depths were found to vary significantly with the change of PG grade of the binder. In general, the higher the binder PG grade, the lower the AC layer rut depth. The variation of rut depths between surface mixes with PG 64-22 and PG 76-28 binders was as high as 21%.
- In regard to the design reliability, significantly lower rut was predicted in the case of *Level 1* analysis compared with *Level 3* analysis. The reduction in rut depth obtained from *Level 3* to *Level 1* input was as much as 36% for the PG 70-28 binder. These findings are in agreement with predicted E* values from the DSR data, which significantly underestimated E* values. Comparatively, at *Level 2* analysis, the AC layers' rut depth was found to be somewhat sensitive to the binder source.
- Fatigue fracture and thermal cracking were not found to be critical distresses in this study. Also, binder type and source did not seem to have significant influence on these distresses.

- IRI was not found to be sensitive to the binder source due to the fact that its major influencing factors (alligator cracking and thermal cracking) were insignificant and they were insensitive to the binder source.

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Table 7.1 Summary of Mix Properties and Aggregate Gradation[1]

Mix Parameter	MixDesign#1			MixDesign#2		
Mix Type	S4 (Oklahoma)			S3 (Oklahoma)		
Binder Grade	PG 64-22	PG 70-28	PG 76-28	PG 64-22	PG 70-28	PG 76-28
V_a (%)	4.3	4.3	4.6	4.8	4.7	4.5
Vb_{eff} (%)	9.4	9.4	9.3	8.4	8.4	8.4
P_b (%)	5	5	5	4.6	4.6	4.6
G_b	1.026	1.0274	1.0288	1.026	1.0274	1.0288
G_{se}	2.649	2.649	2.649	2.671	2.671	2.671
G_{mm}	2.503	2.504	2.504	2.488	2.488	2.488
G_{sb}	2.631	2.631	2.631	2.654	2.654	2.654
VTM (%)	4			4		
VMA (%)	14.9			14.4		
VFA (%)	73.2			72.2		
DP	1.6			0.6		
% Retained ¾″	0			15		
% Retained 3/8″	10			26		
% Retained # 4	22			48		
P_{200}	7.6			2.7		

[1] Volumetric mix design was conducted by Dr. Cross at Oklahoma State University and they was reported in Cross et al. (2007); G_{mm} = bulk specific gravity of compacted mix , G_{sb} = bulk specific gravity of aggregate, G_{se} =effective specific gravity of aggregate, G_b = specific gravity of asphalt binder, P_b = asphalt binder content, VTM = void in total mix, VFA = voids filled with asphalt, DP = dust proportion, VMA = voids in mineral aggregate, V_{beff} = effective asphalt content, % by volume, V_a = air void content, %, P_{200} = % passing #200 (0.075 mm) sieve.

Table 7.2 Continuous PG Grades and MEPDG *Level 1* and *Level 2* Input Parameters of Tested Asphalt Binders

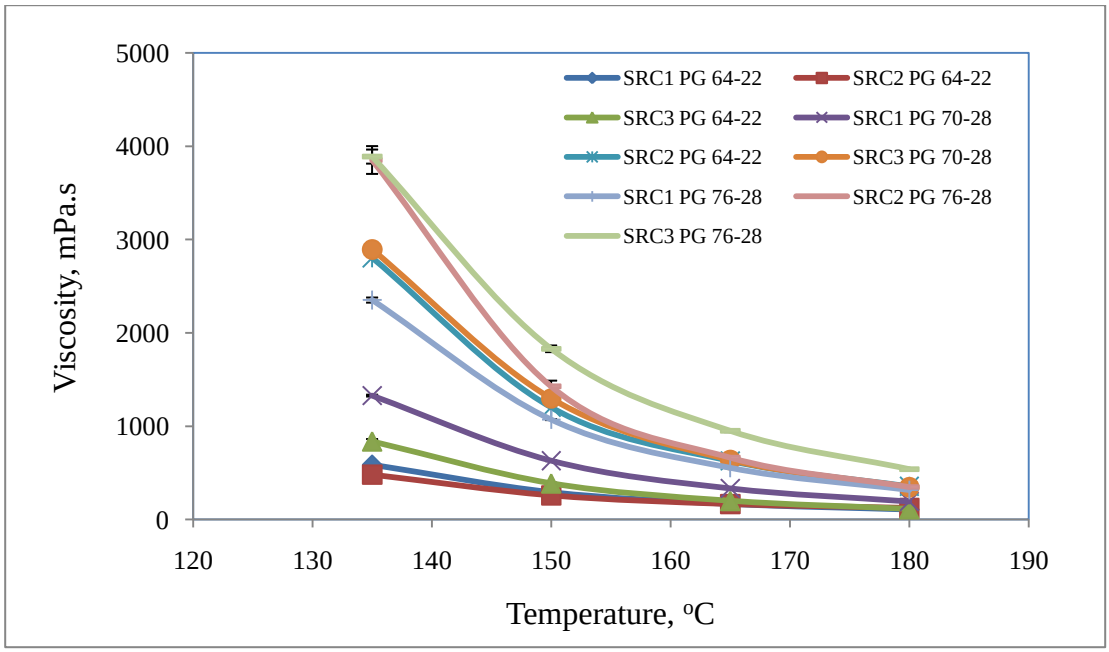
Continuous PG Grade							
Binder Type	Testing Temp. (^oC)	Binder From SRC1		Binder From SRC2		Binder From SRC3	
PG 64-22	Multiple	PG 66.7-22.1		PG 64.9 -23.8		PG 67.6 -22.5	
PG 70-28	Multiple	PG 70.7-28.8		PG 74.8-28.1		PG 73.4-28.1	
PG 76-28	Multiple	PG 76.9-31.3		PG 81.8 -28.6		PG 76.8-28.9	
MEPDG Input Parameters							
Binder Type	Testing Temp (^oC)	Binder From SRC1		Binder From SRC2		Binder From SRC3	
		DSR Test Data		DSR Test Data		DSR Test Data	
		G* (kPa)	δ (deg)	G* (kPa)	δ (deg)	G* (kPa)	δ (deg)
PG64-22	54.4	9.28	80.63	10.32	78.7	13.80	81.2
	46.1	32.47	76.10	34.20	73.6	48.99	76.9
	43.3	46.98	74.70	56.52	71.0	75.55	74.8
	29.4	344.36	63.77	402.11	63.7	407.86	66.6
	21.1	1030.38	60.77	1869.11	45.5	911.32	48.3
	12.7	4870.00	55.9	4574.00	48.8	8606.19	50.8
	4.4	18300.00	53.3	23778.84	47.0	19848.75	49.6
PG70-28	54.4	12.14	65.7	15.54	49.4	12.20	63.3
	46.1	28.31	64.6	32.92	51.3	31.80	63.8
	43.3	40.56	64.2	44.01	51.9	46.27	64.1
	29.4	268.41	60.8	229.39	54.2	333.00	63.5
	21.1	1061.36	54.4	861.58	49.2	1720.00	52.0
	12.7	4040.00	52.2	3796.25	49.1	4155.00	50.60
	4.4	15200.00	50.4	13875.00	48.1	14528.50	48.40
PG76-28	54.4	13.93	59.4	14.09	50.3	12.64	59.9
	46.1	33.39	59.4	30.03	51.9	30.79	61.3
	43.3	47.15	59.4	40.47	52.4	44.05	62.0
	29.4	274.68	58.8	181.40	56.6	322.22	62.9
	21.1	1025.48	52.7	548.47	58.1	1478.04	53.3
	12.7	5010.00	53.8	3287.20	47.5	5823.44	52.3
	4.4	17800.00	51.8	13726.25	46.5	20450.98	46.0

Table 7.3 ASTM A and VTS Parameters of Tested Binders

Basis	Binder Type	ASTM A and VTS Parameter	Binder Source		
			SRC1	SRC2	SRC3
Typical	PG 64-22	A	10.980	10.980	10.980
		VTS	-3.680	-3.680	-3.680
	PG 70-28	A	9.715	9.715	9.715
		VTS	-3.217	-3.217	-3.217
	PG 76-28	A	9.200	9.200	9.200
		VTS	-3.024	-3.024	-3.024
RV	PG 64-22	A	9.051	9.883	8.911
		VTS	-2.986	-3.292	-2.956
	PG 70-28	A	8.714	8.759	9.042
		VTS	-2.857	-2.869	-2.967
	PG 76-28	A	9.883	9.954	8.046
		VTS	-3.292	-3.280	-2.614
DSR	PG 64-22	A	13.885	14.588	12.833
		VTS	-4.833	-5.075	-4.460
	PG 70-28	A	12.887	10.707	10.707
		VTS	-4.465	-3.660	-3.570
	PG 76-28	A	11.997	10.806	12.970
		VTS	-4.135	-3.699	-4.491

Table 7.4 Goodness-of-fit Statistics for the Witczak Model Predictions

	Design No.	Binder Type	Measured/ Predicted From	S_e/S_y		R^2	
				Value	Evaluation	Value	Evaluation
Master Curve	MixDesign#1	PG 64-22	Measured E*	0.11	Excellent	1.00	Excellent
			Typical ASTM	0.00	Excellent	1.00	Excellent
			RV	0.00	Excellent	1.00	Excellent
			DSR	0.00	Excellent	1.00	Excellent
		PG 70-28	Measured E*	0.12	Excellent	0.99	Excellent
			Typical ASTM	0.00	Excellent	1.00	Excellent
			RV	0.00	Excellent	1.00	Excellent
			DSR	0.00	Excellent	1.00	Excellent
		PG 76-28	Measured E*	0.12	Excellent	0.99	Excellent
			Typical ASTM	0.00	Excellent	1.00	Excellent
			RV	0.00	Excellent	1.00	Excellent
			DSR	0.00	Excellent	1.00	Excellent
	MixDesign#2	PG 64-22	Measured E*	0.09	Excellent	1.00	Excellent
			Typical ASTM	0.00	Excellent	1.00	Excellent
			RV	0.00	Excellent	1.00	Excellent
			DSR	0.00	Excellent	1.00	Excellent
		PG 70-28	Measured E*	0.09	Excellent	1.00	Excellent
			Typical ASTM	0.00	Excellent	1.00	Excellent
			RV	0.00	Excellent	1.00	Excellent
			DSR	0.00	Excellent	1.00	Excellent
		PG 76-28	Measured E*	0.07	Excellent	1.00	Excellent
			Typical ASTM	0.00	Excellent	1.00	Excellent
			RV	0.00	Excellent	1.00	Excellent
			DSR	0.00	Excellent	1.00	Excellent
Measured Versus Predicted	MixDesign#1	PG 64-22	Typical ASTM	0.08	Excellent	0.95	Excellent
			RV	0.27	Excellent	0.93	Excellent
			DSR	1.12	Very Poor	0.80	Good
		PG 70-28	Typical ASTM	0.05	Excellent	0.95	Excellent
			RV	0.06	Excellent	0.95	Excellent
			DSR	1.28	Very Poor	0.82	Good
		PG 76-28	Typical ASTM	0.10	Excellent	0.95	Excellent
			RV	0.17	Excellent	0.94	Excellent
			DSR	1.39	Very Poor	0.76	Good
	MixDesign#2	PG 64-22	Typical ASTM	0.17	Excellent	0.93	Excellent
			RV	0.30	Excellent	0.90	Excellent
			DSR	1.02	Very Poor	0.73	Good
		PG 70-28	Typical ASTM	0.05	Excellent	0.95	Excellent
			RV	0.06	Excellent	0.95	Excellent
			DSR	0.97	Very Poor	0.81	Good
		PG 76-28	Typical ASTM	0.06	Excellent	0.95	Excellent
			RV	0.14	Excellent	0.94	Excellent
			DSR	1.08	Very Poor	0.77	Good



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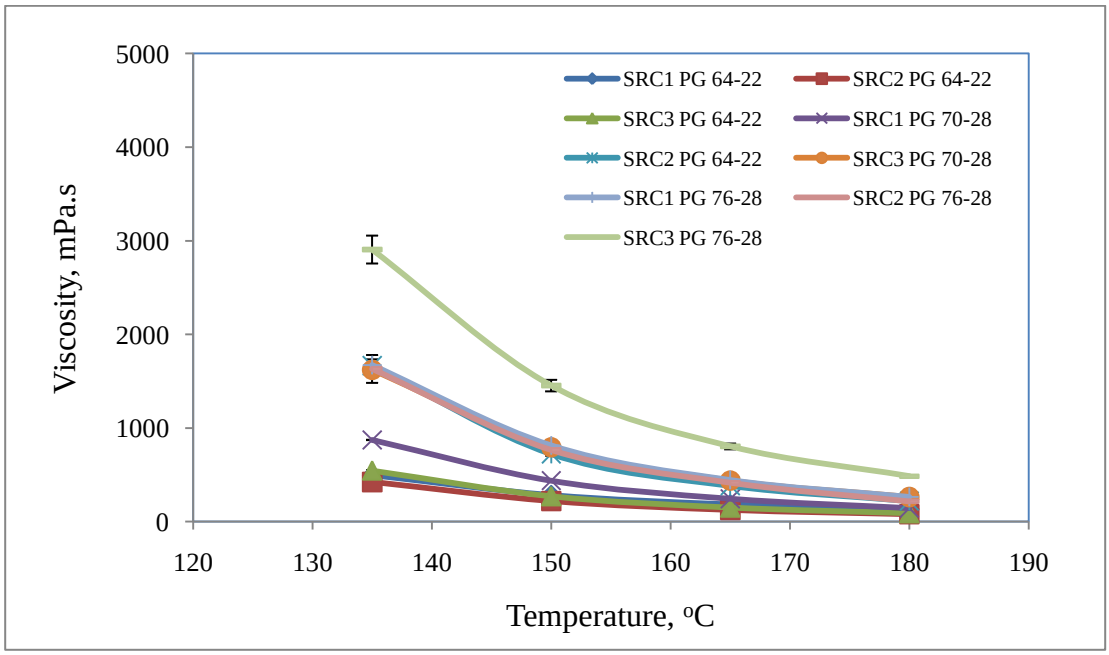

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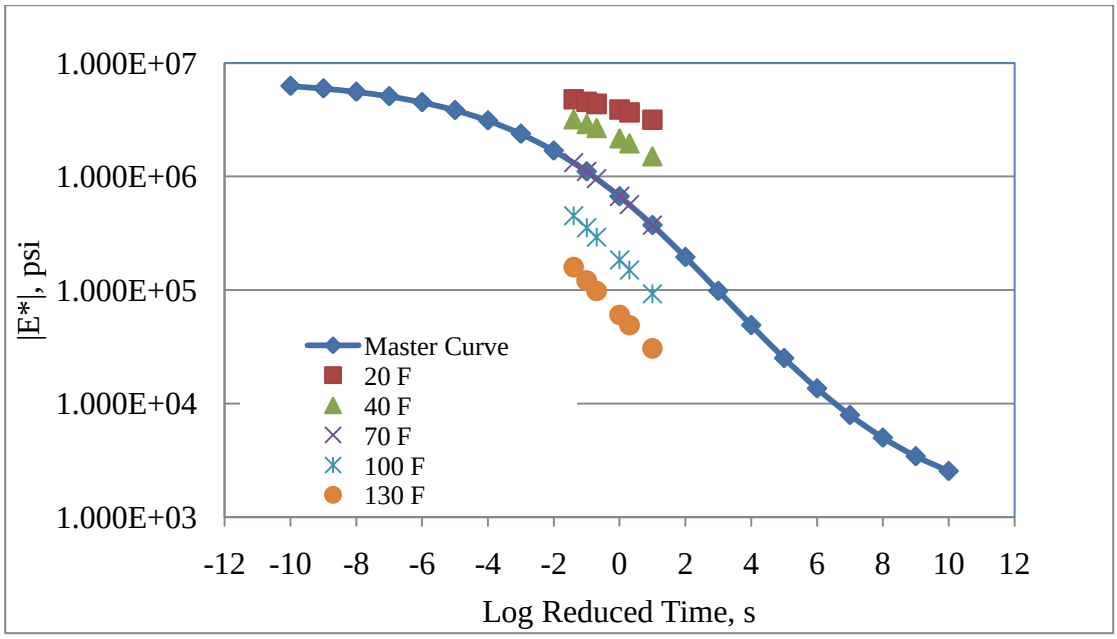
Figure 7.1 Viscosity Test Results of Tested Binders: (a) Unaged, and (b) RTFO-aged.



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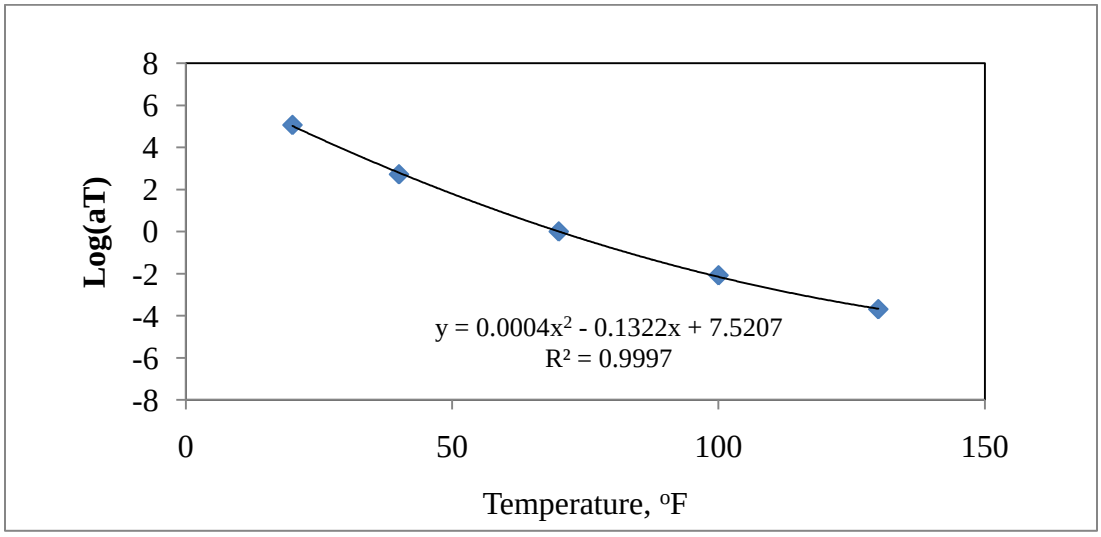
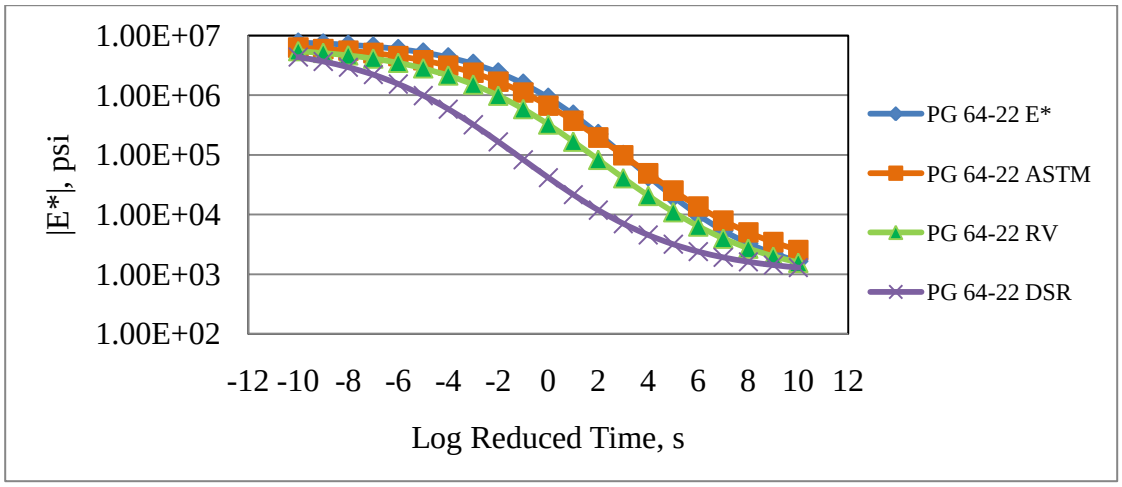


(b)

Figure 7.2 (a) Master Curve for MixDesign#1 Based on Typical ASTM A VTS Parameters of PG 64-22 Binder; and (b) Time-temperature Shift factor for E* Master Curve for MixDesign#1 at Reference Temperature of 70°F.



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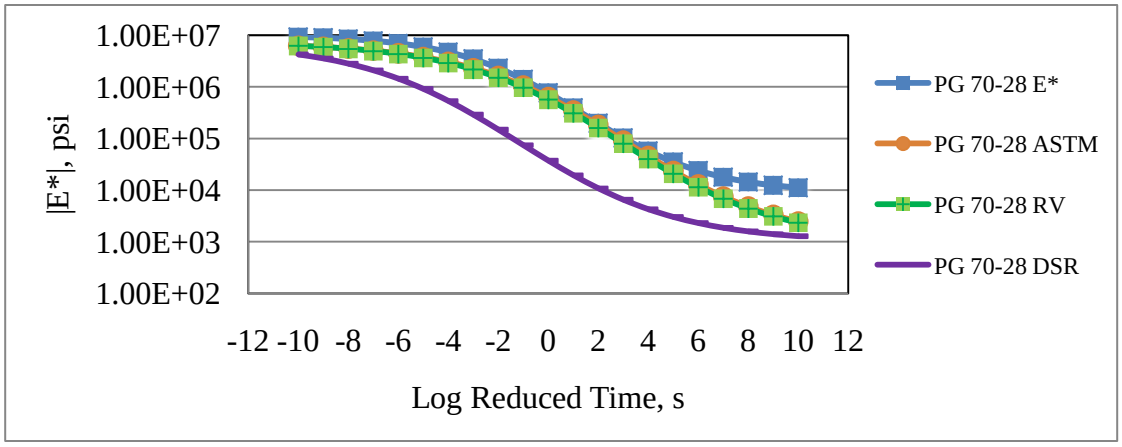

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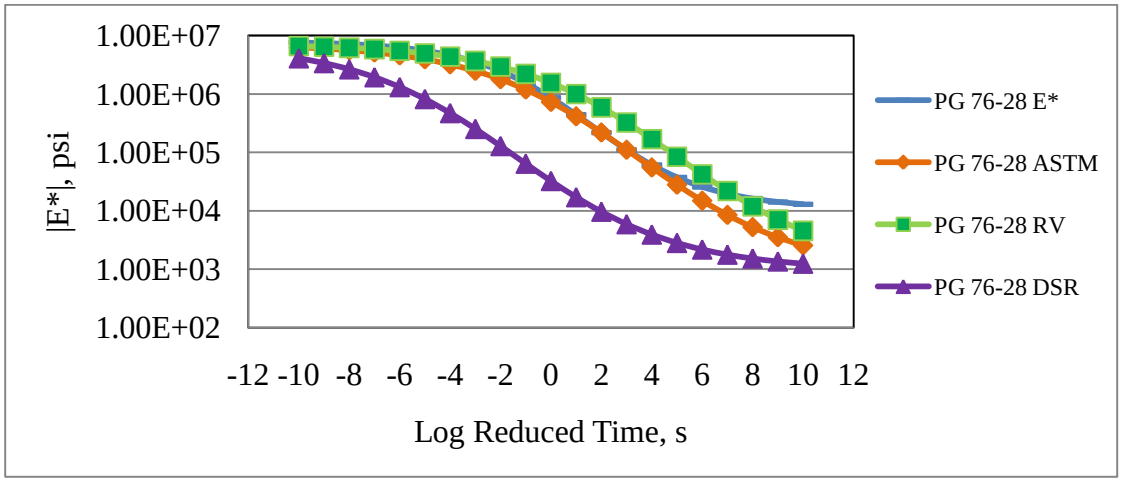
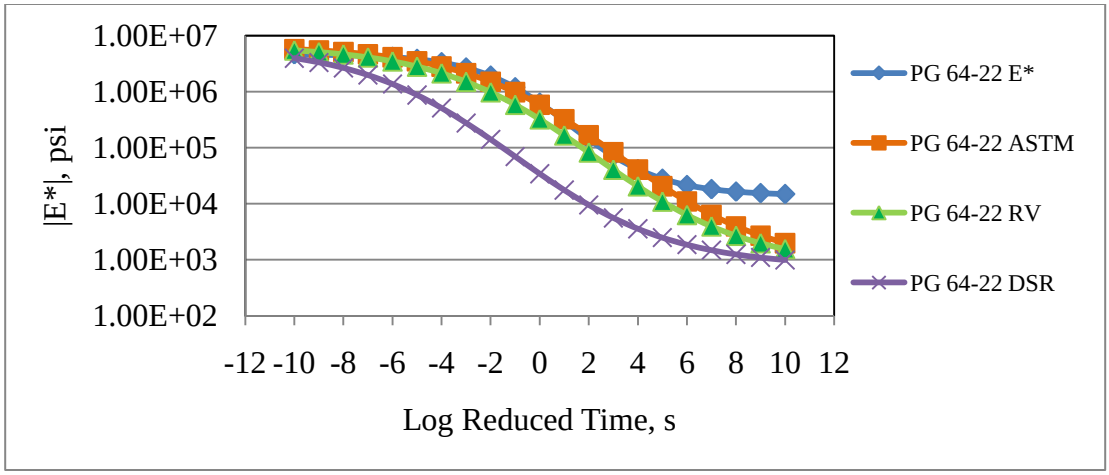
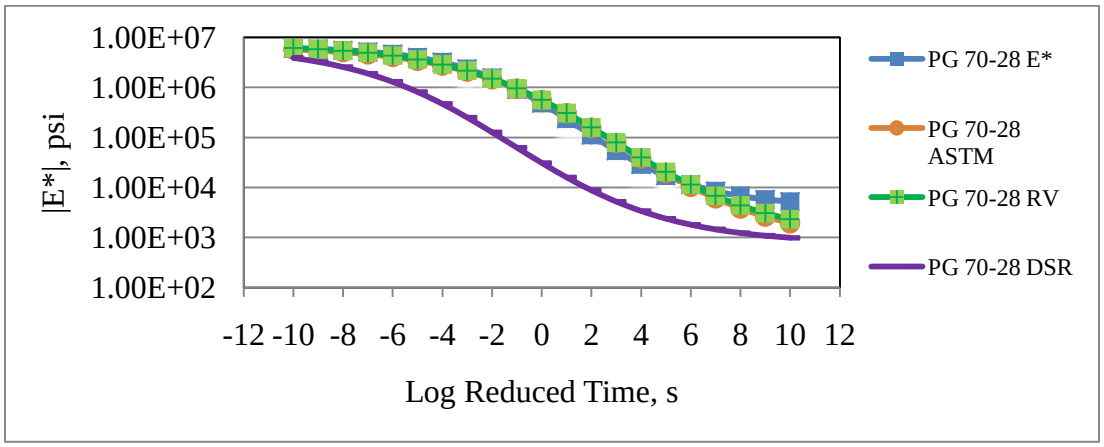


(c)

Figure 7.3 E* Master Curves for MixDesign#1: (a) PG 64-22, (b) PG 70-28, and (c) PG 76-28.



(a)



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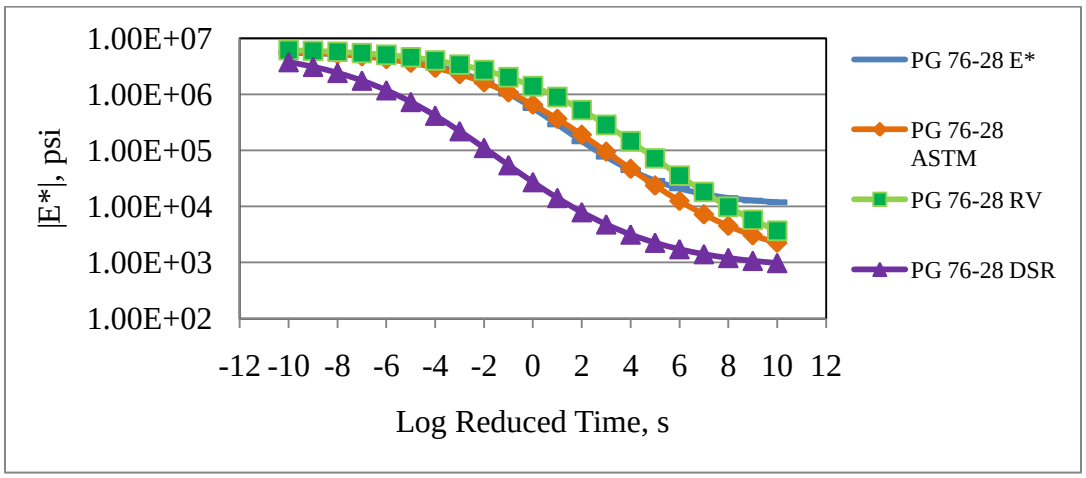
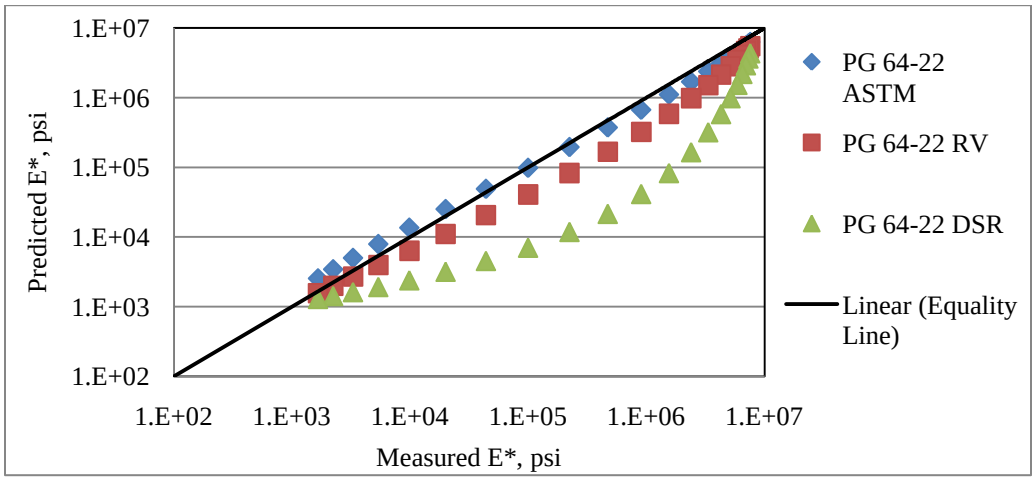


(c)

Figure 7.4 E* Master Curves for MixDesign#2: (a) PG 64-22, (b) PG 70-28, and (c) PG 76-28.



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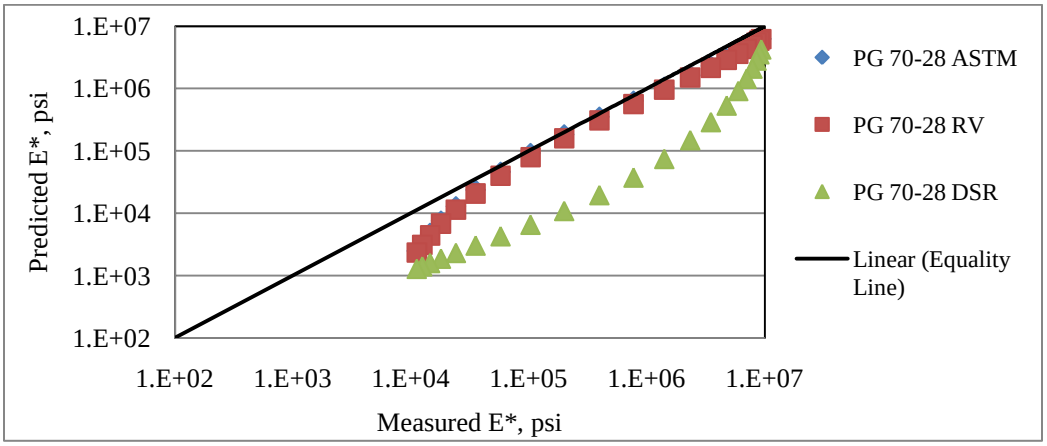

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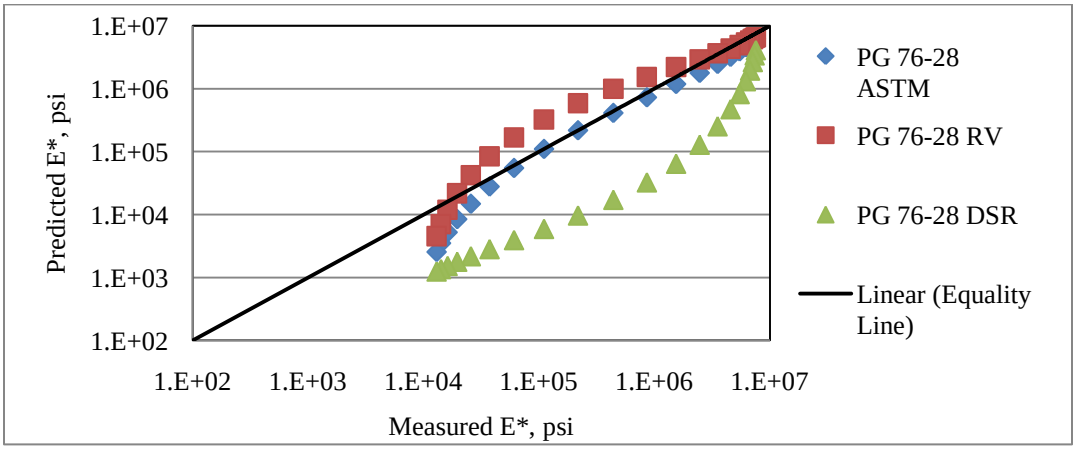
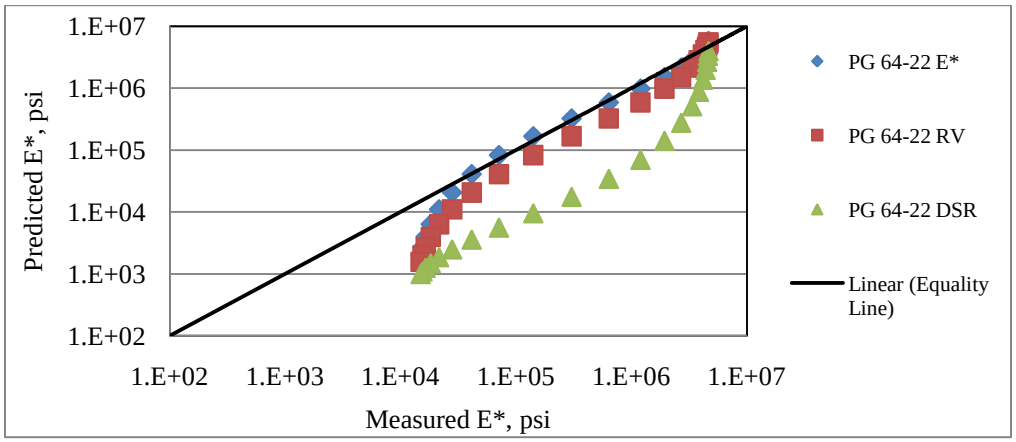


(c)

Figure 7.5 Measured Versus Predicted E* Values for MixDesign#1: (a) PG 64-22, (b) PG 70-28, and (c) PG 76-28.



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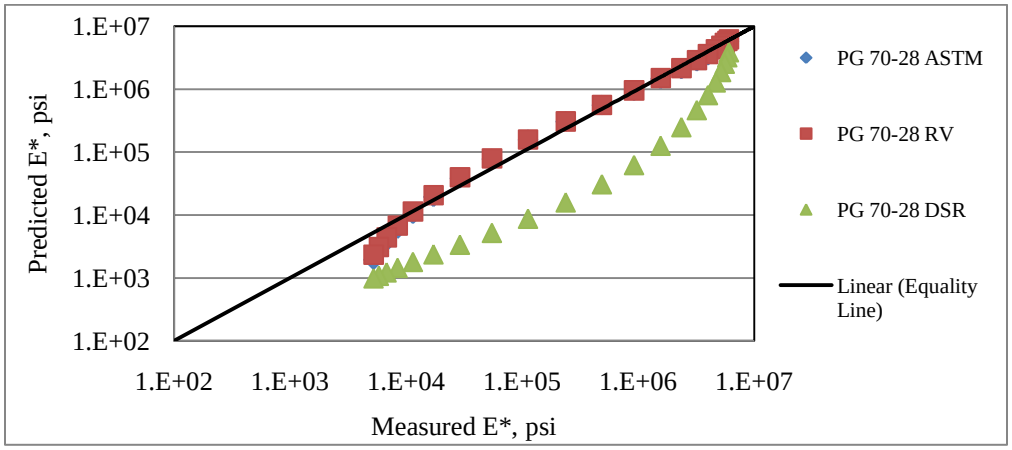
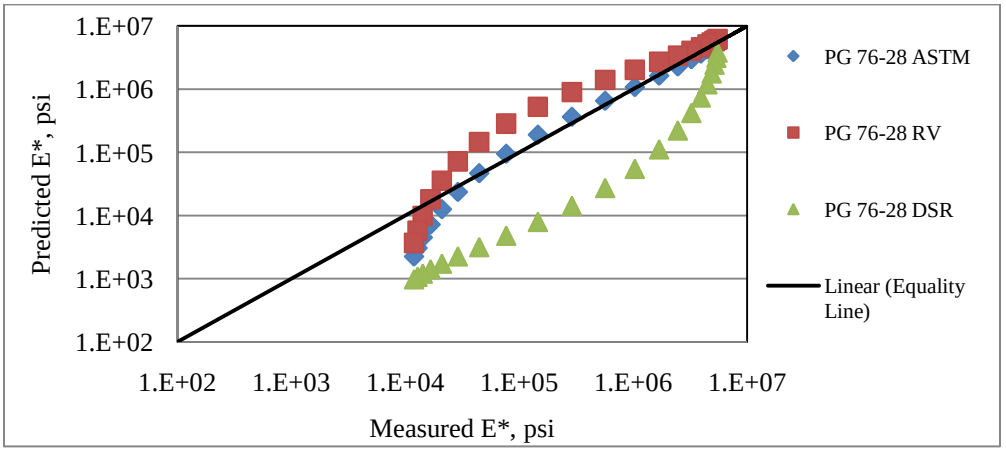
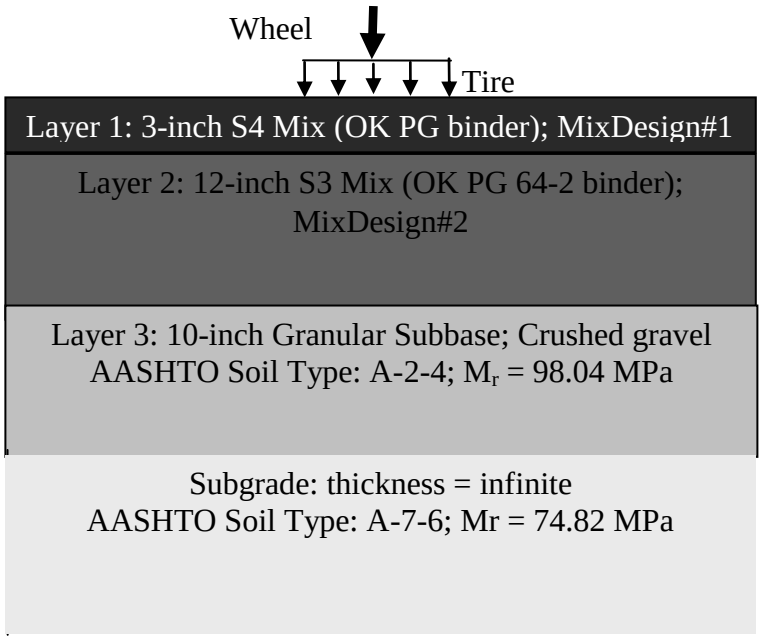


(b)



(c)

Figure 7.6 Measured Versus Predicted E* Values for MixDesign#2: (a) PG 64-22, (b) PG 70-28, and (c) PG 76-28



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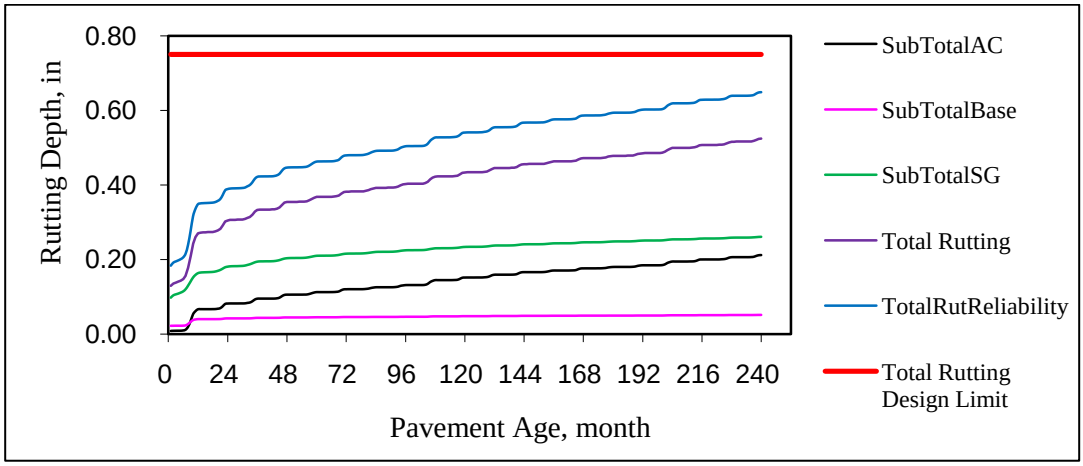
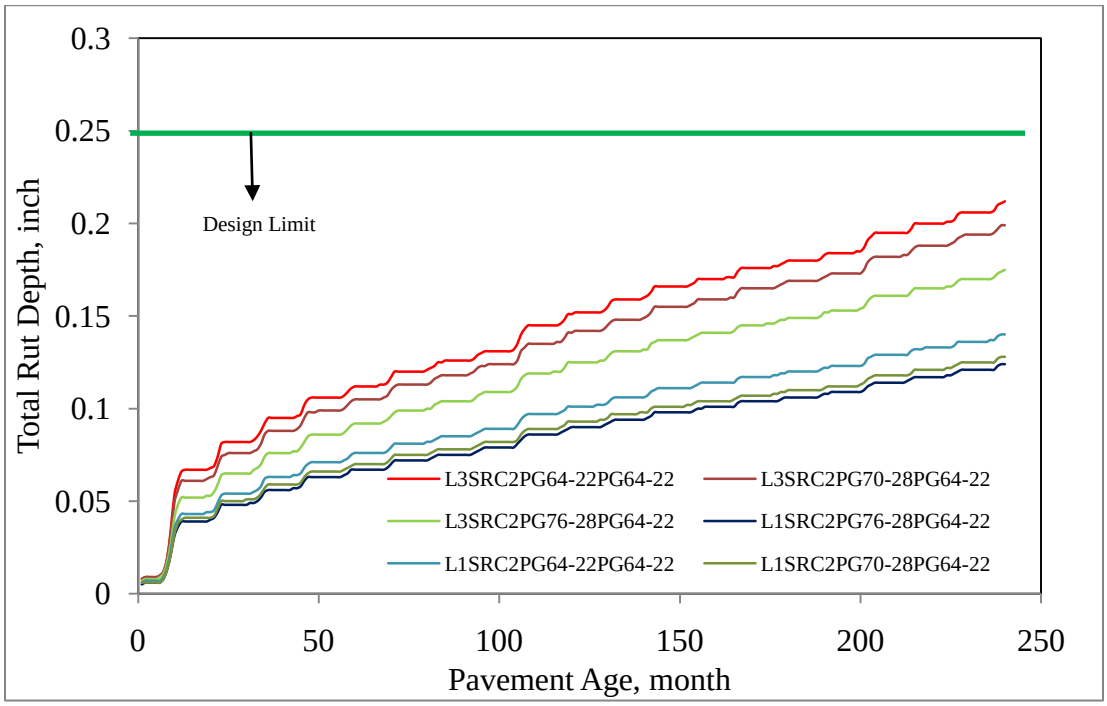


(b)

Figure 7.7 (a) A Typical Pavement Section, (b) Typical Contribution of Different Layers in Total Rut Depth of Project #L1SRC2PG64-22PG64-22.



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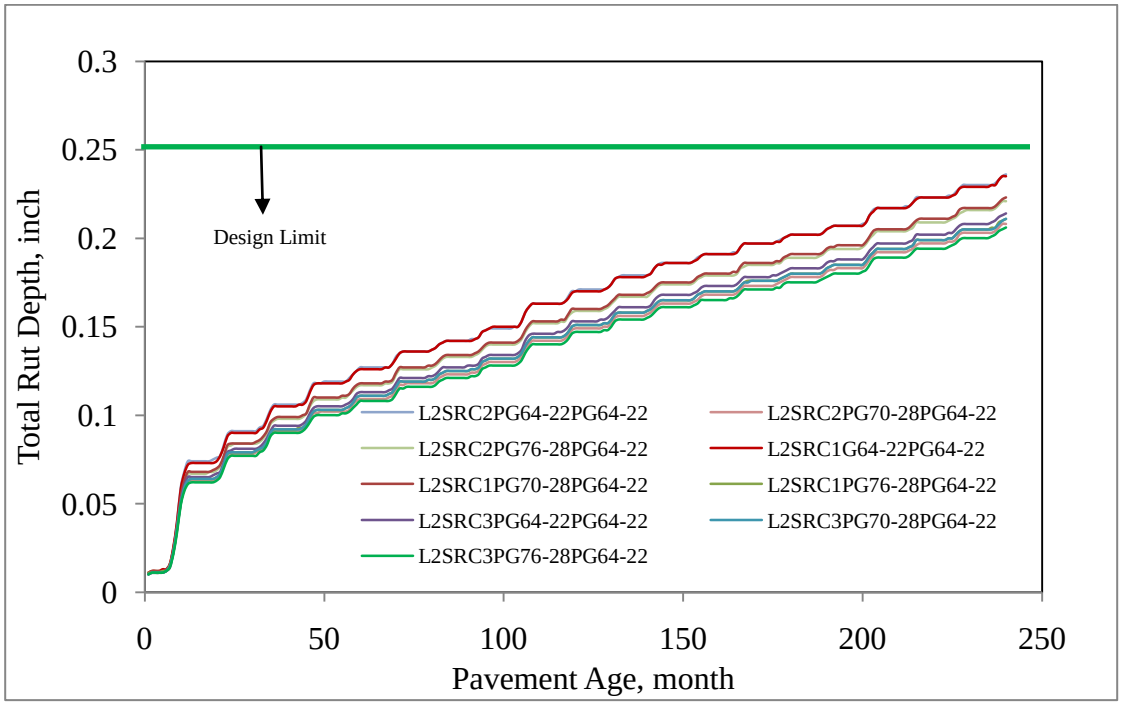
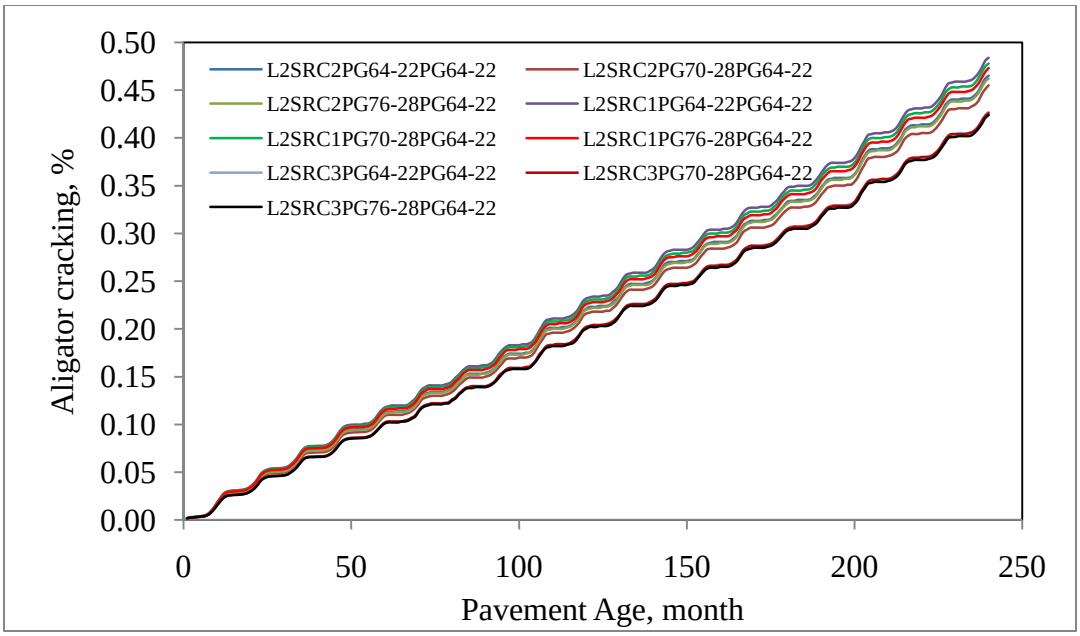
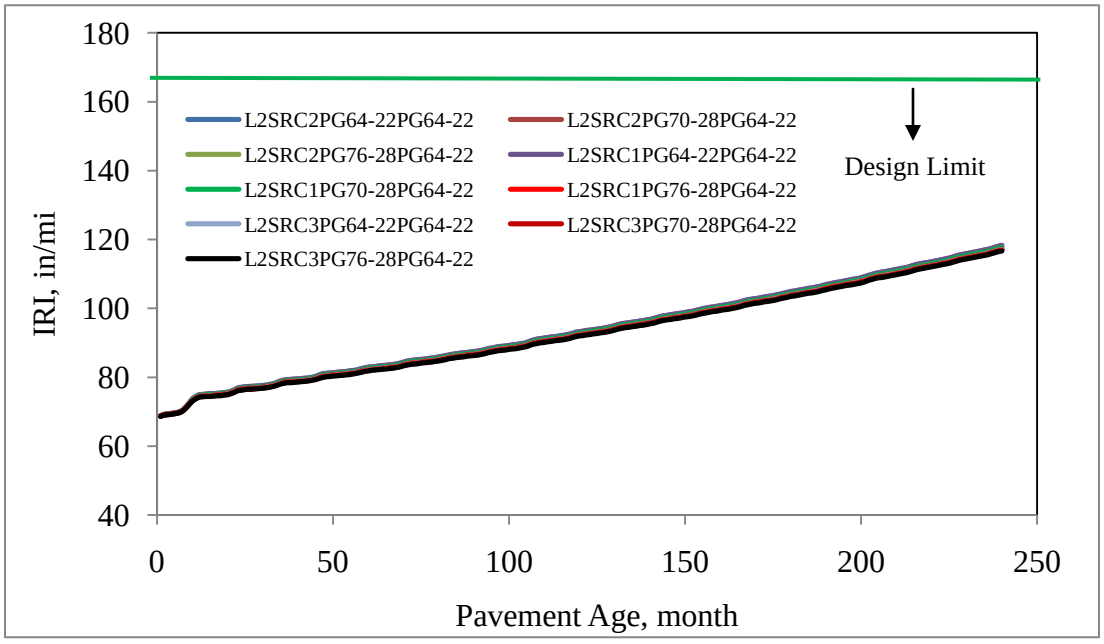


(b)

Figure 7.8 Sensitivity to AC Layer Rut Depth: (a) PG grade and Design Reliability, and (b) Binder Source.



(a)



(b)

Figure 7.9 (a) Bottom Up Alligator (Fatigue Fracture) Cracking, and (b) International Roughness Index.

8 CONCLUSIONS AND RECOMMNENDATIONS

8.1 GENERAL

This study evaluated rheological properties of commonly used unmodified and modified performance grade (PG) binders in Oklahoma. Different dosages of two anti-stripping (AS) agents, namely, AD-here® HP Plus and Perma-Tac® Plus, and two warm mix asphalt (WMA) additives, namely, Advera® and Sasobit®, were blended with a local PG 64-22 binder. The evaluated rheological properties included consistency, linear viscoelastic (LVE) limit, temperature susceptibility, and loading frequency dependency. Specification tests (i.e., viscosity, PG grading) of these binders were conducted according to the Superpave® test methods, which required a rotational viscometer (RV), a dynamic shear rheometer (DSR), a rotational thin film oven (RTFO), a pressure aging vessel (PAV), and a bending beam rheometer (BBR). Non-specification binder tests (e.g., frequency sweep) were conducted by using a dynamic mechanical analyzer (DMA). The rheological data were used to predict mixing temperature, estimate PG grading, and evaluate potential rutting, fatigue fracture and thermal cracking of the modified binders. Selective rheological data were used to predict dynamic modulus (E*) values of asphalt concrete (AC) mixes commonly used in Oklahoma. Besides virgin binders, this study also evaluated recovered binders from recycled asphalt pavement (RAP) materials. Possible effects of the widely used Abson recovery method on the consistency and PG grade of the recovered binders were then evaluated. Finally, this study developed an inventory of the mechanistic-empirical design guide (MEPDG) input parameters for three certified PG binders (PG 64-22, PG

70-28 and PG 76-28) obtained from three different refineries in Oklahoma. The MEPDG input parameters were then used to predict major distress factors (rutting, fatigue fracture, and thermal cracking) of a typical AC pavement section in Oklahoma using the MEPDG version 1.100 software. The major findings of the current study and recommendations for future studies are presented next.

8.2 CONCLUSIONS

Specific conclusions pertaining to specific topics were included in individual chapter. The pertinent overall conclusions are summarized as follows:

- The DMA used in this study was found to be an effective and useful alternative to DSR, especially in evaluating asphalt binders at very low temperatures (12.7^{o}C or below), where the latter poses some compatibility issues. Because of the versatility of the DMA, it was found to be a valuable device for non-specification testing (e.g., strain sweep, frequency sweep, flow test) of asphalt binders.
- Liquid AS agents were found to reduce the rutting resistance of the base binder. The maximum allowable dosage of either of these AS agents (AD-here® Plus or Perma-Tac® Plus), for the tested PG 64-22 binder, was found to be 0.5% (by the weight of the binder). Neither of these AS agents seemed to alter the LVE limit of the base binder up to a strain level of 51%.
- Viscosity test data of the asphalt binder modified with Advera®, a water-bearing WMA additive, revealed that it was not effective in reducing the mixing and compaction temperatures of the binder. This was most likely due to

the fact that the adsorbed crystalline water (about 21%) in Advera® escaped during the heating and aging processes. In fact, Advera® creates foaming effects in the mixing plant by releasing the crystalline water and facilitates adequate coating of aggregates at a reduced temperature. The Superpave® binder test results also revealed that Advera® was found to increase the high temperature stiffness and decrease the low temperature stiffness of the base binder. Thus, the optimum dosage of Advera® was found to be 6% (by the weight of the base binder). This dosage level of Advera® was not expected to change the PG grade of the base binder.

- The RTFO-aging at a reduced operating temperature reduced the rutting factor of the Advera®-modified binder. Asphalt binder samples with 6% Advera®, RTFO-aged at 135°C, failed to meet the PG grade of the base binder. Thus, the poorer rut resistance of Advera® mixes is suspected to be due to the reduced production temperature rather than Advera® itself.

- Addition of 0.5% AD-here® HP Plus did not show any adverse impacts on the performance factors of the Advera®-modified binder. Rather, it improved the fatigue fracture resistance of the Advera®-modified binder. Furthermore, AD-here® HP Plus was found to increase the low temperature thermal cracking resistance of the Advera®-modified binder. However, the PG grade of the Advera®-modified binder remained unchanged with the addition of AD-here® HP Plus.

- Viscosity data showed that significant reduction in production temperatures could be achieved by using the Sasobit® technology. To attain the Superpave® specified target viscosity for proper mixing and compaction, the PG 64-22 binder modified with 3% Sasobit® was expected to reduce the mixing and compaction temperatures by 16°C and 19°C, respectively.
- The LVE limit was found to decrease with an increase in the dosage level of Sasobit®. In the case of 3% Sasobit®-modified binder, the corresponding LVE limits under unaged and RTFO-aged conditions were found to be 16% and 8%, respectively. Thus, for specification testing, it is recommended to maintain a strain level that does not exceed the actual LVE limit of the modified binder, especially with a high dosage of Sasobit®.
- Sasobit® was found to increase the stiffness of the binder at both high and low service temperatures. With 3% Sasobit®, it was observed that the PG grade of the base binder was increased by about one PG grade at both ends, indicating that the tested PG 64-22 binder became a PG 70-16 binder.
- The optimum dosage of Sasobit® was found to be 1.5% (by the weight of the binder). With this optimum dosage of Sasobit®, the continuous PG grade of the modified PG 64-22 binder was found to be PG 68.5-22.0.
- As expected, the reduced RTFO aging led to reduced oxidative age hardening of the Sasobit®-modified binder. The high PG temperature of 1.5% Sasobit®-modified binder, RTFO-aged at 121°C, is expected to be 3.8°C lower than that of the same binder RTFO-aged at 163°C. This indicated that the poorer rut

resistance of the Sasobit®-modified mix was due to the reduced production temperature (i.e., lower aging) rather than Sasobit® itself.

- AD-here® HP Plus (0.5%) did not show any adverse effects on the viscosity of the Sasobit®-modified binder. Rather, AD-here® HP Plus was expected to improve the fatigue resistance (reduced fatigue factor) and low temperature cracking resistance (decreased stiffness and increased m-value) without decreasing the rutting resistance significantly.

- The variations of consistency and stiffness of the binder recovered from the field RAP was found to be comparatively higher than its virgin counterpart, indicating inadequate supervision and quality control guidelines for RAP. Also, it was realized that reproducing the field RAP in the laboratory was practically impossible because of the unavailability of the mix design, exact virgin binder and aggregates from the original pavement.

- The recovered binders from RAPs were found to be significantly stiffer than the virgin binders. The corresponding continuous PG grades of the binders recovered from two simulated RAPs made with a PG 76-28 (continuous grade PG 79.8-33.7) and PG 64-22 (continuous grade PG 64.8-24.0) were found to be PG 94.8-30.6, and PG 86.2-18.6, respectively. Thus, compared to the virgin counterparts, the high critical and low critical temperatures of the recovered binders increased by at least three PG grades and one PG grade, respectively.

- The prolong use of the centrifuge and heat in the Abson recovery method were suspected to harden the recovered binder. It was believed that the extra age-hardening, during the recovery process, caused the high PG and low PG

temperatures of the recovered binder to increase up to 4°C and 3°C, respectively. The polymer-modified PG 76-28 binder was found to be more sensitive to the recovery process than the unmodified PG 64-22 binder.

- The loading frequency was found to have significant influence on the dynamic shear modulus (G*) of the asphalt binder. The G* value reduced as much as 18 times when the loading frequency was reduced from 10 Hz to 0.1 Hz. Likewise, the dynamic compressive modulus (E*) value of the corresponding mix reduced as much as five times.
- While using the MEDPG-adopted Witczak model, it was observed that the DSR test data of the asphalt binder significantly underestimated the E* values of both the control (HMA) and WMA mixes. On the other hand, rotational viscosity (RV) test data of the asphalt binder somewhat overestimate the E* values of these mixes.
- To predict the E* of asphalt mixes, the other commonly used Hirsch model, based on the G* data of the asphalt binder from frequency sweep tests, was found to be better than the Witczak model. The correlation coefficient (R^2) values of the Hirsch model for the control, WMA, and WMA with the AS agent mixes were found to be 0.95, 0.91, and 0.92, respectively. On the other hand, the R^2 values of the Witczak model, based on DSR test data, for the same mixes were obtained as 0.84, 0.83, and 0.81, respectively.
- The estimated E* values of the Sasobit®-modified WMA mix was found to be significantly higher than the control (HMA) mix. The AS agent did show significant change in the E* values of the WMA mix.

- The consistency and stiffness of tested binders varied significantly from one source to another. Consequently, the estimated ASTM A and VTS parameters from the RV and DSR test data of these binders differed significantly from the MEPDG suggested typical ASTM A and VTS values.
- The MEPDG software predicted that about 40% of the total rutting occurred in AC layers of a newly constructed pavement section, of which 80% would occur in the surface course. It also predicted that about 39% of rutting would occur within two years of construction.
- The AC layers' rut depths were found to vary significantly with the change in PG grade of the binder. In general, the higher the binder PG grade the lower the AC layer rut depth. The variation of rut depths between surface mixes with PG 64-22 and PG 76-28 binders was as high as 21%.
- In regard to the design reliability, significantly lower rut was predicted in the case of *Level 1* analysis compared with *Level 3* analysis. The reduction in rut depth obtained from *Level 3* to *Level 1* input was as much as 36% for the PG 70-28 binder. These findings are in agreement with predicted E* values from the DSR test data, which significantly underestimated the E* values.
- From *Level 2* analysis, it was observed that the AC layers' rutting was somewhat sensitive to the binder source, and the variation of the estimated rut depth was as high as 9%.
- Fatigue fracture and thermal cracking were not found to be critical distresses in this study. Also, binder type and source did not seem to have significant influence on these distresses. Consequently, the international roughness index

(IRI) was not found to be sensitive to the binder source due to the fact that its major influencing factors (alligator cracking and thermal cracking) were insignificant and insensitive to the binder source.

8.3 RECOMMENDATIONS

Based on the observations from this study, the following recommendations are made for future studies:

- The current study was limited to the evaluation of a PG 64-22 binder modified with AS agents and WMA additives. A future study pertinent to the viscoelastic analysis of polymer-modified binders (e.g., PG 76-28) modified with AS agents, WMA additives, and a combination of both, can be conducted.
- Hydrated lime is another commonly used AS additive in the United States. The effects of hydrated lime on rheological properties of the modified binder can be evaluated in a future study.
- As mentioned in this study, the usage of RAP has increased in recent years and is expected to be doubled by 2014. At the same time, the usage of WMA technologies is expected to grow significantly in near future. Therefore, a future study focusing on the evaluation of the performance of WMA with RAP, especially with high RAP content, can be conducted.
- This study evaluated binders recovered from RAPs. A future study encompassing the rheological evaluation of the binder recovered from local recycled asphalt shingles (RAS) can be conducted.

- The current study gathered major input parameters for the analysis of asphalt concrete pavements and presented some sensitivity analyses of binder type and source on distress parameters (rutting, fatigue fracture and thermal cracking) using the MEDPG software. However, local calibration factors for these distress parameters need to be determined for the implementation of the MEDPG in Oklahoma. Future studies may use the findings of this study and assess the calibration factors based on field performance of selected pavement sections in Oklahoma

DISCLAIMER

Neither the developers of this work nor the University of Oklahoma assume any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, product or process disclosed in this dissertation.

LIST OF SYMBOLS AND ABBREVIATIONS

List of Symbols

E^*	Elastic modulus
G^*	Dynamic shear modulus
$G^*.\sin\delta$	Fatigue factor
$G^*/\sin\delta$	Rutting factor
G'	Storage modulus
G''	Loss modulus
m	Rate of stress relaxation
δ	Phase angle
S	Stiffness
S_e	Standard error
S_y	Standard deviation of regression model
μ	Absolute viscosity

List of Abbreviations

AADTT	Annual Average Daily Truck Traffic
AASHTO	American Association of State Highway and Transportation Officials
AC	Asphalt Concrete
AS	Anti-stripping
ASTM	American Society of Civil Engineers
BBR	Bending Beam Rheometer
DMA	Dynamic Mechanical Analyzer

DOT	Department of Transportation
DSR	Dynamic Shear Rheometer
EICM	Enhanced Integrated Climate Model
ESAL	Equivalent Single Axle Load
FHWA	Federal Highway Administration
ETC	Environmental Testing Chamber
HMA	Hot Mix Asphalt
HT	High Temperature
IRI	International Roughness Index
IT	Intermediate Temperature
LTPP	Long Term Pavement Performance
LT	Low Temperature
LVE	Linear Viscoelastic
MEPDG	Mechanistic Empirical Pavement Design Guide
NA	Not applicable
NAPA	National Asphalt Pavement Association
NCAT	National Center for *Asphalt* Technology
ODOT	Oklahoma Department of Transportation
OHD	Oklahoma Highway Department
PAV	Pressure Aging Vessel
PG	Performance Grade
RAP	Recycled Asphalt Pavement
RAS	Recycled Asphalt Singles

RTFO	Rolling Thin Film Oven
RV	Rotational Viscometer
SD	Standard Deviation
SF	Shift Factor
SHRP®	Strategic Highway Research Program
SMA	Stone matrix asphalt
SFE	Surface Free Energy
SPSS	Statistical Package for the Social Sciences
Superpave®	Superior Performing Asphalt Pavements
TRB	Transportation Research Board
TSR	Tensile Strength Ratio
TTI	Texas Transportation Institute
TTS	Time Temperature Superposition
VTS	Viscosity Temperature Susceptibility
WMA	Warm Mix Asphalt

SI (METRIC) CONVERSION FACTORS

Approximate Conversions to SI Units					*Approximate Conversions from SI Units*				
Symbol	***When you know***	***Multiply by***	***To Find***	***Symbol***	***Symbol***	***When you know***	***Multiply by***	***To Find***	***Symbol***
		LENGTH					***LENGTH***		
in	*inches*	*25.40*	*millimeters*	*mm*	*mm*	*millimeters*	*0.0394*	*inches*	*in*
ft	*feet*	*0.3048*	*meters*	*m*	*m*	*meters*	*3.281*	*feet*	*ft*
yd	*yards*	*0.9144*	*meters*	*m*	*m*	*meters*	*1.094*	*yards*	*yds*
mi	*miles*	*1.609*	*kilometers*	*km*	*km*	*kilometers*	*0.6214*	*miles*	*mi*
		AREA					***AREA***		
in^2	*square inches*	*645.2*	*square millimeters*	mm^2	mm^2	*square millimeters*	*0.00155*	*square inches*	in^2
ft^2	*square feet*	*0.0929*	*square meters*	m^2	m^2	*square meters*	*10.764*	*square feet*	ft^2
yd^2	*square yards*	*0.8361*	*square meters*	m^2	m^2	*square meters*	*1.196*	*square yards*	yd^2
ac	*acres*	*0.4047*	*hectacres*	*ha*	*ha*	*hectacres*	*2.471*	*acres*	*ac*
mi^2	*square miles*	*2.590*	*square kilometers*	km^2	km^2	*square kilometers*	*0.3861*	*square miles*	mi^2
		VOLUME					***VOLUME***		
fl oz	*fluid ounces*	*29.57*	*milliliters*	*mL*	*mL*	*milliliters*	*0.0338*	*fluid ounces*	*fl oz*
gal	*gallon*	*3.785*	*liters*	*L*	*L*	*liters*	*0.2642*	*gallon*	*gal*
ft^3	*cubic feet*	*0.0283*	*cubic meters*	m^3	m^3	*cubic meters*	*35.315*	*cubic feet*	ft^3
yd^3	*cubic yards*	*0.7645*	*cubic meters*	m^3	m^3	*cubic meters*	*1.308*	*cubic yards*	yd^3
		MASS					***MASS***		
oz	*ounces*	*28.35*	*grams*	*g*	*g*	*grams*	*0.0353*	*ounces*	*oz*
lb	*pounds*	*0.4536*	*kilograms*	*kg*	*kg*	*kilograms*	*2.205*	*pounds*	*lb*
T	*short tons (2000 lb)*	*0.907*	*megagrams*	*Mg*	*Mg*	*megagrams*	*1.1023*	*short tons (2000 lb)*	*T*
		TEMPERATURE (exact)					***TEMPERATURE (exact)***		
°F	*degrees Fahrenheit*	*(°F-32)/1.8*	*degrees Celsius*	*°C*	*°C*	*degrees Fahrenheit*	*9/5(°C)+32*	*degrees Celsius*	*°F*
		FORCE and PRESSURE or STRESS					***FORCE and PRESSURE or STRESS***		
lbf	*poundforce*	*4.448*	*Newtons*	*N*	*N*	*Newtons*	*0.2248*	*poundforce*	*lbf*
lbf/in^2	*poundforce per square inch*	*6.895*	*kilopascals*	*kPa*	*kPa*	*kilopascals*	*0.1450*	*poundforce per square inch*	lbf/in^2