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Warm-Mix Asphalt Study: Evaluation of Hot- and Warm- Mix Asphalt with Respect to Binder Aging

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Partnered Pavement Research Center (PPRC) Contract Strategic Plan Element 4.41.2:
Environmental Impacts and Energy Efficiency of Warm Mix Asphalt

PREPARED FOR:

California Department of Transportation
Division of Research, Innovation, and System Information
Office of Materials and Infrastructure Roadway Research

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DOCUMENT RETRIEVAL PAGE **Research Report: UCPRC-RR-2013-02**

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Caltrans Technical Lead: T. Joe Holland (Caltrans) and Nathan Gauff (CalRecycle)

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Abstract:

Warm-mix asphalt (WMA) technologies are added to asphalt binders to lower production and construction temperatures, reduce emissions, allow for increase haul distances, and improve the workability. Reduced temperatures at the plant and during laydown and compaction are hypothesized to impact long-term oxidative aging behavior. This study attempted to quantify these impacts through characterization of field-aged unmodified and rubber-modified binders extracted and recovered from cores sampled from 13 test sections representing seven different WMA technologies and associated hot-mix controls. A dynamic shear rheometer (DSR) was used to evaluate the binder rheological properties at high temperatures with respect to expected rutting performance. The cup-and-bob DSR testing procedure was assessed as an alternative approach for testing rubberized binders. A bending beam rheometer (BBR) was used to characterize low-temperature properties. Test results did not appear to be influenced by the warm-mix technology chemistry. However, the organic wax additive consistently showed better rutting resistance across all tests, and this was attributed to the residual crystallization wax structure in the binder. All test results appeared to be influenced by production and placement temperatures, indicating that some mixes produced at very low temperatures could be more susceptible to early rutting on pavements experiencing high ambient temperatures and high traffic loading. Air-void content appeared to have very little effect on the rheological properties of the extracted binder over the aging period assessed, which was not expected. The results indicated that zero shear viscosity (ZSV) is a good indicator of the rheological behavior of asphalt binders with respect to rutting performance, as observed from accelerated load testing. ZSV was also found to be more suitable for describing the rutting performance of rubberized binders than the current Superpave G*/sinδ criterion. Viscosity-shear susceptibility is a suitable parameter for understanding the shear sensitivity of rubberized binders. It increases during long-term oxidative aging due to the increased association of polar compounds. The non-recoverable creep compliance and percent recovery parameters obtained from the multiple stress creep recovery test are useful parameters for understanding expected field rutting performance. Bending beam rheometer results indicated that the WMA technologies tested did not result in a grade change with respect to thermal cracking properties at low temperatures, with all binders meeting the Superpave criteria at all ages tested. Performance trends for individual binders were consistent with rutting test results. The warm-mix additives and associated lower production and placement temperatures generally had limited effect on aging kinetics with respect to long-term field aging, with the exception of the organic wax. Laboratory binder aging, specifically the rolling thin film oven test, did not always correspond to field performance.

Keywords:
Warm-mix asphalt, rubberized hot-mix asphalt, warm-mix asphalt, binder aging

Proposals for implementation:
None

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UCPRC Research Reports, RR-2008-11, RR-2009-02, RR-2011-02, RR-2011-03, RR-2013-03

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PROJECT OBJECTIVES

The objective of this study is to determine whether the reduced production and compaction temperatures associated with the use of warm-mix asphalt technologies result in different rheological properties and binder aging kinetics compared to the same binder used in hot-mix asphalt. This will be achieved through the following tasks:

1. Review of the literature
2. Sampling of cores from two warm-mix asphalt test tracks (total of 13 test sections)
3. Extraction and recovery of binders from the cores
4. Evaluation and characterization of the rheological properties of the recovered asphalt binders and comparison of results with field and laboratory rutting performance data

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EXECUTIVE SUMMARY

Warm-mix asphalt (WMA) technologies are added to asphalt binders to lower production and construction temperatures, reduce emissions, allow increased haul distances, and improve workability. Reduced temperatures at the plant and during laydown and compaction are hypothesized to impact long-term oxidative aging behavior. This study attempted to quantify these impacts through characterization of field-aged unmodified and rubber-modified binders extracted and recovered from cores sampled from 13 test sections representing seven different WMA technologies and associated hot-mix controls. A dynamic shear rheometer (DSR) was used to evaluate the binder rheological properties at high temperatures with respect to expected rutting performance. The cup-and-bob DSR testing procedure was assessed as an alternative approach for testing rubberized binders. A bending beam rheometer (BBR) was used to characterize low-temperature properties. The following observations were made from the results:

- Test results did not appear to be influenced by the warm-mix technology chemistry. However, the organic wax additive consistently showed better rutting resistance across all the tests, and this was attributed to the residual crystallization wax structure in the binder.
- All the test results appeared to be influenced by production and placement temperatures, indicating that some mixes produced at very low temperatures could be more susceptible to early rutting on pavements that experience high ambient temperatures and high traffic loading.
- Air-void content appeared to have very little effect on the rheological properties of the extracted binder over the aging period assessed, which was not expected.
- Zero shear viscosity (ZSV) was found to be a good indicator of the rheological behavior of asphalt binders with respect to rutting performance, as observed from accelerated load testing. ZSV was also found to be more suitable for describing the rutting performance of rubberized binders than the current Superpave $G^*/\sin\delta$ criterion.
- Viscosity-shear susceptibility was found to be a suitable parameter for understanding the shear sensitivity of rubberized binders. Viscosity-shear susceptibility increased during long-term oxidative aging due to the increased association of polar compounds.
- The non-recoverable creep compliance and percent recovery parameters obtained from the multiple stress creep recovery test are useful parameters for understanding expected field rutting performance.
- Bending beam rheometer results indicated that the WMA technologies tested did not result in a grade change with respect to thermal cracking properties at low temperatures, with all binders meeting the Superpave criteria at all ages tested. Performance trends for individual binders were consistent with rutting test results.
- The warm-mix additives and associated lower production and placement temperatures generally had limited effect on aging kinetics with respect to long-term field aging, with the exception of the organic wax.
- Laboratory binder aging, specifically the rolling thin film oven test, did not always correspond to field performance.

Current laboratory binder aging protocols do not necessarily provide results that correspond to field aging. The findings of the NCHRP 9-52 study should be reviewed and the recommended changes implemented if appropriate. Since the NCHRP study is not investigating rubberized binders, the applicability of these recommendations to rubberized binder aging should be investigated for a range of binder sources and field aging conditions in California.

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LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
BBR	Bending beam rheometer
Caltrans	California Department of Transportation
CalRecycle	California Department of Resources, Recycling, and Recovery
CA	Carbonyl area
CF	Chemical foam
CS	Chemical surfactant
CT	Commuted tomography
DGAC	Dense-graded asphalt concrete
DMA	Dynamic mechanical analysis
DSR	Dynamic shear rheometer
ESAL	Equivalent standard axle load
FHWA	Federal Highway Administration
G*	Complex modulus
G’	Shear storage modulus
G’’	Shear loss modulus
HMA	Hot-mix asphalt
HVS	Heavy Vehicle Simulator
HWTT	Hamburg Wheel-Track Test
J_{nr}	Non recoverable compliance
LVE	Linear viscoelastic
MF	Mechanical foam
MSCR	Multiple stress creep recovery
η^*	Complex viscosity
NCHRP	National Cooperative Highway Research Program
OW	Organic wax
PAV	Pressure aging vessel
PG	Performance grade
PM	Polymer modified
PPRC	Partnered Pavement Research Center
RHMA-G	Gap-graded rubberized hot-mix asphalt
RSST-CH	Repeated simple shear test at constant height
RTFO	Rolling thin film oven
RWMA-G	Gap-graded rubberized warm-mix asphalt
SHRP	Strategic Highway Research Program
SPE	Strategic Plan Element
TFOT	Thin film oven test
UCPRC	University of California Pavement Research Center
VSS	Viscosity-shear susceptibility
WMA	Warm-mix asphalt
ZSV	Zero shear viscosity

CONVERSION FACTORS

SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
		LENGTH		
in	inches	25.4	Millimeters	mm
ft	feet	0.305	Meters	m
yd	yards	0.914	Meters	m
mi	miles	1.61	Kilometers	Km
		AREA		
in^2	square inches	645.2	Square millimeters	mm^2
ft^2	square feet	0.093	Square meters	m^2
yd^2	square yard	0.836	Square meters	m^2
ac	acres	0.405	Hectares	ha
mi^2	square miles	2.59	Square kilometers	km^2
		VOLUME		
fl oz	fluid ounces	29.57	Milliliters	mL
gal	gallons	3.785	Liters	L
ft^3	cubic feet	0.028	cubic meters	m^3
yd^3	cubic yards	0.765	cubic meters	m^3
		NOTE: volumes greater than 1000 L shall be shown in m^3		
		MASS		
oz	ounces	28.35	Grams	g
lb	pounds	0.454	Kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
		TEMPERATURE (exact degrees)		
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C
		ILLUMINATION		
fc	foot-candles	10.76	Lux	lx
fl	foot-Lamberts	3.426	candela/m^2	cd/m^2
		FORCE and PRESSURE or STRESS		
lbf	poundforce	4.45	Newtons	N
lbf/in^2	poundforce per square inch	6.89	Kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
		LENGTH		
mm	millimeters	0.039	Inches	in
m	meters	3.28	Feet	ft
m	meters	1.09	Yards	yd
km	kilometers	0.621	Miles	mi
		AREA		
mm^2	square millimeters	0.0016	square inches	in^2
m^2	square meters	10.764	square feet	ft^2
m^2	square meters	1.195	square yards	yd^2
ha	Hectares	2.47	Acres	ac
km^2	square kilometers	0.386	square miles	mi^2
		VOLUME		
mL	Milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	Gallons	gal
m^3	cubic meters	35.314	cubic feet	ft^3
m^3	cubic meters	1.307	cubic yards	yd^3
		MASS		
g	grams	0.035	Ounces	oz
kg	kilograms	2.202	Pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
		TEMPERATURE (exact degrees)		
°C	Celsius	1.8C+32	Fahrenheit	°F
		ILLUMINATION		
lx	lux	0.0929	foot-candles	fc
cd/m^2	candela/m^2	0.2919	foot-Lamberts	fl
		FORCE and PRESSURE or STRESS		
N	newtons	0.225	Poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in^2

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

1. INTRODUCTION

1.1 Background

Warm-mix asphalt is a relatively new technology. It was developed in response to the needs for reduced energy consumption and stack emissions during the production of asphalt concrete, to allow longer haul distances, lower placement temperatures, improved workability, and better working conditions for plant and paving crews. Studies in the United States and Europe indicate that significant reductions in production and placement temperatures, and potentially related emissions, are possible.

The California Department of Transportation (Caltrans) has expressed interest in warm-mix asphalt with a view to reducing stack emissions at asphalt plants, to allowing longer haul distances between asphalt plants and construction projects, to improving construction quality (especially during nighttime closures), to improving working conditions during construction, and to extending the annual period for paving. However, the use of warm-mix asphalt technologies requires incorporating an additive into the mix, and/or changes in production and construction procedures specifically related to temperature, which could influence the short- and long-term performance of the pavement, as well as the emissions during production and placement. Consequently, Caltrans identified the need for research to address a range of concerns related to these changes before statewide implementation of the technology is approved.

1.2 Project Objectives

The research presented in this report is part of Partnered Pavement Research Center Strategic Plan Element 4.41.2 (PPRC SPE 4.41.2), titled “Environmental Impacts and Energy Efficiency of Warm Mix Asphalt,” which has been undertaken for Caltrans and the California Department of Resources, Recycling, and Recovery (CalRecycle) by the University of California Pavement Research Center (UCPRC). This study follows an earlier three-phase project (SPE 4.18), which assessed the performance of warm-mix asphalt in laboratory, accelerated loading, and full-scale field trials on California highways *(1-6)*. The objective of the study documented in this report was to undertake a preliminary investigation into the effects of different warm-mix asphalt technologies on binder aging. This was achieved through a study of the relevant literature and a series of laboratory tests on binders extracted from asphalt concrete used in two test tracks constructed to compare the performance of warm mixes, produced using a range of different warm-mix asphalt technologies, against hot-mix asphalt control mixes.

1.3 Structure and Content of this Report

This report presents an overview of the work carried out in meeting the objectives of the study, and is organized as follows:

- Chapter 2 provides an overview of studies on binder aging and the various factors that influence it.
- Chapter 3 summarizes the experiment plan and describes the materials tested in the study.
- Chapter 4 presents the test results and associated discussion.
- Chapter 5 provides conclusions and preliminary recommendations.

1.4 Measurement Units

Although Caltrans recently returned to the use of U.S. standard measurement units, metric units have always been used by the UCPRC in the design and layout of experiments, for laboratory and field measurements, and for data storage. In this report, both U.S. Customary and metric units (provided in parentheses after the U.S. Customary units) are provided in general discussion. Laboratory testing approaches and methods developed specifically in metric (e.g., the asphalt performance grading [PG] procedure) are not converted into English units. In keeping with convention, only metric units are used in Heavy Vehicle Simulator (HVS) and laboratory data analyses and reporting. A conversion table is provided on page x at the beginning of this report.

1.5 Terminology

The term “asphalt concrete” is used in this report as a general descriptor for asphalt concrete surfacings. The terms “hot-mix asphalt (HMA)” and “warm-mix asphalt (WMA)” are used as descriptors to differentiate between the control mixes and the warm-mixes discussed in this study.

2. LITERATURE REVIEW

2.1 Asphalt Aging

The phenomenon of asphalt mixtures becoming stiffer and more brittle with age is known as asphalt aging or age-hardening. The major mechanisms cited in the literature to explain this phenomenon include volatilization, oxidation, polymerization, and separation *(7-9)*. Volatilization and oxidation are generally accepted as the two most important mechanisms.

Asphalt binder components can be grouped into four broad groups for chemical characterization, namely asphaltenes, resins, cyclic aromatics, and saturates.

- Asphaltenes are black solids containing carbon, hydrogen, nitrogen, sulfur, and oxygen. They are considered as highly polar materials of high molecular weight *(10),* and typically constitute between 5 and 25 percent of the weight of asphalt.
- Resins (polar aromatics) are dark-colored, solid or semi-solid materials and act as dispersing agents for the asphaltenes. They typically constitute between 15 and 25 percent of the weight of the asphalt.
- Cyclic aromatics and saturates are converted to resins during oxidation, which are in turn converted to asphaltenes. As the asphaltene content increases, the stiffness of the asphalt increases.
- Saturates comprise the straight and branched-chain aliphatic hydrocarbons present in asphalt. This fraction typically constitutes between 5 and 20 percent of the weight of the asphalt.

2.1.1 Volatilization

Volatilization (or loss of volatiles) is the loss of lighter fractions from the binder. Volatilization mostly occurs at elevated temperatures during mixing and leads to increased binder viscosity and binder hardening. It occurs independently of oxidation *(11)*. During the Strategic Highway Research Program (SHRP), Anderson *(12)* compared four different binders in the thin film oven test (TFOT) and aged the binders under both air (to simulate oxidation and volatilization) and under nitrogen (to simulate volatilization). The study concluded that volatilization contributes significantly to age hardening and that high polar asphalt binders experienced more mass loss than low polar asphalt binders.

2.1.2 Oxidation

Oxidation is the chemical reaction of atmospheric oxygen with the asphalt binder. It occurs throughout the life of the asphalt, including during mixing, placement, and service life. As the asphalt molecules react with oxygen, new polar sites are formed, which increases the stiffness of the binder. Oxidation of the aromatic compounds present in asphalt binder results in stronger association between asphalt components, which increases the asphaltene fraction and in turn leads to a stiffening of the binder in both its elastic modulus and viscosity *(13,14)*. The rate and amount of oxidation depends on factors such as environmental conditions (e.g., high temperatures in summer), the chemical nature of the binder (e.g., different crude sources *[10,15]*), and additives such as polymers, crumb rubber, and warm-mix technologies.

Mixes produced with WMA technologies are hypothesized to experience less oxidation and aging at the plant due to reduced production and placement temperatures.

2.1.3 Polymerization and Separation

Polymerization is a combination reaction in which smaller similar molecules continually combine to form larger molecules that results in a progressive increase in hardness. It has been speculated that polymerization mostly affects the low temperature aging of asphalt, although Roberts *(8)* found no specific evidence that supported its occurrence in asphalt pavements. Separation is the removal of oily constituents, resins, or asphaltenes from asphalt binder and its absorption into porous aggregates.

2.2 Oxidation Kinetics

Asphalt binder is oxidized and hardened during the high temperatures experienced during mixing, placement, and compaction. As binder oxidizes, carbonyl (–C=O) groups are formed, which increase the polarity of their host compounds and render them more likely to associate with other polar compounds *(16)*. During these associations, less soluble asphaltene materials are created, the formation of which leads to asphalt hardening. The binder oxidation rate follows an Arrhenius equation, which implies an initial rapid rate period (initial jump) until a constant rate period is reached, after which oxidation continues at that constant rate *(13,16)*:

$$r_n = \frac{\partial n}{\partial t} = AP^{\alpha} Exp(-E_n / RT) \tag{2.1}$$

Where: n is viscosity or carbonyl area (CA)
A is the pre-exponential factor
P is the pressure
α is the reaction order with respect to oxygen pressure
E is the activation energy
R is the gas constant
T is the absolute temperature.

Figure 2.1 shows a typical aging plot for an unmodified asphalt binder in which η_{ot} is the original viscosity of the binder before injection at the mixing plant. The viscosity after the initial jump (η_{tj}) is defined by the intercept of the constant-rate line. Region A is defined as the time of the initial jump during mixing and placement, and Region B is defined as the constant rate of aging during service.

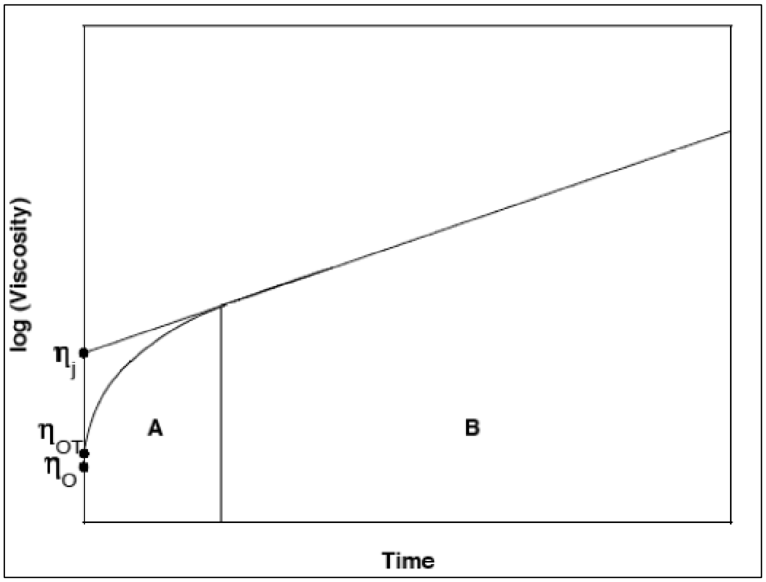


Figure 2.1: Typical response of unmodified asphalt binder to oxidation aging *(17)*.

2.3 Binder Rheology

2.3.1 Effect of Aging Temperature

Oxidation is highly correlated to aging temperature (both during mixing and in service). Numerous studies have demonstrated that temperature significantly affects asphalt aging, both in laboratory and field aging studies *(15,18-20)*. The results of these studies showed strong correlation between maximum pavement temperature and aging in the field. The studies also showed the importance of selecting laboratory temperatures that are as close to the actual aging condition as possible when simulating aging in the laboratory. Anderson *(12)* showed that a 10°C increase in aging temperature resulted in a 100 percent increase in aging-hardening at two pressure aging vessel (PAV) temperatures (60°C and 70°C).

2.3.2 Effect of Air Voids

Air voids in a mixture can significantly affect asphalt aging. Oxygen diffusion in compacted asphalt mixtures is directly related to air-void content, with higher contents resulting in higher rates of oxidative aging. The correlation between air voids and age hardening has been demonstrated by several investigators *(21, 22)*. The results of a study by Martin *(22)* showed that an air-void content increase of about 1.5 percent resulted in an almost tenfold increase in viscosity change over a two-year period after construction. An important conclusion of these studies was that a high air-void content (5 percent or higher) correlates with higher age-hardening because oxygen is more freely available to react with the binder.

2.3.3 Effect of Aggregate Type

Asphalt binder constituents can potentially be absorbed and/or adsorbed into the surfaces of mineral aggregates. Early research by Peterson *(23)* and more recent research done as part of the first Strategic Highway Research Program (SHRP), investigated the differences in aging performance of the same asphalt when mixed with different aggregates. The studies found out that the difference in aging was mainly caused by different adsorption properties of the aggregates, specifically those associated with highly polar fractions. Adsorption of these fractions inhibits their reaction with oxygen and reduces the rate of oxidation. Aggregates showing the lowest adsorption of highly polar compounds (e.g., quartzite) exhibited increased catalytic effects in asphalt oxidation, while those showing the highest adsorption (e.g., limestone) exhibited the lowest catalyst effect.

2.4 Test Methods

2.4.1 Multiple Stress Creep Recovery Test

One of the objectives in the development of the SHRP Performance Grade (PG) asphalt binder specification was to make use of performance-related criteria specific that are specific to distresses related to climate and traffic loading. Meeting this objective implied that test measurements would need to be made at temperatures and loading rates consistent with actual conditions that pavements are subject to. However, in the PG specification, the temperature criteria stay the same for determining the high temperature performance parameter (i.e., complex modulus divided by the sine of the phase angle [$G^*/\sin\delta$]) regardless of the location of the pavement. To show improved rutting performance, a performance grade bump is required to represent a stiffer binder.

While the performance grading concept works well for conventional speed and moderate traffic volumes, recent research has indicated that some refinement is needed for pavements that have slow speed loading and high traffic volumes *(24)*. Rather than change the criteria or test conditions to reflect a change in loading time and traffic volume, researchers elected to simply adjust for traffic speed and volume by grade-bumping or testing at higher temperatures than those indicated by the local climate *(25)*. For example, a designer might specify a PG 58-28 binder for typical traffic in an average climate, but might also specify a PG 70-28 binder for a pavement with high traffic volumes in the same area, even though the pavement temperature would not go above 58°C. One problem with grade-bumping in the PG system, however, is that the performance-related properties of the PG 70-28 binder would be determined at a temperature at least 12°C higher than the highest pavement temperature will experience. This can result in problems during construction at lower temperatures.

Another objective of the PG system was to make performance-related properties blind to modifications, which implies that all asphalt binders of a particular performance grade would be expected to perform identically in the same traffic/environmental conditions regardless of crude source, refining process, or mix production method. Although the G*/sinδ parameter does capture viscous and elastic effects, it is unable to adequately quantify the benefits of elastomeric modification because of the relatively small impact of phase angle on the overall value of G*/sinδ. As a result, additional empirical tests referred to as "plus" tests were developed to insure that the performance of the modified binder is appropriately evaluated *(26)*.

To overcome these issues, a performance-related specification for the high-temperature characterization of binder was developed. The specification was determined using the Multiple Stress Creep Recovery (MSCR) test. The approach was developed during the SHRP program *(27)* and is based on the findings of the Repeated Simple Shear Test at Constant Height (RSST-CH), which is conducted using repeated cycles of 0.1 second shear load followed by a 0.6 second rest period. The MSCR test, undertaken with a dynamic shear rheometer (DSR), uses the well-established relationship between creep and recovery in asphalt binder, and the binder's potential for permanent deformation. In the test, a one-second creep load is applied to the sample, and this is followed by a nine-second recovery period. The test starts with a low stress (0.1 kPa) for 10 creep/recovery cycles, which is then increased to 3.2 kPa and repeated for an additional 10 cycles. Unlike the PG system, the test temperature used for the MSCR test is selected based on actual high pavement temperatures with no grade bumping.

2.4.2 Zero Shear Viscosity

The Strategic Highway Research Program specifies G*/sinδ as a parameter for determining the rutting resistance of the binder. This specification is used for characterizing the rutting resistance of the binder with respect to permanent deformation in the field and is measured at a fixed temperature and frequency of loading (upper limit of PG grade, and 10 rad/sec). This frequency corresponds to a total sinusoidal load without any rest period of 24 to 48 km/h in the field *(28)*. Since a moving wheel load generates a wide range of frequencies in a pavement structure, it is important to study the behavior of flexible pavements subjected to a moving wheel across an applicable range of frequencies to describe the material properties.

The viscosity of the binder also changes with temperature and frequency of loading and consequently the G*/sinδ parameter is considered by some to be ineffective in capturing the rutting performance of the binder, especially that of modified binders *(29)*. Rubberized binders and binders with warm-mix additives are considered as modified binders, which could behave as non-Newtonian materials (viscosity is a linear function of shear strain rate for Newtonian materials). To overcome these concerns, zero shear viscosity

(ZSV) has been suggested as an alternative parameter for describing the rutting resistance of modified binders *(30)* and is defined as the viscosity measured in shear deformation, when the shear rate is approaching zero. In order to calculate the ZSV, the binder should reach a steady state ($d\gamma/dt$ = const.) of purely viscous strain to attain a plateau in the viscosity curve (i.e., the goal is to produce a creep curve showing a constant slope at the end of the creep phase). At least 10 percent of the measuring points of the entire curve should occur within this range of steady state flow. Once the plateau region is reached, the data from the frequency sweep results are fitted to the Cross/Sybilski's model as follows *(31,32)*:

$$\eta^* = \frac{\eta_0}{1+(\kappa\omega)^m} \quad 3.1$$

Where:
η^*= complex viscosity
η_0 = first viscosity after the plateau region
ω = frequency (rad/sec)
K and m are dimensionless parameters

The ZSVs calculated by the Cross/Sybilski's model are conventionally determined by extrapolating the data to a frequency of 0 Hz. However, the approach used in the UCPRC study discussed in the following chapters involved performing a frequency sweep test and determining the ZSV value at the lowest frequency that the DSR is capable of (0.01 Hz) *(33)*. The advantage of this approach is that it does not force formation of a plateau region as does the Cross/Sybilski's model, and the calculated values are more representative of the materials' actual behavior.

The ZSV is said to be an indicator of two rutting-related binder characteristics, namely the stiffness of the binder, and the binder's resistance to permanent deformation under long term loading *(31)*. Although the test was developed as an alternative rutting parameter for polymer-modified binders (including both elastomers and plastomers) *(17,34)*, a well-established method to run the test has not been documented in the literature. In order to understand the rutting resistance properties of the various unmodified and modified binders assessed in the UCPRC study, the test was run at 65°C and 25°C.

Another parameter that has been suggested to describe the shear sensitivity of binders is *viscosity-shear susceptibility* (VSS) *(35)*, which is defined as the slope of the viscosity-frequency log plot. Using this parameter involves calculating the absolute values of the slope and then reporting the reciprocal values. A lower slope value indicates greater sensitivity to shear. The frequency sweep test is run at 25°C on samples aged for different periods.

2.4.3 Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) is conducted within the region of linear viscoelastic response using oscillatory-type testing on asphalt binder samples. DMA allows the viscous and elastic nature of the binder to be determined over a wide range of temperatures and loading times (loading frequencies). The amplitude of the stress is measured by determining the torque transmitted through the sample in response to the imposed strain. Calculated parameters include complex shear modulus (G*), phase angle (δ), shear storage modulus (G'), shear loss modulus (G''), and complex viscosity (η*).

Most empirical tests used to predict rutting resistance (e.g., SHRP performance grade or earlier penetration grade) are often inaccurate in identifying the effects of binder modification because they were developed for neat unmodified binders, and they only quantify the rheological behavior of the binder at a certain temperature and time of loading. This is a particular concern for modified binders such as rubberized asphalt with warm mix additives where the applicability of the time-temperature superposition principle is questionable *(36)*.

In this UCPRC study discussed in the following chapters, results of the dynamic mechanical analysis are reported in "black diagram" plots, which plot complex modulus versus phase angle at different times of loading and temperatures of interest.

2.4.4 Dynamic Shear Rheometer Testing Geometries

In conventional DSR testing, a sample of asphalt binder is sandwiched between two parallel plates, one that is fixed and one that oscillates. In this type of testing, a complete oscillation is one cycle and oscillations are repeated for a number of cycles. The number of cycles completed in one second is the loading frequency. In the UCPRC study discussed in the following chapters, DSR tests were run at a range of frequencies (0.1 rad/sec to 100 rad/sec) to simulate a range of moving wheel loads.

After the asphalt binder is applied to the bottom plate, the two plates are brought together until an appropriate gap setting (1 mm for 25 mm plates and 2 mm for 8 mm plates), along with an extra gap for trimming, is achieved. Next, the sample is trimmed so that the asphalt binder is flush with the edges of the plates. This trimming ensures that the diameter of the test specimen is controlled, enabling an accurate calculation of the complex modulus. After trimming, the extra gap is eliminated. This geometry, however, limits the material that can be tested between the plates. Particles larger than 250 μm cannot be tested due to the possible interaction of the particles with the torque and strain response of the binder *(23)*. Consequently, the test cannot be used with rubberized binders since these contain rubber particles in sizes that can range from 0.5 mm to over 1 mm. A 1 mm rubber particle in a 1 mm gap plate-to-plate geometry would touch both the top and bottom plates at the same time, and consequently the test results would represent the rubber particle and not the rubber modified binder. Therefore a different testing geometry is required.

Most studies that have examined rubberized binders have used viscosity to characterize their rheological properties. However, while viscosity is an important parameter for the workability of the binder and the mix, it does not directly relate to the binder's in-service performance. Also, viscosity measurements alone lack sufficient accuracy to completely describe the complex properties of these binders due to their particulate phase. The Federal Highway Administration (FHWA) has undertaken some work on characterizing terminal blends with the DSR by using plate-to-plate geometry and increasing the gap size from 1 mm to 2 mm *(37)*. Crumb rubber particle sizes in terminal blends are 0.178 mm or finer, which makes it possible to measure the properties of these binders with the standard plate-to-plate measuring systems. However, in California, Arizona, and Alaska, the crumb rubber size in the wet process is in the 1.4 mm (-14 mesh) range, and consequently, the rubber particles would significantly influence the test.

In the UCPRC study discussed in the following chapters, the testing of rubberized binders was conducted with a "cup-and-bob" geometry that uses a 27 mm cup and 14 mm bob to give a 6.5 mm effective gap size. This gap size is more than enough to accommodate the swelled crumb rubber particles. The initial evaluation with the cup-and-bob geometry included comparing the results of the parallel plate geometries to the cup-and-bob geometry. Three binders were evaluated (PG 64-16, PG 64-28PM, and PG 70-10). The percent difference between the results for the three binders was less than the allowable single operator tolerance for DSR results, indicating that the new geometry provided results similar to the existing 1 mm and 2 mm plates.

3. EXPERIMENT DESIGN

3.1 Research Scope

The investigation for this research study was focused on developing a preliminary understanding of the effects of WMA technologies on binder oxidation during mix production, placement, and initial service, with specific focus on how the oxidation is related to lower mix temperatures. This research addressed the following issues:

- The effect of different WMA technologies on the rheological properties of asphalt binders that influence rutting resistance and low temperature cracking in the field.
- The effect of reduced production and compaction temperatures of WMA mixes on long-term oxidative aging kinetics in the field.
- Measurement and interpretation of the rheological properties of rubberized binders with and without WMA technologies in the laboratory.
- Comparison of the laboratory-determined rheological properties of binders to field performance in terms of rutting resistance.

3.2 Research Approach

The analysis of the rheological properties of binders with different WMA technologies with respect to mix performance in the field included both short-term (rutting resistance) and longer-term (fatigue and thermal cracking resistance) issues. The kinetics of binder properties with field aging time were analyzed and compared to results from laboratory and accelerated load testing.

3.3 Materials

Materials used in this research were sourced from loose mix and/or cores sampled from two different construction projects (termed Phase 1 and Phase 2 in this report) as part of a broader research project for Caltrans studying field performance of warm-mix asphalt technologies. The first project (Phase 1) was a conventional dense-graded mix, and cores were taken at three different times; soon after construction and then again after 48 and 52 months. The second project (Phase 2) was gap-graded rubberized asphalt. Two mix designs (termed Mix Design #1 and Mix Design #2 in this report) from two different asphalt plants were assessed. Loose mix was sampled during construction and then cores were taken 14 and 18 months thereafter.

3.3.1 Conventional Mixes (Phase 1)

The first test section was constructed in June 2007 at a quarry near Watsonville. The test sections included a hot-mix control and three different warm-mix technologies (chemical water foaming additive [WMA-CF], chemical surfactant additive [WMA-CS1], and organic wax [WMA-OW]).

A standard DGAC mix design with 19 mm nominal maximum size coarse aggregate was used for these test sections. Two 60 mm lifts were placed. The base asphalt binder was graded as PG 64-16. Mix properties are summarized in Table 3.1. The mix design was not adjusted to accommodate the WMA technologies. Production temperatures for the warm mixes were the same for all technologies (60°F [30°C] below the control mix).

Table 3.1: Mix Properties of Conventional Mixes

Parameter	Control	WMA-CF	WMA-CS1	WMA-OW
Binder Content (%)	5.4	5.4	5.4	4.7
Production Temperature (°C/°F)	153 (308)	120 (248)	122 (252)	121 (251)
Average in-place Air Voids (%)	5.6	5.4	7.1	7.0

3.3.2 Rubberized Mixes (Phase 2)

The Phase 2 mixes were produced at two different asphalt plants in order to accommodate two different mechanical foaming WMA processes. Two hot-mix controls and seven warm mixes were assessed as follows:

- Mix Design #1: Mechanical foaming additive (R-WMA-MF1) and two chemical surfactant additives (R-WMA-CS1 and R-WMA-CS2).
- Mix Design #2: Mechanical foaming additive (R-WMA-MF2), chemical foaming additive (R-WMA-CF), organic wax additive (R-WMA-OW), and chemical surfactant additive (R-WMA-CS3).

The two mix designs both met Caltrans specifications for standard 12.5 mm nominal maximum aggregate size Type-1 gap-graded asphalt rubber. The mix properties are summarized in Table 3.2.

Table 3.2: Mix Properties of Rubberized Mixes

Mix Design #1					
Parameter	**Control**	**R-WMA-MF1**	**R-WMA-CS1**	**R-WMA-CS2**	
Binder Content (%)[1]	7.7	7.9	7.7	7.7	
Production Temperature (°C/°F)	160/(320)	140/(284)	125/(237)	130/(266)	
Paving Temperature (°C/°F)[3]	154/(309)	128/(262)	120/(248)	128/(262)	
Average in-place Air Voids (%)	4.9	6.3	6.2	6.4	
Mix Design #2					
Parameter	**Control**	**R-WMA-OW**	**R-WMA-CF**	**R-WMA-MF2**	**R-WMA-CS3**
Binder Content (%)[2]	7.7	8.0	7.6	8.4	9.0
Production Temperature (°C/°F)	166/(335)	149/(300)	145/(295)	145/(295)	140/(285)
Paving Temperature (°C/°F)[3]	137/(279)	137/(279)	130/(266)	125/(257)	126/(258)
Average in-place Air Voids (%)	11.6	8.5	10.7	9.1	8.4

[1] Target 7.3% [2] Target 8.3% [3] Behind screed

No adjustments were made to the mix designs to accommodate the WMA technologies. The target binder contents for the two mixes (Mix Design #1 and Mix Design #2) were 7.3 percent and 8.3 percent by mass of dry aggregate, respectively. PG 64-16 base binders were used in both mixes, but were sourced from different refineries. The two mix designs contained 18 and 19 percent crumb rubber by weight of the binder. The modified binders were produced using the standard wet process according to Caltrans specifications. The production temperature of each R-WMA mix was set by the technology provider. Haul distances were approximately one hour from the asphalt plant for Mix Design #1 and approximately two hours for Mix Design #2.

3.4 Test Methods

3.4.1 Binder Extraction

Binders were extracted from loose mix and cores using a Rotovapor as per ASTM D2172, *Standard Test Methods for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures*, and ASTM D5404, *Standard Practice for Recovery of Asphalt from Solution Using the Rotary Evaporator*. This procedure uses three successive washes: one of 100 percent toluene followed by two washes of a mixture of 15 percent ethanol plus 85 percent toluene by volume *(38,39)*. After extraction, the solvent is filtered to remove all aggregate particles from the binder solution using a centrifuge. The binder is then recovered from the solvent with a Buchi Rotovap. During recovery, nitrogen gas is introduced into the vessel to drive off any remaining solvent and to prevent contact with oxygen.

3.4.2 Rheological Properties

All test specimens were prepared using the standard test method for determining the rheological properties of asphalt binder (AASHTO TP-5, *Method for Determining the Rheological Properties of Asphalt Binders Using a Dynamic Shear Rheometer).* Rheological properties were obtained at temperatures of 25°C and 65°C and frequencies ranging from 0.1 to 100 rad/sec using a dynamic shear rheometer (DSR) in strain mode. The linear viscoelastic strain limit at each temperature was determined for each sample prior to any frequency sweep to ensure the response remained in the linear viscoelastic (LVE) range of the material.

The two material properties obtained in dynamic mechanical analysis testing are the complex shear modulus (G^*) and the phase angle (δ). The complex shear modulus is defined as the ratio of maximum stress to maximum strain and provides a measure of the total resistance to deformation due to repeated shearing force. It contains elastic and viscous components which are defined as the storage modulus G', and loss modulus G", respectively. These two parameters are related to the complex modulus and to each other through the phase angle, which is the time difference between the applied stress and strain responses. The phase angle is an indicator of the relative amount of recoverable and non-recoverable deformation *(32,40)*.

In addition to dynamic mechanical analysis of the binders, the Superpave rutting parameter G*/Sinδ at 64°C, Zero Shear Viscosity (ZSV), and Multiple Stress Creep Recovery (MSCR) were conducted to determine the performance of the binders at high in-service pavement temperatures. Bending Beam Rheometer (BBR) tests were conducted to assess low-temperature performance. All of the MSCR tests were conducted at 64°C which corresponds to the actual high pavement temperatures that the base binder was designed for. Both percent recovery and non-recoverable creep compliance (J_{nr}) parameters were obtained at two different stress levels (0.1 kPa and 3.2 kPa). The percent difference in non-recoverable creep compliance values were calculated and compared between the control and WMA binders to determine the effect of the WMA technologies on shear stress sensitivity. The BBR test was used to evaluate the binder properties at low pavement temperatures by measuring the creep stiffness (S) and creep rate (m-value). The BBR tests were conducted at -6°C and -12°C.

Baseline testing was conducted on artificially aged neat binders obtained during mix production. Aging was carried out according to standard Superpave test methods. The rolling thin film oven (RTFO) test was used for short-term aging and the pressurized aging vessel (PAV) test was used for long-term aging.

A summary of the experimental testing plan is shown in Figure 3.1.

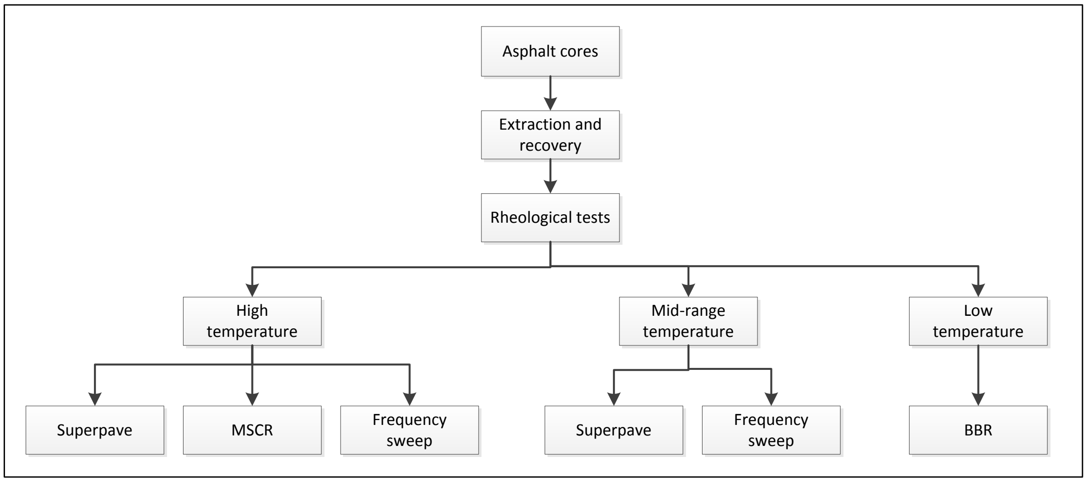


Figure 3.1: Schematic of experimental plan for binder testing.

4. TEST RESULTS

4.1 Zero Shear Viscosity and Viscosity-Shear Susceptibility

The zero shear viscosity (ZSV) values for all the binders tested in this study were calculated from the frequency sweep test results after they reached a plateau region. Since oxidation aging increases the association of polar compounds and, therefore, their interactions with asphaltenes as field aging time increases, the samples extracted and recovered from cores with different field aging times did not show a plateau region. Consequently, ZSV values were only calculated for the samples extracted and recovered from the loose mix at aging time zero. The results are summarized in Table 4.1 and Figure 4.1 and Figure 4.2. Higher ZSV values typically imply better rutting performance.

Table 4.1: Zero Shear Viscosity Results

Mix Type	Additive[1]	ZSV (Pa-S)
Phase 1 Conventional binder	Control	1,120
	CF	900
	OW	1,780
Phase 2 Rubberized binder Mix Design #1	Control	443
	MF1	435
	CS1	420
	CS2	246
Phase 2 Rubberized binder Mix Design #2	Control	578
	OW	630
	CF	450
	MF2	510
	CS3	770

[1] CF – Chemical Foam OW – Organic Wax MF – Mechanical Foam
CS – Chemical Surfactant

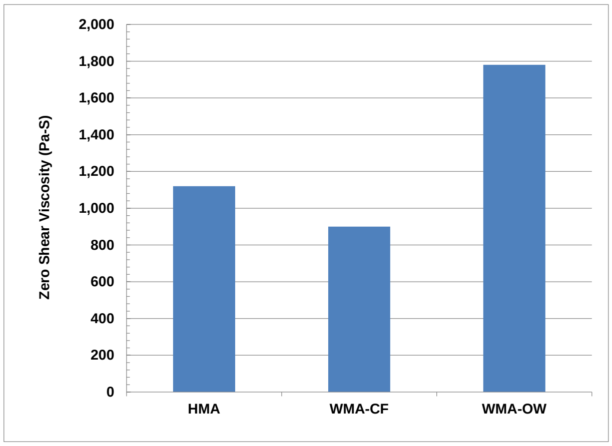


Figure 4.1: Conventional binder: ZSV at zero aging time (loose mix).

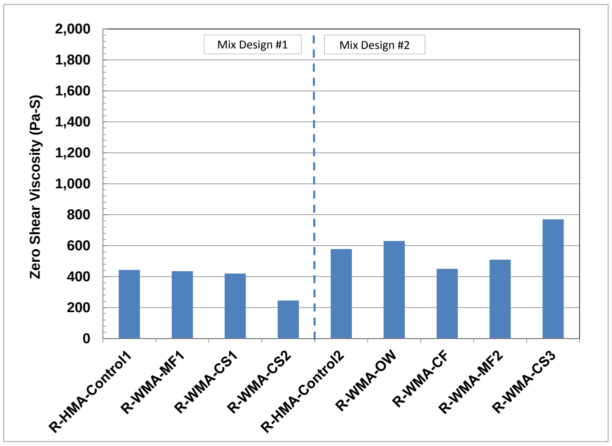


Figure 4.2: Rubberized binder: ZSV at zero aging time (loose mix).

The WMA technologies generally did not influence the results; however, production temperatures appeared to have some influence. Binders with the chemical foam, one of the chemical surfactants, and one of the mechanical foam technologies had lower ZSV values compared to the control binders, implying that the permanent deformation behavior at high temperatures could vary with respect to the zero shear viscosity criterion, depending on the production temperature. In both phases, higher ZSV values were obtained for the organic wax technology compared to the controls, implying that use of this additive could lead to increased rutting resistance performance. This was attributed to crystallization of the long carbon chains present in the binder at in-service temperatures associated with the use of this additive.

The results of the shear sensitivity testing of the binders using the previously defined viscosity-shear susceptibility (VSS) parameter are shown in Figure 4.3 through Figure 4.5. Higher VSS values typically imply better rutting performance.

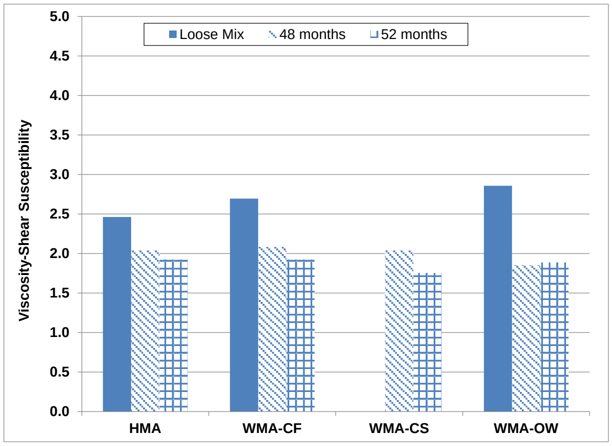


Figure 4.3: Conventional binder: VSS at different aging times.

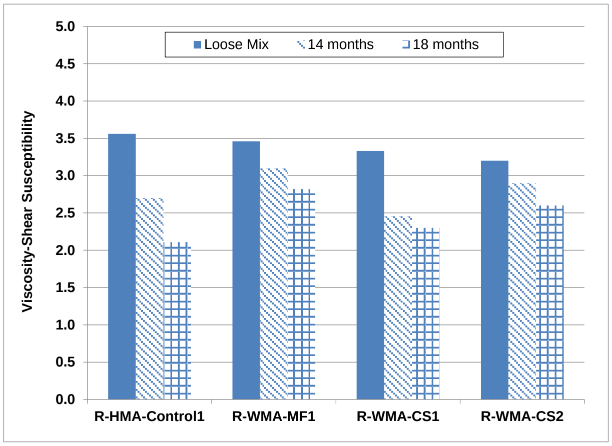


Figure 4.4: Rubberized binder, Mix Design #1: VSS at different aging times.

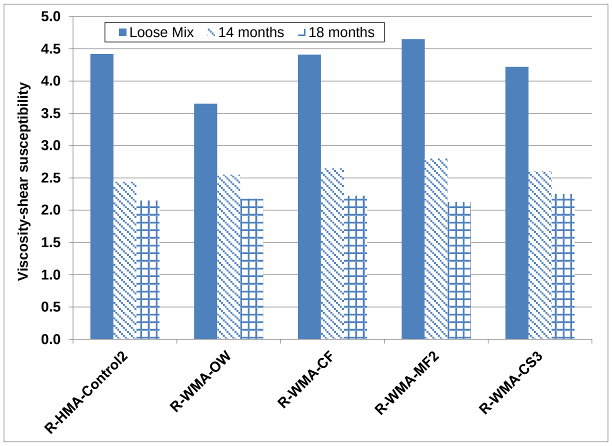


Figure 4.5: Rubberized binder, Mix Design #2: VSS at different aging times.

A lower value of the viscosity-shear indicates more sensitivity to shear. Results show that as oxidation aging of the binders increases the shear sensitivity also increases, which is expected because oxidation aging results in more brittle behavior. Compared to the control mixes, the addition of the WMA technologies and the reduced production and compaction temperatures did not significantly change the shear sensitivity of the binders at any of the testing intervals.

4.2 Multiple Stress Creep Recovery

4.2.1 Non-Recoverable Creep Compliance

Rutting resistance at high temperatures was assessed with the Multiple Stress Creep Recovery (MSCR) test, which was conducted at the highest temperature that the binder would typically experience during its service life. Figure 4.6 and Figure 4.7 summarize the results for binders recovered from samples collected during construction in both phases. The Superpave criterion for standard traffic loading (less than three million ESALs) is a maximum non-recoverable compliance value of 4.0 kPa. This criterion was established after a comparison of binder results from different projects in different states and during research studies done at the Federal Highway Administration Accelerated Loading Facility (*26*). However, subsequent studies have suggested that as traffic increases above three million ESALs the non-recoverable creep compliance value of the binder needs to be lowered, with maximum values ranging between 2.0 kPa and 1.0 kPa for traffic loadings between three and ten million ESALs, respectively.

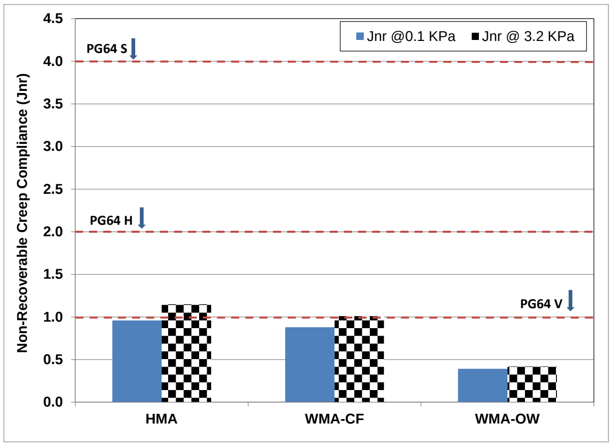


Figure 4.6: Conventional binder: non-recoverable creep compliance.

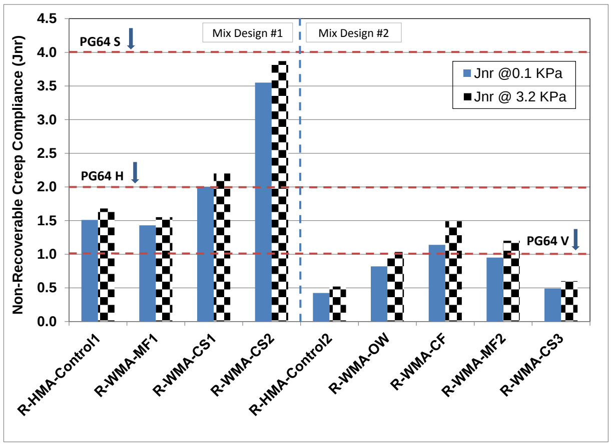


Figure 4.7: Rubberized binder: non-recoverable creep compliance.

(S = Standard Traffic [< 3 million ESALs], H = Heavy Traffic [3-6 million ESALs],
V = Very Heavy Traffic [6-10 million ESALs)

<u>Phase 1: Conventional Binder</u>

In the Phase 1 results, the addition of WMA additives coupled with reduced production and compaction temperatures appeared to have some influence on rutting behavior in terms of non-recoverable compliance values, with production temperatures having the bigger influence on the trend. The binder with organic wax had lower non-recoverable creep compliance values, which implies better rutting resistance compared to the control.

Phase 2: Rubberized Binders

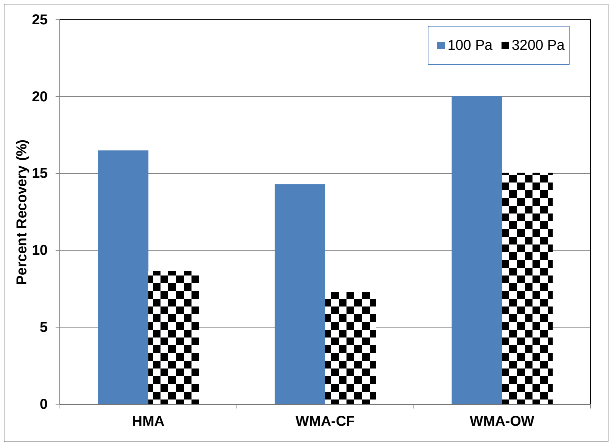
In the Phase 2 results, there was a notable difference between the two mix designs, which was attributed to the different base binders. The binder used in Mix Design #2 showed better properties with respect to percent recovery and non-recoverable creep compliance. The large difference in air-void contents between the two mix designs did not appear to influence the non-recoverable creep compliance. An opposite trend would have been expected if air-void content had influenced the results. (The effect of air void content is discussed in Section 2.3.2.) The results also show that the very low production temperature associated with some WMA technologies could potentially influence rutting behavior in the early stages of service, with some of the binders not meeting the requirements for very heavy traffic.

4.2.2 Percent Recovery

In addition to non-recoverable creep compliance, the MSCR test can be used to determine the amount of recovery (i.e., elasticity) in an asphalt binder during creep-recovery testing. Although this is not part of the Superpave specification, this parameter was used as an indicator to investigate the effect of WMA technologies and reduced production and compaction temperatures on elastic recovery with respect to durability in the field. Table 4.2 lists the percent recovery for the Phase 2 rubberized binders at two different stress levels (0.1 kPa and 3.2 kPa), and Figure 4.8 and Figure 4.9 show the percent recovery values for the binders from the two phases, respectively. Higher values typically imply better rutting performance. There was a significant difference between the two binder sources in Phase 2. The use of WMA technologies had some influence on the elastic recovery of the binders, with trends similar to those observed for the zero shear viscosity results.

Table 4.2: Rubberized Binder: Percent Recovery Comparison Between Mix Design #1 and #2

Source	Mix	Percent Recovery at 64°C	
		0.1 kPa	3.2 kPa
Mix Design #1	Control	9.5	4.7
	MF1	7.5	4.3
	CS1	6.4	2.7
	CS2	3.3	0.7
Mix Design #2	Control	38.7	27.6
	OW	27.0	14.2
	CF	25.2	10.8
	MF2	24.6	12.4
	CS3	32.3	23.6



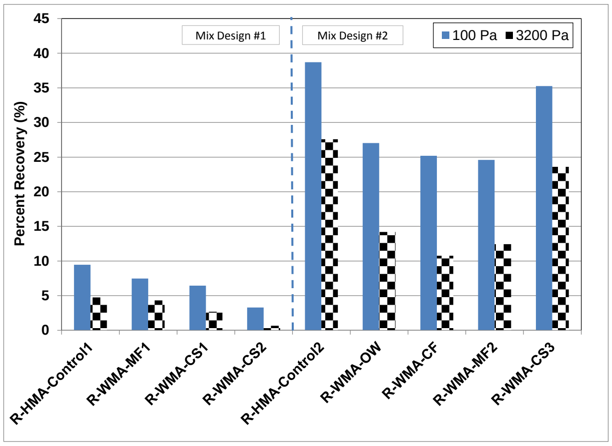


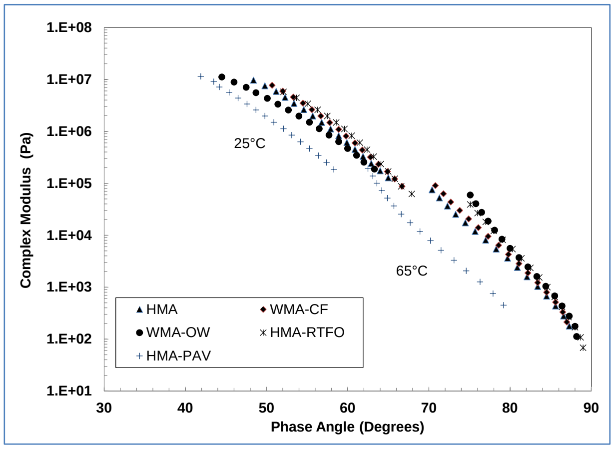
Figure 4.8: Conventional binder: percent recovery.

Figure 4.9: Rubberized binder: percent recovery.

4.3 Dynamic Mechanical Analysis

4.3.1 Phase 1: Conventional Binder

The results of complex modulus tests for the first phase of testing on conventional binders are summarized in Figure 4.10 through Figure 4.12 for three different ages. Results for the short- and long-term laboratory-aged base binders (HMA-RTFO and HMA-PAV, respectively) are also shown for reference. The control and WMA specimens both exhibited lower phase angles at the same complex modulus compared to the RTFO-aged specimen, indicating that more aging occurred during production and initial service in the field than was simulated in the laboratory. Similar results have been documented elsewhere in the United States, prompting debate about the appropriateness of the RTFO aging protocol. The topic is currently being investigated in a National Cooperative Highway Research Program study (NCHRP 9-52).



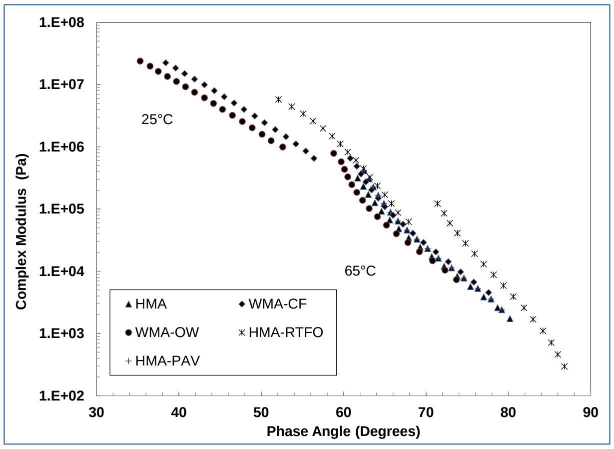


Figure 4.10: Phase 1: Black diagram for binders at zero aging (loose mix).

Figure 4.11: Phase 1: Black diagram for binders after 48 months.

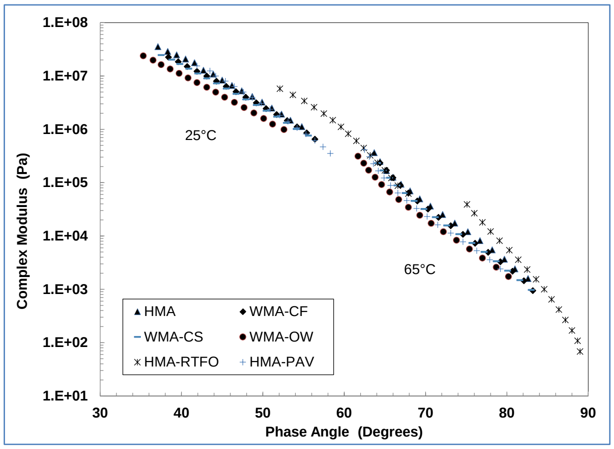


Figure 4.12: Phase 1: Black diagram for binders after 52 months.

Conditioning in a pressure aging vessel supposedly simulates the aging that occurs in the field after five to ten years. After 48 months of field aging, both the control and WMA binders showed aging-induced rheological behavior similar to those conditioned in the pressure aging vessel, which implies that field aging may occur faster than aging in the laboratory simulation. The regular shape of the complex modulus curves towards higher phase angles are similar for the control and WMA binders. The binders showed a shift towards lower phase angles for a given complex modulus after 48 and 52 months in the field, as expected, indicating an increase in the hardening of the binder.

Figure 4.13 through Figure 4.16 show the viscosity at 65°C and 25°C versus angular frequency for the Phase 1 binders at time zero and at 48 months. The addition of the WMA technologies did not result in any significant frequency sensitivity, although the organic wax generally had a higher complex viscosity corresponding to similar trends in other tests. At 25°C, both the control and WMA binders had higher shear susceptibility compared to the 65°C results, as expected, indicating that the lower production and placement temperatures did not appear to change the shear susceptibility of the binders at intermediate temperatures after field aging.

4.3.2 Phase 2: Rubberized Binders

Plots of the complex modulus of the control and WMA binders for the two mix designs are shown in Figure 4.17 through Figure 4.19 (Mix Design #1) and Figure 4.20 through Figure 4.22 (Mix Design #2). There was very little difference in performance at both testing temperatures for the binders used in Mix Design #1, although one of the chemical surfactants (CS1) showed a tendency toward a lower complex modulus over time. This was attributed to the considerably lower production and placement temperatures of this mix compared to the other mixes.

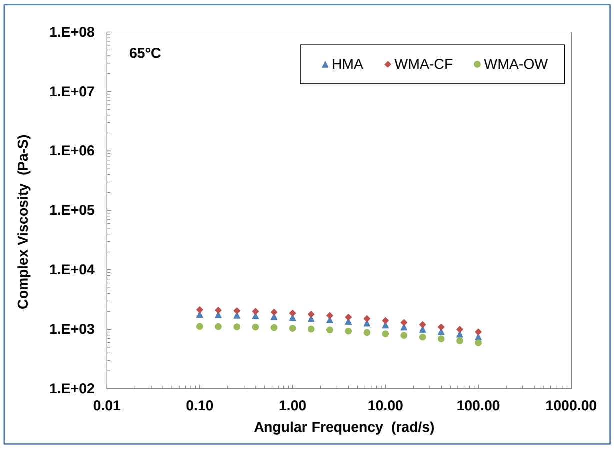


Figure 4.13: Phase 1: Viscosity at 65°C at zero aging time (loose mix).

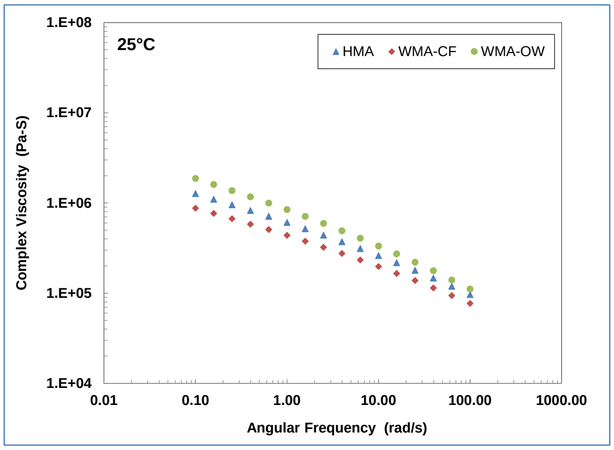


Figure 4.14: Phase 1: Viscosity at 25°C at zero aging time (loose mix).

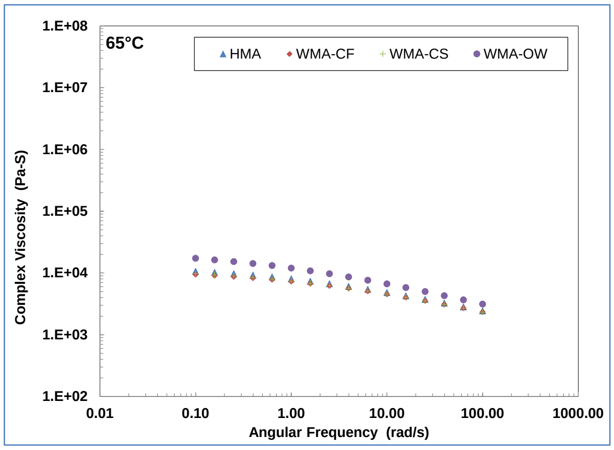


Figure 4.15: Phase 1: Viscosity at 65°C after 48 months.

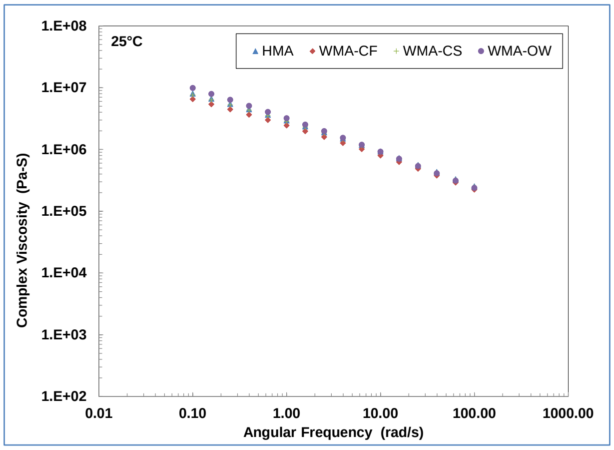


Figure 4.16: Phase 1: Viscosity at 25°C after 48 months.

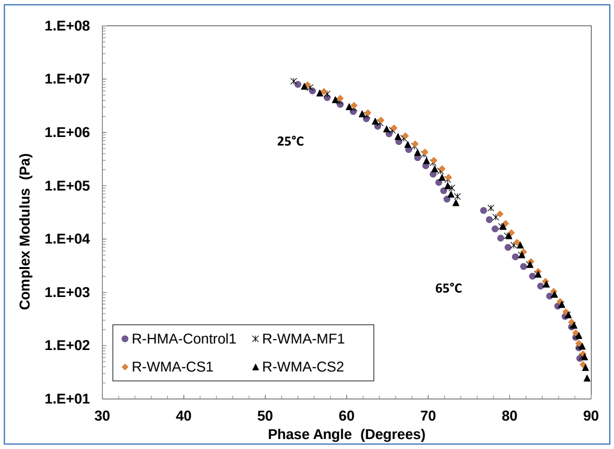


Figure 4.17: Phase 2, Mix Design #1: Black diagram for binders at zero aging (loose mix).

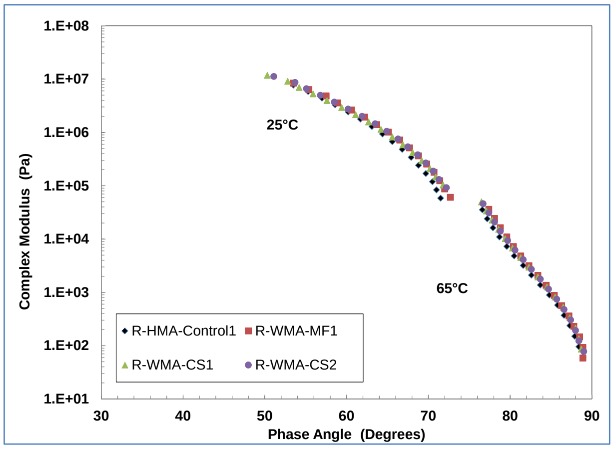


Figure 4.18: Phase 2, Mix Design #1: Black diagram for binders after 14 months.

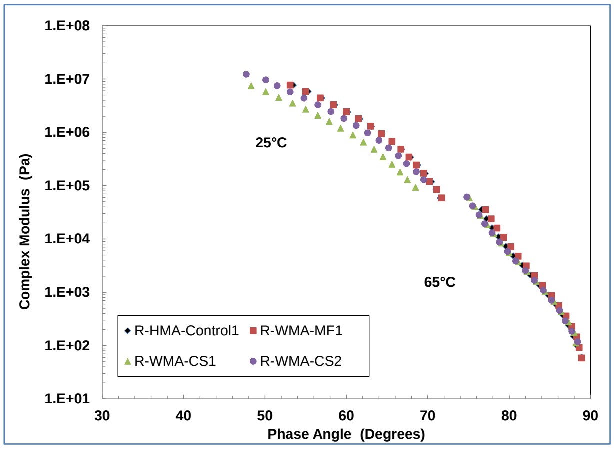


Figure 4.19: Phase 2, Mix Design #1: Black diagram for binders after 18 months.

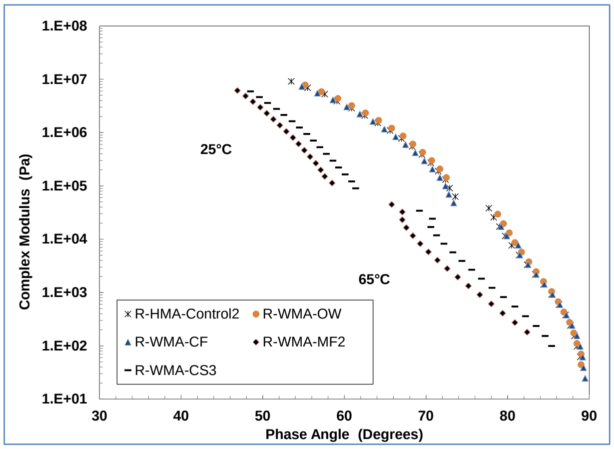


Figure 4.20: Phase 2, Mix Design #2: Black diagram for binders at zero aging (loose mix).

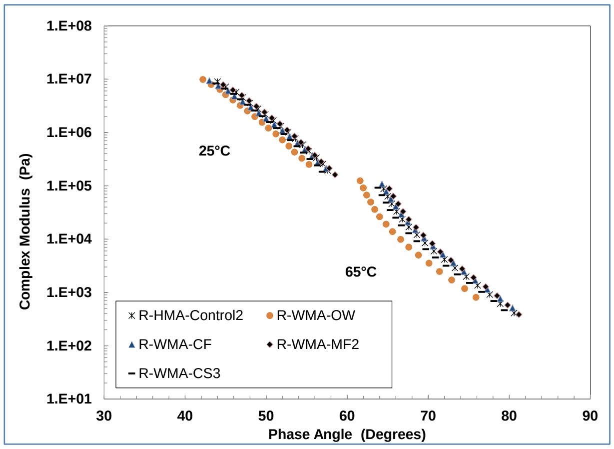


Figure 4.21: Phase 2, Mix Design #2: Black diagram for binders after 14 months.

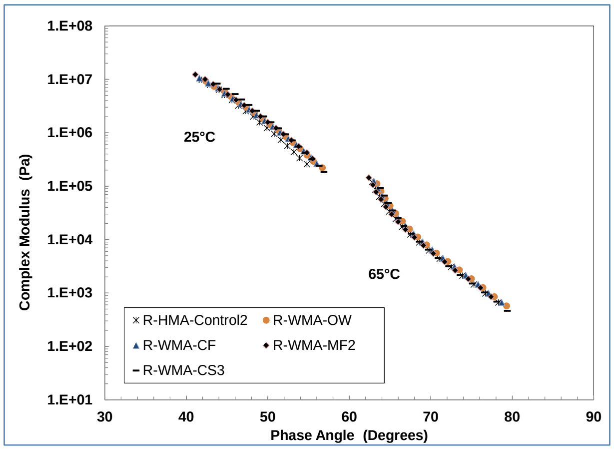


Figure 4.22: Phase 2, Mix Design #2: Black diagram for binders after 18 months.

The binders used in Mix Design #2 showed considerable variation in phase angle at the same complex modulus at zero aging. However, after 18 months of field aging, the results were very similar. The binders with the greatest variation compared to the control binder (MF2 and CS3) had the lowest placement temperatures, which probably influenced the initial performance in a similar manner to the CS1 binder in Mix Design #1.

Figure 4.23 through Figure 4.30 are plots of viscosity versus frequency for the rubberized binders of both mix designs. All of the binders showed similar trends, but with more variation at zero aging compared to the tests conducted after 14 months. This was again attributed to the different production and placement temperatures, which appeared to only influence very early performance.

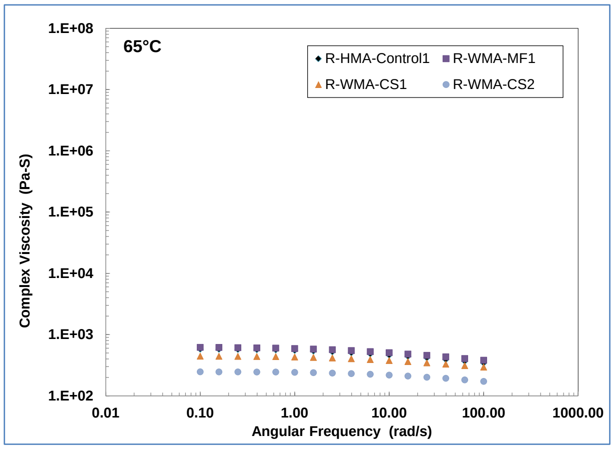


Figure 4.23: Phase 2, Mix Design #1: Viscosity at 65°C at zero aging (loose mix).

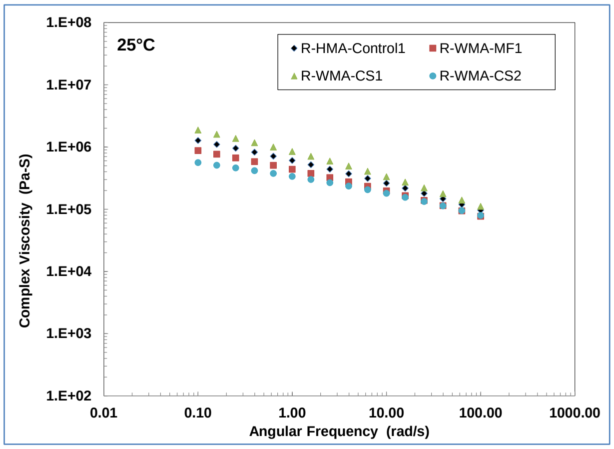


Figure 4.24: Phase 2, Mix Design #1: Viscosity at 25°C at zero aging (loose mix).

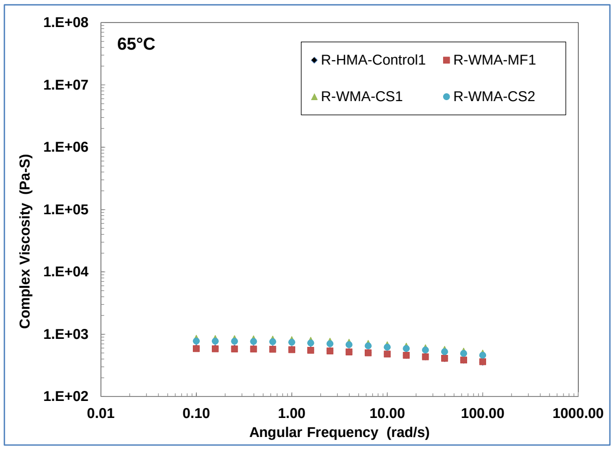


Figure 4.25: Phase 2, Mix Design #1: Viscosity at 65°C after 14 months.

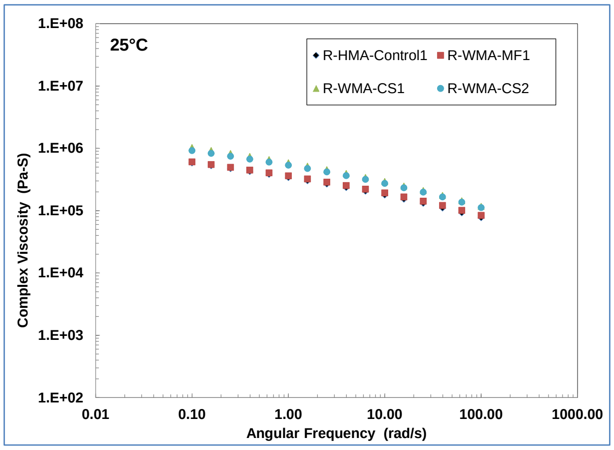


Figure 4.26: Phase 2, Mix Design #1: Viscosity at 25°C after 14 months.

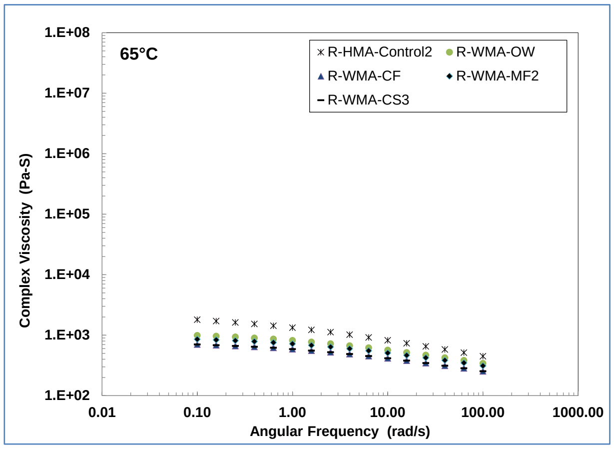


Figure 4.27: Phase 2, Mix Design #2: Viscosity at 65°C at zero aging.

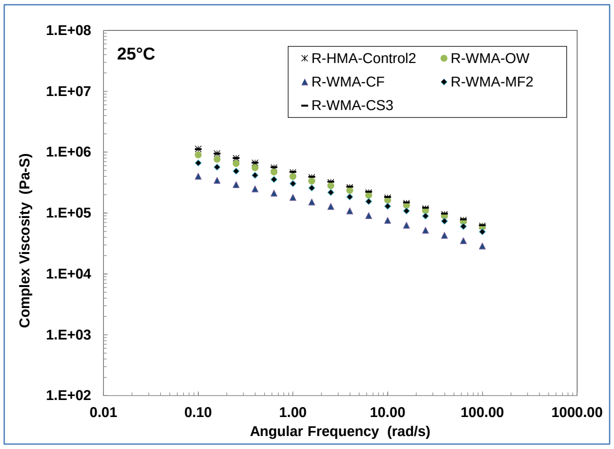


Figure 4.28: Phase 2, Mix Design #2: Viscosity at 25°C at zero aging.

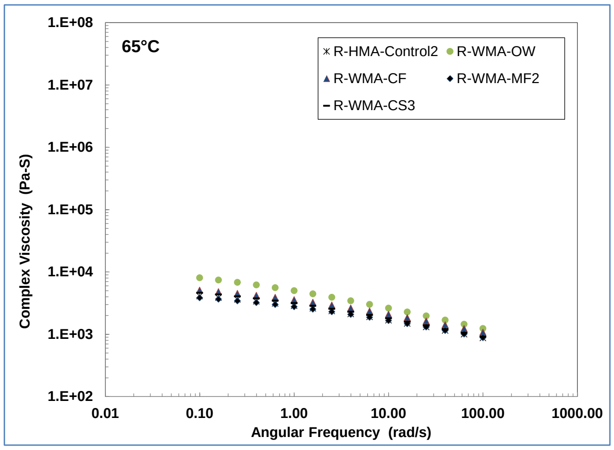


Figure 4.29: Phase 2, Mix Design #2: Viscosity at 65°C after 14 months.

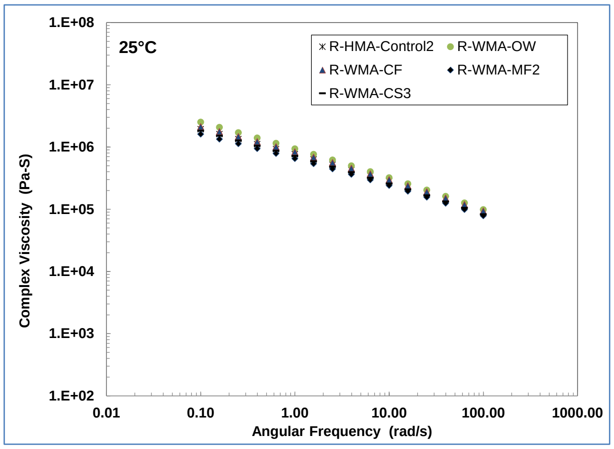


Figure 4.30: Phase 2, Mix Design #2: Viscosity at 25°C after 14 months.

4.4 Oxidation Kinetics Analysis

4.4.1 Viscosity-Phase Angle Relationship

The viscosity of aged binders is a useful parameter for characterizing age hardening, and the phase angle is an important parameter for characterizing flow properties. A certain level of viscous flow is desirable in an oxidized binder at low temperatures to provide for the relaxation of stress. In this research the level of age hardening is of concern when comparing aged control binders and aged warm-mix binders. The lower the phase angle at the same viscosity, the more susceptible the asphalt binder will be to thermal and fatigue cracking at low temperatures. The relationship between viscosity and phase angle therefore provides an indicator for characterizing warm mix-treated asphalt binders after long-term oxidative aging in the field. Figure 4.31 and Figure 4.32 plot the viscosity-phase angle relationship and offer insight into the binders' properties (for Phase 1 and Phase 2, respectively) as they relate to aging kinetics. The figures clearly show the reduction in phase angle as the binder ages.

Phase 1: Conventional Binder

Figure 4.33 shows the complex viscosity at 65°C versus the phase angle at 25°C for the Phase 1 binders at different field ages. As age increases, the complex viscosity increases and the phase angle decreases at an exponential rate, with a higher phase angle at the same viscosity indicating better flow properties. All the binders tested exhibited similar trends with field aging. The binder with organic wax (OW) showed slightly lower phase angles at the same oxidation viscosity, indicating less flow than the control, as expected. The complex viscosity increases and the phase angle decreases at an exponential rate as aging time increases, with a higher phase angle at the same viscosity indicating better flow properties. All binders exhibited similar trends with field aging. The binder with organic wax (OW) showed slightly lower phase angles at the same oxidation viscosity indicating less flow behavior compared to the control, as expected.

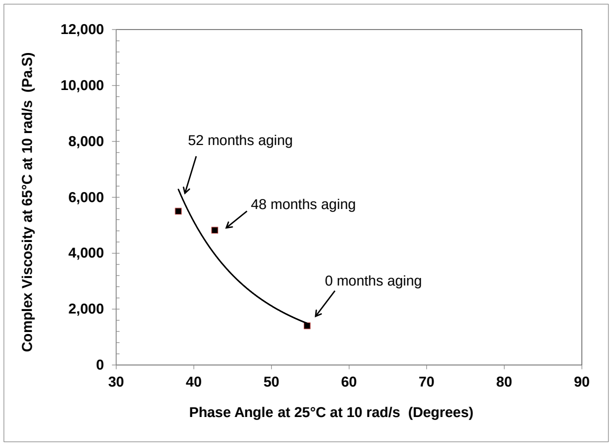


Figure 4.31: Phase 1: Viscosity-phase angle relationship with aging kinetics.

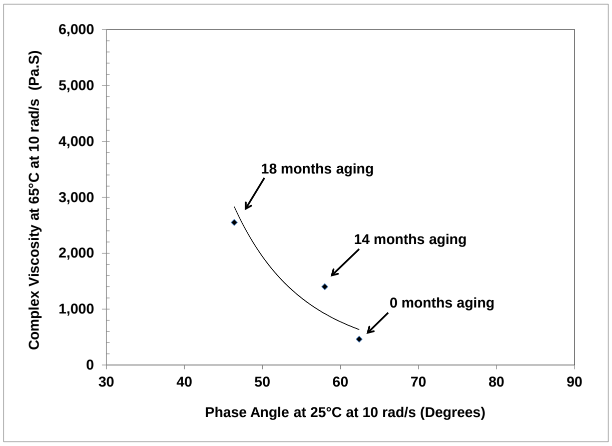


Figure 4.32: Phase 2: Viscosity-phase angle relationship with aging kinetics.

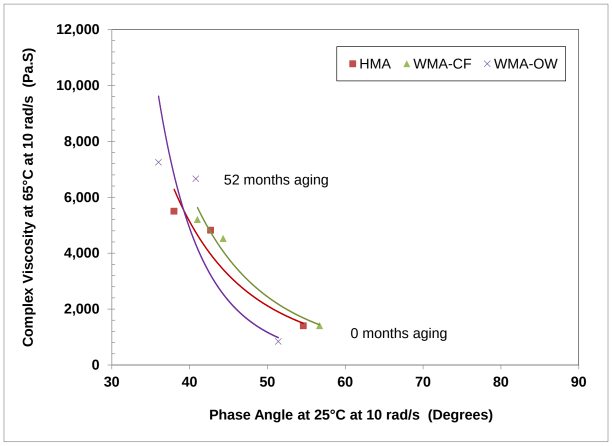


Figure 4.33: Phase 1: Effect of WMA additives on viscosity-phase angle relationship.

Phase 2: Rubberized Binder

Figure 4.34 shows the viscosity-phase angle relationship for the two control binders. The two binders showed similar trends over time; however, the binder used in Mix Design #1 had a lower phase angle at the same oxidation viscosity than the Mix Design #2 binder, indicating that the Mix Design #1 binder had lower flow properties after longer-term oxidative aging. Figure 4.35 and Figure 4.36 show the relationships for all the binders in the two mix designs. The binders in Mix Design #1 all had similar properties at zero aging. The small differences between the control and the mechanical foaming technology and the two chemical surfactants (CS1 and CS2) were attributed to the different production temperatures. Over time, the control and mechanical foam binders showed a significant increase in complex modulus compared to the binders with the chemical surfactants. The mechanical foam binder also showed a higher phase angle at the same oxidation viscosity compared to the control, indicating higher viscous flow. It is not clear why this occurred, but may have resulted from the influence of water that was added to the binder. This was not investigated. The chemical surfactants showed very little change in complex modulus over time, indicating that some effect of the lower production and placement temperatures may still have been evident.

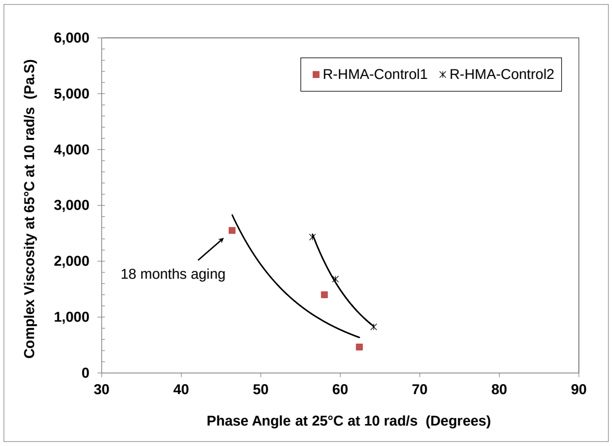


Figure 4.34: Phase 2: Effect of mix design on viscosity-phase angle relationship.

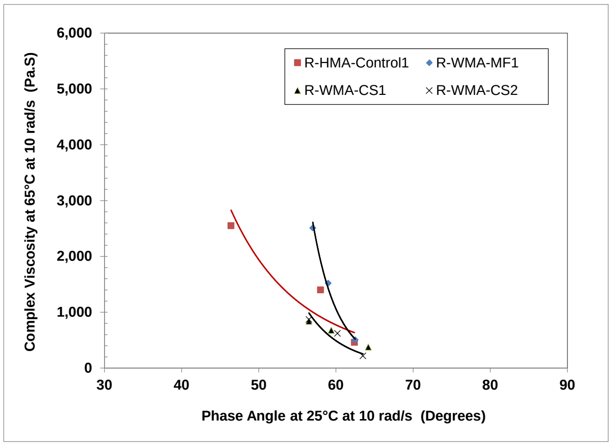


Figure 4.35: Phase 2, Mix Design #1: Effect of WMA additives on viscosity-phase angle relationship.

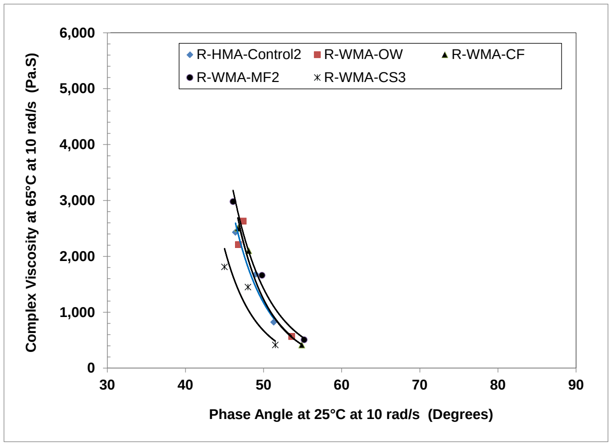


Figure 4.36: Phase 2, Mix Design #2: Effect of WMA additives on viscosity-phase angle relationship.

The Mix Design #2 binders showed very similar trends and properties over time, with the chemical surfactant (CS3) having a slightly lower phase angle compared to the other binders. This was attributed to this mix having the lowest production and placement temperatures.

4.4.2 Short-Term Oxidative Aging

The impact of lower production temperatures on the short-term oxidative aging that occurs at the plant was investigated by comparing the properties of the Phase 1 control and WMA binders to the properties of the original base binder (HMA Original) and the RTFO-aged base binder (the rolling thin film oven test supposedly replicates the oxidative aging that occurs during production, construction and the first one to two years in the field). The results are shown in Figure 4.37. The field sampled binders showed similar performance to the RTFO-aged base binder, with all binders exhibiting lower phase angles at the same complex modulus compared to the unaged original base binder. This implies that the laboratory simulation provided a reasonable indication of field performance, and contradicts the results discussed in Section 4.3.1. Given that this study only considered a very limited dataset, no conclusions can be drawn until more data (asphalt binder sources and asphalt plant variables) are collected and an investigation is undertaken to understand these observations.

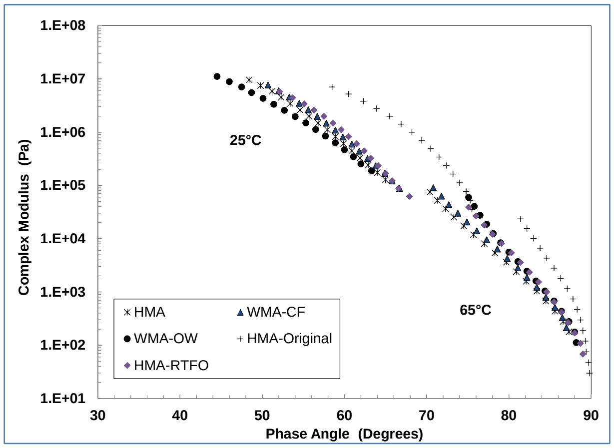


Figure 4.37: Phase 1: Comparison of original, RTFO-aged, and recovered binders.

4.4.3 Oxidative Susceptibility Aging Rate

The *oxidative susceptibility aging rate,* defined as the slope of log viscosity-aging time, was used to compare the aging susceptibility of the WMA binders with that of the control binders. A higher value indicates less susceptibility. The results are listed in Table 4.3. In Phase 1, the first assessment period (zero to 48 months) covered four summers. In Phase 2, only one summer was assessed during the first assessment period. The second period covered one additional summer for each mix. The average maximum monthly temperatures for the two test section locations are shown in Figure 4.38 for reference purposes.

Phase 1: Conventional Binder

The results for Phase 1 binders are shown in Figure 4.39. The control and chemical foam (CF) binders showed similar aging susceptibility rates. The organic wax (OW) binder had a higher rate of aging susceptibility, which is consistent with other observations throughout the study.

Table 4.3: Long-Term Oxidative Aging Susceptibility Analysis

Phase	Mix Type	Phase 1[1]		Phase 2[1]	
		0 – 48 Months	49 – 54 Months	0 – 14 Months	15 – 18 Months
Phase 1	HMA	40.16	35.16	-	-
	WMA-CF	39.15	33.54	-	-
	WMA-OW	23.69	19.56	-	-
Phase 2 Mix Design #1	R-HMA Control	-	-	11.57	10.15
	R-WMA-MF1	-	-	11.58	10.25
	R-WMA-CS1	-	-	22.77	20.57
	R-WMA-CS2	-	-	13.08	12.11
Phase 2 Mix Design #2	R-HMA Control	-	-	17.30	15.85
	R-WMA-OW	-	-	10.55	9.85
	R-WMA-CF	-	-	10.00	9.08
	R-WMA-MF2	-	-	10.12	9.38
	R-WMA-CS3	-	-	11.93	10.85

[1] Higher value indicates less susceptibility

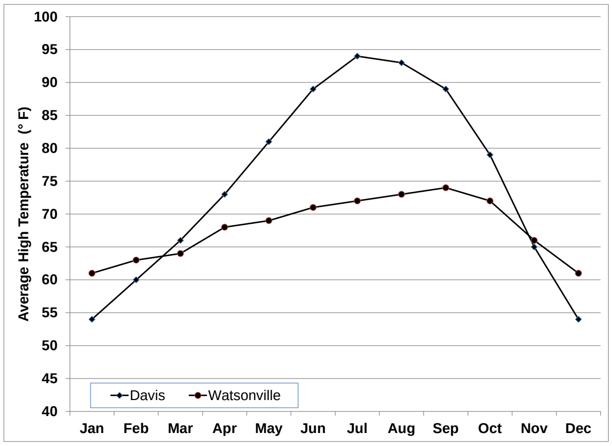


Figure 4.38: Average high temperatures for Watsonville (Ph.1) and Davis (Ph.2).

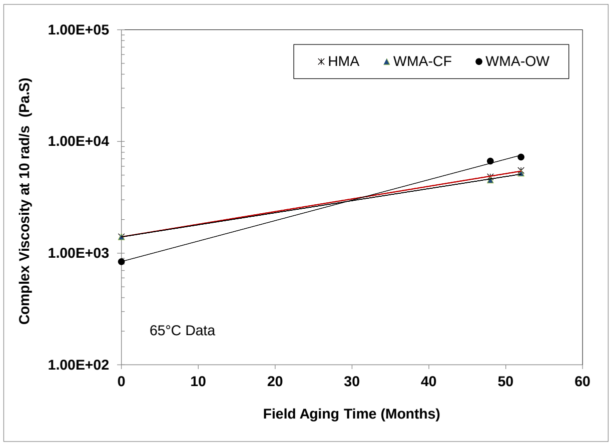


Figure 4.39: Phase 1: Effect of WMA additives on binder aging kinetics.

Phase 2: Rubberized Binders

The rates of oxidation kinetics of the different Phase 2 control binders are shown in Figure 4.40. They showed different kinetics at zero aging but very similar kinetics at the end of the assessment period. Results for all the binders in the two mix designs are shown in Figure 4.41 and Figure 4.42, respectively. Less change was noted in the Phase 2 results compared to those in Phase 1 due to the significantly shorter aging period. Rates appear to be linked to production and placement temperatures for the Mix Design #1 binders, but not for the Mix Design #2 binders, where the control mix showed the lowest susceptibility. A binder with one of the chemical surfactants (CS1) showed considerably different aging susceptibility compared to the other binders, and this was attributed to the much lower production and placement temperatures for this mix. The chemical surfactants used in the Phase 2 Mix Design #1 binders had a different kinetic trend than the control and mechanical foam, and this too was attributed to the lower production and placement temperatures. The binders in Mix Design #2 showed similar temperature-related trends. As expected, the complex viscosity of the binder with organic wax (OW) showed a larger change compared to the other binders.

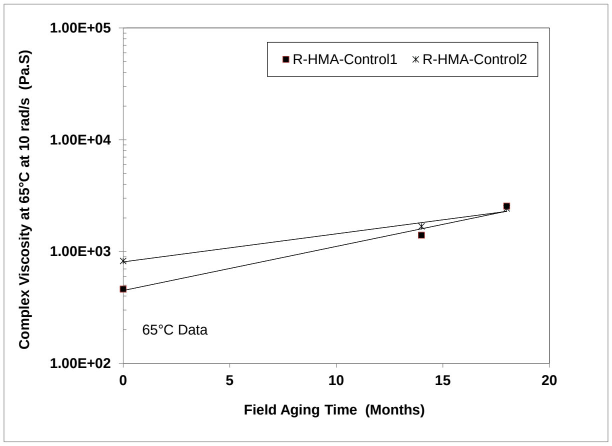


Figure 4.40: Phase 2: Effect of mix design on binder aging kinetics.

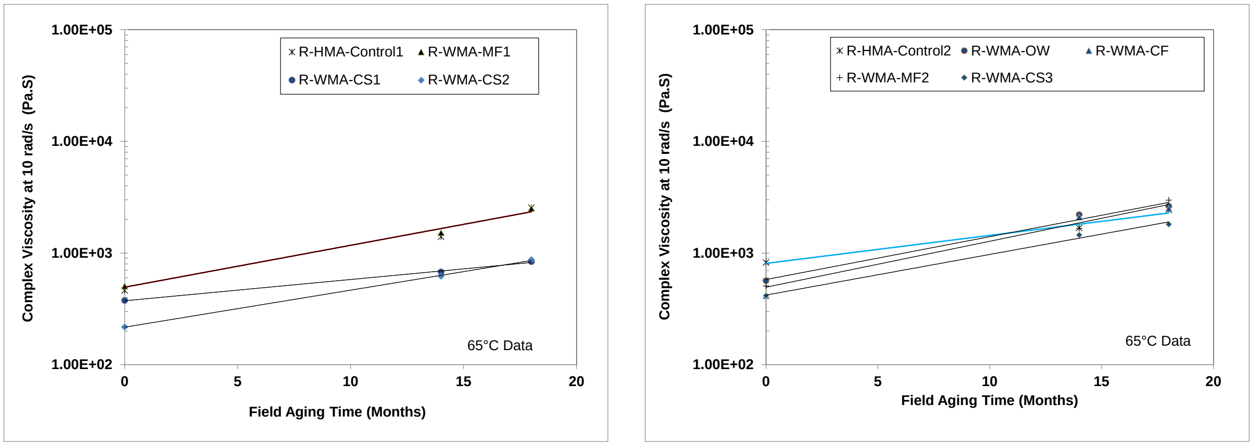


Figure 4.41: Phase 2, Mix Design #1: Effect of WMA additives on binder aging kinetics.

Figure 4.42: Phase 2, Mix Design #2: Effect of WMA additives on binder aging kinetics.

4.5 Effect of Air Voids

As discussed in Section 2.3.2, research has shown that air-void contents of five percent and higher can lead to oxygen being more freely available to react with the asphalt binder in a compacted layer, which can result in oxidative age hardening. To understand the effect of air voids on the binders assessed in this study, the accessible air-voids content in cores was analyzed using X-ray computed tomography (CT). The results were analyzed using the methodology developed by Coleri *(41)*. Figure 4.43 and Figure 4.44 show the distribution of air voids with depth for the two Phase 2 control mixes, and for all the Mix Design #1 mixes. The air void distribution was not uniform with pavement depth but was distributed in an approximate "C" shape for all mixes, indicating higher air-void contents at the top and bottom of the layer compared to the middle. This was attributed to a temperature differential during compaction (colder at the bottom of the lift due to contact with the cold surface, and more rapid cooling at the top due to exposure to cool ambient temperatures). This non-uniform air void distribution could result in a variation in the micro-scale binder properties over the layer depth.

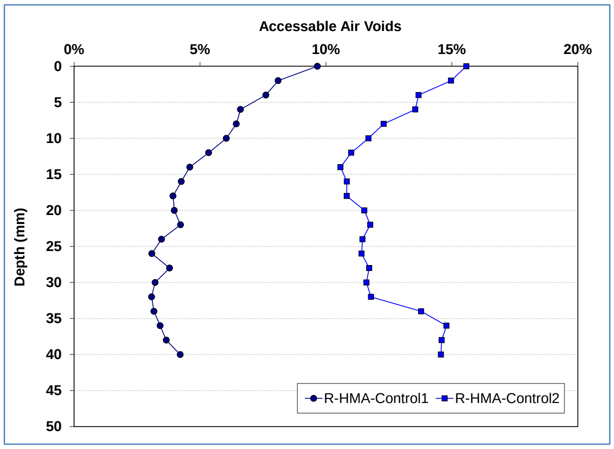


Figure 4.43: Phase 2: Accessible air-void content versus depth for control mixes.

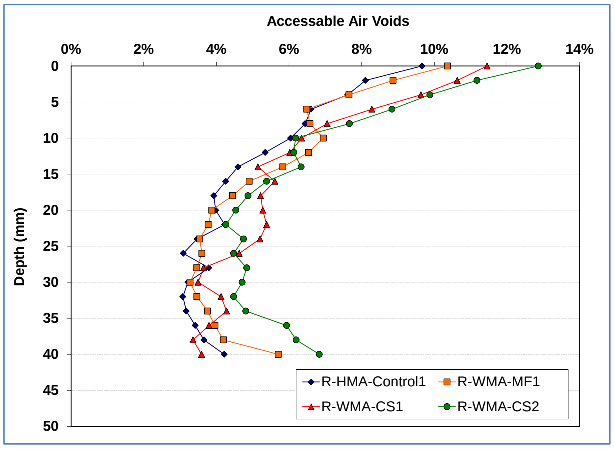


Figure 4.44: Phase 2, Mix Design #1: Accessible air-void content versus depth.

The air-void contents of both control mixes were considerably higher than the five percent level (top 15 mm in Mix Design #1 and full-depth for Mix Design #2). Consequently, the accessible air-void contents were sufficiently high at the surface for oxygen to freely enter and react with the polar compounds in the asphalt binder. However, based on the results discussed in the preceding sections, this did not appear to influence the oxidative aging behavior of the binders in the short-term.

4.6 Effect of Asphalt Film Thickness

The effect of asphalt film thickness on oxidative aging behavior was assessed by calculating the *equivalent film thickness* using the following equation:

$$\text{Percent Asphalt} = \text{Surface Area (SA)} \times \text{Equivalent Film Thickness} \times \gamma\text{asphalt} \times 100 \qquad (4.1)$$

The results of these calculations are listed in Table 4.4. The asphalt film thickness for the Phase 1 samples varied between 2.1 mm and 2.5 mm and between 3.7 mm and 4.3 mm for the Phase 2 samples. According to the literature *(12, 23),* film thickness could affect the binder aging characteristics if the difference between the mixes was greater than or equal to 2 mm. Although the film thickness was dependent on the binder content of the individual mixes, as expected, the 2 mm limit was not exceeded. Based on the results discussed in the preceding sections, film thickness did not appear to influence the oxidative aging behavior of the binders.

Table 4.4: Asphalt Film Thickness for all Mixes Studied

Phase	Mix Type	Binder Content (%)	Film Thickness (mm)
Phase 1	HMA	5.4	2.50
	WMA-CF	5.3	2.45
	WMA-CS1	5.4	2.50
	WMA-OW	4.7	2.05
Phase 2 Mix Design #1	R-HMA-Control1	7.7	3.65
	R-WMA-MF1	7.9	3.75
	R-WMA-CS1	7.7	3.65
	R-WMA-CS2	7.7	3.65
Phase 2 Mix Design #2	R-HMA-Control2	7.7	3.65
	R-WMA-OW	8.0	3.85
	R-WMA-CF	7.6	3.60
	R-WMA-MF2	8.4	3.95
	R-WMA-CS3	9.0	4.25

4.7 Effect of Base Asphalt Binder

The results discussed so far have indicated a difference in the performance of the two rubberized binder sources used in Phase 2. The unmodified binders used in both mix designs were therefore compared to better understand their effect on the observed rheological behavior. Figure 4.45 shows the complex modulus–phase angle relationship for both the unmodified (without rubber) and modified (with rubber) binders. For given complex modulus values, the unmodified and the modified base binders used in Mix Design #2 showed a lower range of phase angles both at 25°C and 65°C, indicating more elastic behavior compared to the binders used in Mix Design #1. The viscosity and angular frequency relationships at 65°C and 25°C are shown in Figure 4.46 and Figure 4.47, respectively, and clearly illustrate how the addition of crumb rubber decreases the range of phase angles (i.e., increases the elastic behavior) at given complex modulus values. These results are consistent with those obtained from the multiple stress creep recovery and zero shear viscosity results discussed in previous sections.

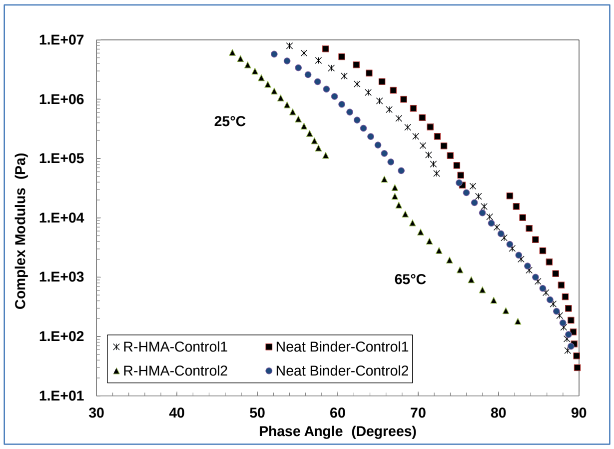


Figure 4.45: Phase 2: Complex modulus–phase angle relationship for neat and modified binders.

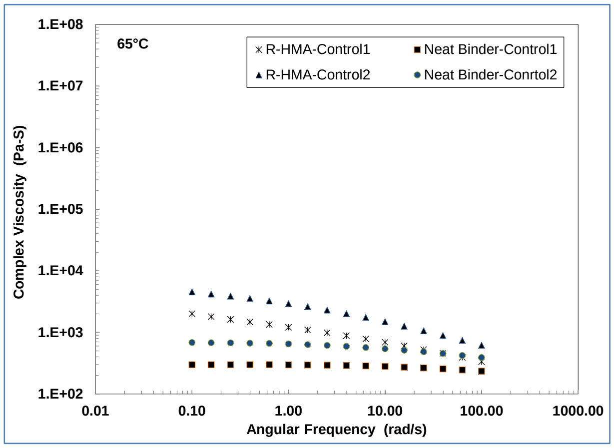


Figure 4.46: Phase 2: Viscosity–frequency relationship at 65°C for neat and modified binders.

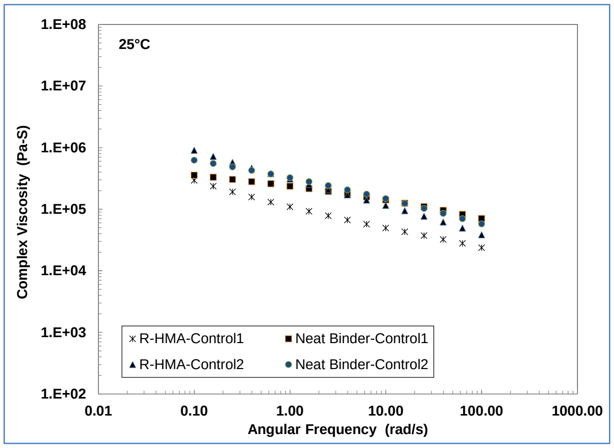


Figure 4.47: Phase 2: Viscosity–frequency relationship at 25°C for neat and modified binders.

4.8 Comparison of Binder Results with HVS and Hamburg Wheel-Track Test Results

To better understand the relationship between the binder test results and actual field performance, the Phase 2 binder test results were compared to rut-depth results measured on Heavy Vehicle Simulator (HVS) test tracks and to laboratory Hamburg Wheel-Track Test results measured on cores removed from the test track.

The rut depth measured after 160,000 HVS load repetitions was used for the analysis. This was the point at which the load was increased from 40 kN to 60 kN (9,000 lbs to 13,500 lbs) if the test section had not reached the experiment terminal rut of 13 mm. HVS results were compared with the non-recoverable creep compliance values obtained from the multiple stress creep recovery test results on the binders recovered from loose mix collected during construction. Figure 4.48 shows an R^2 correlation of 0.6

between the current Superpave criteria for rutting performance of asphalt binders (G*/Sinδ) and the actual rutting that occurred on the test sections after HVS testing. The non-recoverable compliance values measured at 3.2 kPa are plotted versus HVS rut depth for all the tests in Figure 4.49, and for the individual mix designs in Figure 4.50 and Figure 4.51. A stronger correlation (R^2 of 0.76) was observed between non-recoverable compliance values and rut depth in terms of the rutting resistance performance of the binders when all the mixes were compared; however, when the mixes were compared individually, a considerable difference was noted. The results for Mix Design #1 had a relatively strong correlation (R^2 of 0.83), while those for Mix Design #2 had a weak correlation (R^2 of 0.25), indicating that other factors (e.g., production temperature, air-void content, binder content) in addition to the binder properties influenced rutting performance.

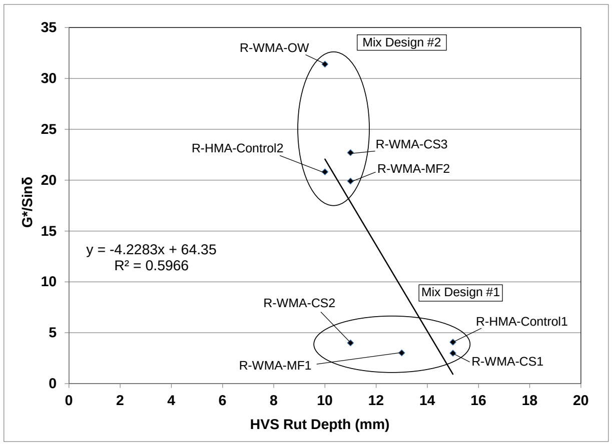


Figure 4.48: Phase 2: G*/sinδ at 64°C versus HVS rut depth.

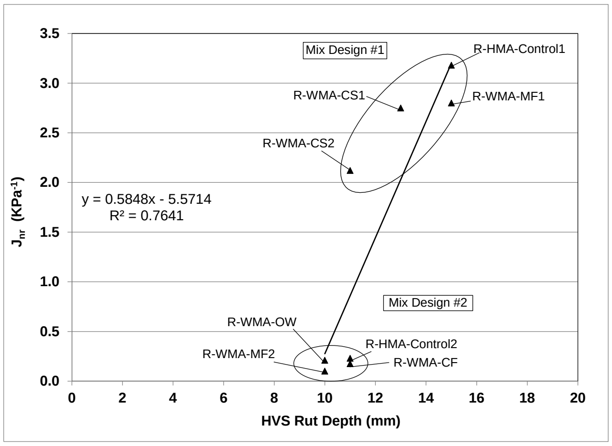


Figure 4.49: Phase 2: Non-recoverable compliance versus HVS rut depth.

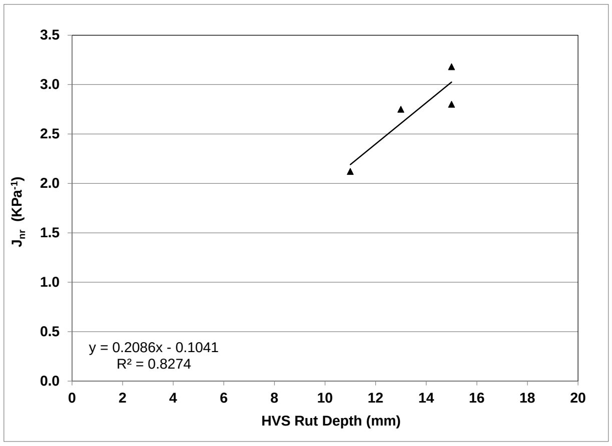


Figure 4.50: Phase 2, Mix Design #1: Non-recoverable compliance versus HVS rut depth.

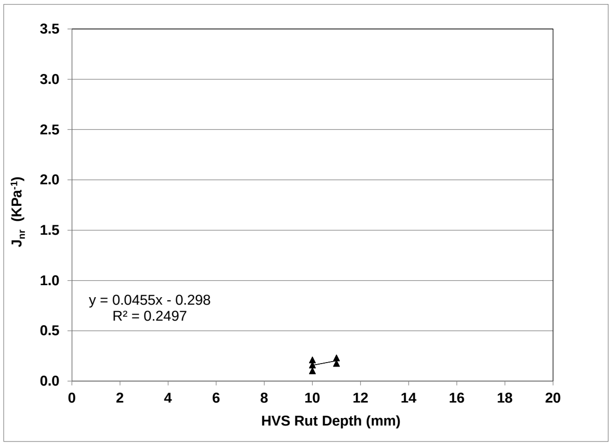


Figure 4.51: Phase 2, Mix Design #2: Non-recoverable compliance versus HVS rut depth.

The non-recoverable compliance values measured at 3.2 kPa are plotted versus Hamburg Wheel-Track tests for Mix Design #2 in Figure 4.52. There was no significant correlation (R^2 of 0.65) between non-recoverable compliance and the Hamburg Wheel-Track Test results, indicating that other factors in addition to binder properties also influenced these results.

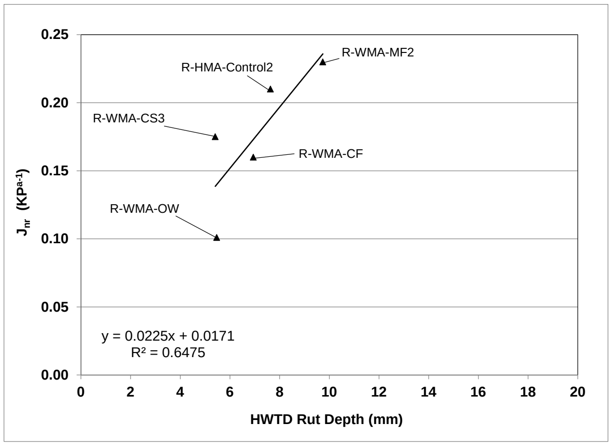


Figure 4.52: Phase 2, Mix Design #2: Non-recoverable compliance versus HWTT rut depth.

Comparisons of the zero shear viscosity and HVS rut depths for all the Phase 2 mixes are plotted in Figure 4.53. Figure 4.54 and Figure 4.55 show the relationships for the two different mix designs, respectively. The results were consistent with those discussed above for non-recoverable compliance, with a weak correlation (R^2 of 0.11) between all the mixes and HVS rut depth, and between the Mix Design #2 results and HVS rut depth (R^2 of 0.15). A stronger correlation (R^2 of 0.73) was observed between the Mix Design #1 results and HVS rut depth.

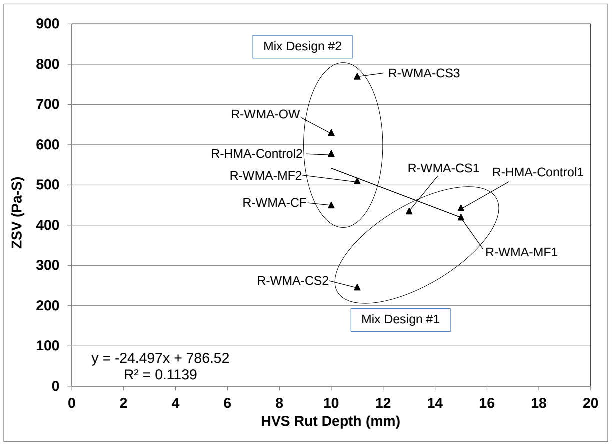
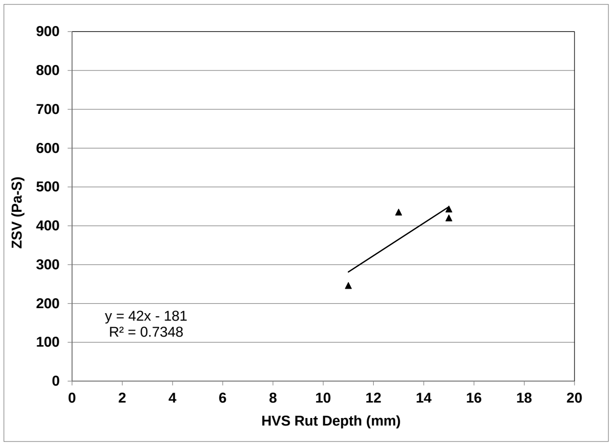


Figure 4.53: Phase 2: ZSV versus HVS rut depth for all mixes.



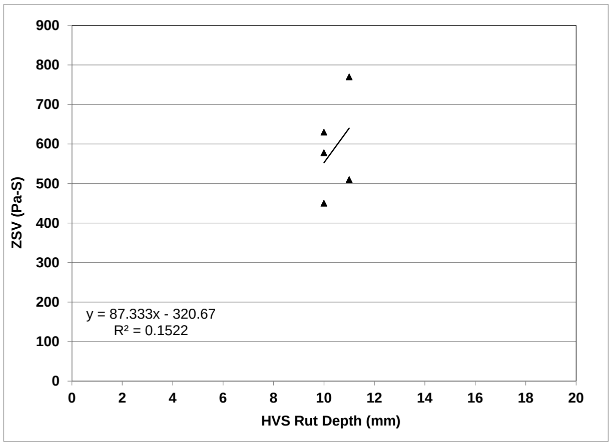


Figure 4.54: Phase 2, Mix Design #1: ZSV versus HVS rut depth.

Figure 4.55: Phase 2, Mix Design #2: ZSV versus HVS rut depth.

4.9 Thermal Cracking Properties

The Bending Beam Rheometer (BBR) was used to determine the rheological response at -6°C, which corresponds to the -16°C critical cracking temperature in the field using time/temperature superposition. The creep stiffness and the slope of log creep stiffness versus log time curve (m-value) at 60 seconds were of interest in this study *(42)*. Test results after 48 and 14 months of aging for the two phases respectively, are listed in Table 4.5 and shown in Figure 4.56 through Figure 4.59. Error bars in the figures represent the 95 percent confidence interval (lower stiffness values equate to better thermal cracking performance). The m-value is a measure of the rate at which the binder relaxes from imposed stresses, with higher m-values typically corresponding to lower thermal cracking. Binders should have an m-value greater than 0.30 and a stiffness value of less than 300 MPa at the design temperature according to the Superpave specification. All the binders were within these criteria and the result trends were generally consistent with the other test results discussed earlier. No thermal cracking was observed on any of the test sections.

Performance of the Phase 2 binders was notably different for the two mix designs, as expected, with binders in Mix Design #1 showing higher stiffness values compared to those in Mix Design #2. Production and placement temperatures appear to have had less of an effect on cracking performance tests than they did on the rutting performance tests.

Pressure-aged samples of the base binders were also tested for reference. These results compared reasonably well with the control for the Phase 1 binders but they compared poorly with the Phase 2 binders. This was attributed to the limited field aging of the Phase 2 binders (14 months compared to 48 months in Phase 1) given that pressure aging is associated with longer-term performance. Rolling thin film oven aging may have been more representative in this instance.

Table 4.5: Bending Beam Rheometer Test Results

Phase	Mix Type	Stiffness (MPa)	m-value	Age (months)
Phase 1	HMA	98.8	0.367	48
	WMA-CF	98.4	0.342	48
	WMA-CS1	92.7	0.372	48
	WMA-OW	115.0	0.325	48
	HMA/WMA	136.0	0.374	PAV
Phase 2 Mix Design #1	R-HMA-Control1	48.2	0.367	14
	R-WMA-MF1	45.8	0.479	14
	R-WMA-CS1	66.1	0.448	14
	R-WMA-CS2	63.1	0.451	14
	R-HMA/R-WMA	120.0	0.345	PAV
Phase 2 Mix Design #2	R-HMA-Control2	40.8	0.412	14
	R-WMA-OW	41.5	0.398	14
	R-WMA-CF	43.1	0.404	14
	R-WMA-MF2	36.6	0.438	14
	R-WMA-CS3	36.3	0.422	14
	R-HMA/R-WMA	95.0	0.354	PAV

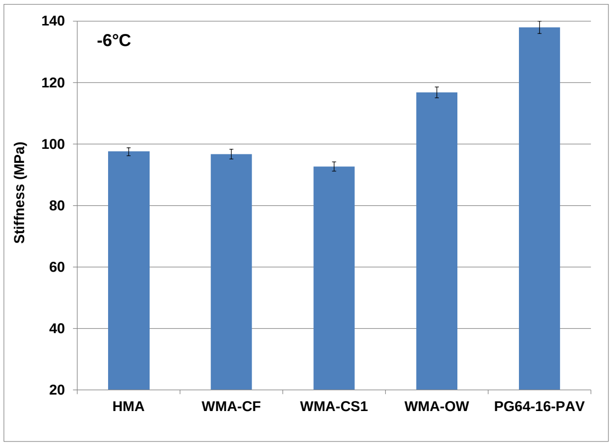


Figure 4.56: Phase 1: Comparison of binder stiffness values at -6°C after 48 months field aging.

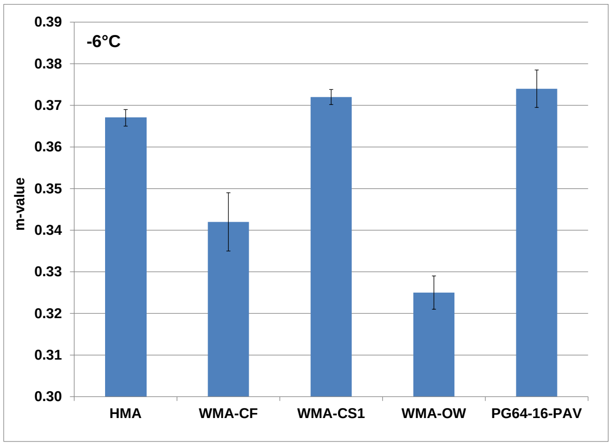


Figure 4.57: Phase 1: Comparison of binder m-values at -6°C after 48 months field aging.

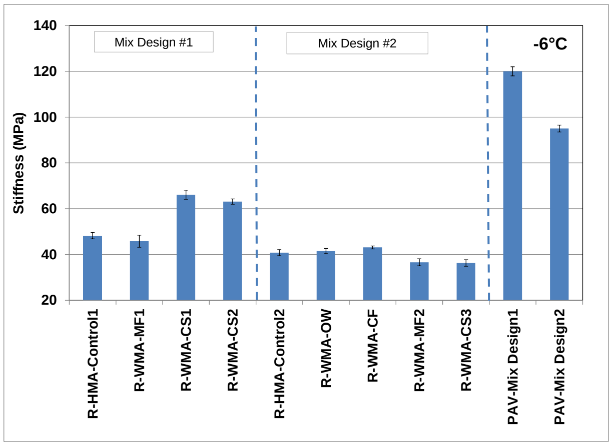


Figure 4.58: Phase 2: Comparison of stiffness values at -6°C after 14 months field aging.

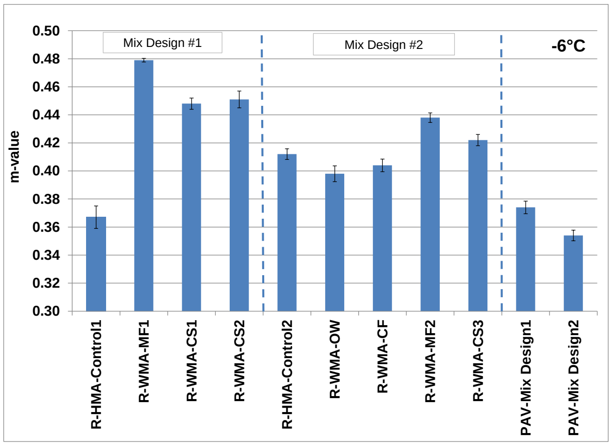


Figure 4.59: Phase 2: Comparison of m-values at -6° C after 14 months field aging.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The following conclusions are made based on the test results discussed in Chapter 4:

- Zero shear viscosity (ZSV) is a good indicator of the rheological behavior of asphalt binders with respect to rutting resistance performance in the field as observed from accelerated load testing results. ZSV was also found to be more suitable for describing the rutting performance of rubberized binders than the current Superpave $G^*/\sin\delta$ criterion.
- Viscosity-shear susceptibility is a suitable parameter for understanding the shear sensitivity of rubberized binders. It increased during long-term oxidative aging due to the increased association of polar compounds.
- The non-recoverable creep compliance and percent recovery parameters obtained from multiple stress creep recovery tests are useful parameters for understanding expected field rutting performance.
- The cup-and-bob geometry testing procedure is more appropriate than the parallel plate geometry test for measuring the rheological properties of rubberized binders with respect to performance-related properties in the field.
- Test results did not appear to be influenced by warm-mix technology chemistry. However, the binder with the organic wax consistently showed better rutting resistance across all the tests. This was attributed to the residual crystallization wax structure in the binder.
- All test results appeared to be influenced by production and placement temperatures, indicating that some mixes produced at very low temperatures could be more susceptible to early rutting on pavements that experience high ambient temperatures and high traffic loading.
- The addition of crumb rubber to two binders with the same PG grading from different refineries increased the rutting resistance properties compared to the neat base binders.
- Bending beam rheometer results indicated that WMA technologies did not result in a grade change with respect to thermal cracking properties at low temperatures. All binders met the Superpave criteria for thermal cracking properties at all ages tested. Performance trends for individual binders were consistent with rutting test results.
- The warm-mix additives and associated lower production and placement temperatures generally had limited effects on aging kinetics with respect to long-term aging in the field, with the exception of the organic wax additive. In all instances, production temperature appeared to have the most influence on performance.
- Air-void content appeared to have very little effect on the rheological properties of the extracted binder over the aging period assessed in this study. This was not expected.
- Laboratory binder aging, specifically in the rolling thin film oven, did not always correspond to field performance.

5.2 Recommendations

Current laboratory binder aging protocols do not necessarily provide results that correspond to field aging of conventional or rubberized binders, or either of these types of binders produced at warm-mix temperatures using a variety of warm-mix technologies. The findings of the NCHRP 9-52 study (completion due in February 2015) should be reviewed and its recommended changes implemented if appropriate. Since the NCHRP study is not investigating rubberized binders, the applicability of any recommendations to rubberized binder aging should be investigated for a range of rubberized binder sources and field aging conditions in California.

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