

Transportation Consortium of South-Central States

Solving Emerging Transportation Resiliency, Sustainability, and Economic Challenges through the Use of Innovative Materials and Construction Methods: From Research to Implementation

Feasibility of Warm Mix Asphalt in Arkansas

Project No. 20BASU24 Lead University: Arkansas State University

> Final Report October 2021

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16. Abstract

The future of Warm Mix Asphalt (WMA) technologies is promising in the U.S. However, the Arkansas Department of Transportation (ARDOT) does not have any specific guidelines to implement them in the field. This research aims to provide necessary baseline data for WMA as a proof of concept. In this study, three ARDOT approved Performance Grade (PG) binders namely PG 64-22, PG 70-22, and PG 76-22 were investigated. Each of these binders was obtained from two different sources. They were modified by varying doses of four selected additives: Sasobit®, Advera®, Evotherm®, and Rediset®. Additionally, four different types of aggregates (sandstone, limestone, gravel, and dolomite) from different quarries in Arkansas were evaluated for their compatibility with modified binders. Empirical test (Penetration test), Superpave Performance tests such as Rotational Viscometer, Rolling Thin-Film Oven (RTFO), Pressure-Aging Vessel (PAV), Dynamic Shear Rheometer (DSR), and Bending Beam Rheometer), PG Plus tests such as Multiple Stress Creep Recovery and Frequency Sweep, chemical analyses (SARA analysis, FTIR, pH), science-based test (Surface Free Energy), and Texas Boiling Test on loose mixture samples were conducted at different aging conditions. Based on the RV test results, reduced mixing and compaction temperatures have been observed for Sasobit®, Evotherm[®], and Rediset[®] modified samples. The DSR test results suggest that both Sasobit[®] and Advera[®] can reduce rut potential. On the other hand, BBR test results indicate that both Evotherm® and Rediset® have the capabilities of improved resistances against fatigue and low-temperature thermal cracking. Binder samples modified by these have also demonstrated minimal stripping in SFE and Texas Boiling Test. Advera® has modified the chemical compositions of the neat binders, which was also observed in the SARA analysis. The findings of this study will help the agency to select the most appropriate WMA additive along with its application rate.

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	SI* (MODER	N METRIC) CONVER	SION FACTORS	
	APPRO	XIMATE CONVERSIONS	FO 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
. 2		AREA		2
in ²	square inches	645.2	square millimeters	mm ²
ft-	square feet	0.093	square meters	m ²
ya	square yard	0.830	square meters	m
ac mi ²	square miles	2 59	square kilometers	km ²
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floz	fluid ounces	29 57	milliliters	ml
dal	gallons	3 785	liters	1
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	e shown in m ³	
		MASS		
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
Т	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
		TEMPERATURE (exact deg	rees)	
°F	Fahrenheit	5 (F-32)/9	Celsius	°C
		or (F-32)/1.8		
		ILLUMINATION		
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m ²	cd/m ²
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ACRONYMS, ABBREVIATIONS, AND SYMBOLS

AASHTO	American Association of State Highway and Transportation Officials
AC	Asphalt Concrete
ARDOT	Arkansas Department of Transportation
ASTM	American Society for Testing and Materials
ASU	Arkansas State University
BBR	Bending Beam Rheometer
DSR	Dynamic Shear Rheometer
DOT	Department of Transportation
FHWA	Federal Highway Administration
FS	Frequency Sweep
FTIR	Fourier Transform Infrared Spectroscopy
HMA	Hot Mix Asphalt
MSCR	Multiple Stress Creep Recovery
min	Minute(s)
mJ/m^2	Unit of Cohesion Energy
mm	Millimeter
m-value	Slope of the Stiffness Curve
NCHRP	National Cooperative Highway Research Program
OCA	Optical Contact Analyzer
OCG	van Oss, Chaudhury, and Good
PAV	Pressure Aging Vessel
PG	Performance Grade
RTFO	Rolling Thin Film Oven
RV	Rotational Viscosity
S1	Source 1
S2	Source 2
SD	Sessile Drop
SFE	Surface Free Energy
S-value	Creep Stiffness
TranSET	Transportation Consortium of South-Central States
TRB	Transportation Research Board
TSR	Tensile Strength Ratio
TTI	Texas Transportation Institute
%	Percent
G*	Complex Shear Modulus
g/gm	Gram (Unit of weight)
hrs	Hours
Hz	Unit of Frequency used in DSR Test
in.	Inch
kPa	Kilo Pascal
lb	Pound
nN	Adhesion Force
°C	Degree Celsius (Unit of Temperature)

°F	Degree Fahrenheit (Unit of Temperature)
mPa.s	Unit of Viscosity used in RV tests
psi	lb/in ²
rpm	Rotation per Minute

EXECUTIVE SUMMARY

Warm Mix Asphalt (WMA) technologies showed promising performances in terms of lowering mixing and compaction temperatures, improved rut and low-temperature fatigue resistances, moisture damage resistance, and so on. The benefits span from less fuel demand, less overhead cost, less greenhouse gas emission, a healthy environment, longer hauling distance, and extended paving season. Many state Departments of Transportations (DOTs), local agencies, private contractors have adopted these technologies, yet the Arkansas Department of Transportation (ARDOT) lacks necessary supportive data towards implementing them in the field. Hence, a series of laboratory tests were conducted and results were analyzed to generate the necessary baseline for ARDOT for practice.

Three ARDOT-certified PG asphalt binders (PG 64-22, PG 70-22, and PG 76-22) from two different sources were selected for this study. The first binder (PG 64-22) was a neat binder and the other two (PG 70-22 and PG 76-22) were polymer modified binders. They were modified by four different WMA additives: Sasobit[®], Advera[®], Evotherm[®], and Rediset[®]. The dosages recommended by the manufacturers of these WMA additives were selected to modify the aforementioned binders. Additionally, four different types of aggregates (sandstone, limestone, gravel, and dolomite) from different quarries in Arkansas have been evaluated for their compatibility with modified binders. An empirical test (Penetration test) was performed for obtaining the stiffness or consistency of the unmodified and modified binder samples. Superpave Performance tests such as Rotational Viscometer (RV), Rolling Thin-Film Oven (RTFO), Pressure-Aging Vessel (PAV), Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR) were performed to evaluate the rheological properties of the binder samples at different aging conditions. PG plus tests such as Multiple Stress Creep Recovery (MSCR) and Frequency Sweep were conducted to capture the effect of polymer in the modified binders. Chemical analyses (SARA analysis, FTIR, pH) were carried out to observe changes in asphalt chemistry due to the addition of selected additives. A science-based test namely the Surface Free Energy method and the Texas Boiling Test were accomplished to assess the compatibility between the aggregates and modified samples, and also to measure their stripping resistance.

Based on the laboratory test results, quantitative comparisons among binder-additive-aggregate combinations were discussed in this report. Overall, all tested binders modified with the optimum dosages of additives (Sasobit[®] 1.5%, Asphamin[®] 6%, Evotherm[®] 0.5%, and Rediset[®] 0.75% by the weight of the neat binder) have met the Superpave criteria for viscosity, rutting factor, and lowtemperature cracking. Specifically, the RV test data suggest that Sasobit[®], Evotherm[®], and Rediset[®] have been able to reduce mixing and compaction temperatures. The DSR test data suggest that Sasobit[®] and Advera[®] can improve rut resistance at the beginning of service life. The BBR test results of Evotherm[®] and Rediset[®] modified PG 64-22 and PG 76-22 binders have shown satisfactory performance against thermal cracking. The SFE and Texas Boiling Test results suggest improved resistances against moisture damage for both Rediset[®] and Evotherm[®] modified binders. The FTIR test results have shown that Advera[®] has introduced a new functional group in all the binder samples. The SARA fraction analysis using IATROSCAN has shown increased asphaltene and reduced aromatics (cyclic) content in Advera[®] modified samples. The Acid number test (pH) results have shown that all Advera® and Rediset® modified samples have acid numbers higher than their corresponding unmodified samples. The findings of this study are expected to reduce the knowledge gap; hence, promote green construction and safeguard the environment in Arkansas.

1. INTRODUCTION

1.1 Problem Statement

Warm Mix Asphalt (WMA) technologies can reduce the mixing and compaction temperatures of asphalt concrete compared to the traditional Hot Mix Asphalt (HMA). The benefits of these technologies are propitious in the U.S., but further study is required to validate their expected outcome in terms of energy savings, reducing pollutions, better workplace environment, and so on. WMA technologies are reportedly able to reduce the mixing and compaction temperatures by 16 to 55 °C. They can also extend the paving season in some places where the construction of HMA is restricted to warmer months. Contractors in Arkansas occasionally produce foam-based WMA mixes, which are often questionable as traces of water used in the process may still be entrapped causing premature distresses. At this point, no performance data regarding additivebased WMAs are not available in Arkansas. Therefore, this study aims to generate such performance data to reduce the research gap. Three ARDOT-approved Performance Grade (PG) binders have been modified by the corresponding manufacturer-recommended dose of four additives: Sasobit[®], Advera[®], Rediset[®], and Evotherm[®]. An empirical test (Penetration), Superpave Performance tests (e.g., Rotational Viscosity, Dynamic Shear Rheometer, and Bending Beam Rheometer), PG Plus tests (Multiple Stress Creep Recovery and Frequency Sweep), chemical analysis (SARA analysis, FTIR, and pH), and a science-based test (Surface Free Energy), and Texas Boiling Test have been performed on both aged and unaged binders. The collective results will suggest the agencies and asphalt producers choose the right WMA technology for their future pavements.

1.2 Background

The WMA technologies allow mixing the asphalt binders with aggregates at temperatures typically lower than the HMA mixtures. Bonaquist (1) reported that this reduction was around 28 °C and Newcomb (2) found that this could vary between 16 to over 55 °C. This temperature reduction provides several environmental and health benefits including less emission of greenhouse gases, lower fuel consumption, and a better workplace environment (3, 4). The lower production and mixing temperatures reduce short-term binder aging, provide extended hauling distance, and allow paving in the cold region (1). Also, lower production temperature reduces the overhead cost by 30-50 % (5). The above benefits allow the mixing plant to be located near paving zone i.e., shorter haul distances; thus, shorter construction period that will allow the authority to open the road for traffic sooner.

In 2002, personnel from several agencies e.g., National Asphalt Pavement Association (NAPA), Federal Highway Administration (FHWA), state agencies, and some municipal agencies formed a technical committee to investigate some WMA field trials in the United States (6). The Asphalt Pavement Association of Oregon reported that lower plant mixing temperatures of WMA could lead to a 30% reduction in fuel consumption (7). It was reported that low mixing and compaction temperatures led to a better workplace environment, and low greenhouse gas emissions (8, 9, 10). Similarly, low production temperatures save budget by reducing fuel demand, by 30-50% overhead cost due to less emission (3). WMAs were reportedly extended hauling distance and construction season due to low construction temperatures (11). Moreover, these technologies reduced thermal cracking and block cracking, and prevents the mix to be tender when placed due to less aging during construction.

WMA technologies are relatively new processes and products, which can be used to reduce the shear resistance of the asphalt mixture at relatively low production temperatures (Hossain et al. 2012). WMA additives are divided into three main groups: organic additives, foaming processes and additives, and chemical additives. Another method is the combination of two or more aforementioned methods to reduce the viscosity of binders (12). Many additives such as Sasobit®, Aspha-Min®, Radiset[®], Evotherm[®], etc. have been used in WMA technologies for several years. Hurley and Prowell evaluated Sasobit[®], Aspha-min, and Evotherm[®] on PG binders based on mixtures density, strength gain, rutting performance, and moisture resistance (11, 13, 14). They described Sasobit[®] as a product of Sasol Wax. Abraham et al. (2002) defined Sasobit[®] as a fine crystalline, long-chain aliphatic polymethylene hydrocarbon produced from coal gasification using the Fischer-Tropsch (FT) process. They also mentioned that Sasobit[®] can act as an asphalt flow improver during mixing and construction. Moreover, Sasobit[®] has a congealing temperature of approximately 102°C and is completely soluble in asphalt binder at temperatures higher than 120°C. It reportedly forms crystalline network structures in the binder at temperatures below its melting point, which leads to added stability (15, 16, 17). Hurley and Prowell (13) showed a potential temperature reduction of 12.2°C of typical production temperatures for Aspha-min modified samples. Similar to Aspha-Min®, Advera[®] is another water-bearing additive and is marketed by PQ Corporation in the U.S. Radiset[®], is supplied as a liquid form and added at the rate of 0.3-1.0% by weight of the asphalt binder (18). Another additive, Evotherm[®], manufactured by Ingevity (formerly part of MeadWestvaco), is another family of chemical additives that have successfully been used in construction projects in Texas, Oklahoma, and Iowa (19, 20, 21). The Iowa study used Evotherm[®] 3G for producing WMA and reported a 25°C reduction of mixing temperature and a 15°C reduction of compaction temperature while the former performed the same as HMA (21). foamed (water) based WMA technologies (e.g., Double-Barrel Green and Terex WMA System) require significant plant modifications. Only a few asphalt plants in Arkansas are equipped with the production of foam-based WMA technologies, which inject water into the mixing plant.

2. OBJECTIVES

The main goal of this study is to assess the feasibility of selected additives in producing WMA binders in Arkansas. In particular, their effects on the viscoelastic and chemical properties of selected asphalt binders will be determined in laboratories. The specific objectives of this study are listed below:

- Evaluate changes in asphalt binder's viscosity due to the addition of WMA additives;
- Determine the mixing and compaction temperatures of WMA-additive modified binders;
- Determine changes in binder grade of the selected asphalt binder due to the addition of WMA additives;
- Determine the effect of WMA additives on the oxidation of the selected asphalt binder;
- Evaluate the effect of short-term aging, using the rolling thin film oven (RTFO), on binders with and without additives at a reduced temperature; and
- Determine changes in SARA fractions, indicators of mechanical properties and stability, of the asphalt binder modified with different WMA additives.
- Evaluate the compatibility of different aggregates and asphalt binders modified with WMA additives in presence of water (e.g., stripping resistance).

3. LITERATURE REVIEW

Hurley and Prowell (13) investigated the effect of Aspha-min[®] on PG 58-22 and PG 64-22. They performed resilient modulus, APA (Asphalt Pavement Analyzer) rut, moisture sensitivity, and HWTD tests to evaluated this additive. The mixes were compacted at 300, 265, 230, and 190 °F; and corresponding mixing temperatures were 35 °F higher than their compaction temperatures. It was found that Aspha-min[®] slightly improved the density of the samples. It was also observed that Aspha-min[®] could lower the mixing and compaction temperatures by one grade. The addition of Aspha-min[®] slightly improved resilient modulus. ANOVA (Analysis of Variance) on APA rut depth results showed that Aspha-min[®] did not influence the rutting potential. Overall, limestone imparted an improved rut potential. The authors reported the redundancy for curing time for Aspha-min[®] modified binders. However, they put a temperature threshold for pavement for Aspha-min[®] modified mixes, which was at or below 120 °F of. ITS test showed that aspha-min[®] reduced TSR values for all compaction temperatures which was also confirmed by Hamburg Wheel Tracking Device (HWTD) test. But the field performances for both the control and Aspha-min[®] modified mixes were similar.

Hurley and Prowell (11) also evaluated the effect of Sasobit® and Sasoflex® on PG 64-22 and PG 58-28 binders. High-temperature PG grade of PG 58-28 binder increased by one by the addition of 2.5 % of Sasobit[®] and two by the addition of 4 % Sasoflex[®]. Also, the addition of 4 % of Sasoflex[®] to PG 64-22 binder produced PG 76-22 binder. They reported that Sasobit[®] reduced aging and improved compaction. However, it could not improve density for SBS (Styrene Butadiene Styrene) modified binders. On the other hand, the ANOVA test on resilient modulus could not identify the influence of Sasobit[®] on it rather combined effect of aggregate types and Sasoflex[®] was found to be influential. Similarly, interaction plots identified limestone as influential on resilient modulus rather than the presence of the additive. From the ANOVA test on APA rut potential, it was observed that binder containing Sasoflex[®] offered less rut depth but Sasobit[®] deteriorated rut resistance of the binders by reducing the aging. It was concluded by the authors that Sasobit[®] modified pavements needed no curing time before allowing traffic on them. Sasobit[®] failed to meet Superpave TSR (Tensile Strength Ratio) specifications (TSR > 0.80) until an ASA (anti-stripping agent) was added along with it. Moreover, the Sasobit[®]-modified samples exhibited very low unsaturated and saturated ITS (Indirect Tensile Strength) values. On the other hand, Hamburg Wheel-Tracking Device (HWTD) test showed improved results for the samples modified by Sasoflex[®].

Similar to Aspha-min[®] and Sasobit[®], Evotherm[®] was also evaluated in the NCAT study (14). The researchers used the same materials and methodologies to evaluate the performance of this additive on mixtures density, resilient modulus, rutting, and moisture sensitivity. The results indicated that Evotherm[®] reduced optimum asphalt content but improved compaction compared to control mixtures. Improved compaction of course results in a higher resilient modulus. Thus, Evotherm[®] modified samples demonstrated a higher modulus compared to their respective control samples. Though, these modulus values decreased as compaction temperature decreased as sample density decreased. Both APA and HWTD test results showed that Evotherm[®] could reduce the rut depth which was explained due to improved compaction. The HWTD test results also showed that Evotherm[®] modified samples possessed improved moisture resistance comparing to control mixes. The researchers also reported that pavements containing Evotherm[®] modified mixtures could be opened for traffic without delaying for curing as sufficient strength could be achieved right after the construction.

Biro et al. (22) investigated rheological properties of PG 64-22 binders from five different sources (Venezuela, Mexico, Texas, Canada, and Rocky Mountain) using Sasobit[®] and Aspha-min[®] at the intermediate temperature range. The dosages of Aspha-min[®] and Sasobit[®] were 0.3% weight of the mixture and 1.5 % weight of the binder, respectively. The addition of Sasobit[®] decreased the distribution of Large Molecule Size (LMS) of the Venezuelan binder significantly, thus more prone to rutting. The other Sasobit[®] modified binders and all Aspha-min modified binders did not show such alternation. The Aspha-min[®] modified binders acted as a Newtonian fluid at 60 °C unlike Sasobit[®] modified binders. Both additives increased viscosity at the test temperature. Aspha-min® increased viscosity due to its filling effect, and Sasobit® increased viscosity because of recrystallization at 60 °C. The frequency sweep test results showed that Sasobit® modified binders were more rut resistant than both neat and Aspha-min modified binders. Creep test results showed lower compliance values for Sasobit® modified binders, thus improved stiffness at midrange temperatures. They also observed that Sasobit[®] modified showed the lowest maximum deformation and was able to recover fully in creep recovery tests which were consistent with the repeated creep recovery test. However, the performance of Aspha-min® was controlled by its filling nature in all performance tests. The temperature sweep test showed that the complex shear modulus was almost insensitive to the presence of additives over the test temperature (25-80 °C). But Sasobit[®] was proven to provide some elasticity to the base binders.

Wasiuddin et al. (23) investigated Superpave criteria for PG 64-22 and PG 70-28 modified by Sasobit[®] and Aspha-min[®]. The dosages of Sasobit[®] and Aspha-min[®] were the following: 2, 3, and 4% by the weight of binder; and 0.2, 0.3, and 0.4% by the weight of mixer, respectively. The RV test results showed that Sasobit[®] brought a significant reduction in viscosity for both binders, whereas Aspha-min could not notably reduce the viscosity. For both additives, the reduction of viscosity increased with their increased dosages. Authors found that Sasobit® increased the rutting parameter $(G^*/\sin(\delta))$ for both unaged and RTFO-aged binders and high-temperature grade of unaged binders, which increased as the Sasobit[®] dosages increased. On the other hand, all Asphamin[®] dosages decreased $G^*/sin(\delta)$ and high-temperature grade for both unaged and RTFO-aged binders. But, Sasobit[®] modified binders failed to meet the minimum m-value specified in Superpave and the authors suggested further study in this regard. However, most of the Asphamin[®] modified samples passed the minimum requirement. Both additives made the aged PG 64-22 binder more prone to cracking (higher G^* .sin(δ) values) at their higher dosages. On the other hand, aged PG 70-28 became more susceptible to cracking when were mixed with Aspha-min but Sasobit[®] reduced the cracking susceptibility of this aged binder. The APA test results showed a good correlation ($R^2 = 0.8$) with the rutting parameter. In terms of rut depth reduction, Sasobit[®] performed better than Aspha-min[®] for all samples. It was also reported that a decrease in mixing and compaction temperature would decrease the rutting potential.

Mohammad et al. (24) conducted performance tests on Sasobit[®] (1%) modified PG 76-22 samples. They also used Permatac[®] 99 anti-strip agent (an amount of 0.6 by weight of the binder). The authors reported lower ITS values for both un-aged and aged Sasobit[®] modified samples than the conventional mix. Also, the toughness index (TI) for modified samples was reported to be lower than the conventional mix. The SCB test results showed that Sasobit[®] slightly improved the fracture resistance of the PG 76-22 binder. The P-statistics showed a statistically significant difference between phase angles of modified and conventional samples only at high temperatures and frequencies, whereas no statistically significant difference was observed between E* values within the testing parameters. The crack resistance parameter (E* × Sin\delta) was observed to be lower

for Sasobit[®] modified samples, although p-statistics did not show any significant difference except at 10 Hz and 4°C. Another Superpave specification, $E^*/Sin\delta$ was found to be insensitive to the mix type within the test premise.

Workability and compatibility of additive modified PG 76-22 binder were investigated by Bennert et al. (8). The binder used in this was pre-modified by 11-13% of SBS (Styrene Butadiene Styrene). Later, it was modified by varying dosages of Evotherm[®] 3G, Sasobit[®], and Rediset[®]. The RV test results exhibited an impractical binder mixing (>300 °F) and compaction (>287 °F) temperatures. which would be typical for WMA. The frequency sweep test results also showed similar results; thus, they were not used for ranking. The authors observed that viscosity decreased exponentially as the rotation of the Lubricity Test increased. The authors stated PG 76-22 as the second-worst binder without any additive. In this study, workability and compactibility were assessed using asphalt workability device (AWD), Marshall compaction, and gyratory compaction. It was observed that both 2% Rediset[®] and 0.6% Evotherm[®] 3G showed the least torque value, thus easier coating at temperatures as low as 190 °F. 0.6% Evotherm[®] 3G also resulted in lower air voids when was compacted in the gyratory compactor. It was observed that the compaction rate decreased as the compaction temperature decreased (from 300 to 260 °F) even with the presence of the additive but increased again when the temperature decreased further (216 °F). At 216°F compaction temperature, all mixture had sufficient flowability due to less aging. The results were in favor of 2.0 % Rediset[®] and 1.5% Sasobit[®] modified samples. Authors wrapped up their work by crediting 2% Rediset[®] and 0.6% Evotherm[®] as overall best performers.

Koc et al. (25) investigated the wettability of Sasobit[®] modified PG 64-22 asphalt binder using the sessile drop (SD) method. The surface free energy (SFE) component was calculated using Good-van Oss-Chaudhury (GVOC) approach. The authors used the following dosages of Sasobit[®]: 0.5, 1.0, 1.5, 2.0, 4.0, and 8.0%. The probe liquids used in this study were water, ethylene glycol, and diiodomethane. The compatibility was tested between the modified aggregates and Davis limestone and Snyder granite. The authors found that Sasobit[®] decreased the average contact angles (average of six measurements) of the neat binder and this reduction had a positive relation with Sasobit[®] dosages. In other words, the more Sasobit[®] is added, aggregates will be coated more by the binders. The authors also noted a slight increase in SFE as the Sasobit[®] dosage increased. The energy ratio also increased for higher Sasobit[®] dosages and the mixtures lost resistance against moisture for Sasobit[®] dosage 2% or lower than that. However, it is worth mentioning that the manufacturer recommends keeping this additive amount at or below 3% considering the resistances against moisture as well as rutting and other distresses.

Malladi et al. (26) investigated the moisture damage resistances of PG 64-22 binder modified by Sasobit[®], Advera[®], and a foamer. Their dosages were 1.5% by weight of the binder, 0.25% additive, and 2% water by the weight of the mixture, respectively. The control (HMA) and WMAs had mixing and compaction temperatures of 163 and 135 °C, and 149 and 120 °C, respectively. Volumetric properties analysis showed that all WMAs had similar workability compared to the HMA even with lower mixing and compaction temperatures. Modified Lottman test results revealed that both Advera[®] WMA and foamer could not pass the NCDOT (North Carolina DOT) TSR requirement of more than 0.85 even doubling the ASA dose could not make them achieve the threshold value. Contrariwise, both HMA and Sasobit[®] WMA achieved TSR values greater than 0.85. However, both conditioned and unconditioned Sasobit[®] WMA samples exhibited tensile strengths lower than even the conditioned HMA samples. It was also observed that Advera[®] WMA samples went through moderate to severe stripping indicating their high susceptibility to moisture-

induced damage. The APA test data showed that all the mixtures had rut depth less than 6 mm, which is less than the NCDOT failure criterion (9.5 mm), and the WMA samples performed better than HMA.

Rahmad et al. (27) studied the effect of Rediset[®] LQ-1106 on the SFE of the PG 76 binder. The dosages of the additives used in this study are the followings: 0% (control mix), 1%, 2%, 3%, 4%, and 5%. The authors evaluated the anti-stripping feature of Rediset[®] using boiling water test, and the results were analyzed visually using a digital image analyzer software. This test showed good adhesion between the aggregates and the binder qualitatively. The adhesive forces were quantified from the SFE data and an AFM (Atomic force microscope) observation. The SFE was calculated by the Owens, Wendt, Rabel, and Kaelble (OWRK) method. The contact angles of water reduced drastically with the dosage increment. The other liquids (formamide and glycol) could not establish any such trend. This study proved that the binder has a positive wettability up to the additive dosage of 3%. The AFM data revealed that 2% of Rediset[®] modified samples had average adhesive force and the highest grain number, whereas SFE test results showed that 4% of Rediset[®] possessed the highest adhesive force.

Hossain et al. (28) evaluated the rheological properties and chemical composition of PG 64-22 modified by Sasobit[®] and Aspha-min[®] through various mechanical and chemical tests. The RV tests showed that Sasobit[®] could lower mixing temperature and the maximum reduction was 11 °C for 3% Sasobit[®]. On the other hand, Aspha-min[®] increased viscosity during blending and testing temperatures. The DSR test data showed that Sasobit[®] increased both high and low PG temperatures. On the other hand, Aspha-min[®] detrimentally increased the low PG temperature. The NMR (nuclear magnetic resonance) spectroscopy data showed that Sasobit[®] dose. However, aliphatic content in the binder, while this increment ceased at a higher Sasobit[®] dose. However, aliphatic content in the binder increased as Aspha-min[®] doses increased. From FTIR tests, the authors noticed higher carbonyl absorption for the Sasobit[®] modified binders and lower absorption for Aspha-min modified binders than the base binder. In general, higher absorption indicates higher complex shear modulus values. Elemental Analysis proved higher carbon and lower oxygen content in Sasobit[®] modified binder, which indicated higher G*. This test also showed a slight increase in hydrogen content in the modified binder, thus a slight change in G* values.

Syed et al. (29) investigated the rut resistance of SPS-10 (Specific Pavement Study) asphalt binders by conducting the HWTD, Frequency Sweep, Zero Shear Viscosity (ZSV), and MSCR tests. Four different additives were employed to modify the binder: Terex® Foaming, Evotherm[®], Cecabase®, and Cecabase®+ polymerized. From the analysis of the HWTD test, moisture damage was found to be insignificant. Cacebase® with polymer showed significant rutting improvement whereas Evotherm[®] could not bring such improvement. Also, the rut depths for the control mix and Evotherm[®] were 4.72, and 3.99 mm, respectively. The Superpave rutting parameter (G*/Sin\delta) was also found to be increased by 1.75 times for Evotherm[®] modified samples. The authors suggested that the use of polymer and anti-stripping agents might improve rutting resistance. Similar results were observed from frequency sweep and ZSV test data. The MSCR test showed that the Evotherm[®] modified binder showed the least improvement.

Kassem et al. *(30)* focused on the surface energy of both aged and unaged binders and aggregates and used this energy to evaluate the potential for fatigue cracking and moisture damage. Two different binders: modified PG 76-22 and unmodified PG 64-22 produced by Qatar Fuel were used in this study. The additives used in this study were the following: Sasobit[®], Evotherm[®] MA3, and

Rediset[®] LQ. The surface energy of the binder and aggregates was evaluated using the Wilhelmy Plate method and Universal Sorption Device, respectively. It was noticed that all the additives reduced the surface energy to some extent and, Sasobit[®] modified binder exhibited the lowest surface energy for PG 76-22. As the tests were performed at room temperature, it was expected that SFE would get lower further at mixing and compaction temperatures. The authors suggested revising the recommended dosages of Evotherm[®] and Rediset[®]. The modified PG 76-22 binder was found to be stiffer indicating higher fracture resistance offered by its polymer network. Only neat and Sasobit[®] modified PG 76-22 binders showed higher cohesive bond energy after aging, which means that PG 76-22 binders offered more resistance to fracture. It was observed that a Sasobit[®] dose of 2% or higher decreased the adhesive bond energy, thus more susceptible to fatigue cracking. Both Rediset[®] and Evotherm[®] improved adhesive energy with one exception (0.5% Evotherm[®]). The better performance of Evotherm[®] and Rediset[®] might result from the presence of an anti-stripping agent in the Evotherm[®] and Rediset[®]. The bond energy ratio calculation showed that Rediset[®] improved the moisture resistance for all types of binders, whereas Evotherm[®] enhanced only modified PG76-22 binders' moisture resistance property. Sasobit[®], on the other hand, impaired the resistance when its dosages exceeded 1.5%.

Hossain et al. (31) assessed moisture resistance and compatibility of a group of binder-aggregate systems using Surface Free Energy of binder-aggregate systems and Texas boiling tests. The researchers used three PG binders: PG 64-22, PG 70-22, and PG 76-22 from two different crude sources; and four aggregates: limestone, sandstone, dolomite, and gravel were used in their study. They found that polymer-modified asphalt binders (PG 70-22 and PG 76-22) had higher work of cohesion than unmodified binders, which means polymer-modified binders had stiffer bonding and better resistance to moisture damage. They also found that aging made asphalt binder more prone to cracking. Finally, the researchers ranked dolomite and limestone as highly compatible with tested binders, and sandstone was ranked as the worst aggregates under this study premise.

4. METHODOLOGY

In this project, three ARDOT approved Performance Grade (PG) binders were collected from two different sources and they were subsequently modified by four additives. Based on an extensive literature review and past experiences, one dose of each additive was selected. This study encompassed a wide variety of tests, which were shown in Figure 1.



Figure 1: Project plan towards achieving goals.

4.1 Materials

4.1.1 Asphalt Binders

Three asphalt binders (PG 64-22, PG 70-22, and PG 76-22) were collected from two different sources: Ergon Asphalt and Emulsion at Memphis, TN (S1); and Marathon Petroleum at Memphis, TN (S2). Their designations are tabulated in Table 1.

Table 1: Asphalt bi	nder types, designa	ations, and sources.
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Asphalt Binder Designation	Designation	Asphalt Binder Source
PG 64-22	B1	Source 1: Ergon Asphalt & Emulsion, Inc.
		Memphis, TN
PG 70-22	B2	Source 2: Marathon Petroleum
		Corporation, Memphis, TN
PG 76-22	B3	

4.1.2 Aggregates

Four aggregates: Limestone, Sandstone, Dolomite, and Gravel were collected from different ARDOT-approved sources, which are tabulated in Table 2. These aggregates represent a wide variety of durability, mineralogy, and chemical composition.

Aggregate Type	Source	General Characteristics			
Sandstone	APAC-Central- Preston Quarry, Van Buren, AR	Sandstones are siliciclastic sedimentary rocks, whose principal mineral constituents are quartz, feldspar, and rock fragments. Common heavy minerals include zircon, tourmaline, rutile, garnet, and magnetite.			
Gravel	Capital Quarries Company, Pocahontas, AR	Gravels are mostly quartz (silicon dioxide, SiO ₂) grains that are formed from weathering of rocks such as granite.			
Limestone	White River Materials Inc., Cord, AR	Limestones are carbonate sedimentary rocks. They are mostly composed of calcite and aragonite minerals, which are different crystal forms of calcium carbonate (CaCO ₃).			
Dolomite	Capital Quarries Company, Pocahontas, AR	Dolomites are anhydrous carbonate minerals that are composed of calcium magnesium carbonate, CaMg(CO ₃) ₂ .			

Table 2: Aggregate types and sources.

4.1.3 Warm Mix Additives

Sasobit[®] was collected from Sasol Chemicals (USA) LLC. It is an asphalt flow improver, which is produced from coal gasification using the FT (Fischer-Tropsch) process. It is a fined crystalline, long-chain aliphatic polymethylene hydrocarbon with a varying chain length. Below its melting point (> 90 °C), Sasobit[®] forms a lattice structure that provides additional stability to the asphalt binder (*15*). It also provides improved rut resistance at service temperature (*15*, *16*, *17*).

Advera[®] 401PS Aluminosilicate is a water-based additive. It was obtained from PQ Corporation, Malvern, PA. It is also known as Hydrated zeolite sodium (Na₂Al₂Si₂O₈.xH₂O) powder. The water in the lattice structure is released at a temperature range of 85-182 °C, which causes volume expansion of binder thus increases the workability of binders. This vaporization process happens for a long period until the mixture is cooled down below 100 °C; hence, the mixture remains workable across entire mixing and compaction processes. Also, zeolite accommodates an arrangement of interconnected large vacant spaces, which allow transportation of large cations even relatively large molecules and cation groups across it (4).

Rediset[®] LQ-1102C is a heat-stable adhesion promoter with built-in anti-stripping properties. It is a chemical package containing proprietary alkoxylated fatty polyamines, proprietary polyamines, and Diethylene glycol ($C_4H_{10}O_3$) collected from Nouryon (18).

Table 3: Physical, chemical, and dosage information of additives (18, 32, 33, 34).

Additives		Sasobit [®]	Asphamin®	Rediset [®] LQ – 1102C	Evotherm [®] P25	
D			(Advera [®] 401 PS)			
Types		Asphalt flow improver	Water based	Heat stable adhesion promoter, built-in anti- stripping effect	Adhesion promoter, built-in anti-stripping effect	
Physical	Appearance	Solid	Powder	Liquid	Liquid	
	Color	Off-white to Yellow	White	Dark brown	Tan. Brown.	
	Odor	Odorless	Odorless	Amine-like	Odorless (slight)	
	Density	0.9 g/cm ³ @ 25° C	$0.4 - 0.48 \text{ g/cm}^3$	1.0 g/cm ³ @ 20 °C	0.99 g/cm ³ (relative)	
	Viscosity (Dynamic)	9.9 mPa.s @ 135 °C	NA	1700 mPa.s @ 20 °C	487 mPa.s	
Chemical	Melting point	>90 °C	>1000 °C	NA	<-16°C	
	Pour point	NA	NA	2 °C	NA	
	Boiling point	>180 °C	NA	215 °C	>100°C	
	Flash point	>180 °C	NA	165 °C	181°C (Closed cup)	
Auto-ignition		>450 °C	NA	NA	NA	
	Decomposition temperature	NA	NA	NA	NA	
	pН	NA	10.1 - 11.4	10 @ 0.1% solution	2.3	
Solubility	Water	Insoluble	Insoluble	Dispersible	Insoluble	
	Other	Asphalt binder (>120 °C)	NA	NA	NA	
Congealing ASTM D92	g point 38	101 °C	NA	NA	NA	
Penetration ASTM D1	a 25°C 321	1	NA	NA	NA	
Chemical composition		FT hard wax, C_nH_{2n+2} , n= 45 - >100, apprx. 1000 g=mole	Zeolite (78 – 82 %), water, Na ₂ Al ₂ Si ₂ O ₈ .xH ₂ O	Proprietary alkoxylated fatty polyamines, Proprietary polyamines, and Diethylene glycol (C ₄ H ₁₀ O ₃); Amine value (540-640 mg KOH/g)	Modified tall oil fatty acid (\geq 75% - \leq 90%), Proprietary Alkyl acid phosphate (\geq 25% - \leq 41%)	
Dosages		0.8 -3.0% by weight of binder (Manufacturer)	0.05 – 0.3 % by weight of the asphalt mix	0.3 – 1.0 % by weight of basis binder (Manufacturer)	0.25–0.50% (Unmodified asphalt) 0.30– 0.75% (PMB	

Evotherm[®] P25 is also an adhesion promoter with built-in anti-stripping properties. According to Ingevity, it can replace lime and is pumpable. It can reduce production temperature up to 90° F and can be added to the mix plant and asphalt terminal *(32)*. Other information and dosages of these additives were summarized in Table 3. Table 4 contains sample nomenclatures of asphalt binders used in this study.

Table 4: Sample nomenclature.

	Madification (by weight	Sample	Sample
Base Binder	of asphalt hinder)	Nomenclature	Nomenclature
	of asphalt officer)	(Source 1)	(Source 2)
	-	S1B1A0	S2B1A0
	Sasobit [®] 1.25%	S1B1A1	S2B1A1
	Sasobit [®] 1.5%	S1B1A2	S2B1A2
	Sasobit [®] 1.75%	S1B1A3	S2B1A3
	Advera [®] 4%	S1B1A4	S2B1A4
	Advera [®] 6%	S1B1A5	S2B1A5
PG 64-22	Advera [®] 8%	S1B1A6	S2B1A6
	Evotherm [®] 0.25%	S1B1A7	S2B1A7
	Evotherm [®] 0.5%	S1B1A8	S2B1A8
	Evotherm [®] 0.75%	S1B1A9	S2B1A9
	Rediset [®] 0.5%	S1B1A10	S2B1A10
	Rediset [®] 0.75%	S1B1A11	S2B1A11
	Rediset [®] 1.0%	S1B1A12	S2B1A12
	-	S1B2A0	S2B2A0
	Sasobit [®] 1.25%	S1B2A1	S2B2A1
	Sasobit [®] 1.5%	S1B2A2	S2B2A2
	Sasobit [®] 1.75%	S1B2A3	S2B2A3
	Advera [®] 4%	S1B2A4	S2B2A4
	Advera [®] 6%	S1B2A5	S2B2A5
PG 70-22	Advera [®] 8%	S1B2A6	S2B2A6
	Evotherm [®] 0.25%	S1B2A7	S2B2A7
	Evotherm [®] 0.5%	S1B2A8	S2B2A8
	Evotherm [®] 0.75%	S1B2A9	S2B2A9
	Rediset [®] 0.5%	S1B2A10	S2B2A10
	Rediset [®] 0.75%	S1B2A11	S2B2A11
	Rediset [®] 1.0%	S1B2A12	S2B2A12
	-	S1B3A0	S2B3A0
	Sasobit [®] 1.25%	S1B3A1	S2B3A1
	Sasobit [®] 1.5%	S1B3A2	S2B3A2
	Sasobit [®] 1.75%	S1B3A3	S2B3A3
	Advera [®] 4%	S1B3A4	S2B3A4
	Advera [®] 6%	S1B3A5	S2B3A5
PG 76-22	Advera [®] 8%	S1B3A6	S2B3A6
	Evotherm [®] 0.25%	S1B3A7	S2B3A7
	Evotherm [®] 0.5%	S1B3A8	S2B3A8
	Evotherm [®] 0.75%	S1B3A9	S2B3A9
	Rediset [®] 0.5%	S1B3A10	S2B3A10
	Rediset [®] 0.75%	S1B3A11	S2B3A11
	Rediset [®] 1.0%	S1B3A12	S2B3A12

4.2 Laboratory Tests

4.2.1 Blending of WMA additives and asphalt binder

The hand-blending protocol used in this study was developed by Hossain et al. (28). They developed a manual protocol for blending WMA additives and RAP (Reclaimed Asphalt Pavement) with a neat binder, which was followed in subsequent studies (35, 36). They expect that this laboratory-based protocol will simulate the large-scale field blending procedure. Figure 2 shows the stirring process of a mixture following the blending protocol in this study.



Figure 2: Blending of additives with the neat binder.

This laboratory blending protocol comprises of following steps:

- a) The required amount of binder is calculated and heated in the aluminum can at 150 °C for one hour.
- b) The required amount of additive (depending on dosage) is calculated and poured into the base binder can.
- c) The binder-additive can is put back into the oven at 150 °C for nine minutes.
- d) A pre-cleaned and pre-heated glass rod are used to stir the mixture vigorously for one minute.
- e) Then, the mixture is kept back in the oven again for nine minutes at 150 °C.
- f) This "nine minutes heating + one-minute stirring" cycle is repeated for a total count of six times, resulting in 60 minutes of blending time for each mix.
- g) The interior wall of the sample container is scrapped periodically while stirring to prevent the accumulation of mixtures to the wall.
- h) The mixing procedure ends with heating. Then, it is kept at room temperature with a lid to cool down.

4.2.2 Penetration Test

The penetration test is one of the most commonly used asphalt testing methods before the viscosity-based grading system. This test was conducted in this study according to AASHTO T 49. The penetration test device used in this study is shown in Figure 3. The test was performed on water-cured samples at 25 °C. The final reading was the average of at least three measurements and reported in 1/10 of a millimeter.



Figure 3: Penetration Testing Device.

4.2.3 Rotational Viscosity (RV) Test

The RV test was performed according to AASHTO T 316. A DV-II+ Pro rotational viscometer from Brookfield Engineering Inc. (Figure 4) was used in this study to perform this test. This test measures the workability, pumpability, and mixability of the asphalt binders. The amount of torque required to maintain this constant speed (20 rpm) of the cylindrical spindle indicates the viscosity of the binder at the test temperature. The RV test is performed from 135 °C to 180 °C at a 15 °C interval to measure the viscosity of the binder. According to Superpave specification, the viscosity for unaged asphalt binder should be \leq 3 Pa.s at 135 °C.



Figure 4: DV-II+ Pro rotational viscometer.

4.2.4 Rolling Thin Film Oven (RTFO) aging

By nature, asphalt binders get oxidized over time, even at ambient temperature. This phenomenon is known as aging. This aging accelerates when binders are mixed with aggregates and compacted in the field at a high temperature. RTFO simulates the aging during the mixing and compaction procedures. In this study, AASHTO T 240 guideline was used except the aging temperature. As WMAs are mixed with aggregates at around 150 °C; the aging temperature was selected as 150 °C throughout the study. A similar strategy was used by other researchers (37, 38). The instrument used in this study for this purpose is shown in Figure 5.



Figure 5: Rolling Thin-Film Oven (RTFO).

4.2.5 Pressure Aging Vessel (PAV) aging

While in service, asphalt binders expose to a wide range of temperatures and longer oxidation periods. This aging, although slow, stiffens the binder over time and makes it susceptible to low-temperature fatigue cracking. To simulate this phenomenon, a Pressure Ageing Vessel (PAV) was used in this study following AASHTO R 28 specifications. It is assumed that a single PAV cycle (20 hours) can simulate 7 to 10 years of service life. The following Figure 6(a) depicts PAV used in this study. A regular PAV-aging (20 hours) on RTFO-aged samples was adopted in this study. Once the aging was complete, samples were degassed using a vacuum degassing oven (Figure 6(b)) at 170 °C.



Figure 6: (a) Pressure Aging Vessel (PAV) (left), and (b) Degassing oven (right).

4.2.6 Dynamic Shear Rheometer (DSR) Test

Asphalt binders are viscoelastic material i.e., they act partly as an elastic material and partly as a viscous material. A Dynamic Shear Rheometer (DSR) can characterize such behaviors of a viscoelastic material. Thus, DSR has been adopted in Superpave PG binder specification to

characterize both elastic and viscous components of asphalt binders at medium to high temperatures. It measures two properties of the specimen: complex shear modulus (G*), and phase angle (δ). G* is the total resistance of the specimen when repeatedly sheared; mathematically it is the ratio between the absolute value of peak-to-peak shear stress and the absolute value of peak-to-peak shear strain. Phase angle (δ) is the delayed response between applied shear stress and resulting shear strain measured in radians. Higher the δ value, the more viscous the material. For a purely viscous material $\delta = 90^{\circ}$, and a purely elastic material $\delta = 0^{\circ}$. Phase angle usually is the measure of viscous component of asphalt binder and complex shear modulus is the combined measure of both elastic and viscous moduli. Figure 7 shows a graphical illustration of these two properties.



Figure 7: Phase angle (δ) and complex shear modulus (G*) of asphalt binder (39).

In this study, this test is being conducted following AASHTO T 315 specifications. The DSR device used in this study is MCR 302. It is a strain-controlled device manufactured by Anton Paar (Figure 8). The strain was applied to the specimen and corresponding stress was measured. The whole operation was controlled by RHEOPLUS software v3.2. At high service temperature (for unaged and RTFO-aged), the test was conducted using 25 mm plate geometry (Figure 9(a)) and at intermediate service temperature (for PAV-aged), 8 mm plate geometry (Figure 9(b)) was used. Superpave rutting factor ($G^*/Sin\delta$) and fatigue factor ($G^*/Sin\delta$) govern in high service temperature and intermediate service temperature tests, respectively. Superpave rutting parameters are presented in Table 5.



Figure 8: Dynamic Shear Rheometer.



Figure 9: (a) 25 mm parallel plate geometry (left), and (b) 8 mm parallel plate geometry (right).

Binder Sample	Value	Test Temperature (°C)	Specification
Unaged binder	G*/sinð	High Service	≥ 1.00 kPa (0.145 psi)
RTFO-aged binder	G*/sinð	High Service	≥ 2.20 kPa (0.319 psi)
PAV-aged binder	G*.sind	Intermediate Service	≤ 5000 kPa (725 psi)

Table 5: Superpave specifications for DSR test.

4.2.7 Bending Beam Rheometer (BBR) Test

During service life, asphalt binders get oxidized over time, which increases the solid fraction of the binder. This results in grade bump or stiffening of the binder; thus, the binder becomes more susceptible to low-temperature fatigue cracking. In the BBR test, these phenomena are mimicked and low-temperature stiffness and relaxation properties of asphalt are measured. AASHTO T 313 was followed in this study to conduct a BBR test and AASHTO PP 42 was used to determine the low PG temperature of asphalt binder in the laboratory. This test was conducted on PAV-aged samples, and creep stiffness (S-value) and slope of the stiffness curve (m-value) were measured.

In this study, the test temperatures were -9 and -12 °C. Table 6 shows Superpave parameters for the BBR test. The following Figure 10 shows a typical BBR machine and test setup for a sample. BBR tests were performed at Bituminous Laboratory in Lyles School of Civil Engineering at Purdue University.

Parameters	Test Temperature (°C)	Specification
"m-value" at 60 second	Low Service Temperature +10 °C	≥ 0.300
Stiffness at 60 seconds	Low Service Temperature +10 °C	≤ 300 MPa

Table 6: Superpave specifications for BBR test.



(c)

(d)

Figure 10: a) Bending Beam Rheometer, b) poured samples, c) test samples, and d) Loading Arrangement.

4.2.8 Multiple Stress Creep Recovery (MSCR) Test

This test method uses a well-established creep and recovery test concept to evaluate the permanent strain in the asphalt binder. In the MSCR test (AASHTO T 350) method, one-second creep is applied using the DSR machine which is followed by a nine-second recovery from the applied creep load. The stresses used were 0.1 kPa and 3.2 kPa, and the test temperature was 64 °C. The following Figure 11 shows the load application pattern in the MSCR test. Two important output is measured in this test: non-recoverable creep compliance (Jnr) and percent recovery (%R). Jnr value is the amount of residual strain left in the binder specimen within the linear and non-linear viscoelastic range at high temperature and high-stress levels. Mathematically, it is residual strain after a creep/recovery cycle divided by applied stress. %R is the measurement of the binder's recovery to its original position after stress is released.



Figure 11: Determination of the percent recovery and Jnr value (40).

4.2.9 Frequency Sweep

This test is performed to measure the viscoelastic properties of asphalt binders in the linear viscoelastic region using DSR. This test is run at a varying temperature at a constant 0.1 % oscillatory shear loading over a frequency range of 0.2-30 Hz. The obtained results can be used to plot a rheological master curve using the time-temperature superposition principle. The results from each test temperature are shifted to align with the results from a reference temperature (Figure 12) resulting in a master curve. One can determine the mechanical properties of binder from this curve over a wide range of reduced (shifted) frequencies.



Figure 12: Master curve using frequency sweep test data (41).

4.2.10 SARA Analysis

The acronym SARA stands for Saturates, Aromatics, Resin, and Asphaltenes. These four constituents make the whole binder. They are not a single compound rather a group of different compounds thus removes the complexity of studying the chemistry of asphalt binders. Any changes in the rheological properties of the binder are due to certain chemical alterations or arrangements of these chemical components. These components are briefly explained next.

Saturates: This fraction was not absorbed in activated alumina and eluted with n-heptane solvent. This is the first constituent that comes out of column chromatography.

Aromatics (Naphthene aromatics/cyclics): They are absorbed in activated alumina in the presence of n-heptane and eluted with toluene. This is the second fraction that is obtained from column chromatography.

Resins (polar aromatics): This group desorbed from calcined CG-20 alumina absorbent after the saturates fraction and naphthenic aromatics fraction has been removed, using toluene: methanol (50:50, by volume) and trichloroethylene eluate. This is the last group that is eluted from the column.

Asphaltene (alkane insoluble): This is the solid fraction of asphalt binder that is separated after the digestion of the asphalt in n-alkane (here, n-heptane).

Maltenes (petrolens): These are the constituents of asphalt that are soluble in n-alkanes/branched alkanes (here, n-heptane).

Other than the aforementioned four SARA components, Napthene is another minor component. Napthenes are a group of hydrocarbon ring compounds of the general formula, CnH₂n, derivatives of cyclopentane, and cyclohexane, found in certain petroleum stocks.

In this study, an IATROSCAN was used to conduct the SARA analysis. It quantitatively analyzes organic mixtures separated on thin-layer chromatography (TLC) and detected by Hydrogen Frame Ionization System (FID). At first, SARA components are separated on an exclusive thin layer chromatography media e.g., CHROMAROD (thin layer quartz rod). Then, they are charged as both negative and positive by the energy of the Hydrogen Flame. The negative ions (-) flow to the Burner and the positive ions (+) flow to the Collector Electrode due to the electric field loaded between the FID electric poles. This phenomenon initiates a current flow between the Burner and the Collector, which is proportional to the mass of components being ionized in the Hydrogen Flame. The ion current is amplified by the FID circuit, and the components are quantitatively measured and recorded (42). Figure 13 illustrates the working principle of an IATROSCAN.



Figure 13: IATROSCAN working procedure (42).

4.2.11 FTIR Test

FTIR test is a spectroscopy technique applied on an asphalt binder to detect any presence or change in quantities of functional groups that might have occurred due to the modification (43). When the natural vibrational frequency of a specific molecule matches the frequency of the IR radiation, the molecule absorbs the energy and increases the amplitude of the vibrational motion, and is detected as a peak in the interferogram. Figure 14 shows the mapping tool that lists the functional groups and the wavenumbers of their peak occurrences. Figure 15 displays a schematic diagram of the FTIR spectrometer and its working principle.



Figure 14: The approximate regions where various common types of bonds absorb (stretching vibrations only) (44).



Figure 15: Schematic diagram of FTIR spectrometer (44).

The sample preparation was the important step of this test. Improper preparation of the sample could result in an erroneous result (45). In this study, disposable Real Crystal IR cards were used for preparing the samples. The IR cards contained a KBr substrate (15 mm). The steps involved in preparing the samples were as follows:

- Asphalt binder was heated at 150°C to make sufficiently fluid. A speck of asphalt binder was dropped right outside the aperture and dragged over the KBr substrate for being completely coated.
- For this study, a KBr beam splitter from a spectrum range of 350 to 7400cm-1 was used. The samples were run over 50 scans at 4cm-1 resolution for 30 seconds. The test was executed at a relative humidity below 5%. Before starting the test, a blank card was scanned to have a background. A Nicolet 8700 spectrometer was used in this study

(Figure 16). The data acquisition and analysis were done using the Omnic 6.2 software. The absorbance spectrum was used in this for analysis.



Figure 16: Nicolet 8700 spectrometer.

4.2.12 Sessile Drop (Optical Contact Angle) Test

The Sessile Drop test was conducted to determine the contact angles of glass-coated asphalt binders with the three reference solvents (water, ethylene glycol, and formamide). The SFE parameters (work of cohesion, work of adhesion, compatibility ratio, etc.) of different aggregate and binder systems were then estimated by using the Good-van Oss-Chaudhury theory and the Young-Dupre equation (46). In this method, a droplet of a reference liquid was placed on a solid surface (aggregate surface or glass plate coated with asphalt binder). The shape of the drop and contact angle between the liquid and solid surface was measured by an OCA. For each drop, more than 100 contact angles on each side of the drop were measured to get a very precise measurement. The volume of the drop was regulated and the same drop volume was used for all specimens. The following Figure 17 shows the Sessile drop test setup used in this study.



Figure 17: Sessile Drop test set up.

4.2.12 Texas Boiling Test

The Texas Boiling Test is a simple and quick method of evaluating the moisture susceptibility of an asphalt mix. In this test, the stripping of asphalt binders is measured by visual observation after an asphalt mix is subjected to heat in the presence of water for a specified time. ASTM D 3625 was followed to perform this test. The aggregate size used in this test was passing 3/8 inch retained on No. 4. Figure 18 shows a few steps from the performed tests. Figure 19 shows guidelines given by the Texas Transportation Institute (TTI) for determining what percentage of asphalt was remaining on the surface of the aggregates, which was followed in this study (47).



Figure 18: Texas Boiling Test; separating coated aggregates (left), boiling the sample (middle), and air drying after boiling (right).



Figure 19: Rating board for Texas Boiling Test (47).

4.2.13 Acid Number Test

All the binder samples were tested for measuring their acid number values (i.e. pH). Asphalt binders may inherently contain some organic acids and also Evotherm[®] P25 and Rediset[®] have acid components in them. The methodology followed here was originally proposed by the researchers at the Western Research Institute (WRI) *(48)*. About 5 gm of binder sample was dissolved in 30 ml of Toluene (HPLC grade) at first. The solution was eluted with 30 ml of water in two steps (15 ml each). The aqueous portion was separated and used to measure the acid number of the sample. Before taking readings for every sample, the probe was calibrated using three buffer solutions. Figure 20 explains a few steps followed in this study.



Figure 20: Acid number test: sample preparation (left), separation of aqueous layer from asphalt layer (middle), and measuring acid number (right).

At the beginning of this study, the research team selected three dosages of each of the additives for blending them with each of the three asphalt binders from two sources. Preliminary test results suggested that each of these additives had a dosage value beyond which the reduction in viscosities was intuitively insignificant. For example, viscosities for Source 1 PG 70-22 blended with 0, 1.25,

1.5, and 1.75% of Sasobit[®] at 135 °C were 1195.89, 1147.56, 1075.11, and 1096.89 mPa.s, respectively. Thus, the optimum dosage of Sasobit[®] was selected to be 1.5 % by weight of the binder, which was initially designated as A2. Similarly, the optimum dosages of Advera[®], Evotherm[®], and Rediset[®] were selected as 6.0, 0.5, and 0.75 % by weight of the binder, respectively. The designations of Advera[®], Evotherm[®], and Rediset[®] modified binders with their optimum dosages include "A5", "A8", and "A11", respectively, at the end of their nomenclatures. All of these dosages were within the recommended dosages of the additive manufacturers. The reason for choosing RV test results as a prefatory study was to comply with one of the main goals of this study i.e., reduction in mixing and compaction temperatures. Thus, the researchers focused on further testing the selected WMA additives at their optimum dosage levels. Consequently, laboratory test results of samples ending with A0, A2, A5, A8, and A11 are presented and discussed thoroughly in Chapter 5. Moreover, test results further revealed that viscoelastic and mechanistic properties of binders from the two selected sources did not vary significantly. Therefore, some of the laboratory tests (BBR, and SARA fractional analysis) of this study focused on the neat (PG 64-22) and one polymer-modified (PG 76-22) binders from Source 1.

5. ANALYSIS AND FINDINGS

5.1 Penetration Test

Penetration tests were completed for all the samples from both sources. The results of Source 1 and Source 2 binder samples are graphically represented in Figures 21 and 22, respectively. It is evident that at 25° C, the binders get stiffened upon the addition of the additives with a few exceptions. Sasobit[®] stiffened the binders most followed by Advera[®]. Both Evotherm[®] and Rediset[®] modifications showed similar stiffness for all samples. It is expected that stiffer binders would exhibit better moisture resistance.



Figure 21: Penetration test results of source 1 samples.



Figure 22: Penetration test results of Source 2 samples.

5.2 RV Test results

The RV tests were completed for all binders from both sources. This test was performed from 135 to 180 °C at a 15 °C interval. Figures 22 through 28 show the results from the RV test for both sources. PG 64-22 and PG 70-22 binder samples from Source 2 were softer than Source 1. However, PG 76-22 binder samples from Source 1 were softer. Test results showed that both Sasobit[®] and Rediset[®] reduced the viscosity for all the binder samples from both sources compared to the unmodified samples. These reductions increased as the binders grades got high. Figures 22 through 28 also suggested that the reductions in viscosities were noticeable around mixing and compaction temperature ranges typical for WMA. The Advera[®] modified samples had a higher viscosity than unmodified binders in all cases. Because water molecules in its lattice structure evaporated during blending action. It was found in the literature that foam-based WMA technologies increased binders viscosity as the water evaporated at the time between mixing and viscosity test (*38, 49*). Evotherm[®] reduced the viscosity to a lesser extent.



Figure 23: Viscosity temperature graphs for Source 1 PG 64-22 samples.



Figure 24: Viscosity temperature graphs for Source 1 PG 70-22 samples.



Figure 255: Viscosity temperature graphs for Source 1 PG 76-22 samples.



Figure 26: Viscosity temperature graphs for Source 2 PG 64-22 samples.



Figure 27: Viscosity temperature graphs for Source 2 PG 70-22 samples.



Figure 28: Viscosity temperature graphs for Source 2 PG 76-22 samples.

The mixing and compaction temperatures of HMA are expressed in terms of mixtures viscosities which are described in AASHTO T 312. In this study, the viscosity values for mixing and compaction temperatures are selected as 170 ± 20 and 280 ± 30 mPa.s, respectively. The determination of these temperature ranges is explained in ASTM D2493 entitled "Standard Viscosity-Temperature Charts for Asphalts." According to the ASTM specifications, these temperatures are to be determined from the intersections of viscosity-temperature line, viscosity ranges of 170 ± 20 mPa.s and 280 ± 30 mPa.s. Figure 29 illustrates a typical graph to determine these temperatures.

Figures 30 and 31 show the mixing and compaction temperatures obtained from viscosity test results. From Figure 25 it is obvious that Sasobit[®], Evotherm[®], and Rediset[®] reduced mixing temperature ranged in 1-5 °C except for PG 64-22 binder from Source 1. For this binder, these temperatures increased it by 1 °C. Advera[®] in all cases increased this value by 1-2 °C. Similarly, compaction temperatures for Sasobit[®], Evotherm[®], and Rediset[®] modified samples got reduced by 2-4 °C. Advera[®] increased this value by 1-2 °C for all the samples. It is also noted that the reductions in mixing and compaction temperatures are more prominent at higher graded binders.



Figure 29: Sample Graph for Determining Mixing and Compaction Temperatures.



Figure 30: Mixing and compaction temperatures for Source 1 samples.



Figure 31: Mixing and compaction temperatures for Source 2 samples.

5.3 DSR Test result

5.3.1 Rutting Factors

Figures 32 through 37 show DSR test results for all unaged binder samples from both sources. Overall, Sasobit[®] modified samples showed the highest G*/Sinδ value for all Source 1 and S2B3 samples. For S2B1 and S2B2 samples, Advera[®] modification showed the highest G*/Sinδ value. Overall, Sasobit[®] and Advera[®] modified samples were more rut resistant than other additive modified samples. Rediset[®] modified samples had the lowest G*/Sinδ values, therefore, more prone to rutting. In general, Advera[®] either increased this value except for S1B3 where it decreased. Evotherm[®] generally decreased G*/Sinδ for the binder samples besides S1B2 samples.



Figure 32: G*/sin δ vs temperature graph for Source 1 PG 64-22 samples.



Figure 33: G*/sin δ vs temperature graph for Source 1 PG 70-22 samples.



Figure 34: G*/sin δ vs temperature graph for Source 1 PG 76-22 samples.



Figure 35: G*/sin δ vs temperature graph for Source 2 PG 64-22 samples.



Figure 36: G*/sin δ vs temperature graph for Source 2 PG 70-22 samples.



Figure 37: G*/sin δ vs temperature graph for Source 2 PG 76-22 samples.

Table 7 contains pass/fail temperatures obtained from the DSR test data. All the failing temperatures were higher than their corresponding unmodified samples' high PG temperatures. All Sasobit[®] modified samples had higher failure temperatures comparing to their unmodified samples true high PG grade. All Rediset[®] modified samples had lower high PG temperatures comparing to their unmodified samples true high PG grade. Advera[®] reduced true high PG temperatures to only PG 76-22 binders from both sources. Evotherm[®] had a mixed effect on high PG temperatures e.g., it increased the high PG temperature of S1B2 by 0.90 °C, whereas it decreased this temperature of S2B2 by 0.15 °C. However, all binder samples modified with the WMA additives comfortably passed the Superpave criteria for rutting parameter when tested at the unaged condition (G*/sin $\delta \ge 1.00$ kPa).

Sample Name	High PG Temperature Before Modification (° C)	True High Critical Temperature (° C)	Sample Name	High PG Temperature Before Modification (° C)	True High Critical Temperature (° C)
S1B1A0	64.0	70.10	S2B1A0	64.0	69.90
S1B1A2	64.0	74.00	S2B1A2	64.0	70.40
S1B1A5	64.0	70.40	S2B1A5	64.0	70.93
S1B1A8	64.0	69.00	S2B1A8	64.0	69.40
S1B1A11	64.0	68.70	S2B1A11	64.0	68.35
S1B2A0	70.0	77.90	S2B2A0	70.0	77.65
S1B2A2	70.0	79.60	S2B2A2	70.0	78.40
S1B2A5	70.0	78.70	S2B2A5	70.0	79.90
S1B2A8	70.0	78.80	S2B2A8	70.0	77.50
S1B2A11	70.0	75.60	S2B2A11	70.0	76.90
S1B3A0	76.0	81.20	S2B3A0	76.0	83.80
S1B3A2	76.0	82.60	S2B3A2	76.0	85.63
S1B3A5	76.0	78.50	S2B3A5	76.0	83.70
S1B3A8	76.0	80.10	S2B3A8	76.0	83.95
S1B3A11	76.0	76.50	S2B3A11	76.0	82.25

Table 7: Pass/fail temperatures from DSR test on unaged samples from both sources.

5.3.2 Fatigue Factors

This test was performed only on a limited number of PAV-aged binder samples using an 8 mm plate geometry setup. Figure 38 shows that both Evotherm[®] and Rediset[®] modified samples passed intermediate temperature (IT) (25 °C). However, Sasobit[®] and Advera[®] modified samples passed 28 °C. Similar observations were found in literature and it was reported that Sasobit[®] adversely affected the intermediate temperatures by increasing G*.sinδ value (*11,50*). The critical IT for unmodified, Sasobit[®], Advera[®], Evotherm[®], and Rediset[®] modified samples were 24.95, 25.05, 26, 24.35, and 23.3 °C



Figure 38: |G*|.sin & vs temperature graph for Source 1 PG 64-22 samples.

5.4 Low-Temperature Cracking

BBR tests were conducted to measure the low-temperature stiffness and stress relaxation properties of asphalt binders. Two parameters, S-value (creep stiffness) and m-value (the slope of the stiffness curve) were determined at the 60s. Since all three binders had a low PG temperature of -22°C, the neat binder (PG 64-22) and one polymer modified binder (PG 76-22) were investigated for low-temperature resistance. Figures 39 and 40 show stiffness vs. temperature plots for both S1B1 and S1B3 samples. It was observed that Advera[®] in all cases increased unmodified binders' S-value, whereas other additives could not exhibit such a trend. However, they all met Superpave S-value (< 300MPa) criterion. For PG 64-22 and PG 76-22 samples, the lowest creep stiffnesses were observed for Rediset[®] and Sasobit[®] modified samples, respectively. Figures 41 and 42 show m-value vs temperature plots for S1B1 and S1B3 binder samples. Only S1B1A2 and S1B1A5 could not meet the Superpave m-value criterion (≤ 0.300) at -12 °C, which was obvious in Figure 41. For PG 64-22 and PG 76-22 samples, the highest m-values were observed for Rediset[®] and un-modified samples, respectively. Similarly, the lowest m-values were observed for Sasobit[®] and Evotherm[®] modified samples, respectively. Table 8 shows BBR extrapolated failure temperatures along with both S- and m-values for S1B1 and S1B3 samples. Failure temperatures were calculated using linear extrapolation for both S- and m-values. The highest temperature was chosen for samples with low PG temperature. For example, failure temperatures for S1B1A2 samples were calculated at -28.50 and -21.36 °C based on S- and m-values, respectively. Then, -21.36 °C was taken as low PG temperature for the S1B1A2 sample. It was observed that Rediset[®] and Advera® decreased low PG temperatures for S1B1 and S1B3 binder samples, respectively.



Figure 39: Stiffness vs temperature plot for Source 1 PG 64-22 samples.



Figure 40: Stiffness vs temperature plot for Source 1 PG 76-22 samples.



Figure 41: m-value vs temperature plot for Source 1 PG 64-22 samples.



Figure 42: m-value vs temperature plot for Source 1 PG 76-22 samples.

Table 8: BBR test results for tested samples.

Sample	Low PG Temperature Before Modificationon (° C)	True Low PG Temperature (°C)
S1B1A0	-22.0	-22.41
S1B1A2	-22.0	-21.36
S1B1A5	-22.0	-21.59
S1B1A8	-22.0	-22.22
S1B1A11	-22.0	-23.45
S1B3A0	-22.0	-26.10
S1B3A2	-22.0	-25.63
S1B3A5	-22.0	-26.75
S1B3A8	-22.0	-23.80
S1B3A11	-22.0	-25.32

5.5 Creep Recovery

For RTFO-aged binders, AASHTO M 332 specifications consider the J_{nr} values instead of G*/sin δ based on the traffic condition. Table 9 shows the specific traffic loading conditions for specific Jnr limits. In Table 9, the high and low critical temperatures of the asphalt binders are represented by XX and YY, respectively. Traffic conditions are represented by S (Standard), H (Heavy), V (Very Heavy), and E (Extreme).

J _{nr} (kPa ⁻¹)	Criteria MSCR Grading
$J_{nr} \le 4.5 \text{ and } > 2.0$	PG XXS-YY (S: Standard)
$J_{nr} \le 2.0 \text{ and } > 1.0$	PG XXH-YY (H: Heavy)
$J_{nr} \leq 1.0$ and > 0.5	PG XXV-YY (V: Very Heavy)
$J_{nr} \leq 0.5$	PG XXE- YY (E: Extreme)

Table 9: Minimum Jnr value range for MSCR grading.

Jnr,diff	Stress Sensitivity (AASHTO M 332 Criterion)
$\leq 75\%$	Yes
> 75 %	No

According to AASHTO M 332, if the Equivalent Single Axle Load (ESAL) is less than 10 million then the traffic condition is called Standard. For an ESAL value between 10-30 million, traffic condition is denoted as Heavy. If it is greater than 30 million, then it is called Very Heavy, and the last one is designated as Extreme traffic condition, where ESAL is greater than 30 million along with standing traffic. According to AASHTO M 332, the stress sensitivity predicts the performance of binders at high-stress levels, which is the changes in J_{nr} values at 0.1 kPa and 3.2 kPa relative to J_{nr} at 0.1 kPa. According to AASHTO M 332, the binder is stress-sensitive if this change is less than or equal to 75%, otherwise stress insensitive. Equation 1 was used to calculate the stress sensitivity of the tested samples. Table 10 represents the stress sensitivity criteria of the MSCR test method.

$$J_{nr,diff} = (J_{nr,3.2kPa} - J_{nr,0.1kPa}) / J_{nr,0.1kPa} * 100$$
(1)

The MSCR tests were conducted at 64 °C at two different stress levels. Figure 28 shows the percent recovery vs. stress curve for Source 1 samples. The percent creep recovery (%R) measures the extent of asphalt specimen returns to its original position after the load has been removed. In all cases, Advera[®] modification decreased %R than the unmodified binders. Both Sasobit[®] and Evotherm[®] increased this parameter comparing to unmodified samples. Only for PG 64-22 and PG 70-22 binder samples, Rediset[®] decreased the %R value. The highest percent recovery was observed for all Sasobit[®] modified samples, and the lowest recovery was observed for Rediset[®] modified samples except for the S1B3 sample. Figures 29 shows the non-recoverable creep compliance vs stress curve for Source 1 samples. The J_{nr} value is the amount of residual strain left in the binder within the linear and nonlinear viscoelastic range at high temperatures and stress levels. Figure 29 shows that Sasobit[®] modification in all cases reduced both J_{nr,0.1kPa} and J_{nr,3.2kPa} for all samples. All other modifications (except S1B3A8) increased them compared to their respective unmodified samples.



Figure 43: Percent recovery vs stress for Source 1 samples.



Figure 44: Non-Recoverable Creep Compliance Jnr vs stress for Source 1 samples.

The stress sensitivity was estimated using Equation 1 and data is presented in Table 10. From Table 10, it is observed that all samples met the AASHTO M 332 stress sensitivity criteria besides S1B3A2. Thus, almost all of these modified asphalt binders are not excessively stress-sensitive to unexpected heavy loads or unusually high temperatures. Table 11 shows that all PG 64-22 samples failed to meet the %R criterion according to AASHTO T 350, while both PG 70-22 and PG 76-22 samples meet this criterion. They were also graded according to their corresponding to their J_{nr} values following AASHTO M332 guidelines. All the PG 64-22 samples were graded as PG 64S-22 regardless of modification. For both PG 70-22 and PG 76-22 samples, both Advera[®] and Rediset[®] degraded ESAL capacity from 'Extreme' to 'Very Heavy,' other modifications did not change the ESAL capacity.

Sample name	Jnr, 0.1 kPa	Jnr, 3.2 kPa	Jnr, diff	Stress Sensitivity (AASHTO M 332)	R100	R3200	Rdiff	MSCR line equation: 29.371*(Jnr3.2kPa) ^(2633)	% Recovery (Meets AASHTO T 350)	MSCR Grade
S1B1A0	2.19	2.39	9.07	YES	4.03	0.80	80.11	31.14	NO	PG 64S-YY
S1B1A2	1.75	2.14	22.38	YES	11.01	1.71	83.91	25.49	NO	PG 64S-YY
S1B1A5	1.97	2.13	8.59	YES	3.91	0.79	79.71	31.23	NO	PG 64S-YY
S1B1A8	2.25	2.47	9.91	YES	4.27	0.71	83.47	32.20	NO	PG 64S-YY
S1B1A11	2.58	2.96	9.11	YES	3.19	0.22	93.10	43.97	NO	PG 64S-YY
S1B2A0	0.29	0.34	17.79	YES	62.19	56.85	8.59	10.14	YES	PG 64E-YY
S1B2A2	0.19	0.25	30.49	YES	67.11	60.51	9.84	9.97	YES	PG 64E-YY
S1B2A5	0.45	0.52	17.39	YES	52.54	45.79	12.85	10.73	YES	PG 64V-YY
S1B2A8	0.33	0.40	20.35	YES	61.08	54.95	10.04	10.23	YES	PG 64E-YY
S1B2A11	0.73	0.93	28.20	YES	49.97	39.32	21.32	11.17	YES	PG 64V-YY
S1B3A0	0.33	0.43	30.47	YES	48.90	36.81	24.74	11.37	YES	PG 64E-YY
S1B3A2	0.12	0.22	80.32	NO	65.09	47.33	27.28	10.64	YES	PG 64E-YY
S1B3A5	0.38	0.51	34.54	YES	49.42	35.83	27.60	11.45	YES	PG 64V-YY
S1B3A8	0.26	0.33	27.88	YES	53.55	43.45	18.82	10.88	YES	PG 64E-YY
S1B3A11	0.50	0.72	44.11	YES	53.22	37.31	29.90	11.33	YES	PG 64V-YY

Table 11: MSCR database for WMA modified Source 1 binders at 64 °C.

5.6 SARA analysis

The percent of SARA fractions were reported in Figure 45 as a stacked chart. SARA fractions were determined by IATROSCAN for unaged S1B1 and S1B3 binder samples. Sasobit[®] increased saturates fraction in PG 76-22 binder but did not change any fractions in PG 64-22 binder. Whereas, Advera[®] increased the asphaltene fraction and reduced the aromatics fraction for both binders. These results conformed with DSR test results on unaged samples, as Advera[®] modified samples showed higher G*/Sin\delta values. Both Evotherm[®] and Rediset[®] increased Resin content in PG 76-22 binder only.



Figure 45: IATROSCAN test for SARA fraction.

5.7 FTIR test results

FTIR tests were performed on all the binder samples from both sources. Test results are illustrated in Figure 47. Sasobit[®], Evotherm[®], and Rediset[®] WMA additives could not introduce any new groups to neat binder samples. Only changes in concentration of already existed indices were observed. However, Advera[®] modification makes sulfoxide (S=O) groups (1030 cm⁻¹) to neat binders, which was consistent in all Advera[®] modified samples. Another peak was observed at around 3400 cm⁻¹, which indicated the formation of the hydroxyl (-OH) group in modified samples. The Advera[®] modified samples had also few peaks below 650 cm⁻¹, which was outside of this study premise.



Figure 46: Absorbance spectra for all samples from FTIR test.

5.7 Sessile Drop (Optical Contact Angle) Test results

Optical contact angles were measured using the Sessile Drop (SD) method for all the modified and neat binders for both sources. The main principle of the SD technique is that the probe liquid creates a vertically symmetric drop on the sample surface and the shape of the drop is governed by gravity and interfacial surface tension (46). If the contact angle is close to zero, which means solvent spreads over the sample i.e., complete wetting. If it is close to 90 °, then the wetting is good; and a contact angle higher than 90 ° indicates poor wetting (51, 52). Three probe liquids: water, ethylene glycol, and formamide were used to measure the contact angles. For every drop, more than 100 contact angles were measured and the final measurement was an average of three angles for every liquid-sample combination. Figures 47 and 48 show contact angles for both sources. For all the samples, contact angles for water were between 95-100 ° and for other probe liquids, these angles varied between 78-82 °.



Figure 47: Optical contact angle measurement using Sessile Drop method Source 1 samples.



Figure 48: Optical contact angle measurement using Sessile Drop method Source 2 samples.

Figures 49 and 50 show acid, base, Lifshitz-van der Waals components, and total SFE for Source 1 and 2 binder samples, respectively. For all the samples, the Lifshitz-van der Waals component was much higher than the other components. Similar results were also observed in the literature (25, 53). No trend for acid and base components was observed. Total SFE was calculated using the three aforementioned components. Almost in all cases, higher SFE was observed for either Evotherm[®] or Rediset[®] modified asphalt binders for both sources besides the S1B3 binder, where the highest SFE was calculated for Advera[®] modified sample. The lowest SFE was calculated for Sasobit[®] modified samples even SFE got reduced comparing to their respective unmodified samples. Higher SFE indicates higher adhesive bond strength i.e., a strong bond between asphalt binder and aggregates. So, both Evotherm[®] and Rediset[®] modified samples had strong moisture resistance, which bolstered the presence of an anti-stripping agent in them. Whereas, Sasobit[®] decreased the moisture resistance upon modification.



Figure 49: Surface free energy components for Source 1 samples.



Figure 50: Surface free energy components for Source 2 samples.

Work of cohesion was also calculated from SFE values and presented in Figures 49 and 50. Work of cohesion is twice of SFE and higher this value indicates more works need to be done to break such bond, which indicates less moisture susceptibility. All the additives besides Sasobit[®] offered higher work of cohesion. Both Evotherm[®] and Rediset[®] modified samples possessed quite higher work of cohesion comparing to unmodified samples. The Advera[®] additive also increased the work of cohesion in most of the samples, which means Advera[®] modified samples also had better moisture resistance compared to unmodified samples.



Figure 51: Work of cohesion for Source 1 samples.



Figure 52: Work of cohesion for Source 2 samples.

5.8 Texas Boiling Test results

Texas Boiling Test was performed on a total of 120 samples yielded from four different aggregates e.g., Limestone, Sandstone, Dolomite, and Gravel; and 30 binder samples. It is a quick test for determining moisture damage compared to other testing methods. The moisture damage was

inspected visually using the chart provided by TTI (Figure 19) in terms of % of asphalt retention. Figure 53 shows the percent of asphalt retention in the Texas Boiling Test performed on Source 1 samples. Similarly, Figure 54 shows asphalt percent retention for Source 2 samples.



Figure 53: Texas Boiling test results for Source 1 samples.



Figure 54: Texas Boiling test results for Source 2 samples.

Regardless of the aggregates, all the Rediset[®] and Evotherm[®] modified samples showed very high asphalt retention (> 80%) at the end of the test. According to their manufacturers, they both contain anti-stripping, which seemed to work well against moisture damage. Similar results were also observed in the contact angle measurement test. Both Sasobit[®] and Advera[®] showed mixed results. For example, Sasobit[®] improved moisture resistance for the S1B2 binder, whereas for the S2B2 binder it deteriorated moisture resistance. Overall, they both deteriorated the moisture resistance of the base binder. Among four aggregates, Limestone performed better followed by Dolomite. Because they are both basic, which makes them compatible with acidic asphalt binder. Gravel was found to be the worst aggregate followed by Sandstone.

5.9 Acid Number Test results

Acid number tests were performed and the results for both sources were shown in Figure 55. This test showed that all the Advera[®] modified samples had higher pH values compared to their corresponding unmodified samples except S1B2A5, where it decreases. It is expected that Advera[®] will increase the pH values of the binder because of its basic ingredient (Na₂Al₂Si₂O₈.xH₂O). However, Rediset[®] increased pH values compared to the pH of unmodified samples. For example, S2B3A0 had a pH value of 5.972, and it climbed up to 8.368 when mixed with Rediset[®] (S2B3A11) though it contains fatty acids. On the other hand, Evotherm[®] expectedly reduced pH for all unmodified binders, as it contains fatty acids. It can also be expected that all Evotherm[®] modified samples would have good compatibility with basic aggregates. Similar results were observed both from the contact angle measurement test and Texas Boiling Test. Sasobit[®] reportedly decreased pH values for all PG 64-22 and PG 70-22 binder samples, whereas it increased pH values for both PG 76-22 binder samples. These variations were minute for all the samples except for the S1B1 sample. This can be presumed because Sasobit[®] is wax containing a long alkane chain.



Figure 55: Acid number test results for all samples.

6. CONCLUSIONS

The main objective of this study is to assess the feasibility of selected additives in producing WMA binders in Arkansas. To achieve this objective, three ARDOT approved Performance Grade (PG) binders (PG 64-22, PG 70-22, and PG 76-22) were collected from two different sources (S1 and S2). Only PG 64-22 was a neat binder, and PG 70-22 and PG 76-22 binders were modified with polymer. Four different aggregates e.g., Limestone, Sandstone, Dolomite, and Gravel were also collected from ARODT approved local sources. Six different asphalt binders were modified by four additives: Sasobit[®], Advera[®], Evotherm[®], and Rediset[®].

To encompass a wide range of data from different viewpoints, empirical tests, Superpave tests, PG plus tests, chemical analysis, and some fundamental advanced science-based tests were performed in this study. Asphalt binders and additives were blended at 150 °C for two hours (one-hour preheating followed by one-hour blending). The doses for all additives followed in this study were as per manufacturers' recommendations, preliminary test data, and prior experiences of the research team and other researchers. The empirical test included the Penetration test. Superpave tests such as Rotational Viscometer (RV), Rolling Thin-Film Oven (RTFO), Pressure-Aging Vessel (PAV), Dynamic Shear Rheometer (DSR), and Bending Beam Rheometer (BBR) were performed to evaluate the rheological properties of the samples. PG plus test includes only Multiple Stress Creep Recovery (MSCR) test. Asphalt chemistry was evaluated using SARA fraction analysis using the IATROSCAN tool, FTIR analysis, and acid number test. To evaluate moisture susceptibility of binder samples Optical Contact Angle (OCA) was measured using the Sessile Drop technique, and Texas Boiling Test was also performed. All the tests were performed as per their corresponding standards. The slight modifications adapted in this were necessary to fulfill the project's goal. Based on the aforementioned test results following conclusions can be drawn:

- 1. Unmodified PG 76-22 binders were stiffest followed by PG 70-22 and PG 64-22 binders. Sasobit[®] stiffens all the binders the most, and it is followed by Advera[®].
- 2. Both Rediset[®] and Sasobit[®] decreased the viscosity of unmodified samples. The highest reduction was observed for Rediset[®], and it was followed by Sasobit[®]. Thus, binders modified by both of these binders can easily coat aggregates.
- 3. For all modified binder samples, mixing and compaction temperatures decreased by 1-5 °C. Higher reductions were observed for both Sasobit[®] and Rediset[®] modified samples, and lower reduction was observed for Advera[®] followed by Evotherm[®] modified samples. Therefore, both Sasobit and Rediset[®] have a good potential to decrease production costs.
- 4. Both unaged Sasobit[®] and Advera[®] modified samples showed higher |G*|/sinδ values compared to their unmodified samples, therefore more resistant to rutting. Unaged Rediset[®] modified samples showed least |G*|/sinδ among all the tested samples i.e., high rut potential. In most cases, Evotherm[®] reduced this rutting parameter. No grade bump was not observed compared to unmodified binders' true grade.
- 5. Rediset[®] modified samples performed better in terms of fatigue cracking at intermediate temperature, and it was followed by Evotherm[®].
- 6. All the samples met Superpave stiffness criteria (≤ 300 MPa). Only Evotherm[®] and Rediset[®] modified PG 64-22 samples have an m-value higher than 0.300 at -12 °C. Thus, both these additives will have better resistance against low-temperature fatigue cracking.

- 7. None of the additives changed MSCR grading for PG 64-22 binder. The unmodified PG 76-22 binder sample could sustain 'Extreme' traffic conditions, which were unaltered upon the addition of Sasobit[®] and Advera[®] only.
- 8. The Advera[®] additive increased asphaltene and reduced aromatics contents for both PG 64-22 and PG 76-22 binder samples. Thus, it made the sample stiffer resulting in a higher |G*|/sinδ value i.e., higher rut resistance. Both Evotherm[®] and Rediset[®] could not bring any alternation in SARA fractions. Sasobit[®] increased saturates and decreased resin contents only for PG 76-22 binder samples.
- 9. Advera[®] introduced sulfoxide (S=O) groups (1030 cm⁻¹) to unmodified binders. Other additives changed the concentration of the groups only.
- 10. Contact angles for water were highest among all three probe liquids. Total SFE was governed by the Lifshitz-van der Waals component for all the samples.
- 11. Higher SFE, as well as work of cohesion, were determined for both Rediset[®] and Evotherm[®] modified samples. Thus, both of them reduced the moisture susceptibility of the samples. The lowest SFE and work of cohesion was calculated for Sasobit[®] modified samples, thus Sasobit[®] modified samples would be more prone to moisture damage.
- 12. All the Evotherm[®] and Rediset[®] modified samples showed very good moisture resistance in Texas Boiling Test. Sasobit[®] and Advera[®] modified samples showed poor resistance against moisture damage. Both Limestone and Dolomite showed better performance overall i.e., increased resistance against moisture damage. Gravel was the least performed aggregates followed by Sandstone.
- 13. pH value decreased as the binder grade increased. Advera[®] increased pH for all binders. Evotherm[®] decreased pH; however, pH values ascended upon the addition of Rediset[®]. Lower pH values indicate acidic samples, which will be more compatible with basic aggregates. Thus, Evotherm[®] modified samples are expected to have better moisture resistance.

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APPENDIX A: Binders Rheological Properties

		Viscosity	(mPa.s)	N	lixing	Compaction		
Samples	125% 0	150° a	165%	100 ° a	temp	eratures	temp	eratures
	135 C	150 C	165 C	180 C	Low	High	Low	High
S1B1A0	457.29	218.75	104.17	50.00	153	158	143	148
S1B1A2	435.42	221.88	113.54	60.42	154	159	143	148
S1B1A5	554.17	262.50	131.25	68.75	157	162	146	151
S1B1A8	473.96	233.33	116.67	62.50	155	160	145	149
S1B1A11	458.33	228.13	116.67	62.50	154	160	144	149
S1B2A0	1195.89	568.06	291.67	163.89	176	183	164	169
S1B2A2	1075.11	518.06	273.61	154.17	175	181	162	167
S1B2A5	1202.67	590.28	301.39	162.50	176	182	165	170
S1B2A8	1241.56	586.11	298.61	162.50	175	180	164	169
S1B2A11	1025.33	498.96	259.38	139.58	173	179	161	164
S1B3A0	1675.22	883.33	480.56	281.94	191	198	177	184
S1B3A2	1482.00	807.29	456.25	266.67	189	196	176	182
S1B3A5	1638.33	938.54	538.54	317.71	195	202	181	187
S1B3A8	1571.00	839.58	469.79	279.17	191	198	177	184
S1B3A11	1267.83	677.08	388.54	239.58	187	195	172	179
S2B1A0	481.25	243.75	125.00	68.75	156	161	145	150
S2B1A2	431.25	210.42	106.25	50.00	153	157	142	147
S2B1A5	539.58	262.50	137.50	68.75	157	163	147	152
S2B1A8	460.42	217.71	108.33	52.08	153	159	143	147
S2B1A11	436.46	221.88	104.17	50.00	154	158	143	148
S2B2A0	1074.47	522.22	268.06	151.39	174	181	162	167
S2B2A2	879.17	431.25	229.17	125.00	169	176	158	163
S2B2A5	1119.67	545.83	287.50	162.50	175	184	164	169
S2B2A8	981.25	480.21	256.25	146.88	173	180	161	166
S2B2A11	903.13	435.42	220.83	125.00	169	176	158	162
S2B3A0	1651.67	795.97	412.50	237.50	186	193	173	179
S2B3A2	1497.83	720.83	375.00	212.50	183	189	170	176
S2B3A5	1931.33	892.71	469.79	262.50	189	195	176	182
S2B3A8	1619.58	814.58	410.42	228.13	185	191	172	178
S2B3A11	1514.83	729.17	381.25	215.63	184	190	171	177

A_Table 1 Mixing and compaction temperatures

APPENDIX B: Results for OCA test



B_Figure 1: A few representative samples for OCA test

B _	Table 1	Optical	Contact Angles	s for	Source 1	binder	samples
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Acabalt	Contact Angles (Degree) for different Probe Liquids							
Binders	Water	St. Deviation	St. Ethylene		Formamide	St. Deviation		
		Deviation	giycol	Deviation	01.56	Deviation		
S1B1A0	98.48	0.30	81.22	0.20	81.56	1.15		
S1B1A2	94.20	0.83	81.16	0.49	81.84	1.68		
S1B1A5	93.32	0.83	82.28	0.87	81.53	0.78		
S1B1A8	94.62	0.50	82.74	0.78	81.77	1.46		
S1B1A11	96.44	1.23	81.67	1.46	78.03	0.82		
S1B2A0	98.97	0.19	81.82	0.05	81.08	0.26		
S1B2A2	98.94	0.41	79.58	0.32	80.48	0.26		
S1B2A5	99.13	0.21	81.96	0.05	81.33	0.04		
S1B2A8	98.53	0.24	81.73	0.21	80.16	0.26		
S1B2A11	97.42	0.18	82.78	0.11	80.05	0.24		
S1B3A0	98.60	0.22	80.96	0.35	81.13	0.28		
S1B3A2	99.21	0.63	79.26	0.34	81.98	0.19		
S1B3A5	98.40	0.21	81.08	0.34	80.58	0.35		
S1B3A8	98.29	0.33	81.73	0.23	80.15	0.28		
S1B3A11	97.84	0.48	81.04	0.23	80.04	0.21		

Asphalt	Contact Angles (Degree) for different Probe Liquids						
Binders	Water	St. Deviation	Ethylene glycol	St. Deviation	Formamide	St. Deviation	
S2B1A0	100.25	0.02	83.90	0.31	83.25	0.36	
S2B1A2	99.75	0.13	82.09	0.05	82.37	0.13	
S2B1A5	99.32	0.14	83.30	0.14	81.36	0.34	
S2B1A8	95.07	0.35	80.74	0.12	80.53	0.40	
S2B1A11	98.95	0.11	83.45	0.22	81.52	0.28	
S2B2A0	98.76	0.27	83.18	0.15	81.36	0.23	
S2B2A2	98.49	0.25	81.75	0.16	82.23	0.35	
S2B2A5	99.19	0.20	83.18	0.13	81.10	0.25	
S2B2A8	99.07	0.36	83.32	0.27	81.29	0.36	
S2B2A11	97.99	0.21	79.81	0.26	77.54	0.80	
S2B3A0	99.04	0.21	83.22	0.25	80.70	0.34	
S2B3A2	99.19	0.20	81.88	0.11	82.15	0.10	
S2B3A5	98.55	0.13	82.97	0.31	80.28	0.17	
S2B3A8	98.50	0.09	83.42	0.32	81.28	0.09	
S2B3A11	98.77	0.07	79.79	0.10	78.80	0.44	

B_Table 2 Optical Contact Angles for Source 2 binder samples

APPENDIX C: Samples for FTIR test



C_Figure 1 FTIR test samples