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16. Abstract Since the introduction of warm mix asphalt (WMA) in the United States, a variety of different technologies and processes have been developed and used to achieve proper mixing and compaction at reduced temperatures compared to conventional hot mix asphalt. A better understanding of the effect of WMA additives and reduced aging on rheology of asphalt binders is a crucial step towards the successful implementation of WMA. This report presents the preliminary findings from a study conducted to investigate the influence of chemical WMA additives and reduced aging on the viscosity, stiffness, susceptibility to permanent deformation, fracture resistance, and thermal cracking resistance of asphalt binders. Short-term aged WMA binders have reduced stiffness compared to conventional binders due to the reduced mixing temperatures. However, preliminary results indicate that certain WMA additives tend to exacerbate the reduced stiffness of WMA while other WMA additives tend to compensate for this effect. In most cases, long-term aged WMA binders had a similar stiffness but similar or reduced strength compared to conventional binders at intermediate temperatures. Also, the long-term aged WMA binders had similar or slightly reduced resistance to low-temperature cracking compared to conventional binders. In most cases, the use of WMA with recycled asphalt rendered the asphalt binder slightly more susceptible to low-temperature cracking.			
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Interim Report: Binder Rheology and Performance in Warm Mix Asphalt

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Chapter 1. Literature Review

1.1 Introduction

Warm mix asphalt (WMA) refers to asphalt mixtures that are produced at temperatures that are between 35°C and 55°C lower (63 and 99°F lower) than those used to produce typical hot mix asphalt (HMA) (D'Angelo et al., 2008). The use of WMA has several advantages such as reduced energy and greenhouse gases to produce the asphalt mixture. A variety of different technologies have been developed in the recent past to lower the viscosity of the asphalt binder and allow proper mixing and compaction at these reduced temperatures.

Previous laboratory and field research has identified several likely benefits associated with the use of WMA mixtures. Chowdhury and Button (2008) present a summary of these benefits that include but are not limited to:

- significantly lower production and placement temperatures;

- less fuel/energy consumption, thus lowering fuel/energy costs;

- less aging of binder during plant mixing and placement, thus improving longevity of pavement life;

- reduced thermal segregation in the mat;

- decreased emissions/odors at the mixing plant and at the construction site during placement;

- decreased dust production due to lower temperatures and shorter heating time;

- extended paving season (i.e., paving during cooler weather);

- extended mix haul distance providing expanded market areas;

- paving in non-attainment areas;

- improved compaction, which is beneficial for stiff mixes, reclaimed asphalt pavement (RAP) mixes, low-temperature paving, and reducing compaction effort;

- improved working conditions for plant/paving crew;

- diminished consternation of public over emissions; and

- easier for plant site obtain permits that are close to urban areas.

Notwithstanding the benefits, successful implementation and use of WMA technology is contingent on the expectation that these mixtures must have the same or better long-term durability and performance as compared to an equivalent HMA. WMA was introduced in Europe in the late 1990s and in the United States in early 2004. Within the state of Texas, as of 2008, approximately 300,000 tons of WMA mixture was placed in approximately 8 districts (Rand, 2008). However, due to the short time span since the introduction of this technology, little or no evidence is available for the durability and long-term field performance of WMA. Furthermore, no studies have investigated the long-term effects of the chemical additives or modification processes used to produce WMA.

In general, the additives or technologies used to produce WMA fall into four broad categories:

- wax-based additives,

- emulsion- or surfactant-based additives,

- water-bearing additives, and

- direct foam

1.2 WMA Additives

The following is a summary from a literature review conducted to evaluate the four different warm-mix technologies and their effects on the durability of the WMA mixtures.

1.2.1 Wax-Based Additives

Wax- or fatty amide-based additives reduce binder viscosity when heated to temperatures above the additive's melting point and increase binder stiffness at service temperatures typically below the additive's melting point. One of the most commonly used additives in this category is a Fischer-Trop wax produced by Sasol with the commercial name Sasobit®.

According to Sasol (2008), Sasobit is a fine crystalline long chain aliphatic hydrocarbon, or simply wax. However, they claim that the Sasobit is different from the wax that is naturally found in asphalt binders. Butz et al. (2001) report that the predominant chain lengths of the hydrocarbons in Sasobit ranges from 40 to 115 carbon atoms, whereas those in bituminous paraffin waxes range from about 25 to 50 carbon atoms yielding lower melting points than Fischer-Trop waxes. They also report that the smaller crystalline structure of the Fischer-Trop wax compared to the paraffin wax reduces the brittleness at low pavement service temperatures.

Sasobit has a melting point range of 85°C–115°C (185°F–239°F) and is completely soluble in asphalts at temperatures above 115°C (239°F). It is available as large prills, small prills, or in a flaked form. Sasobit lowers the viscosity of the binder and also acts as a flow modifier in the mix. In the liquid state this allows the aggregates to move freely within the binder. Upon cooling, Sasobit crystallizes and forms a uniform network within the binder.

Sasobit can be blended with the asphalt binder and the modified binder can then be stored and used to produce asphalt mixtures at lower temperatures. Alternatively, the additive can also be added to the mix during production of the mixture. Typically the additive is added at the rate of 1.5% by weight of the binder to achieve a reduction in the mixing temperatures by 10°C (reduction of 18°F).

Hurely and Prowell (2005a) conducted a detailed investigation on WMA mixtures produced using Sasobit. They reported that the additive reduced the viscosity of the mix at temperatures lower than conventional mixing and compaction temperatures. The addition of Sasobit lowered the measured air voids in the gyratory compactor. Sasobit improved the compactability of the mixtures in both the SGC and the vibratory compactor. In terms of the mechanical properties, addition of Sasobit did not affect the resilient modulus of an asphalt mix compared to mixtures having the same PG binder. Hurley and Prowell (2009) also reported that the gyratory compactor was not sensitive to reduction in temperature, suggesting that the energy requirements for compaction of samples are not completely reduced.

In another study, Kristjansdottir (2006) reported that RTFO-aged binders with Sasobit were aged less as compared to conventional binders. The addition of Sasobit improved

compaction in the vibratory compactor over the control mixture for all binder, aggregate, and temperature combinations except for four cases that involved the use of Sasoflex mixes at various temperatures. Those four exceptions are believed to be due to the elastomer part of the Sasoflex, which may have stiffened the binder enough to increase air void levels.

Kristjandottir (2006) also reported that the addition of Sasobit had mixed results in terms of the moisture susceptibility compared to the corresponding control mixture. The addition of anti-stripping agent along with Sasobit produced the lowest rutting rate. This study also reported that the addition of Sasobit to the test mixes resulted in a marginal decrease in the failure potentials due to rutting, fatigue, and thermal cracking. Simply stated, there were no tangible benefits or differences between control mixes and the mixes with Sasobit or Sasoflex, with the exception of the decrease in the energy consumed for the preparation of mix. The findings were similar for both surface course mix and base course mix, i.e., this additive only acts as a compaction aid. The study reported that Sasobit modification increased the haulage time with an increase in the amount of additive added.

Diefenderfer and Hearon (2008) compared HMA with WMA mixtures produced using Sasobit for two different Superpave mixtures with a PG 64-22 asphalt binder. The authors also conditioned the aggregates at high temperatures in a high relative humidity environment for 0, 4, and 8 days. Results based on the tensile strength ratio test for moisture damage indicated that statistically the moisture damage resistance of WMA mixtures was not very different from HMA mixtures. This was also confirmed using the Hamburg wheel tracking device. The rutting and fatigue cracking resistance of the WMA and HMA mixtures was statistically the same based on the results from the APA and beam fatigue tests, respectively. Wasiuddin et al. (2007) and Hurley et al. (2009) reported that addition of Sasobit improved the rutting resistance of the WMA mixtures based on APA testing. Hurley et al. (2009) reported that WMA mixtures with Sasobit had similar indirect tensile strength ratios as compared to HMA mixtures when tested in accordance with AASHTO T-283. Consistent with these findings, Gandhi (2008) reported that $G*/\sin\delta$ of asphalt binders modified using Sasobit generally increased as compared to the neat asphalt binder.

Kanitpong et al. (2007) reported the use of Sasobit with neat and polymer modified asphalt binders. According to this study, the use of Sasobit improved the rutting resistance of the mixture. There was very little difference in the moisture damage resistance of the WMA mixtures as compared to the HMA mixtures.

Mallick et al. (2008) reported the use of Sasobit to produce WMA mixtures with high RAP content (75%). The air voids in the HMA and the WMA mixtures were very similar when measured after compacting the specimens using a Superpave gyratory compactor (SGC) at the same compaction level. Based on the results from APA testing the rutting resistance for the two types of mixtures was not significantly different.

Another commercially available wax-based additive is Asphaltan B, produced by Amsdorf of Germany. The mechanisms by which Asphaltan B facilitates the production of WMA mixtures are similar to that of Sasobit. The recommended dosage for Asphaltan B is 2 to 4% by weight of the asphalt binder. Rowe et al. (2009) reported that the reduction in viscosity using montan waxes such as Asphaltan B was similar to the reduction in viscosity achieved using Fischer-Trop waxes such as Sasobit.

1.2.2 Emulsion- or Surfactant-Based Additives

These additives typically contain a chemical agent such as an emulsifier or surfactant that improves binder workability during mixing and compaction by reducing viscosity and/or interfacial friction. The additives may also contain agents that promote adhesion between the binder and the aggregate.

Two common additives in this category are Evotherm ET and Evotherm DAT produced by MeadWestvaco, where ET stands for emulsion technology and DAT stands for dispersed asphalt technology. More recently Evother 3G (third generation) was also developed as a water-free version of Evotherm that can be introduced in the asphalt binder or at the mixture production plant.

According to MeadWestvaco, Evotherm ET (emulsion technology) is a binder-rich water-based emulsion with approximately 70% solids and is capable of reducing temperatures by approximately 38°C (reduction of 68°F). The water is released in the form of steam during mixture production. The steam may further facilitate the mixing and compaction process. Similar to conventional asphalt emulsions, the emulsifiers in the Evotherm adsorbs onto the aggregate surface with a long hydrocarbon tail extending beyond the aggregate surface (Chowdhury & Button, 2008). This promotes interfacial adhesion between the binder and the aggregate. According to MidWestvaco Evotherm DAT has the same chemistry as Evotherm ET and can be directly injected into asphalt line at the plant. Due to the nature of this process, specific adhesion promoting agents can be tailored for different types of aggregates and added to the additive.

In another study, Kristjansdottir (2006) demonstrated the use of Evotherm in cold regions with benefits such as reduced stripping, improved mechanical properties, and reduced compaction temperatures. Diefenderfer and Hearon (2008) reported the use of Evotherm ET in one WMA test project in Virginia. The WMA mixture was based on a Superpave mixture design with PG 70-22 grade asphalt binder and 20% RAP. Based on the results of the indirect tensile strength ratio test (AASHTO T-283) the authors reported a slight decrease in the moisture damage resistance of the WMA mixtures as compared to the HMA mixtures. Also, the indirect tensile strength of the unconditioned WMA specimens was approximately 65% that of the control HMA specimens. Some of these differences were attributed partially to the fact that the WMA mixtures had higher binder content as compared to the control specimens (6.06% vs. 5.83%). Testing with the APA indicated that the WMA mixtures were more rut susceptible than the control HMA mixtures.

Hurley et al. (2009) reported findings that were consistent with the above. In a study of mixtures from field trials in Wisconsin, the authors reported that mixtures with Evotherm ET had typically lower rutting resistance when compared to HMA mixtures tested using the APA. They also reported mixtures with Evotherm ET to have lower (albeit above the required specification value of 80%) tensile strength ratio as compared HMA mixtures when tested in accordance with AASHTO T-283. The Evotherm ET mixtures were also reported to have stripping when tested with the Hamburg wheel tracking device. Kvasnak et al. (2009) reported similar findings for Evotherm DAT based on test sections in Tennessee.

The second example of an additive in this category is the Rediset WMX produced by Akzo Nobel. Rediset WMX is a surfactant-based additive. The hypothesized mechanism for this type of additive is that it reduces the interfacial friction between thin films of the asphalt binder and coated aggregates thereby improving workability and allowing for mixing and compaction at reduced temperatures. It is available in the form of free flowing prills and is added to the binder prior to mixing or to the mixing unit. The prills melt at about 185°F and completely blend with

the asphalt binder. Akzo Noble recommends that the additive be added 2% by weight of the binder. Also, according to Akzo the additive includes anti-strip agents to promote interfacial adhesion between the aggregate and the binder.

The third example of a chemical additive is Cecabase RT produced by CECA of France. The additive is primarily a surface active agent composed of at least 50% renewable raw materials. The hypothesized mechanism to produce WMA is the same as that of surfactants such as Rediset.

Gonzalez-Leon et al. (2009) reported the performance of binders and mixtures using Cecabase RT. According to their study, this additive improved workability of the mixtures at reduced temperatures allowing the production of WMA mixtures. Also, addition of Cecabase RT did not significantly change the rheological properties of the binder or the mechanical properties of the mix when compared to a conventional hot mix.

1.2.3 Water-Bearing Additives

The most common water-bearing additive to produce WMA is synthetic zeolite. Addition of zeolite during the mixing process results in the formation of a very fine foam. The extremely fine foam creates micropores that in turn help increase workability of the mix (Barthel, Marchand, and von Devivere 2004). A critical point to be considered when using zeolite for mixing is the release timing of moisture. Step-wise release of water is required to ensure consistent workability for longer times (necessary for mix transportation). Barthel et al. (2004) also reported that a mix was compactable until the temperature reached 99°C (210°F), in which case a step-wise release of moisture can be achieved. Tests conducted by Barthel et al. (2004) on warm mixes alleviated concerns that zeolite-modified mixes are prone to moisture damage (stripping). No separation of the binder from the aggregate during the mixing stage was reported in this study. All binder grades were reported to be compatible with synthetic zeolite (specifically, the Aspha-min® brand). A reduction in energy consumption by 30% was reported (i.e., only 5.6 ltrs was sufficient as compared to 8 ltr of oil per ton of mix). The study also reported that fume emission and release of particulate matter was lowered because of low temperature mixing.

Chowdhury and Button (2008) reported that the use of Aspha-min reduced the air voids in the mix. Reduction in air voids also implies better in-place densities of WMA in comparison with HMA. However, reduced air voids at the same level of compaction might affect the optimum binder content and overall mix performance and durability. To offset the disadvantages of air voids, Chowdhury and Button (2008) bumped up the binder grade.

Commercially available brands of synthetic zeolite are Advera (produced by PQ Corporation) and the aforementioned Aspha-min. Hurley and Prowell (2005b) reported that Aspha-min is a large hydrothermally crystallized zeolite, which can trap water within its structure and release it with a slight increase in temperature. In their study, the addition of Aspha-min zeolites lowered the measured air voids in the gyratory compactor but did not affect the resilient modulus or rutting potential of an asphalt mix. They also reported no evidence of differing strength gain with time for the mixes containing zeolite as compared to the control mixes. In another study Sousa Filho et al. (2006) reported that the percentage of zeolite that produced the best volumetric results was 0.5%.

Kristjansdottir (2006) reported that the addition of Aspha-min lowered the binder grade by one grade in terms of compaction effort, air voids, and viscosity in mixing phase. The reduction of compaction energy was more pronounced in case of stiffer binder. Similar to other

studies, this study also concluded that the addition of zeolite did not affect the resilient modulus, i.e., the zeolite did not increase or decrease the stiffness of the mixtures for any compaction temperature. But the resilient modulus decreased as the compaction temperatures decreased and air voids increased. Kristjandottir (2006) also reported that hydrated lime was an effective agent to improve strip resistance and also decrease the rutting potential of mix.

Goh and You (2008) conducted tests and asphalt binders and mixtures modified using Aspha-min with different levels of dosage. They used PG 52-34 and PG 64-28 grade asphalt binders. Dynamic shear rheometer (DSR) testing indicated that addition of Aspha-min reduced the G*/sinδ value at high temperatures as compared to the neat asphalt binders (about 80 to 90% of the value for unmodified binder). This was true for both unaged and short-term aged asphalt binder. Similarly the G*sinδ values increased at low temperatures as compared to the neat asphalt binders after long-term aging. The bending beam rheometer (BBR) tests were also conducted on the long-term aged binder. The creep stiffness decreased slightly for the modified binder and the m-value increased slightly. Contrary to these findings, in another study Gandhi (2008) reported that G*/sinδ for three different asphalt binders was almost the same or increased. Similarly, Akisetty (2008) also reported an increase in the high temperature grade of the crumb rubber modified asphalt binders when used with Aspha-min. You and Goh (2009) reported that addition of another synthetic zeolite, Advera, did not change the binder grade. The BBR results reported by Gandhi (2008) were consistent with Goh and You (2008) in that the m-value was reported to decrease with the addition of Aspha-min.

Goh and You (2008) also reported results from mixture testing. The WMA mixtures with Aspha-min had similar resilient modulus as that of the control mix at different temperatures. Similar results were also reported by Akisetty (2008) for crumb rubber modified asphalt binders with Aspha-min additive. You and Goh (2009) reported an increase in resilient modulus and rutting resistance of mixtures with the Aspha-min additive.

Contrary to the results based on DSR tests, the dynamic modulus and rutting resistance (measured using an asphalt pavement analyzer [APA]) of WMA mixtures with Aspha-min was similar or better than that of a comparable HMA mixture (Goh and You 2008).

1.2.4 Direct Foaming

In the direct foaming process, the main emphasis is laid on the introduction of moisture, which in turn improves mix workability. The most important characteristic of foaming is that it does not modify the chemical make-up of the asphalt binder and causes the binder to foam, which makes the coating of aggregate easier irrespective of the condition of aggregate.

Foamed asphalt has been in use for a few decades now. For example, Csanyi (1957) used foamed asphalt binder for use as a soil stabilizer. Since then, various industries including Mobil Oil of Australia and Conoco developed the techniques for using foamed asphalt binder. Foamed asphalt was reported to have a very sticky and rubbery texture, with high cohesive and adhesive strength. Consequently, the mix using foamed asphalt is presumed to have better binder aggregate interaction and hence better performance.

Ruckel et al. (1983) reported that the criteria used for the design of cold mixtures were also suitable for foamed asphalt. They reported that the major advantage of foamed asphalt over cold mixes was that foamed asphalt could be immediately compacted without any time allotted for aeration or extraction of entrapped water.

This study emphasized that the physical properties of foam to be considered are its expansion and half-life, which are important for aggregate coating and workable time

respectively. In addition, they highlighted that moisture was helpful in breaking up agglomeration of aggregates.

Koenders et al. (2000) reported that WMA foams were found to produce lesser fumes and dusts when compared to HMA's. They quantified the energy savings obtained at 99°C (210°F) to be approximately 25 to 30% but depended on the moisture content of the aggregates.

Jenkins et al. (2002) reported that the asphalt binder behaved as a strain hardening material in both HMA and WMA. They also reported that foamed asphalt displayed better cohesive strength than HMA mixes at higher temperatures. Half warm foamed mixes, produced at temperatures lower than the boiling point of water, lead to enormous energy consumption as the energy barrier of water (latent heat of vaporization) is not exceeded. However, half warm mixes did not exhibit same strength as that of HMA at lower temperatures less than 25°C (77°F), but were almost equivalent at higher temperatures.

Johnston (2006) reported the use of a foaming-based technology to construct test sections in Calgary, Canada. According to this study, the WMA mixture was more susceptible to rutting as compared to an equivalent hot mix (WMA had 11 mm [0.43 inches] rutting as compared to 8.7 mm [0.31 inches] rutting at 8000 cycles when tested using an APA). The resilient modulus for the HMA was 20% higher than the resilient modulus for the WMA at 20°C (68°F). Fatigue life of the WMA was reported to be three times the fatigue life of the HMA when tested using a beam fatigue setup. The WMA mixture also had higher moisture susceptibility as compared to HMA using the indirect tensile test. The tensile strength ratio for the WMA was 50% as compared to 85% for the HMA.

Middleton and Forfylow (2009) reported the evaluation of WMA mixtures produced using the double barrel green process in Vancouver. APA was used in dry and wet tests to evaluate the rutting and moisture damage resistance of the WMA mixtures. The dry rut depths ranged from 4.1 mm (0.16 inches) to 5.2 mm (0.20 inches) and the wet rut depths ranged from 5.2 (0.20 inches) to 7.9 mm (0.31 inches) at 8000 cycles (the usual criterion is 8 mm (0.31 inches) at 8000 cycles). A slight but not significant decrease in rutting was observed with increase in the RAP content. The authors also measured the resilient modulus of the WMA mixtures but there was no reference HMA to compare the results. Results from the AASHTO T-283 indirect tensile strength ratio indicated that all WMA mixtures had a tensile strength ratio of above 80% except the WMA mixture without RAP which had s tensile strength ratio of 77.5%.

Hodo et al. (2009) also reported the properties of WMA mixtures produced using the double barrel green process in Tennessee. Properties of a WMA mixture with no RAP and a WMA mixture with 50% RAP were reported. No reference HMA mix was reported. The average rut depths for the two mixtures using the Hamburg wheel tracking device were 3.4 mm (0.13 inches) and 4.4 mm (0.17 inches), although stripping inflection points were observed for both mixes. The tensile strength ratio in accordance with AASHTO T-283 was around 80% for both mixtures.

Contrary to the findings above, Wielinski et al. (2009) reported the properties of HMA and WMA produced using the double barrel green technology in California. They reported that the tensile strength ratio for the HMA and WMA was very low when tested in accordance with AASHTO T-283. Further the tensile strength ratio for the WMA was consistently lower than the HMA (about 35% vs. about 45%). Based on APA results they also reported that the rutting resistance of WMA mixtures was lower than the rutting resistance of HMA.

1.2.5 Summary

Although several studies have evaluated the influence of additives and processes used to produce WMA, very few studies have evaluated the performance of these mixtures after long-term aging. In other words, the performance of WMA mixtures in terms of its resistance to rutting, cracking, and moisture-induced damage was highly dependent on the WMA technology and binder properties being used. This is because tests conducted on WMA mixtures use only short-term aged and compacted specimens. This inherent limitation of mixture testing precludes results that demonstrate the mixtures' resistance to fatigue and low temperature cracking after long-term aging. Based on the studies reported thus far, it appears that the influence of the WMA additive is highly dependent on the type of binder used.

1.3 Considerations Related to the Design and Use of WMA Mixtures

Recent National Cooperative Highway Research Program (NCHRP) Project 9-43 investigated the mix design practices for WMA mixtures. The objective of this NCHRP research was to specifically develop a mix design procedure for WMA mixtures based on the Superpave methodology. The mix design method was designed to include performance tests to ensure that the WMA mixtures will perform satisfactorily in the field and was intended for all WMA technologies. A summary of findings and preliminary recommendations from the interim report of this project is presented here.

- Higher initial mixing and compaction temperatures increase oxidative aging of asphalt binder, making it not only difficult to compact but also reducing its fatigue life considerably. This problem can be offset by using WMA technologies that allow production and mixing of mixtures at reduced temperatures and consequently save energy as well as improve the fatigue performance of pavement.

- The interim report recommends that the most important change in the design of WMA mixtures is the selection of asphalt binder. Other aspects of the mixture design such as aggregate gradation, performance analysis, and volumetric analysis do not need modifications.

- As mentioned, binder selection is the most important aspect of WMA mixture design and needs some modification irrespective of the process employed to produce WMA. This is because the WMA mixing temperatures are typically lower when compared to HMA mixing temperatures, resulting in reduced aging of the asphalt binder and thereby increasing the rutting potential. To offset this effect, the selection of a stiffer binder is recommended. However, this must be carefully balanced so as not to adversely influence the fatigue cracking life of the mixture (the NCHRP study is currently developing guidelines for this). It must also be noted that the effect of reduced aging on reducing the stiffness of the binder in a WMA is more prominent during the early life of the material. This difference may become less distinguishable between HMA and WMA mixes as the binder ages during the service life of the pavement. Binder grade bumping for warm mix asphalts will be based on the production temperature. For example, if the production temperature of the WMA results in a decrease in the high temperature continuous grade of more than 3°, then the binder grade should be increased by one grade.

- Lower temperatures might limit the functionality of several aspects of design, such as additives used to reduce stripping and effective incorporation of RAP.

Following are the general steps recommended for the design of WMA mixtures:

i) WMA process selection: In this step, the type of additive or process to be employed in the design of the mix is determined. Many factors such as cost, performance data, and availability are taken into consideration.

ii) Material selection: In this step, the basic materials such as binder, aggregate, and/or inclusion of RAP are selected based on criteria such as strength required, material available, and performance. As mentioned previously, binder grade may have to be adjusted to mitigate the adverse effect of reduced aging.

iii) Design of aggregate structure: In this step, the aggregate matrix design is finalized. The design process includes calculation of quantity and quality of aggregate to be used in mixture, specimen fabrication, volume of voids to be allowed, etc. This step is the same as that used for the design of HMA.

iv) Performance prediction and evaluation: This final step includes quality check, conformity of regulations both structurally and functionally, and recommendations on maintenance.

The preliminary recommendations are to evaluate moisture sensitivity and rutting resistance using the same tests for WMA as those used for HMA.

In most cases, WMA mixtures are designed to incorporate RAP. This typically mitigates the increased susceptibility to rutting of WMA mixtures without increasing the binder grade. Following are some of the findings from the NCHRP 9-43 study on the use of RAP in WMA.

- Low compatibility of RAP with neat binder might result in structural and elastic failures. Atomic force microscopy was used to investigate compatibility of different aged and unaged asphalt binders in WMA. The interface mixing and compatibility experiment demonstrated that new binder and RAP binder do mix when subjected to elevated temperatures. The results demonstrated that the process of mixing continues even after the completion of mixing. The compatibility of RAP/WMA blends decrease with increase in RAP content.

- Usually, less compatible binders exhibit more elastic behavior and are less ductile when compared to more compatible binder.

- Dynamic modulus test is very sensitive to stiffness of the binder. Adding RAP increased the dynamic modulus significantly when the binders used were compatible.

- Lab studies have shown that it is reasonable to expect a good amount of mixing between a very stiff binder (worst case scenario) and new binder at typical warm mix temperatures.

- The phenomena of mixing in RAP is more a function of viscosity than of temperature, which implies that if a minimum viscosity of RAP and virgin binder is achieved using an additives, then mixing/fusing is possible.

Finally, quantifying the change in workability at reduced mixing temperatures is the key to all WMA mixtures. The following devices were investigated in the NCHRP project to measure workability:

- UMass Workability device

- Gyratory shear stress

- Nynas workability device

- University of New Hampshire Workability device

1.4 Considerations Related to the Performance of WMA Mixtures

Based on review of the literature, this section summarizes the primary concerns for different WMA technologies with respect to expected performance of WMA mixtures.

1.4.1 Fatigue Cracking and Low Temperature Cracking

Organic additives are typically wax- or fatty amide-based. Sasobit supplies the most commonly used organic additive in the United States: a Fischer-Tropsch wax produced from coal gasification. The presence of wax in asphalt binders is associated with low temperature cracking (Lu & Redelius, 2007). However, it is claimed that Sasobit is not the same as the wax found naturally in asphalt binders (Sasol, 2008). Sasobit is considered to have a smaller crystalline structure and longer chain compared to wax that is naturally found in asphalt binders. Because of the microcrystalline structure, Sasobit is claimed to be not as susceptible to fracture or low temperature cracking (Lee, Amirkhanian, Park, & Kim, 2008). However, Hurley and Prowell (2005a) also report that WMA mixtures produced using Sasobit had lower indirect tensile strength in some cases. Haggag et al. (2011) also reported no significant difference in the fatigue cracking resistance between HMA and WMA mixtures made with Sasobit and Evotherm 3G. Other evidence in the literature also indicates that addition of Sasobit results in a decrease in the resistance to low temperature cracking. For example, addition of Sasobit resulted in an increase in stiffness and reduction of m value (slope of the stiffness curve) from the Superpave BBR test—both of which indicate a reduction in cracking resistance (Goh et al. 2008) and (Lee et al., 2008). Rand (2008) also reported that mixtures with Sasobit demonstrated a lower fatigue cracking life on an overlay tester compared to the control mix. The slightly increased susceptibility to low temperature for some binders with Sasobit was later demonstrated and verified in this study.

1.4.2 Permanent Deformation

WMA is produced and compacted at lower temperatures compared to conventional HMA. This results in reduced short-term aging of the asphalt binder and an increased susceptibility to rutting, which is detrimental for early pavement performance. Hurley and Prowell (2005a) and (2005b) concluded that Aspha-min did not increase permanent deformation potential of the mix, while Sasobit and Evotherm decreased the permanent deformation potential.

They also reported that permanent deformation potential increased with decreasing mixing and compaction temperatures, which may be related to the decreased aging of the binder. In this study, we later demonstrate that both reduced aging as well as the presence of the additive itself (in many cases) is responsible for the initially increased susceptibility to rutting. NCAT (2005) recommended bumping the high-temperature grade of the asphalt by one grade to offset any increase in permanent deformation potential that may occur when using a WMA product. A study conducted by Zelelew et al. (2011) concluded that Advera, LEA, and Gencore increased the permanent deformation potential of the mixes evaluated. They also showed that WMA mixtures were more susceptible to moisture damage than their HMA counterparts. Hence, successful implementation of WMA requires mix design procedures that properly consider the effects of reduced production and compaction temperatures on materials and pavement performance

1.4.3 Moisture Damage

Durability and moisture damage are major concerns in the production of WMA. Lack of durability or sensitivity to moisture damage could be due to the combination of one or more of the following: (i) incomplete wetting and coating of aggregates, (ii) incomplete drying of the aggregate, (iii) residual moisture in the binder (foaming process), (iv) reduced absorption of binder into certain types of aggregates that may result in poor mechanical interfacial bonds between the binder and the aggregate, (v) deleterious chemical interactions between the additive and the binder, and (vi) possible rehydration of additives such as zeolite particles over time.

Previous studies on WMA have indirectly evaluated the proper coating of aggregates by comparing the engineering properties (e.g., resilient modulus) of the WMA mixture to that of an equivalent HMA. In a recent FHWA study, Bhasin et al. (2007) investigated the effect of incomplete drying of the aggregates on the adhesive bond between the binder and the aggregate. The quality of the adhesive bond is directly related to moisture sensitivity and durability of the mixtures. Using a combination of physio-chemical and mechanical tests, they were able to demonstrate that, for siliceous and non-porous aggregates, the difference in bond strength due to incomplete drying at WMA temperatures was not significant. However, Bhasin et al. (2007) also reported that porous aggregates such as limestone were not included in the study. They speculated that porous aggregates retain moisture within the bulk due to incomplete drying at WMA temperatures that may later cause durability problems. This concern was addressed in this study (Chapter 2).

Mogawer et al. (2011) evaluated the effect of WMA technologies (Advera, Evotherm, Sasobit, and Sonne Warmix) on the moisture susceptibility of a mixture and adhesive characteristics of the asphalt binder used in it using HWTD and bitumen bond strength (BBS) tests. The HWTD test results indicated that the moisture resistance of mixtures improves with aging time or temperature and the BBS test results indicated that only Sasobit showed a significant effect on the adhesive strength of binders. Mogawer et al. (2011) also conducted an internet-based survey of all 50 states regarding moisture susceptibility of WMA. In the survey, 73% of the respondents stated that their state has a moisture damage requirement for WMA mixtures.

In a study by Buss et al. (2011), WMA mixes were reported to be more susceptible to moisture damage. In most cases, WMA mixtures had a TSR value that was lower than a similar HMA mixture. The dynamic modulus results showed that the interaction of the mix, compaction type, and moisture conditioning were statistically significant. This suggests that the combination

of all three factors play a role in determining material response. Xiao et al. (2010) also evaluated rut depth of mixtures containing moist aggregate and concluded that such mixes generally satisfy the demand of pavement performance without additional treatment. Hurley and Prowell (2005b), (2005a) also reported that lower mixing and compaction temperatures can cause incomplete drying of the aggregate and that water trapped in the coated aggregate may result in moisture damage.

The specific physio-chemical interactions between the binder and the WMA additives may also be a cause for reduced durability or moisture sensitivity. For example, certain additives contain sodium ions. Previous research studies have demonstrated that such ions can form water-soluble salts with the binder and create moisture sensitivity problems. Also, introduction of divalent cations such as calcium can reduce this deleterious action. Addition of the normally recommended amounts of sodium-bearing warm mix additives resulted in failure of sand-asphalt mixture specimens upon moisture conditioning even before a test could be conducted (Bhasin, Vasconcelos, et al., 2007). The mechanism was verified by measuring the amount of sodium that was found in the water used for moisture conditioning the specimens. They also demonstrated that this problem was mitigated with the use of hydrated lime.

Per TxDOT special provision 341-018 (05-08), the use of liquid anti-strip agents is mandated for all WMA mixtures. However, certain chemical WMA additives (e.g., Evotherm 3G) are already designed to promote adhesion between the binder and the aggregate. In such cases, the mandated use of a liquid anti-strip agent may not benefit the mix or even prove to be harmful. Also, in the case of foaming agents such as zeolite particles, the use of a liquid anti-strip agent may not be of much benefit because of its inability to address the source of moisture sensitivity. For example, Hurley and Prowell (2005a) demonstrated that the use of hydrated lime is more effective with zeolite-based additives compared to other anti-strip agents. Results described in the previous paragraph support these findings.

1.4.4 Time Limitations in the Use of WMA Mixtures

One of the advantages of using WMA technology is the ability to haul the mixes over longer distance or store them for longer durations of time and achieve compaction on site. However, maintaining a loose mix at elevated temperatures for long durations increases its oxidative aging. This aging may negatively affect the durability of the WMA mixture and must be investigated.

A strong precedent for foaming has been its use for in-place recycling and stabilization for over five decades (Csanyi, 1957). However, unlike WMA, there is a very small time gap between mixing and compaction during in place stabilization. The foaming process (either direct or using additives) is used to improve binder workability and coating during the production of the WMA. The beneficial effect of foaming at the time of compaction is readily seen. However, the existing literature does not make clear how long the improved workability due to foaming will last. Previous studies indicate that foamed bitumen (direct injection with nozzle) has a half-life of only 20 seconds in a 1 gallon container (Ruckel et al., 1983). Similarly, a preliminary study demonstrated that, at typical warm mix temperatures, uncoated zeolite particles release the moisture that they carry within 30 to 40 minutes (Bhasin, Vasconcelos, et al., 2007). This period may be longer for particles coated with asphalt binder and transported in bulk. In any case, it is not clear how foaming at the production site facilitates compaction that takes place at reduced temperatures several minutes or hours after the mixing is complete. While continued foaming is important to facilitate compaction, one may also argue that most of the water causing foaming

must be released before compaction to avoid trapping moisture in the pavement structure. This research project will address the issue of timing the placement of WMA mixtures after leaving the production site. This problem is very important for the foaming process, although it is less critical for the chemical or organic wax-based additives. The organic wax-based additives are likely to be effective as long as the additive is above its melting point. In the case of chemical additives, the issue of timing may be related to the strength gained over time. For example, the emulsion-based additive Evotherm demonstrates improved mechanical properties when subjected to mechanical tests after certain duration of time. This aspect must also be taken into consideration in the development of the detailed experiment design.

1.5 Scope of this Report

This report summarizes the findings from the study conducted to evaluate the influence of chemical warm mix additives on the rheology and performance characteristics of asphalt binders. One of the objectives of this study was to evaluate the potential of physic-chemical interactions between the chemical additives and the asphalt binder. Chapter 2 of this report presents the methods that were used to screen and select asphalt binders used by the TxDOT based on their natural acid content and wax content. Chapter 3 of this report presents the tests that were conducted to evaluate the influence of different warm mix additives on the rheology of the asphalt binders and the findings from these tests. More specifically, this chapter evaluates the following attributes of the asphalt binders with and without the warm mix additives:

- viscosity,
- resistance to permanent deformation,
- resistance to fracture and fatigue cracking,
- resistance to low temperature cracking,
- rate of oxidative aging, and
- influence of adding aged binder (similar to binder from recycled asphalt pavement) on the rheology of the asphalt binders.

Chapter 4 of this report presents concluding remarks and discussion based on these results.

Chapter 2. Material Selection and Modification of Asphalt Binders

2.1 Introduction

The main objective of this research was to investigate potential interactions between the WMA additives and asphalt binder as well as the impact of these interactions, if any, on the performance characteristics of the asphalt binders. Asphalt binders demonstrate a very broad range of chemical properties depending on the source of crude oil and processes used to produce the asphalt binder. The chemical makeup of the asphalt binder eventually dictates the stiffness of the asphalt binder, resistance of the binder to fracture and plastic deformation, and overall performance of mixtures that incorporate the asphalt binder. In addition, the chemical makeup of the asphalt binder changes over time as it continues to react with atmospheric oxygen. In the case of a WMA, chemical additives are added to the asphalt binder to allow the production and placement of asphalt mixtures at lower than conventional temperatures. Depending on the chemical makeup of the asphalt binder and WMA additives, one can expect chemical or physio-chemical interactions that influence the performance of the asphalt binder and mixture. In order to achieve the objectives of this study, it is extremely important that the materials (asphalt binders, WMA additives, and aggregates) are selected to represent not only a range of mechanical properties but also a range of chemical properties.

This chapter presents a description of materials used in this study, the rationale for using specific properties that were the basis for material selection, and the test procedures employed to determine these material properties.

2.2 Selection and Use of WMA Additives

Several technologies and processes are used to produce WMA mixtures. Table 2.1 presents a summary of these technologies and products grouped based on their generic mode of use. At least one chemical WMA additive from each technology was selected for this study. A brief description of the chemical additives, recommended mixing procedure, and dosage is presented below.

2.2.1 Evotherm® DAT

Evotherm DAT (Dispersed Asphalt Technology) is a product of MeadWestvaco Asphalt Innovations, Charleston, South Carolina. Evotherm DAT is a water and chemical mixture that is pumped directly into the asphalt supply line at the mix plant. It is typically used at 5% by weight of the asphalt binder and can achieve a reduction of up to 55°C (reduction of 99°F) in plant temperatures.

Table 2.1: Summary of Warm Mix Additives

Generic Class	Summary of Mechanism	Products (Producer)	Remarks
Organic additives	In general, most of these additives are wax- or fatty amide-based. The additive reduces binder viscosity above its melting point and increases binder stiffness at operating temperatures below its melting point.*	• Sasobit FT Wax (Sasobit) • Asphaltan-B Wax (Romonta) • Cecabase RT (Ceca)	Sasobit FT Wax is approved for use by TxDOT.
Chemical additives	These additives typically contain an adhesion promoting chemical agent that improves binder workability during mixing and compaction by reducing viscosity and/or interfacial friction. At service temperatures this agent improves interfacial adhesion.*	• Evotherm ET (MeadWestVaco) • Evotherm DAT (MeadWestVaco) • Rediset WMX (Akzon Nobel)	Evotherm is approved for use by TxDOT.
Water-bearing additives for foaming	Water-bearing fine particulate additives, such as zeolites, are added to the binder. Water released by the additive causes foaming and improves workability during mixing and compaction. The additive remains in the mix as a particle filler.	• Aspha-min (Eurovia) • Advera (PQ Corp.)	Both Advera and Aspha-min are approved for use by TxDOT.
Direct foaming during production	A foaming nozzle is used to inject foam during mixing and compaction to improve workability of the mix.	• Double Barrell Green • Ultrafoam GX	Double Barrell Green and similar technologies are approved for use by TxDOT.***
Other mixed techniques (Koenders et al. 2000), (Larsen et al. 2004)	This class includes typically combining wet and as-is fine aggregates at ambient temperature with dry coarse aggregates then mixing them with the binder. The heat from the dry aggregates causes the moisture from the fine aggregates to vaporize, foam the binder, and improve workability. Low Energy Asphalt (LEA) is created through the process of using sequential coating of aggregates. Other techniques include combining a soft binder with a hard foamed binder to achieve the desired workability.	• McConnaughay Technologies	As of this report's date, the LEA has been used only in very few locations in the United States.

* Note: The exact mechanism may vary from one proprietary product to another.
*** This technology is well received by contractors because of the low capital cost to modify the mixing plant. However, it does not rely on chemical modification.

2.2.2 Evotherm® 3G

Evotherm 3G (Evotherm 3rd Generation) is also a product of MeadWestvaco Asphalt Innovations, Charleston, South Carolina. It is a water-free version (chemical-based) of Evotherm (*MeadWestvaco Asphalt Innovations*). It can be added at the terminal of the asphalt binder supplier or at the binder supply line of an asphalt mix plant. It is typically added at 0.5% by weight of the asphalt binder and can achieve reductions of up to 50°C (reduction of 90°F) in plant temperatures.

2.2.3 Sasobit®

Sasobit is a product of Sasol Wax, South Africa. It is a fine crystalline, long chain aliphatic hydrocarbon produced from coal gasification using the Fischer-Tropsch process (Chowdhury and Button 2008, Hurley and Prowell 2005). It is typically mixed directly with the asphalt binder at 1.5% by weight and can lower plant production temperatures by 10°C–30°C (reduction of 18°F–54°F). Sasobit melts between temperatures of 85°C–115°C (185°F–239°F), and is completely soluble in asphalt binders above 115°C (239°F). At temperatures below its melting point Sasobit forms a lattice structure in asphalt binders that is a basis for the stability of asphalts modified with Sasobit.

2.2.4 Rediset™ WMX

Rediset WMX is a pelletized additive from Akzo Nobel, Netherlands, and classified as both a viscosity reducer and surfactant-based WMA technology. It can be added at the terminal of the asphalt binder supplier or at the mix plant right after the addition of binder (Chowdhury and Button 2008). The recommended rate is typically 1.5 to 2% by weight of the asphalt binder and can achieve a reduction of about 35°C (reduction of 63°F) in mixing and compaction temperatures.

2.2.5 Cecabase® RT 945

Cecabase RT 945 is a chemical-based WMA technology from Ceca Arkema Group, France (Ceca Arkema). It is typically added at 0.4% by weight of asphalt binder and can achieve a reduction of up to 40°C (reduction of 72°F) in the mixing and compaction temperatures.

2.2.6 Advera® WMA

Advera is a product available in a very fine white powdered form in 25 or 50 kg bags or in bulk for silos. Advera WMA, a manufactured synthetic zeolite (Sodium Aluminum Silicate), is a product of PQ Corporation. The percentage of water held internally in its crystalline structure is 18% to 21% by mass and can be released at temperatures above 100°C (212°F). When Advera is added to the mix at the same time as the binder, a very fine water foam is created. This release of water creates a volume expansion of the binder that results in asphalt foam and allows increased workability and aggregate coating at lower temperatures. Advera is typically added at a rate of 0.25% by weight of the mix. It can result in a typical production and mixing temperatures that are 30°C–40°C (54°F–72°F) lower than those needed for conventional HMA. In this study, a dehydrated Advera WMA was also used as a reference to evaluate its influence on binder performance.

2.3 Asphalt Binders

Based on a review of the WMA additives, the researchers speculated that the alkaline or crystallizable nature of some of the additives could interact with the asphalt binders and potentially cause deterioration of its mechanical properties. For example, WMA additives that contain wax could possibly create additional nucleation sites for the crystallizable material naturally present in asphalt binders and reduce its ability to relax thermal stresses. Similarly, alkaline additives may interact or react with weak or strong natural acids in asphalt binders affecting its performance. At the time of this study, researchers did not find evidence of any investigation on the potential chemical interactions between the additives and asphalt binder in the literature. Most studies focused either on limited number of asphalt binders or used asphalt binders that were selected based solely on their PG grade. Accordingly, for this study, selecting asphalt binders that demonstrated not only different PG grades but also a range of different chemical properties was important. The two attributes that were used to make these selections were (i) the amount of naturally crystallizable material (*herein after referred to as the natural wax content for brevity*) and (ii) the amount of acids present naturally in the asphalt binder. The significance of these two attributes is further described below.

2.3.1 Influence of Wax on WMA Binders

Several studies have investigated the influence of wax naturally present in asphalt binders on its rheological properties and pavement performance. High crystalline wax content in asphalt binders has been associated with a sudden increase in viscosity during compaction of asphalt mix, reduced ductility, increased brittleness at low temperature, reduced cohesion of the asphalt binder, and deterioration of adhesion to aggregates. Because different waxes have different temperatures at which they undergo phase transition (crystallization and melting), the range of service temperatures experienced by the asphalt binder dictate the influence of wax on the performance of the mix. At low temperatures the waxes in asphalt binders tend to crystallize and increase the stiffness of the asphalt binder. While an increase in stiffness may be beneficial for aspects such as rutting resistance, increased stiffness also translates into higher tensile stresses at low temperatures. At higher temperatures the crystal structures may break down and reduce the stiffness or viscosity of the asphalt binder.

Some of the disadvantages due to the presence of wax in asphalt binders are the following:

- High wax contents usually tend to make the bitumen very soft at higher temperature, which may lead to increased rutting. This fact is more clearly elucidated by the fact that wax softening temperatures tend to reduce a lot if they are doped into bitumen in comparison with the same wax in its normal state. The best example of this is paraffin wax, which has a melting point of 50°C–70°C (122°F–158°F) that reduces to 21°C–32°C (70F°–90°F) if present in bitumen (Wong & Li, 2009).

- Crystallization of wax at low temperature may lead to cracking, which can aggravate other forms of distresses such as moisture damage (Xiaohu & Redelius, 2007).

- Waxes (other than FT-paraffin/Montan/polyethylene wax) sometimes lead to physical hardening, thereby leading to decreased fatigue life (Xiaohu & Redelius, 2007).

- Waxy bitumen may have poor adhesive bonding with aggregate, which may lead to more moisture damage (Xiaohu & Redelius, 2007).

Xiaohu and Redelius (2007) also reported that the negative influence of wax on mixture performance was not consistent for all binders or types of wax. The influence of wax was reported to be very much dependent on chemistry and rheology of both wax and binder. Merusi et al. (2010) also showed that the effect of wax on mixture performance is dependent on the type of wax and asphalt binder. Results of contact angle and adhesion measurements performed on bitumen wax blends indicated that changes in asphalt-water affinity occurred according to the type and content of wax.

In fact, Edwards (2009a) and Giavarani and Pochetti (2005) suggested that conducting dynamic mechanical analyzer (DMA) tests was more informative than fixing some maxima values for wax contents in order to avoid prejudices based purely on numbers.

2.3.2 Influence of Acid on WMA Binders

Very limited research is available on the influence of the natural acids present in asphalt binders on the binders' rheological properties and ultimately on pavement performance. However, a few research studies have been conducted on the influence of acid modifiers on the performance of asphalt mixtures. For example, research conducted by the NCAT National Center for Asphalt Technology (NCAT) indicated that acid modification of a binder to improve permanent deformation interacts adversely with anti-stripping agents added to control moisture damage (NCAT 2005). In the case of WMA additives, some of the additives may be alkaline in nature and may adversely interact with the natural acids present in the asphalt binder.

2.3.3 Screening of Asphalt Binders Based on Natural Wax Content

Samples of 34 asphalt binders were collected from the TxDOT laboratory at the Cedar Park campus in Austin. The samples were from asphalt refineries in and around the state of Texas, and represent the different binders typically used in pavement construction in Texas. The natural wax content in these asphalt binders was quantified using a differential scanning calorimeter (DSC). The test procedure and results are briefly described in this section.

A Mettler TA Instruments DSC was used to determine the natural wax content of the 34 asphalt binders. The test procedure followed the heating and cooling procedures described by Michon et al. (1999). The DSC was calibrated for temperature and enthalpy using Indium. Measurements were conducted using approximately 10 mg sample sealed in an aluminum sample pan using as a reference a similar empty pan with cap. The sample was equilibrated at a high temperature of 165°C (329°F), after which it was gradually cooled to -90°C (-130°F) at a rate of 5°C/min (9°F/min) and re-heated to 165°C (329°F) at a rate of 20°C/min (36°F/min). Figure 2.1 illustrates the Mettler TA DSC test set up along with the autosampler with the samples of the asphalt binders. The DSC experimental test result for PG76-22 HW is presented in Figure 2.2. Table 2.2 presents the DSC test results of all the 34 binders initially considered in this screening process. The information provided in Table 2.2 also demonstrates the range of natural wax content present in the asphalt binders typically used in Texas. The endothermic area

in the last heating segment of the experiment was used to estimate the wax content. The crystalline wax content in mass percent is the endothermic area in the last heating segment of the experiment in J/g divided by 1.8 (Michon et al. 1999).
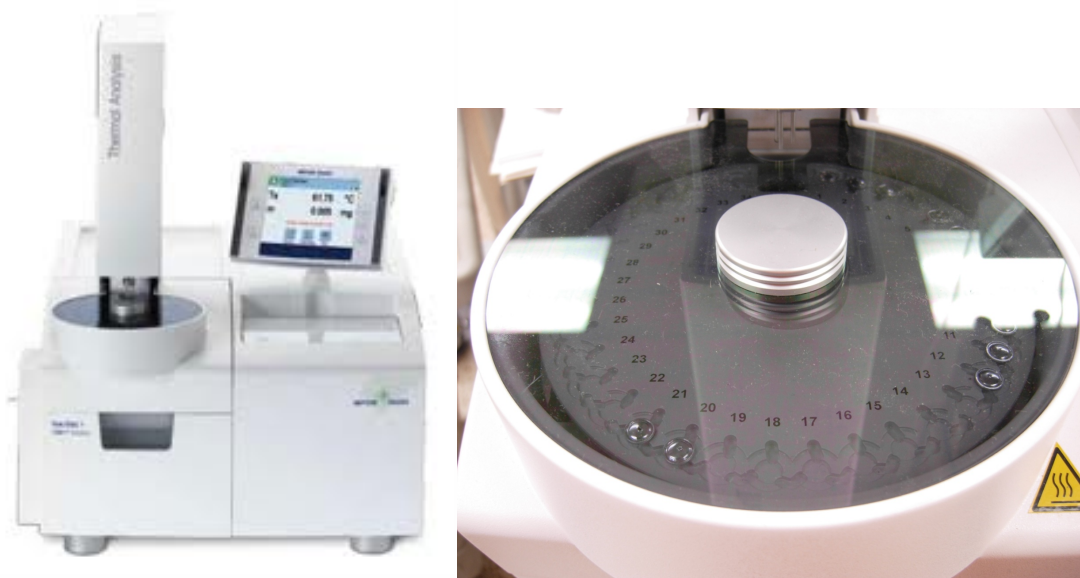
Figure 2.1: A Mettler DSC apparatus (left) with autosampler (right)
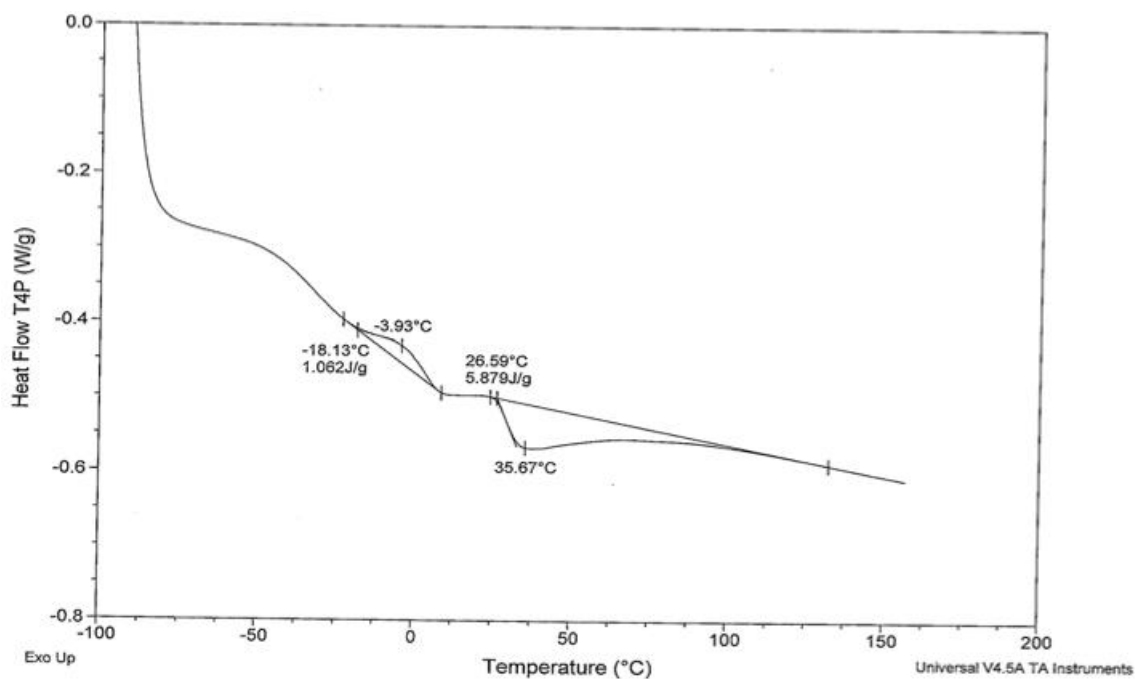
Figure 2.2: Wax content determination of the DSC experimental test for PG76-22 HW
The endothermic area in the last heating segment of the experiment was used to estimate the wax content.

Table 2.2: Crystalline Fraction of Asphalt Binders

Sample Name	Crystallization Onset °C	Crystallization Exotherm, J/g	Melt Midpoint °C	Melt Endotherm J/g (proportional to natural wax content)
PG64-22A	47.44	1.71	26.15	6.19
PG76-22A	46.16	2.69	27.66	5.93
PG76-28A	46.65	4.47	27.41	5.33
PG64-22B	46.16	3.27	27.63	5.27
PG70-28A	46.23	4.01	27.34	4.99
PG64-28A	45.21	3.45	27.17	4.79
PG70-22A	43.97	2.2	27.85	4.56
PG76-22B	48.65	2.43	28.52	4.18
PG64-22C	41.26	1.12	29.39	4.16
PG64-22D	41.6	1.42	29.45	4.1
PG64-22E	42.94	1.99	28.97	4.03
PG70-22B	44.31	1.62	29.04	3.91
PG70-22C	47.95	2.1	29.6	3.89
PG70-22D	43.1	2.3	29.27	3.86
PG64-22F	47.45	1.42	29.38	3.83
PG64-22G	43.04	3.16	28.59	3.82
PG70-22E	43.25	2.59	29.49	3.73
PG76-22C	44.72	1.78	29.02	3.66
PG76-22D	41.92	1.33	29.86	3.6
PG76-22E	42.54	1.79	28.8	3.59
PG64-22H	39.07	0.81	30.76	3.11
PG76-22F	40.22	1.26	30.23	2.95
PG64-22I	43.88	2.06	30.81	2.92
PG70-22F	47.1	1.87	29.54	2.85
PG70-22H	32.02	1.07	29.67	2.68
PG70-22I	33.53	0.65	31.01	2.64
PG76-22G	37.28	0.76	29.84	2.55
PG64-22J	33.17	0.59	30.53	2.48
PG70-22J	39.17	1.42	30.33	2.46
PG64-16A	33.38	0.59	30.82	2.42
PG76-22H	34.11	0.79	30.66	2.36
PG64-22K	41.41	1.76	30.58	2.25
PG64-22L	30.51	0.3	31.22	2.14
PG64-28B	33.67	1.4	33.47	1.62

2.3.4 Screening of Asphalt Binders Based on Natural Acid Content

Acid titrations were conducted on the same set of 34 binders used for the determination of wax content. The objective of this exercise was to identify at least two binders with the highest acid content and two binders with the lowest acid content.

The titration was carried out using 0.1M TBAH (Tetrabutylammonium hydroxide) as the titrant following the procedure developed by Western Research Institute, WY. The titrant was standardized with benzoic acid every day before performing the titrations. The asphalt binder was dissolved in an organic solvent (a mixture of ethyl alcohol and chlorobenzene) and a pH electrode was used to measure the potential change of the system during the titration. The titration data was curve-fit using a five-parameter sigmoid function and the first derivative of this function was used to determine inflection points. Total acid numbers were calculated based on the amounts of titrant consumed at the inflection points. Figure 2.3 shows the setup used to conduct the titration test and Figure 2.4 illustrates the typical results from the titration test. Table 2.3 presents the titration results of all the 34 binders initially considered in this screening process. The information provided in Table 2.3 also demonstrates the range of acid contents in asphalt binders that are typically used in Texas.
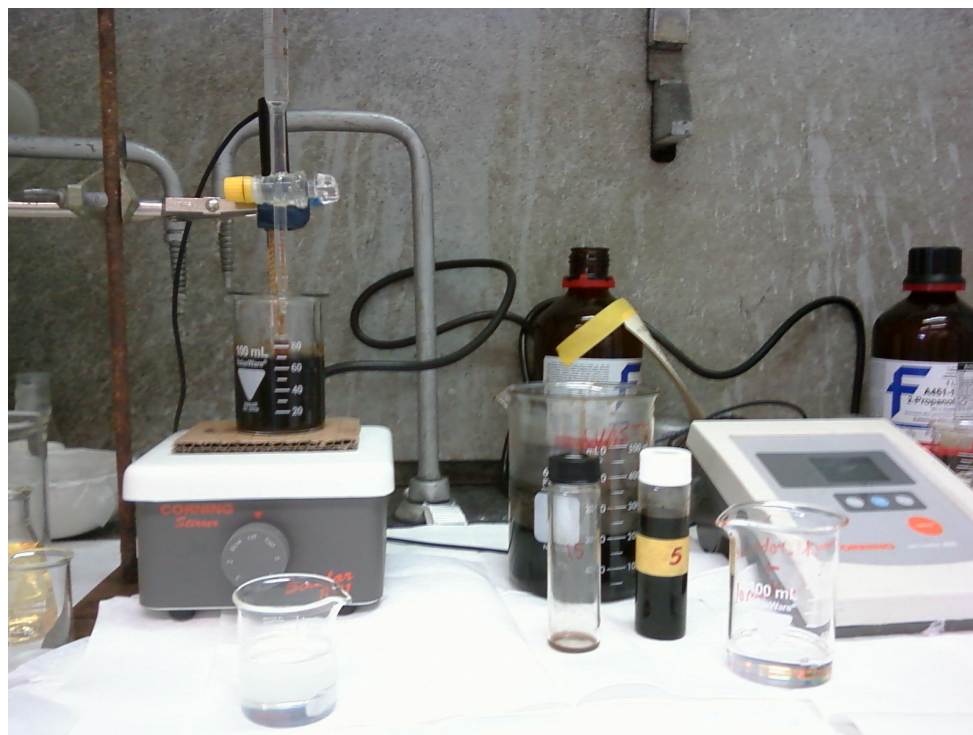
Figure 2.3: Titration test apparatus
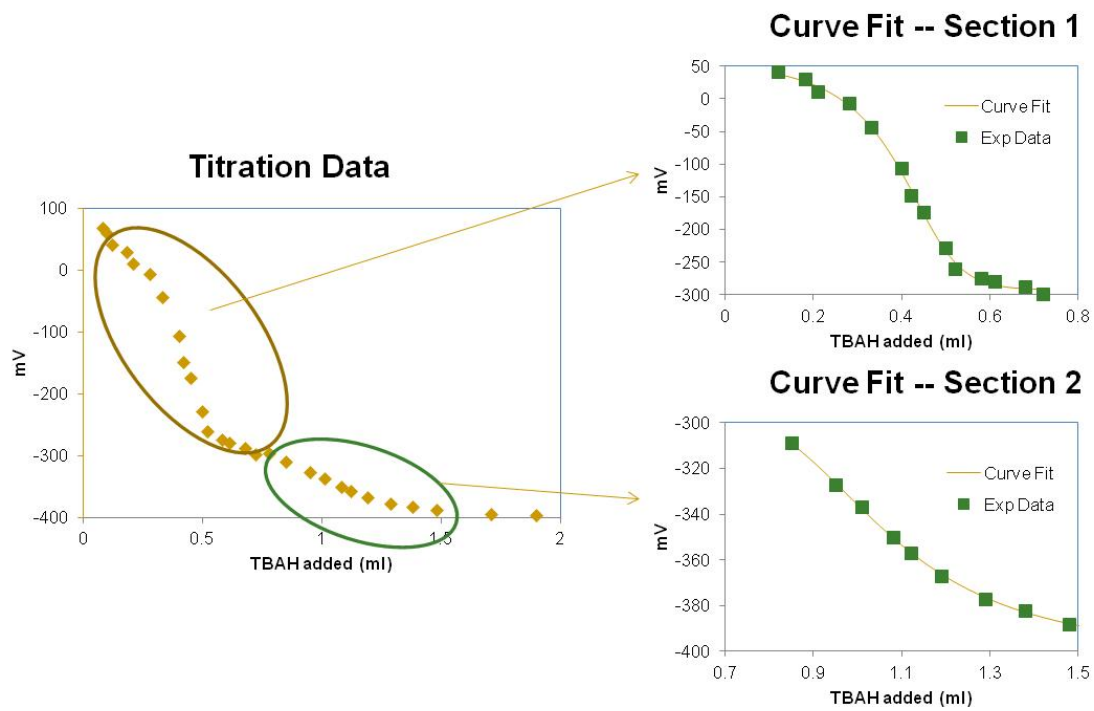
Figure 2.4: Weak acid and strong acid curve fitting of the titration experiment

Table 2.3: Natural Acid Content in Asphalt Binders

Sample Name	Acid Content, mmol/g
PG70-22D	0.002
PG64-22G	0.003
PG70-22B	0.004
PG70-22C	0.005
PG64-28A	0.006
PG76-22C	0.006
PG76-22E	0.008
PG70-22A	0.009
PG70-22E	0.009
PG64-22I	0.009
PG64-22C	0.01
PG76-22F	0.01
PG76-22A	0.011
PG70-22I	0.011
PG76-22D	0.012
PG70-22H	0.012
PG70-22J	0.012
PG64-22D	0.014
PG70-22F	0.014
PG64-22E	0.015
PG64-22B	0.017
PG64-22H	0.041
PG76-22B	0.05
PG70-28A	0.078
PG64-22A	0.09
PG76-22G	0.103
PG64-16A	0.105
PG64-22F	0.11
PG64-22J	0.11
PG76-28A	0.134

2.3.5 Final Selection of Asphalt Binders

The results from the DSC and acid titrations were used to select two groups of binders for further investigation to better understand the interactive effects of WMA additives and asphalt binder chemical composition.

The first group consisted of two binders with the highest wax content and two binders with the lowest wax content. The two asphalt binders with the lowest wax content had a performance grade of PG 64-22 and were labeled as PG 64-22A LW and PG 64-22B LW (the suffix A and B do not correspond to Tables 2.2 and 2.3 and are only used to distinguish between the two PG 64-22 binders from different sources). The two asphalt binders with the highest wax contents had performance grades of PG 76-22 and PG 76-28 and were labeled as PG 76-22 HW and PG 76-28 HW. It must be noted that the two binders with the high wax content were also the two binders with the higher high temperature PG grade. The researchers speculated that the higher wax content may be reason for the higher high temperature PG grade and separating the influence of the two factors may not be possible.

Two binders with the highest acid content and two binders with the lowest acid content were selected among the 34 binders for the second group. The two asphalt binders with the highest acid contents had performance grades of PG 76-28 and PG 64-22 and were labeled as PG 76-28HA and PG 64-22CHA. The two asphalt binders with the lowest acid content had a performance grade of PG 64-22 and PG70-22 and were labeled as PG 64-22DLA and PG 70-22LA. PG76-28HA was also the same PG76-28 with high wax designated as PG76-28HW. Tables 2.4 and 2.5 present detailed description of the materials selected based on their wax and acid contents respectively.

Table 2.4: Properties of Binders Selected Based on their Crystalline Wax Content (Group I)

Binder label	PG76-22HW	PG76-28HW	PG64-22ALW	PG64-22BLW
Original Binder				
Enthalpy of crystallization, J/g	8.62	9.80	4.01	2.44
Viscosity, Pa-s(135°C)	1.38	1.17	0.47	0.60
RTFO Residue				
$G^*/\sin\delta$, KPa +	7.447	4.767	5.844	3.608
Jnr, 100Pa +	0.56	0.08	2.89	2.55
Jnr, 3200Pa +	1.13	0.07	3.23	2.58
PAV Residue				
$G^* \sin\delta$, KPa ++	1564	1426	1984	4609
Stiffness (60), MPa +++	170	165	235	266
m-value(60) +++	0.290	0.283	0.299	0.285
PG Grade	PG76-22	PG76-28	PG64-22	PG64-22

[+] *Indicates properties measured at respective high temperature*
++ Indicates properties measured at respective intermediate temperature
+++ Indicates properties measured at 10°C (18°F) above its respective low temperature

Table 2.5: Properties of Binders Collected Based on their Acid Content (Group II)

Binder Label	PG76-28HA	PG64-22CHA	PG64-22DLA	PG70-22sLA
Original Binder				
Acid Content, mmol/g	0.134	0.110	0.003	0.002
Viscosity, Pa-s(135°C)	1.17	0.57	0.98	0.83
RTFO Residue				
G*/sinδ, Kpa +	4.767	2.502	7.375	2.43
Jnr, 3200 +	0.08	3.6	0.10	0.25
Jnr, 100 +	0.07	3.7	0.11	0.34
PAV Residue				
G* sinδ, Kpa ++	1.426	3.133	2.661	1.736
Stiffness (60) +++	165	254	290	192
m-value(60) +++	0.302	0.316	0.305	0.315
PG Grade	PG76-22	PG64-22	PG64-22	PG70-22

$+$ *Indicates properties measured at respective high temperature*
++ Indicates properties measured at respective intermediate temperature
+++ Indicates properties measured at 10°C (18°F) above its respective low temperature

Five additives (Sasobit, Evotherm DAT, Evotherm 3G, Cecabase RT 945, and Rediset WMX) were evaluated with binders selected based on their crystalline wax content, and two additives (Cecabase RT 925, and Advera WMA) were evaluated with binders selected based on their acid content. In addition to the WMA additives, two controls (Control A and Control B) with different short-term aging were used as a reference to evaluate the effect of reduced aging and additives on binder performance. The additives were blended with the asphalt binder using an RW 20 Digital Overhead Mixer equipped with a four-blade propeller. Chapter 3 presents further details on the specifics of the test matrix.

2.4 Modification of Asphalt Binders with Additives

2.4.1 Mixing Procedure

The following procedure was used to blend the WMA additive into the asphalt binder. The procedure was based on recommendations provided to the researchers by producers of the individual additives.

The additive was blended into the asphalt binder using an RW 20 Digital Overhead Mixer equipped with a four-blade propeller. The binder was heated in the oven in quart-gallon can at its respective storage temperatures for 30 minutes. Subsequently, the can was inserted into a thermoelectric temperature-controlled enclosure and maintained at the mixing temperature of the asphalt binder. The suppliers of all additives recommend the use of conventional (hot mix) mixing temperatures to blend the additive with the asphalt binder. This is because during the production of the WMA, the aggregate temperature is reduced, resulting in significant fuel/energy savings. Before adding the additive, the binder was stirred for 2 minutes in order to obtain a uniform temperature distribution; the addition was performed manually and slowly to attain a homogenous distribution of the additive. Based on the recommendations of the additive

producer, the binder was stirred in the overhead mixer at a constant speed for 15 to 30 minutes to allow complete homogenization of the WMA additive. The mixing apparatus is shown in Figure 2.5.
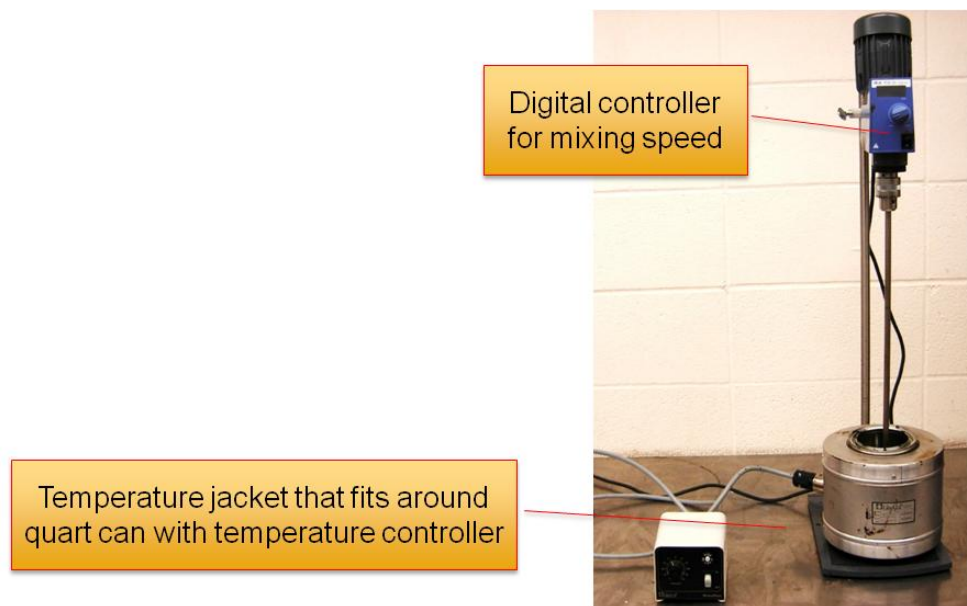

Figure 2.5: Binder and additive mixing apparatus

2.4.2 Aging of Controls and Asphalt Binders with Additives

Asphalt binders used in a WMA are typically heated to mixing temperatures used for the conventional hot mix asphalt prior to the addition of the chemical additives. The energy savings are realized by reducing the temperature to which the aggregates are heated during mixing. Once the modified binder comes in contact with the aggregates during mixing, the temperature of the binder rapidly reduces and equilibrates to the temperature of the aggregate. Therefore, the short-term aging of the asphalt binder in a WMA would occur at this reduced temperature. Based on this rationale, laboratory aging of the asphalt binder using a rolling thin film oven (RTFO) must be carried out at a reduced temperature compared to the temperature used for the HMA (163°C [325°F]). Gandhi et al. (2010) reported that an RTFO temperature of approximately 145°C (293°F) can be used to reflect the aging condition of asphalt binder used in a WMA. In the absence of established protocols for short-term age asphalt binders use in WMA, it was decided to use a temperature of 143°C (289°F) to RTFO age the binders that were modified using WMA additives. This temperature is 20°C lower (36°F lower) than the conventional temperature used for RTFO aging, which is also the typical reduction in mixing temperatures that is achieved with the warm mix technology.

All asphalt binders with and without the WMA additives used in this study were aged in the RTFO to simulate short-term aging. The RTFO aging was carried as per ASTM D2872, where 35 grams (1.2 oz.) of the binder was poured into the RTFO bottles, and aged in the RTFO for 85 minutes. All asphalt binders that were modified with the WMA additives were RTFO aged at 143°C (289°F). In addition, two controls were established for each of the four asphalt binders. For each asphalt binder, Control A represents the unmodified binder RTFO aged at 163°C (325°F) and Control B represents the unmodified binder that was RTFO aged at 143°C (289°F).

The use of the two control binders is necessary to distinguish between the influence of reduced short-term aging and the influence of the WMA additive on the properties of the asphalt binders. More specifically, the following three comparisons can be made.

1. The *influence of reduced short-term aging* on the properties of the binder in a WMA is quantified by comparing the properties of Control B (RTFO aged at 143°C [289°F]) to the properties of Control A (RTFO aged at 163°C [325°F]).

2. The combined *influence of both reduced aging as well as presence of WMA additives* is quantified by comparing the properties of the modified binders (WMA additives and RTFO aged at 143°C [289°F]) to the properties of Control A (RTFO aged at 163°C [325°F]).

3. The *influence of the WMA additive* on the properties of the binder is quantified by comparing properties of the modified binders (WMA additives and RTFO aged at 143°C [289°F]) to the properties of Control B (RTFO aged at 143°C [289°F]).

Unless otherwise indicated, the RTFO-aged binder residues were further aged in the pressure aging vessel (PAV) to simulate long-term aging of the binders. The PAV aging process was carried out as per ASTM D6521, where 50 grams (1.8 oz.) of the RTFO residue was poured into the PAV pans and aged in the PAV for 20 hours at a temperature of 100°C (212°F). Because WMA and HMA are subjected to similar service conditions after being placed in the field, the researchers decided not to change the PAV aging conditions for warm asphalt binders.

An additional sample of the asphalt binders was RTFO aged for twice the standard time, i.e., 170 minutes, to simulate extended short-term aging that may occur when the asphalt mixture is stored in heated silos for a prolonged period or hauled over long distances before being placed and compacted in the field.

2.5 Selection of Aggregates

Aggregates used in WMA are not heated to the same extent as aggregates used in a conventional HMA. The researchers speculated that absorptive aggregates, such as the various limestones commonly found in Texas, may not completely dry when used in a WMA. One of the objectives of this study was to investigate whether or not absorptive aggregates retain moisture that may be detrimental to the performance of the WMA. A short study was carried out using two limestone aggregate samples dried at two different temperatures (130°C [266°F] and 150°C [302°F]) to investigate whether or not reduced drying temperatures had any influence on the retention of absorbed moisture. The test procedure and results from this short study are described in this section.

The AASHTO T85 protocol was followed, with slight variations, to determine the moisture content in aggregates. Approximately 1500 g (53 oz.) of aggregates were immersed in water for 24 hours to saturate the surface and bulk of the aggregates with water. The aggregate sample was then removed from water and the surface of the aggregate particles was dried to determine the SSD (saturated surface dry) weight of the sample. Two samples of the aggregates were placed in ovens at (130°C [266°F] and 150°C [302°F]) to simulate the mixing temperatures in WMA and HMA, respectively. Researchers recognize that laboratory drying in an oven does not simulate the drying of aggregates in a drum mix plant. However, oven drying at the mixing temperatures is the most practical laboratory practice to approximately simulate the drying process in the drum mix plant. The moisture content of the aggregates was determined by

weighing the aggregates after 2, 4, 6, 12, and 24 hours. The dry weight after 24 hours was considered as the ultimate dry weight of the aggregate.

Test results indicated that at both temperatures the aggregates did not lose any additional moisture merely after 2 hours of drying. In addition, there was no significant difference in the moisture lost by the aggregates dried at 130°C (266°F) as compared to the aggregates dried at 150°C (302°F). Therefore, the researchers concluded that even with the reduced mixing temperatures in a WMA were still high enough to dry the moisture absorbed or adsorbed in the aggregate particles. Figure 2.6 illustrates the weight of aggregates cured in the oven at the two temperatures and different drying times. The results were consistent with another recent study by Xiao et al. (2010), which demonstrated that mixtures containing moist aggregates generally had similar indirect tensile strength values compared to other mixtures regardless of the type of mixture (HMA or WMA). Based on the findings from this short study, other results from Task 3 and inputs from TxDOT, the researchers decided that the experimental design for the mortars should be refined to emphasize aspects such as the influence of aging on fatigue cracking resistance in lieu of aspects such as incomplete drying of absorptive aggregates in WMA.
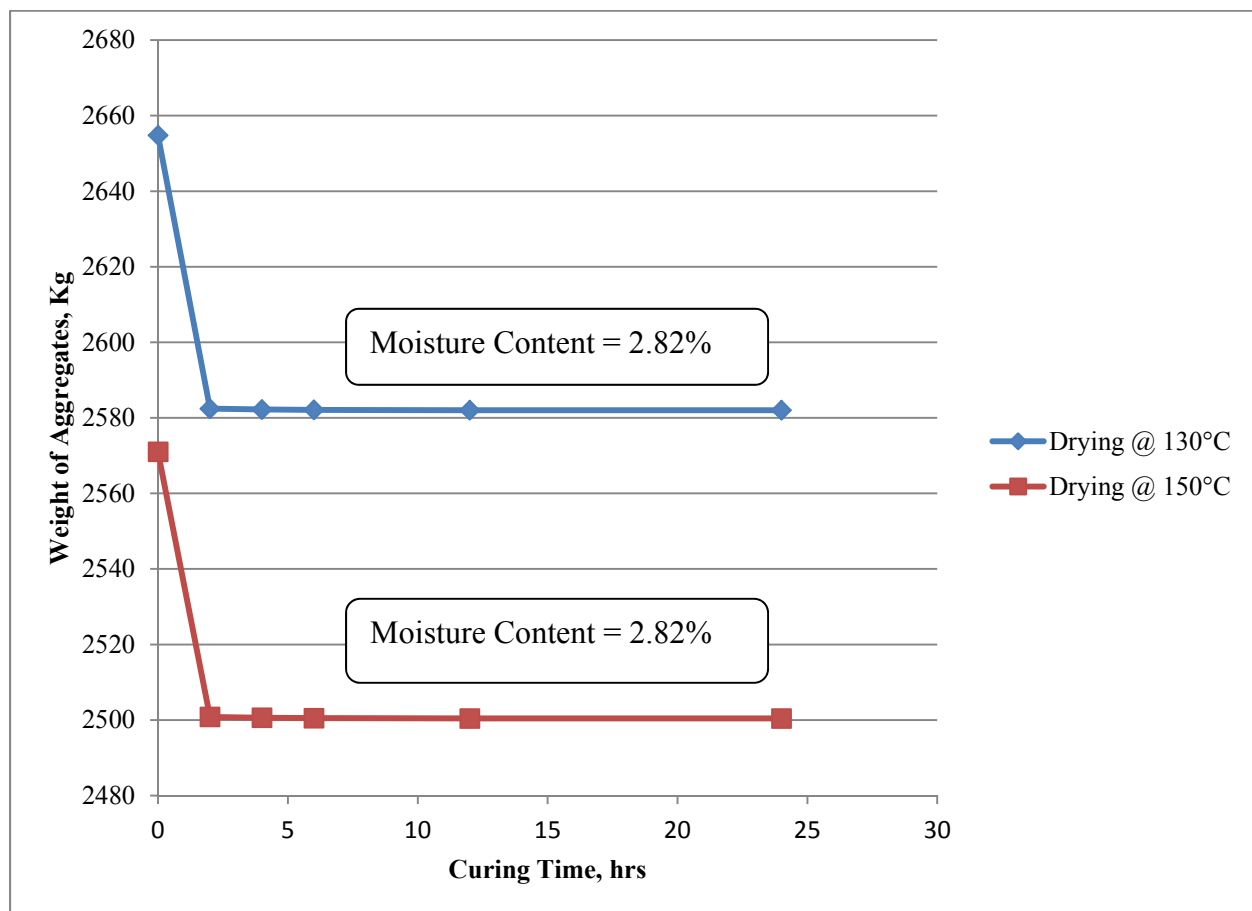

Figure 2.6: Influence of mixing temperature on moisture content of aggregates

2.6 Summary

In summary, 7 different asphalt binders were selected from a pool of 34 binders commonly used in Texas. In addition to the different PG grades, the binders were also carefully selected to represent a range of chemical properties such as wax content and total acid content. Selection of binders based on these properties was warranted to ensure that interactions between chemical WMA additives and asphalt binders, if any, could be detected using the laboratory tests. Six different WMA additives were selected based on the broad categories of chemical WMA additives and the usage. Finally, a short study was conducted to investigate whether or not absorptive aggregates completely dried when subjected to reduced mixing temperatures. Results from this short study indicated that 2 hours of oven drying resulted in no significant difference between the aggregates dried at 150°C (302°F) compared to aggregates dried at a reduced temperature of 130°C (166°F).

Chapter 3. Rheology of Binders in WMA Mixtures

3.1 Introduction

Based on a review of the existing literature on the chemical additives used to produce WMA, crystalline wax content and acid content of binders were identified as prominent binder properties that could potentially interact with these additives and influence rheological properties of binders. Addressing Task 3 of this research project, Chapter 3 evaluates the influence of the chemical makeup of asphalt binders (crystalline wax content, and acid content), WMA additives, and reduced short-term aging temperature on the viscosity, stiffness, susceptibility to permanent deformation, fracture resistance, and thermal cracking resistance of asphalt binders.

Test procedures used in this Task include measurement of viscosity using a Brookfield viscometer, measurement of complex modulus using a DSR, measurement of creep compliance at low temperatures using a BBR, and measurement of extent of oxidation using a Fourier Transform Infra-red (FT-IR) spectroscopy. The viscosity testing was used to determine the influence of the aforementioned materials properties on the viscosity of binders at different aging times and temperatures. The DSR was used to run frequency sweep and multiple stress creep recovery (MSCR) tests using short-term and long-term aged binders. The frequency sweep test was used to evaluate rutting, fatigue cracking, and thermal cracking resistance of the asphalt binder. The MSCR test was used to further evaluate rutting resistance of the asphalt binder. The BBR test was conducted to evaluate the low temperature properties of the asphalt binder.

This Task was divided into four phases. For each phase, a set of objectives were defined and a separate experimental plan was developed. This chapter of the report presents objectives, research approach, and description of phases undertaken to accomplish the objectives of this Task.

3.2 Phase I: Influence of WMA Additives and Reduced Aging on the Rheology of Asphalt Binders with Different Natural Wax Contents

3.2.1 Objectives

The main objectives of the first phase of this Task were to

1. quantify the individual and combined influence of reduced aging and interactions between the WMA additive and the asphalt binder on the viscosity and rheological properties of the binder after short-term and long-term aging,

2. quantify the impact of longer storage or haul times on the viscosity of waxy asphalt binders with varying natural wax content at compaction temperatures, and

3. determine whether naturally crystallizable materials (natural wax) present in the asphalt binder interact with certain WMA additives and influence the rheological properties after short-term and long-term aging.

To accomplish these objectives, the research team developed a testing plan that included four binders, five WMA additives, and two controls. The 4 binders were screened from 34 binders based on DSC tests conducted as a part of Task 2 of this project (Chapter 2). The two binders (PG76-22HW and PG76-28HW) have the highest wax content among the 34 binders

tested, and the other two binders (PG64-22A and PG64-22B) have the lowest wax content. The five WMA additives were selected after a review of the existing literature about WMA additives used in practice. The use of the two control binders (Control A and Control B) is necessary to distinguish between the influence of reduced short-term aging and the influence of the WMA additive on the properties of the asphalt binders. Figure 3.1 illustrates a summary of the tests that were conducted for each binder. At least two test replicates were conducted for each test.
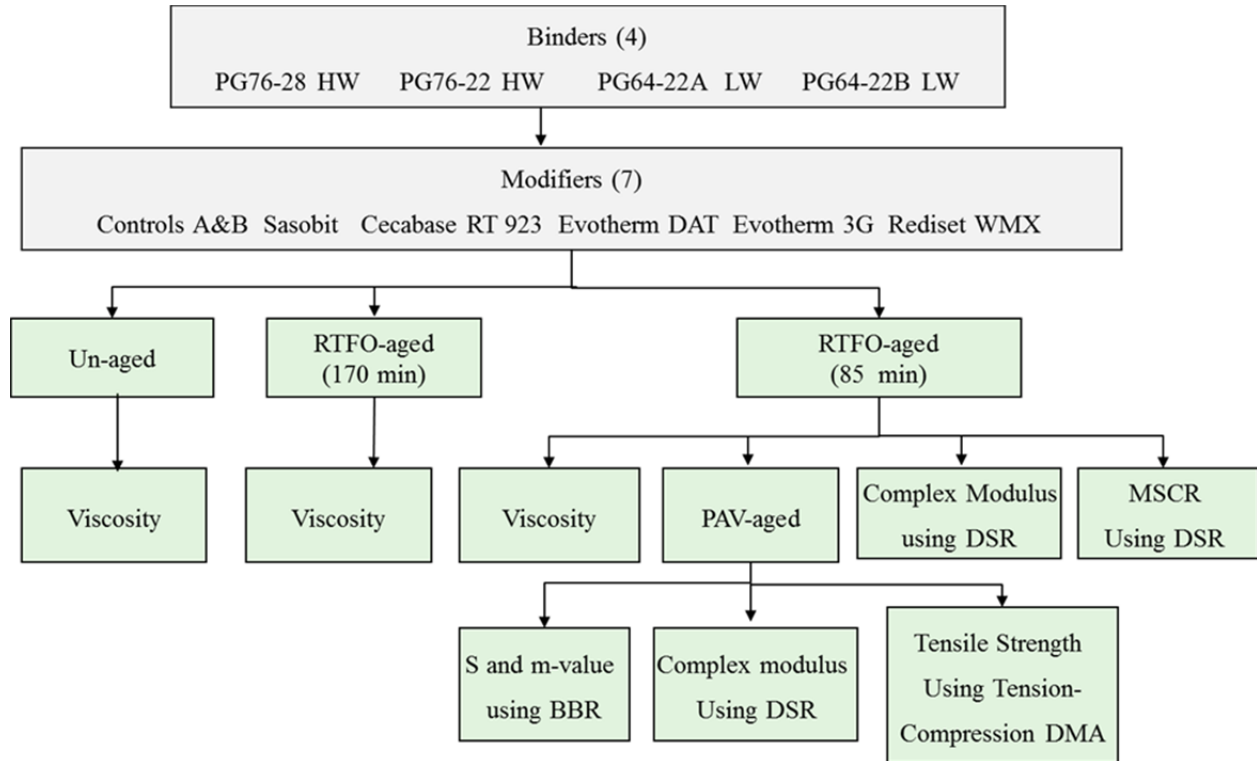

Figure 3.1: Summary of materials and test procedures

3.2.2 Viscosity

In most cases there is a time difference between production and placement of asphalt mixtures. This time difference is typically assumed to be about 2 hours for the purposes of simulating short-term aging of asphalt binders and mixtures. In some cases, such as when the mixture is produced in large quantities and stored in a heated silo or transported over longer distances (as may be the case for WMA), the time gap between production and placement is significantly longer. While reduced viscosity is not the only mechanism by which improved workability is achieved in WMA (Hanz et al. 2010), it still provides a valuable indicator to monitor the changes in the workability of the binder as it ages from the time of production to the time of placement. Consequently, the viscosity of the asphalt binders was measured after 0, 85, and 170 minutes of RTFO aging. Viscosity of the binders was measured using a Brookfield viscometer at 135°C (275°F) and a rotational speed of 20 rpm with the Brookfield spindle number 27. Measurements were taken after the viscosity reading stabilized.

Figures 3.2 through 3.6 illustrate the viscosity after being RTFO aged for 0, 85, and 170 minutes for four different binders, with two controls and five modifiers for each binder. Recall that Control A for each binder was RTFO aged at 163°C (325°F) and Control B for each binder

was RTFO aged at 143°C (289°F). As the focus of the study was to compare the effect of reduced aging and modifiers on the properties of the asphalt binder, all results are normalized with respect to the viscosity of Control A and Control B. Figure 3.2 illustrates the viscosity of the binder at the time of production (prior to RTFO aging), consequently the viscosity of Control A is the same as Control B.
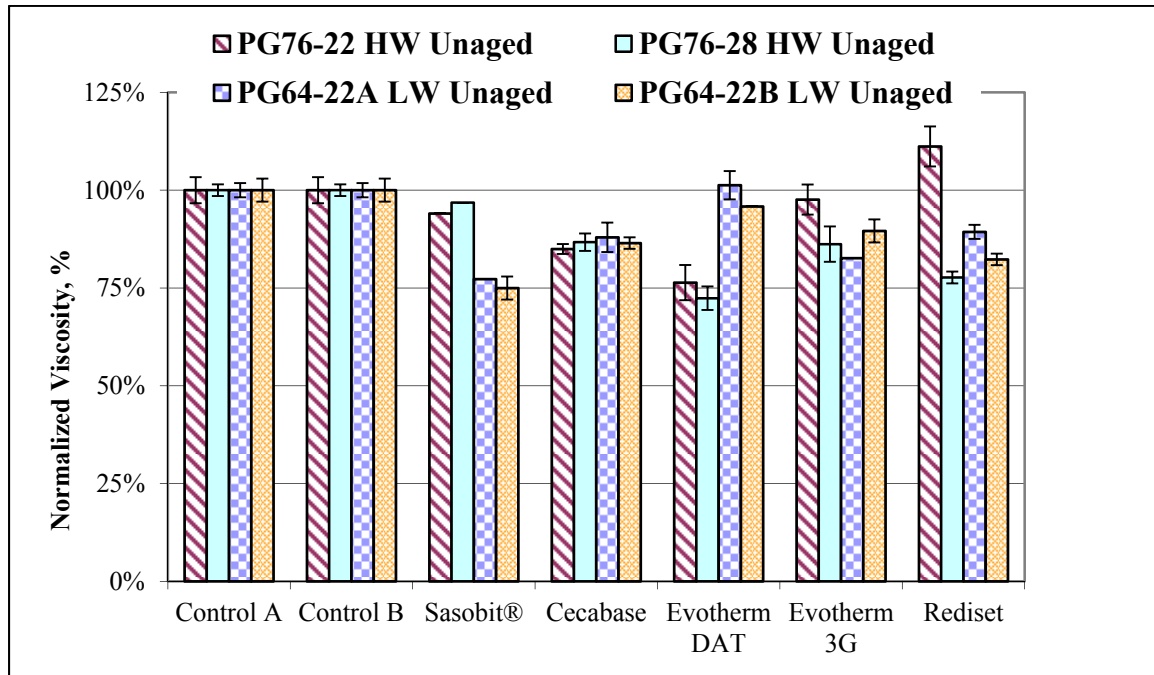

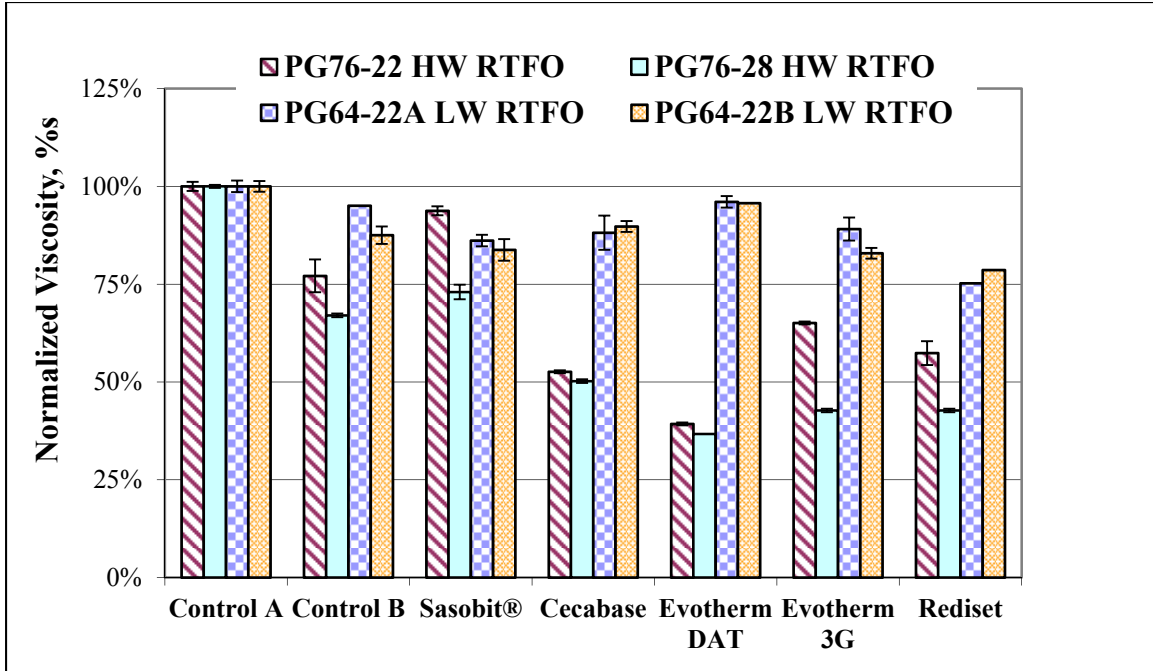*Figure 3.2: Influence of WMA additives on viscosity of un-aged binders measured at 135°C*

Figure 3.3: Influence of reduced short-term aging and WMA additives on viscosity of RTFO-aged binders measured at 135°C (results normalized by Control A)

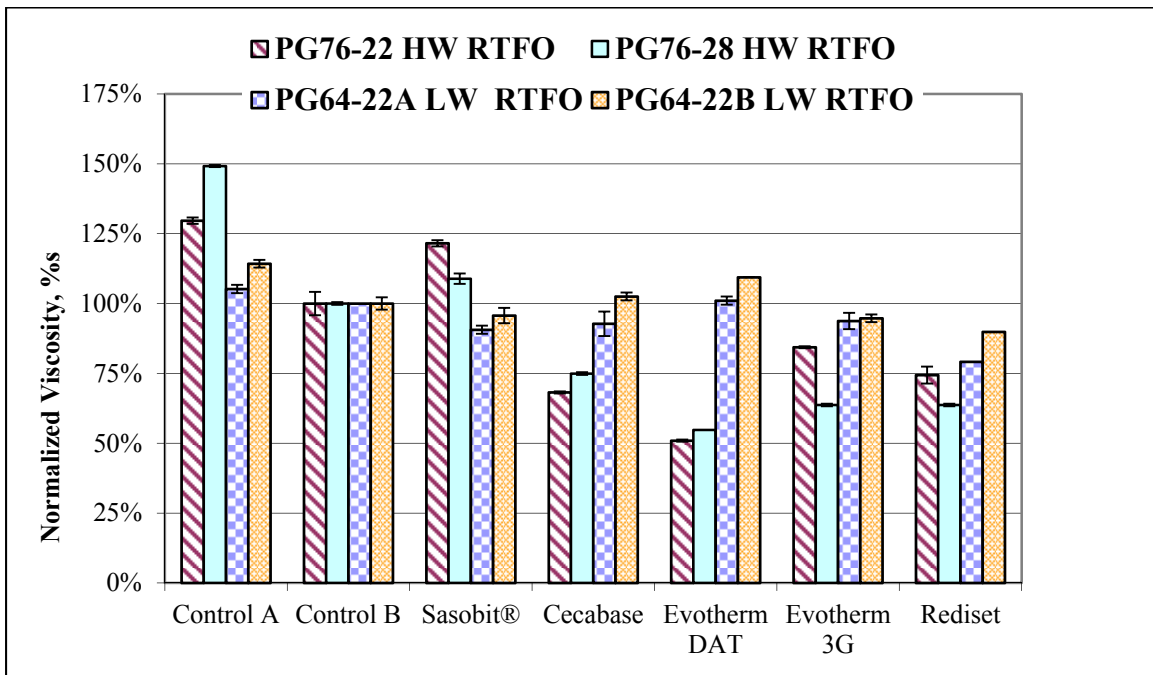

Figure 3.4: Influence of WMA additives on viscosity of RTFO-aged binders measured at 135°C (results normalized by Control B)
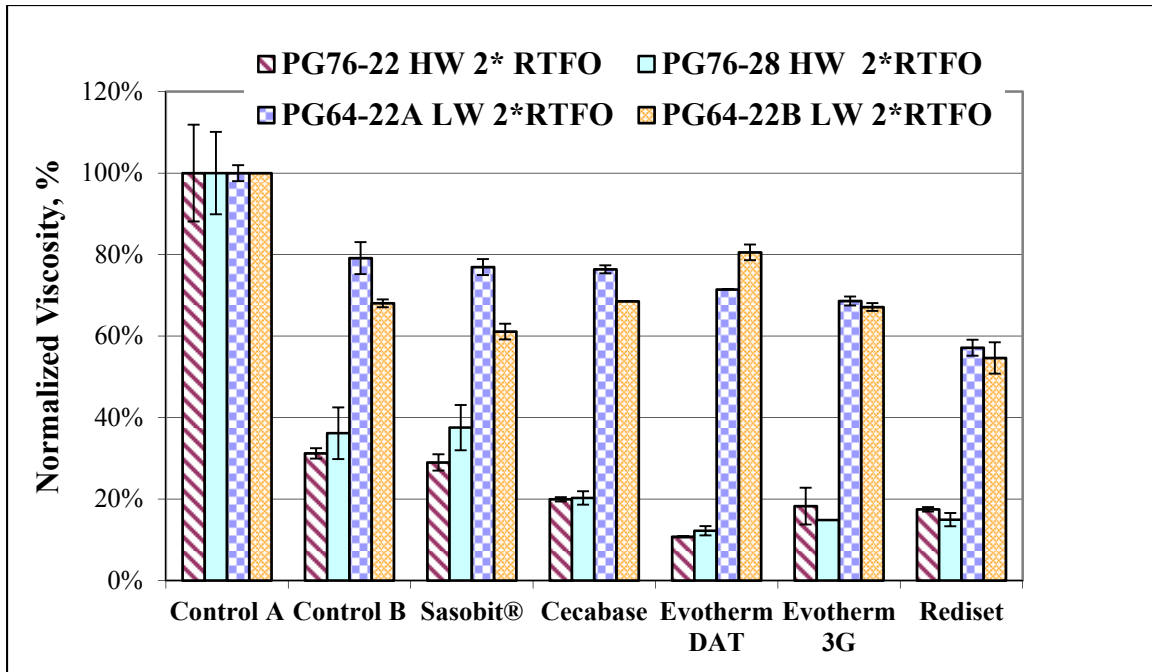
Figure 3.5: Influence of reduced short-term aging and WMA additives on viscosity of extended RTFO-aged binders (results normalized by Control A)
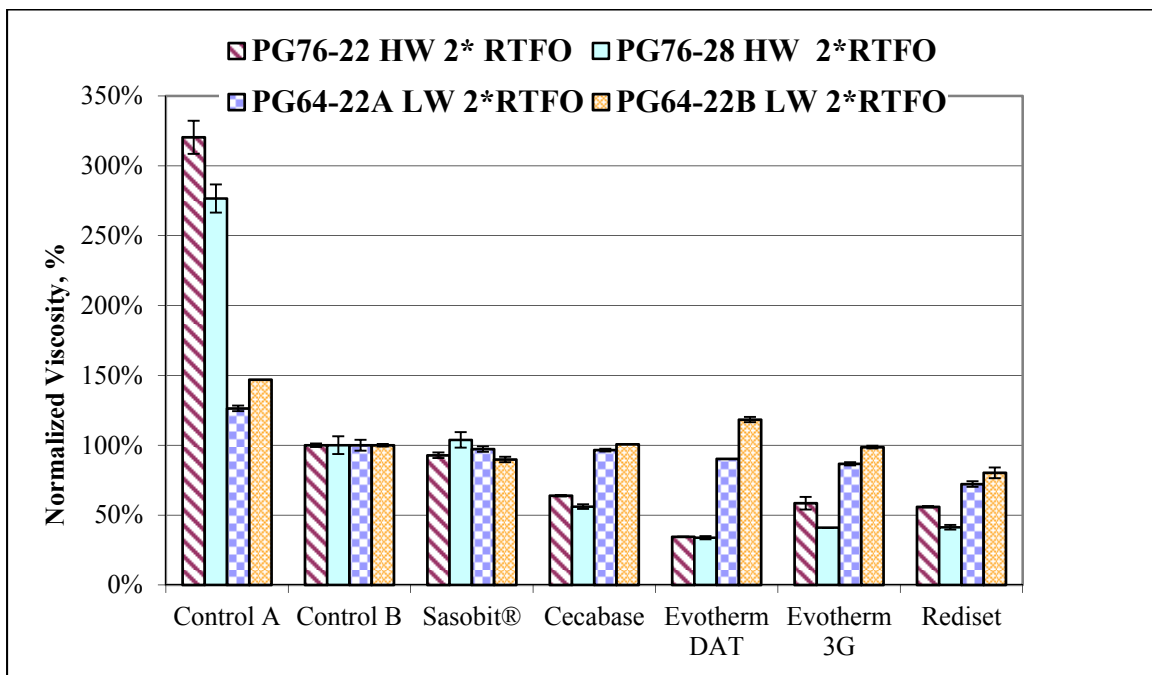

Figure 3.6: Influence of WMA additives on viscosity of extended RTFO-aged binders (results normalized by Control B)

Most of the results are self-explanatory from the figures but the following conclusions can be drawn based on these results.

- For the unaged binders (0 RTFO aging time), with minor exceptions, all additives reduced the viscosity of the binder measured at 135°C (275°F).

- For Cecabse, Evotherm, and Rediset additives, the reduced viscosity of the binders compared to the hot-mix binder (Control A) was not only due to reduced short-term aging at WMA temperatures but also due to the presence of the additive itself (Control B vs. binders with additives from Figures 3.4 and 3.6). This effect was more pronounced for binders with higher natural wax content as compared to binders with lower wax content. However, for the Sasobit additive, the reduced viscosity of the binders compared to the hot-mix Control A is mostly attributed to reduced short-term aging at the WMA temperatures (Control B vs. binders with Sasobit from Figures 3.3, 3.4, 3.5, and 3.6). Unlike other additives, this effect was more pronounced for binders with lower natural wax content as compared to binders with higher natural wax content.

- Binders with high natural wax content demonstrated a significant increase in the viscosity when RTFO aged for longer durations at the standard temperature of 163°C (325°F) compared to binders aged for the same duration at 143°C (289°F) with or without additives (Control A vs. Control B and others from Figures 3.5 and 3.6). The two binders with the high wax content were also the two binders with the higher high temperature PG grade. It is speculated that the higher wax content may be a reason for the higher temperature PG grade and it may not be possible to separate the influence of the two factors without a significantly larger test matrix that was beyond the scope of this work. Although this study incorporates a very limited number of binders, these results suggest that caution must be exercised while storing hot mix in heated silos, especially if the mix contains binders with high natural wax content.

3.2.3 Permanent Deformation

Stiffness of RTFO-Aged Binders

The high temperature performance of binders was investigated based on the Superpave $G^*/\sin\delta$ parameter at 10% strain, 10 radians/second frequency, and at the high PG temperature of the asphalt binder. Figures 3.7 and 3.8 present the test results normalized by the values of Control A and Control B. As expected, reduced temperatures for short-term aging resulted in reduced values for $G^*/\sin\delta$ (Control A vs. Control B in Figure 3.7 or Figure 3.8).

When compared to Control B, results showed similar or increased values of $G^*/\sin\delta$ for all binders modified using Sasobit and a similar or reduced values $G^*/\sin\delta$ for all binders modified with either chemical- or surfactant-based additives. This indicates that while Sasobit tends to compensate for the reduced stiffness due to reduced short-term aging, other additives either do not interact or tend to further reduce the stiffness of the binder.
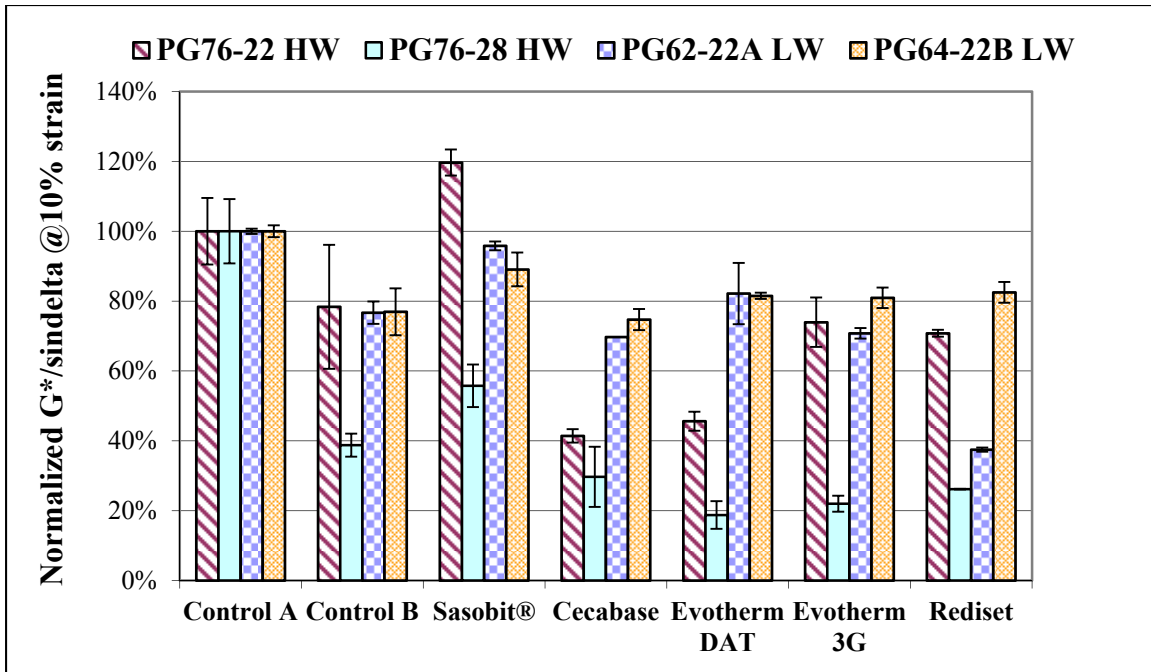
Figure 3.7: Influence of reduced short-term aging and WMA additives on resistance to permanent deformation (results normalized by Control A)
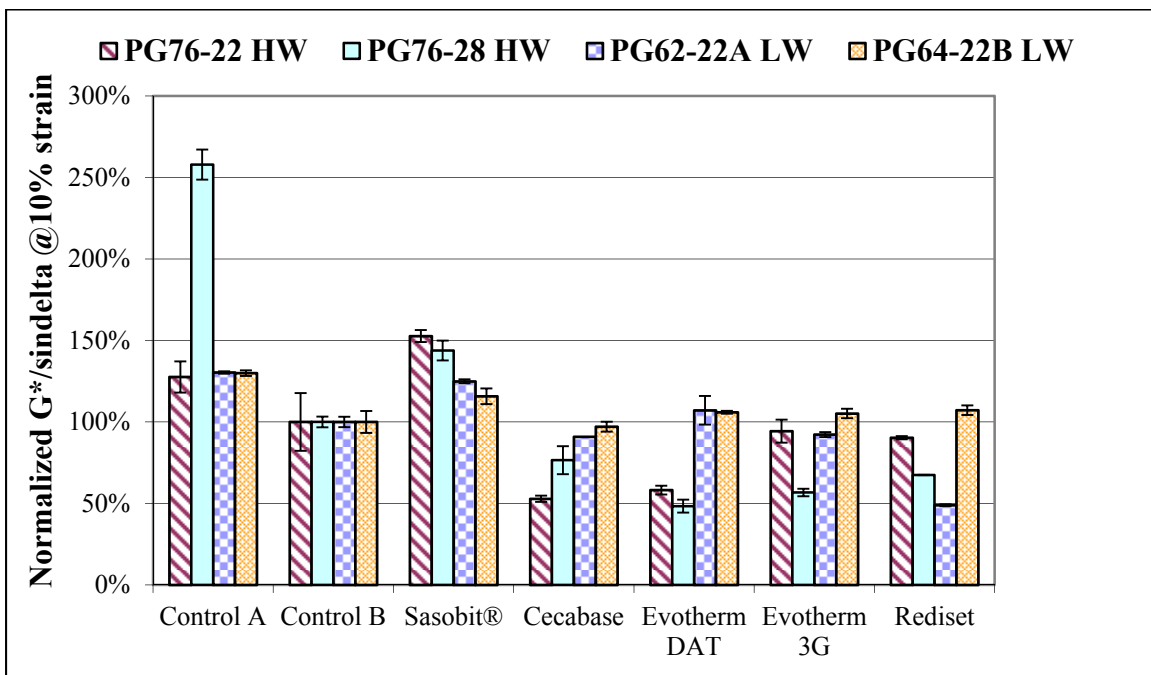

Figure 3.8: Influence of WMA additives on resistance to permanent deformation (results normalized by Control B)

Multiple Stress Creep and Recovery (MSCR) Test

The permanent deformation characteristics of the asphalt binder are critical but not the only drivers of permanent deformation in asphalt mixtures. In this phase of the study, in addition to G*/sinδ the MSCR test was also used to compare the permanent deformation characteristics of the modified binders compared to their respective control binders. The MSCR test was carried out as per AASHTO TP 70-07, where a 25 mm (1 inch) diameter and 1 mm (0.04 inches) thick asphalt specimen is subjected to 10 cycles of 1 second creep loading followed by 9 seconds rest period at stress levels of 100 Pa and 3200 Pa at the high PG temperature using the DSR. Tests were conducted at 64°C (147°F) for the binders with the PG 64 grade and at 76°C (169°F) for the binders with the PG 76 grade.

Figures 3.9 and 3.10 present the non-recoverable creep compliance (*Jnr*) values following the MSCR protocol at stress levels of 3200 Pa. Results using the stress level of 100 Pa were similar to the results using the stress level of 3200 Pa and are not included in this report. The results in Figure 3.9 are normalized by the *Jnr* values of Control A and results in Figure 3.10 are normalized by the *Jnr* values of Control B for each asphalt binder.

Figure 3.9: Non-recoverable compliance (Jnr) from MSCR testing of binders tested at the high PG temperature and 3200 Pa loading (results normalized by Control A)

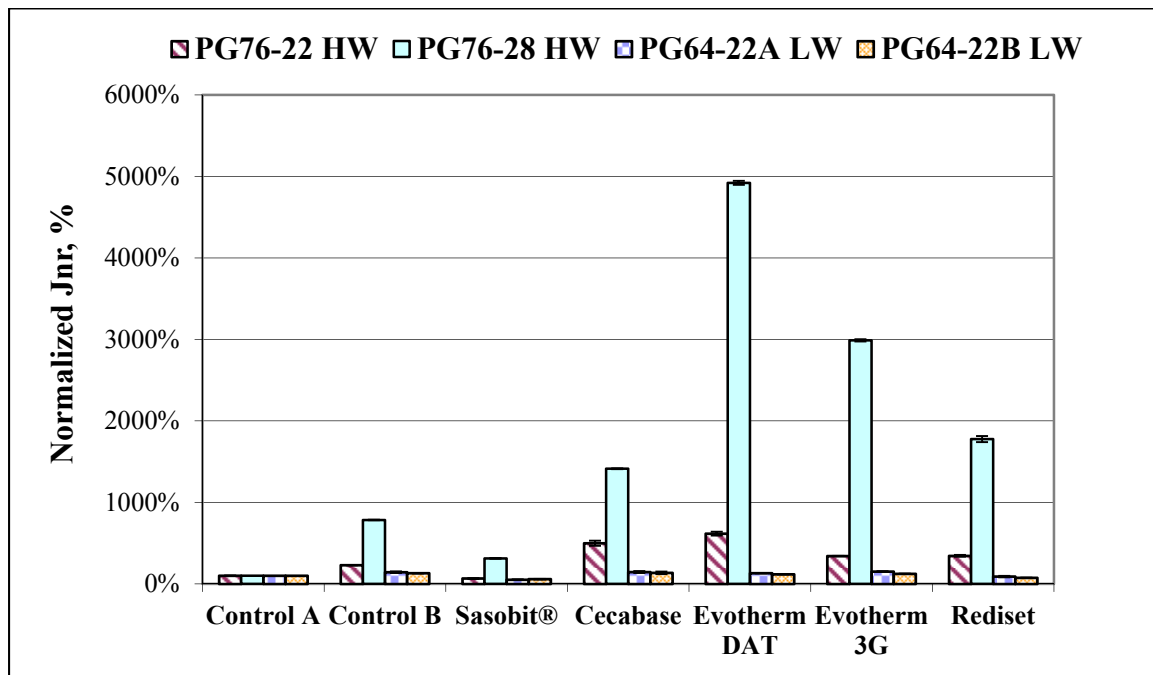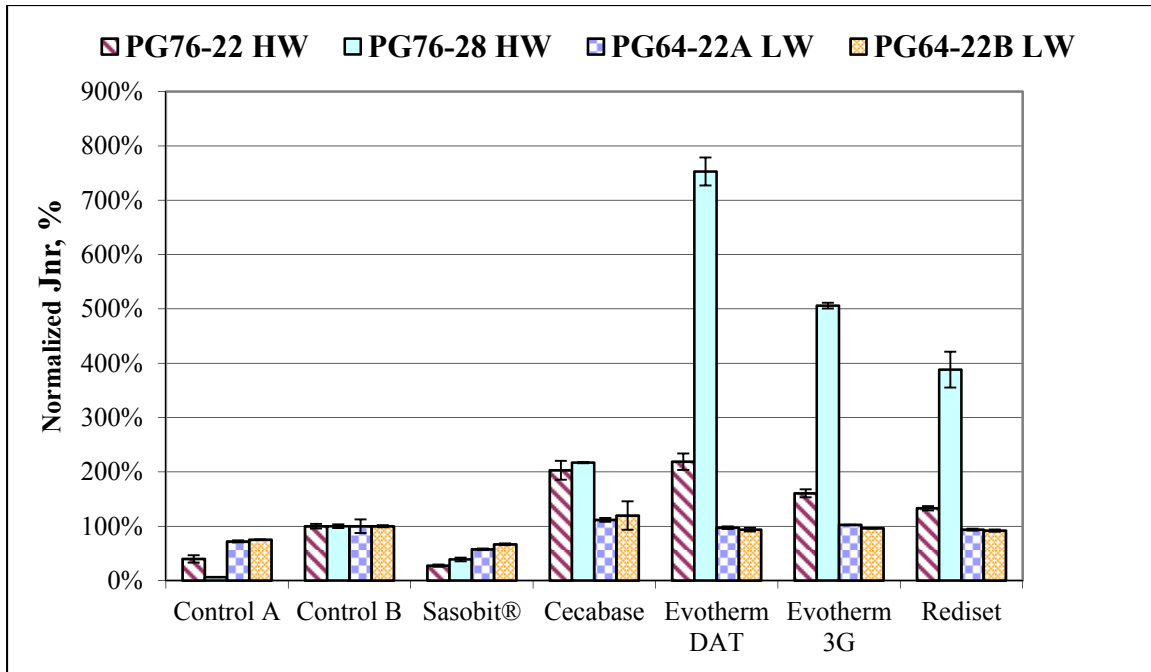*Figure 3.10: Non-recoverable compliance (Jnr) from MSCR testing of binders tested at the high PG temperature and 3200 Pa loading (results normalized by Control B)*

A comparison of Control A to Control B in Figure 3.9 illustrates that reduced aging does contribute to an increase in the permanent deformation of the asphalt binder. In the case of Sasobit, this effect was more than compensated due to the presence of the additive. Therefore, in most cases the net permanent deformation of the asphalt binder with Sasobit was similar to that of the binder representing the hot-mix (Control A). In the case of additives other than Sasobit, the *Jnr* of the binders with low wax content (PG 64-22A and 64-22B) was typically higher than Control A but almost similar to Control B, indicating that the decrease in resistance to permanent deformation was mostly due to the reduced aging. However, when these additives were used with the two binders with high natural wax content, the *Jnr* was significantly higher even when compared to Control B, indicating the possibility of a strong interaction between the additive and natural wax in the asphalt binder.

3.2.4 Fracture

Stiffness of PAV-Aged Binders

Intermediate-temperature performance of the PAV-aged binders was investigated using the Superpave $G^*\sin\delta$ parameter at 1% strain and 10 radians/second frequency measured using the DSR. Figures 3.11 and 3.12 illustrate the test results normalized by values of Control A and Control B. The influence of reduced short-term aging and WMA additives on the stiffness of PAV-aged binder was dependent on the type of binder and the additive. Although no specific trends were observed in terms of this parameter, some general observations are as follow. After PAV aging, the $G^*\sin\delta$ for binders with Sasobit was either similar to or slightly greater than Controls A and B. The only exception to this was the PG 76-28 binder. Similarly, the $G^*\sin\delta$ for binders with other WMA additives was either similar to or less than Controls A and B.

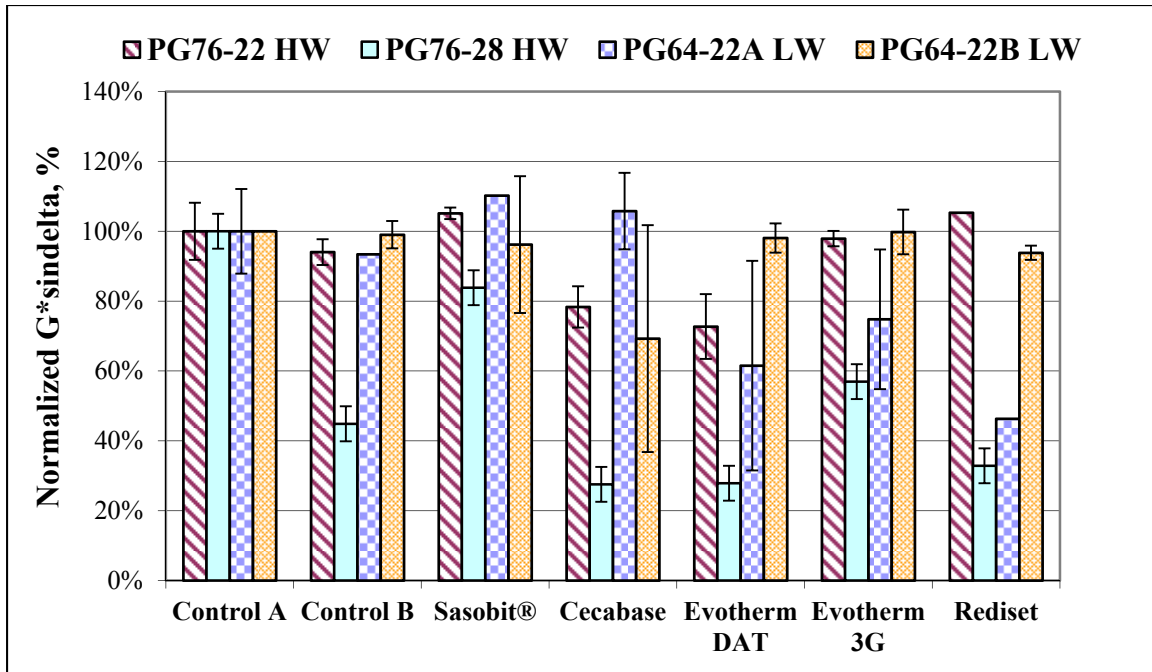*Figure 3.11: Influence of reduced short-term aging and WMA additives on G*sinδ of PAV-aged binders (results normalized by Control A)*
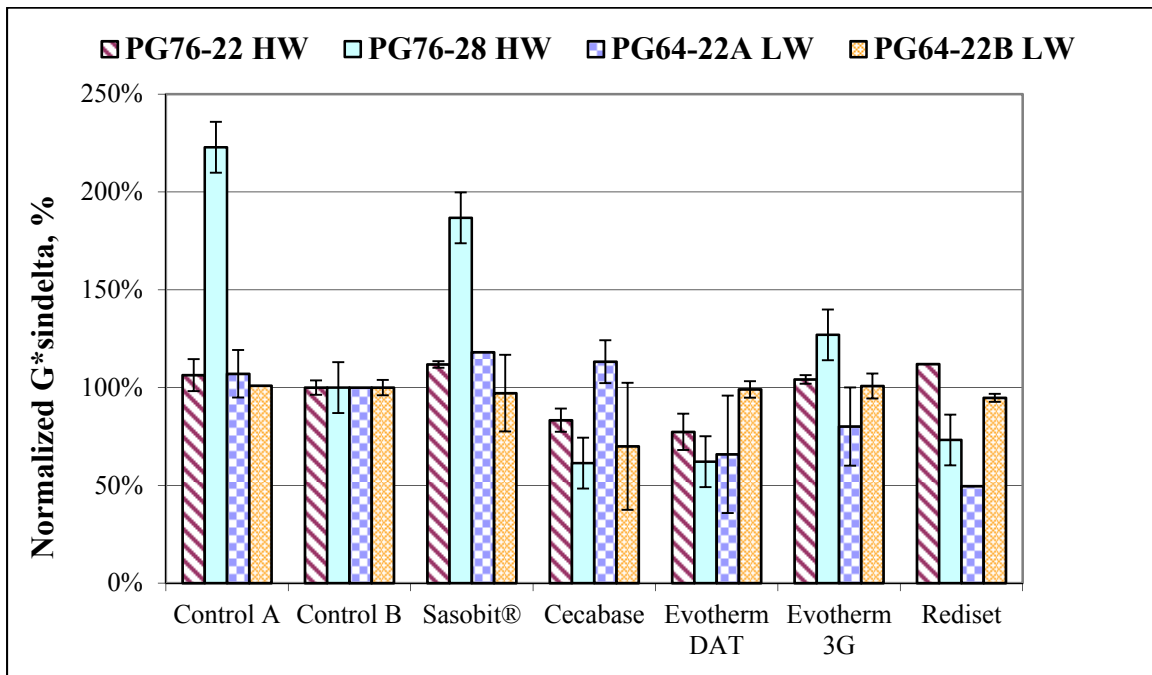

Figure 3.12: Influence of WMA additives on G*sinδ of PAV-aged binders (results normalized by Control B)

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Tensile Strength of PAV-Aged Binders

The G*sinδ parameter reflects only the stiffness of the asphalt binder and is not a direct measure of the inherent fracture or fatigue cracking resistance of the binder. In order to further investigate the fracture properties of the asphalt binder, a tension-compression DMA was used to measure the linear viscoelastic properties of the asphalt binder as well as its tensile strength. A summary of the procedure used to measure tensile strength and results obtained are presented here.

Disc-shaped specimens of PAV-aged asphalt binders with 14.7 mm (0.58 inches) diameter and 300 micron thickness were prepared between two metal end plates. The end plates were then clamped to the tension-compression DMA and a displacement controlled monotonic load at a rate of 100 μm/ min was applied at a temperature of 25°C (77°F) until tensile failure. The area under the stress-strain curve until yielding, referred to as the *critical stain energy density* (Jonson et al., 2009), was measured for different asphalt binders. The testing apparatus is shown in Figure 3.13. Figures 3.14 and 3.15 present the results from these tests on the PAV-aged binders.
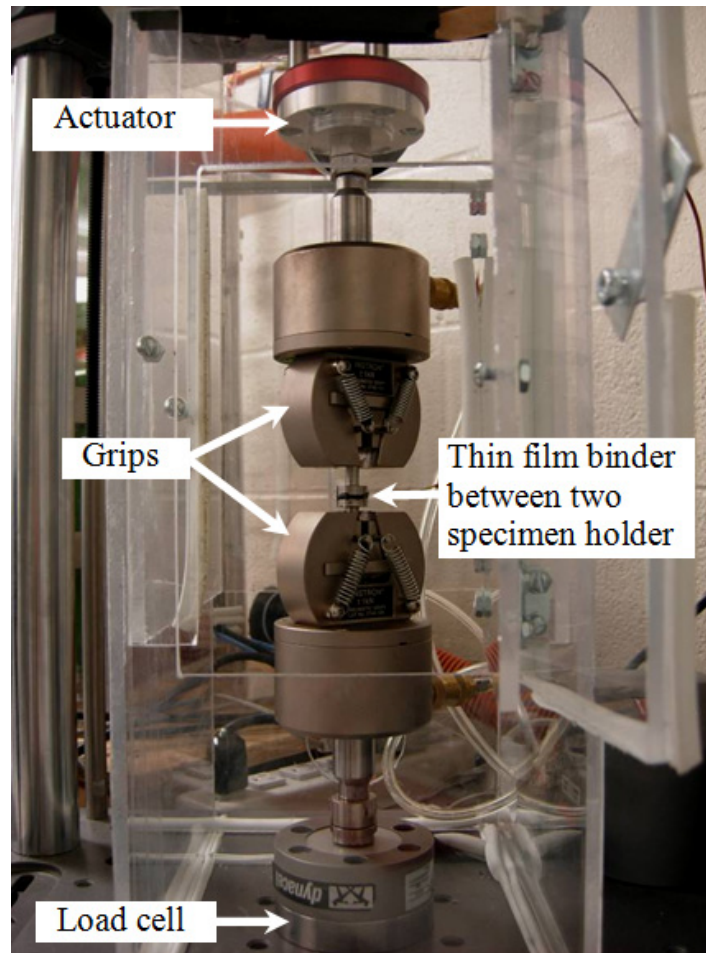

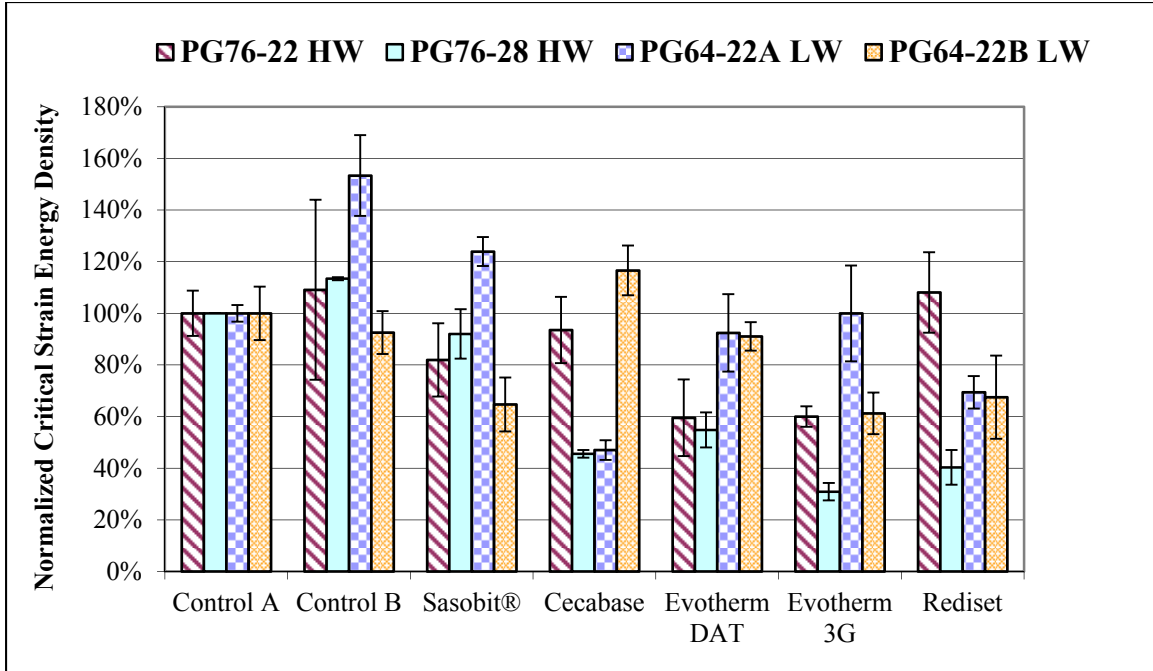*Figure 3.13: Thin film tension test equipment/configuration*

Figure 3.14: Influence of reduced short-term aging and WMA additives on fracture properties of PAV-aged asphalt binders (results normalized by Control A)
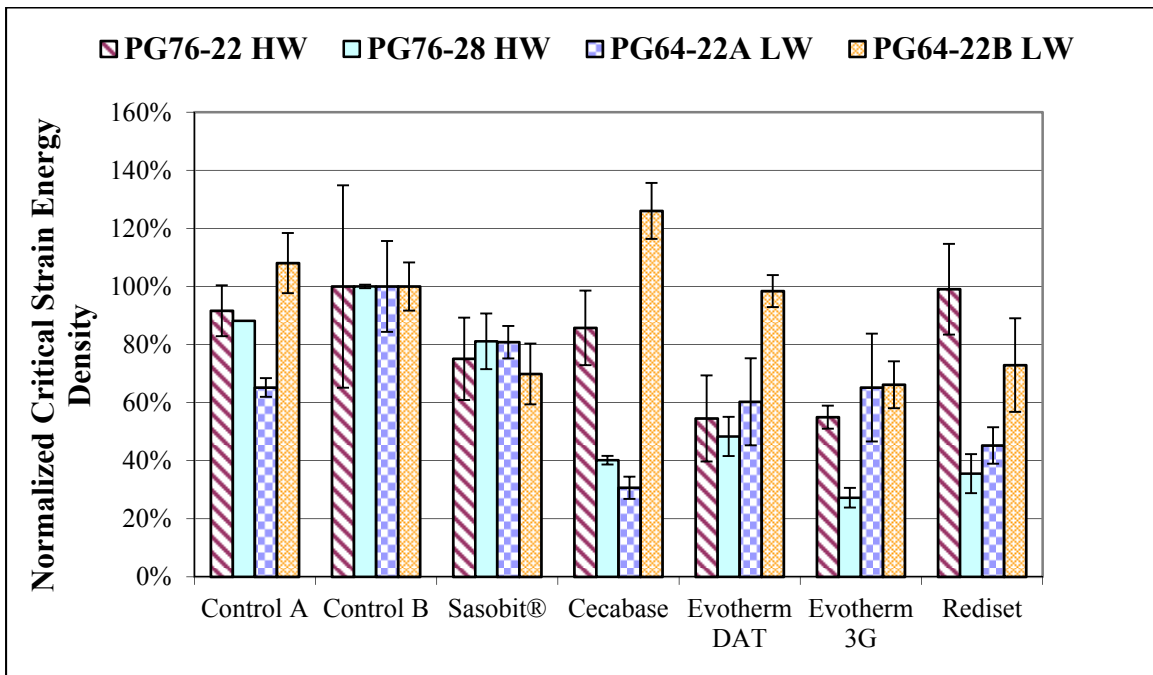

Figure 3.15: Influence of WMA additives on fracture properties of PAV-aged asphalt binders (results normalized by Control B)

The results from this monotonic fracture test provided better insight into binder performance as compared to the results based on G*sinδ. For 10 out of 20 binder additive combinations, the strain energy until failure was at least 20% lower than the strain energy until failure for Control B (binder subjected to reduced short-term aging temperature). This suggests that, after long-term aging, the fracture resistance of modified binders in WMA is likely to reduce depending on the specific combination of the binder and additives. This reduction can be attributed to interaction between the binder and the additive.

3.2.5 Low Temperature Cracking

The effect of additives on the low-temperature properties of the binder was investigated based on the stiffness and m-value measured using the BBR. The BBR test was carried out as per AASHTO T313 on the PAV-aged residues. BBR testing was also used to investigate any interactions between the WMA additives and natural wax in asphalt binders at low-temperatures. Figures 3.16 through 3.19 present the stiffness and m-value of these four asphalt binders after normalizing based on the values of Control A and Control B. Results clearly indicate that the stiffness of the PAV-aged modified binders at low temperatures was either similar to or slightly higher than the stiffness of Control B. In addition, the m-value of the PAV-aged modified binders was either similar to or slightly less than the m-value of Control B. When used with Sasobit and Rediset WMX additives, binders with higher natural wax content were more susceptible to an increase in stiffness and a decrease in m-value as compared to the binders with low natural wax content. These results are consistent with the results based on stiffness and fracture energy of PAV-aged WMA modified and control binders.

Figure 3.16: Normalized creep stiffness of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control A)
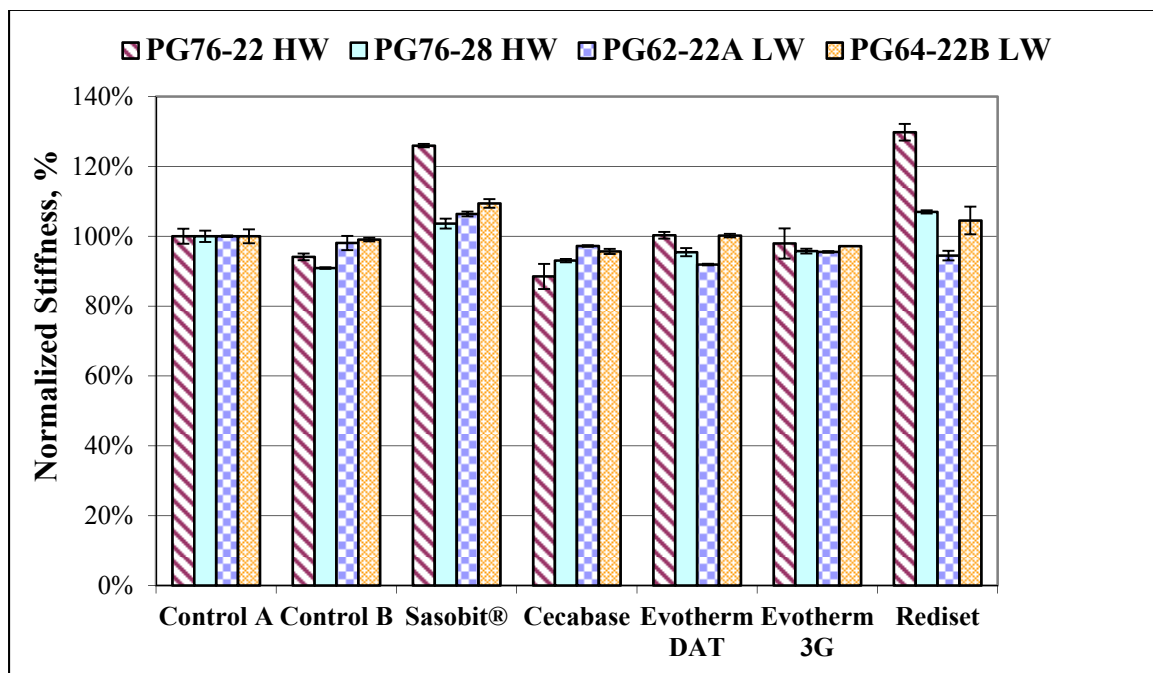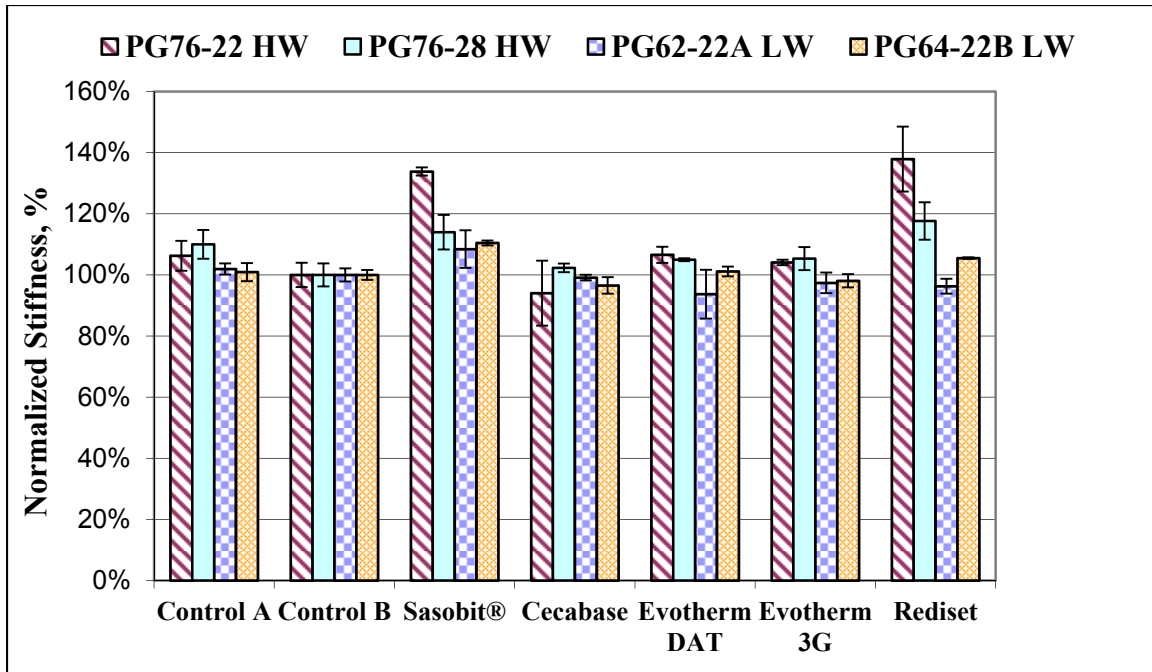
Figure 3.17: Normalized creep stiffness of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control B)
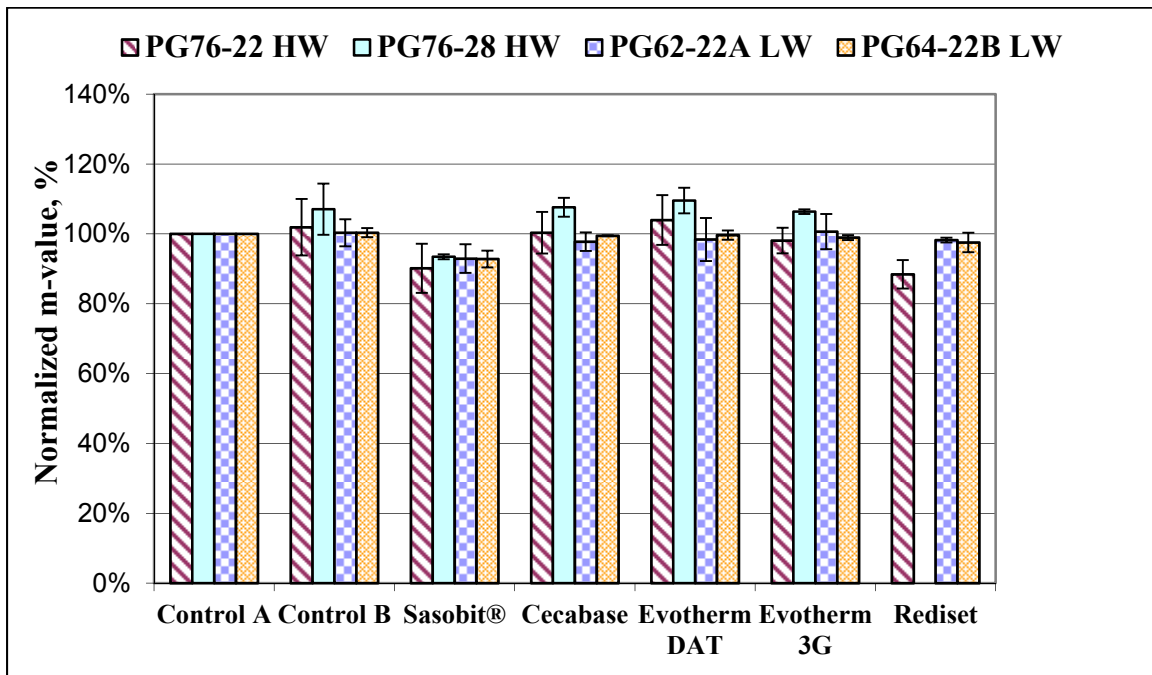

Figure 3.18: Normalized m-value of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control A)
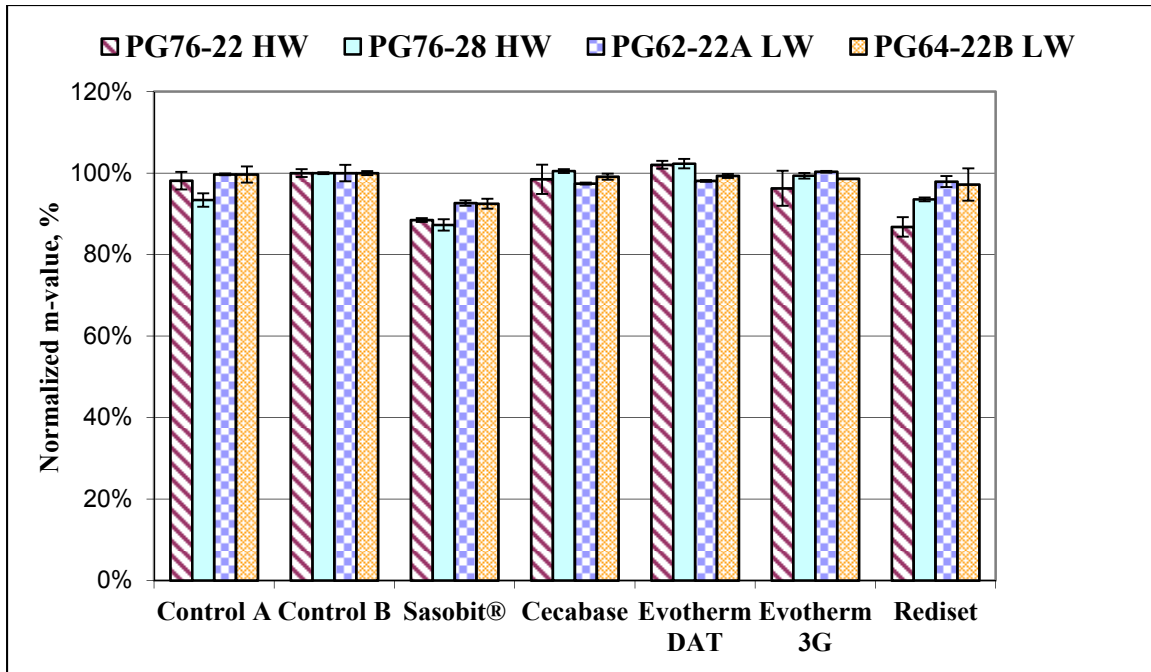
Figure 3.19: Normalized m-value of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control B)

3.2.6 Summary of Results from Phase I

Analysis of the test results collected in Phase I of this Task led to the following findings.

- Reduction in viscosity of the short-term aged binder is mostly due to the reduced aging of the binder at WMA temperatures. *In addition to reduced aging, WMA additives other than Sasobit further reduce the viscosity of short-term aged binders*, especially when used with binders with high natural wax content. This difference was more significant when binders were subjected to a longer duration of short-term aging. Moreover, *viscosity testing results showed that warm asphalt mixtures can be stored or hauled for longer amounts of time when compared with hot mix asphalt mixtures.*

- Binders used in WMA are likely to have reduced stiffness and increased susceptibility to permanent deformation (as quantified based on the MSCR protocol) due to the reduced temperatures during short-term aging. *The Sasobit additive compensates to varying degrees for this reduced stiffness and increased susceptibility to permanent deformation. Certain WMA additive-binder pairs demonstrate chemical interactions that further reduce stiffness of the binder and increase its susceptibility to permanent deformation. It must be noted that this reduced stiffness (or increased susceptibility to permanent deformation) due to chemical interactions is in addition to the reduced stiffness that is due to reduced short-term aging.* Binders with high wax content demonstrate a strong interaction with all WMA additives except Sasobit resulting in excessive permanent deformation that cannot be attributed solely to reduced short-term aging.

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- The cumulative effect of reduced short-term aging followed by PAV aging on the stiffness of the asphalt binders was dependent on the type of the binder and the WMA additive. *For most combinations of the PAV aged binders and WMA additives, the G*sinδ parameter was similar to that of the hot mix control binder.*

- *Results from the monotonic tensile strength of asphalt binders indicate that in many cases the strain energy until failure for PAV-aged binders with WMA additive reduced aging was dependent on the binder additive pair.* In other words, the PAV residues of approximately half of the WMA additive-binder pairs demonstrated a decrease in tensile strain energy when compared to the tensile strain energy of a similar binder representing a HMA. This indicates the possibility of chemical interactions between the additive and the binder that ultimately influence its performance in a WMA.

- Results show that reduced short-term aging does not appear to have a significant effect on either the stiffness or m-value of binders after PAV aging indicating a similar resistance to thermal cracking. In other words, *binders with WMA additives (other than Sasobit) that were subjected to reduced short-term aging followed by PAV aging had similar or slightly reduced thermal cracking resistance.*

- The results suggest that the WMA mix design procedure must incorporate measurement of properties of asphalt binders modified using the WMA additives for the following two reasons. First, several WMA additive-binders demonstrated interactions that are dependent on the binder chemistry as well as the additive. Second, current tests that evaluate cracking resistance of asphalt mixtures are conducted on short-term aged mixtures. This highlights the importance of measuring properties of long-term aged binders that are modified using WMA additives.

- *Strategies to compensate for the reduced initial stiffness in WMA (e.g. using recycled asphalt materials) must be tailored to the specific kind of binder and additive that is used in the mixture.* For example, certain WMA additives such as Sasobit may not result in reduced initial stiffness or susceptibility to permanent deformation and consequently may not be used with RAP. More importantly, in several cases PAV residues of asphalt binders that were subjected to reduced short-term aging had fractures, and low temperature properties similar to or lower than PAV residues of asphalt binders that were subjected to conventional short-term aging. Consequently, there is a possibility that the use of recycled asphalt or other methods to overcome the reduced initial stiffness in WMA may adversely affect fracture and low temperature properties in the long-term if not properly accounted for during the mixture design process. This affect is investigated in section 3.4 of this report.

It is important to note that the conclusions related to the natural wax content in the asphalt binders were based on limited number of binders that were included in the tests. It is possible that other factors common to the binders with high or low wax content might influence these conclusions.

3.3 Phase II: Influence of WMA Additives and Reduced Aging on the Rheology of Asphalt Binders with Different Natural Acid Contents

3.3.1 Objectives

The main objectives of the second phase of this Task were to

1. quantify the individual and combined influence of reduced aging and interactions between the WMA additive and the asphalt binder selected based on natural acid content on the viscosity and rheological properties of the binder after short-term and long-term aging, and

2. determine whether acids present in the asphalt binder interact with certain WMA additives and influence the rheological properties after short-term and long-term aging.

To best accomplish these objectives, the research team followed the methodology described in Figure 3.20. Selection of materials and development of testing plan for this phase was based on the results of Phase I of this task. The testing plan included four binders, two WMA additives, and three controls (including dry Advera) to accomplish these objectives. The four binders were screened from 34 binders based on titration tests conducted in Task 2 of this project. The two binders (PG64-22C and PG76-28) have the highest acid content, and the other two binders (PG70-22s and PG64-22D) have the lowest acid content amongst the 34 binders tested. The two WMA additives selected were Cecabase and Advera. The two additives were selected based on the potential interactions of these two additives with natural acids present in the binder. In addition to the two WMA additives, two control binders (Control A and Control B), and a dehydrated Advera were used to distinguish between the influence of reduced short-term aging and the influence of the WMA additive on the properties of the asphalt binders. Advera was dried in the oven at 150C for 24 hours to produce the dehydrated Advera. Advera is a particulate additive and therefore in addition to its potential chemical or physio-chemical interactions, it also directly contributes to the mechanical properties of the binder and the mix. Due to the filler stiffening effect of Advera on binders, it was decided to use binders modified with dry Advera for comparison with binders modified with Advera. It is also important to note that dry Advera only has a filler effect without serving as a WMA agent.

The test plan was developed to evaluate the impact of reduced aging, acid content of binders, and WMA additives on viscosity, resistance to permanent deformation, fatigue cracking, and thermal cracking of binders selected based on their acid content.
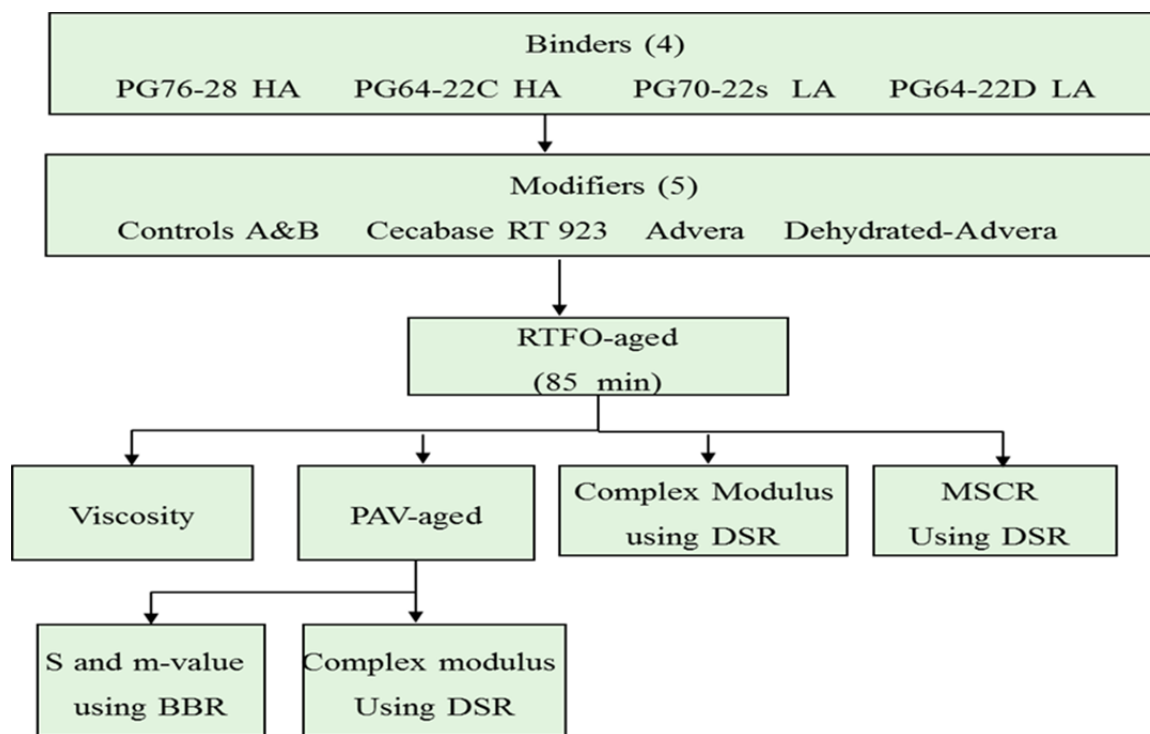
Figure 3.20: Summary of materials and test procedures

3.3.2 Viscosity

The viscosity test was used to monitor the changes in the workability of the binder as it ages from the time of production to the time of placement. The viscosity of the asphalt binder was measured after 85 minutes of RTFO aging. Viscosity tests were conducted using Brookfield viscometer at a testing temperature of 135°C (275°F) and rotational speed of 20 rpm with spindle number 27. The test results presented in Figures 3.21 and 3.22 show the differences in viscosity between modified binders and controls after RTFO aging. As expected, Control A has higher viscosity than the modified binder. PG76-28HA exhibits lower viscosity for all the additives compared to Control B. For all the other modified binders, the viscosity essentially remained the same as Control B. It is important to note that PG76-28HA has high acid and wax contents. Binders modified with Advera and dehydrated Advera exhibit higher viscosity compared to Control B; it is speculated that the increase in viscosity is due to filler stiffening effect. However, an important observation here is that viscosity of binders modified using a dehydrated zeolite was not very different from the viscosity of the binders modified using hydrated or regular zeolite. This suggests that perhaps mechanics, rather than viscosity reduction due to foaming, are responsible for the actions of this WMA additive. The results presented in Figures 3.21 and 3.22 do not seem to indicate any specific trends of interactions between acid content and WMA additive.

Analysis of the viscosity test results led to the following findings.

- For all the binders, except PG76-28HA, the reduced viscosity of the binders compared to Control A was due only to reduced short-term aging at WMA temperatures. In fact, binders modified with Advera and dehydrated Advera showed a slight increase in viscosity for most binders due to the filler stiffening effect.

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- The amount of acid present in binders does not seem to have a significant or consistent interaction with the WMA additive, as reflected by the viscosity.
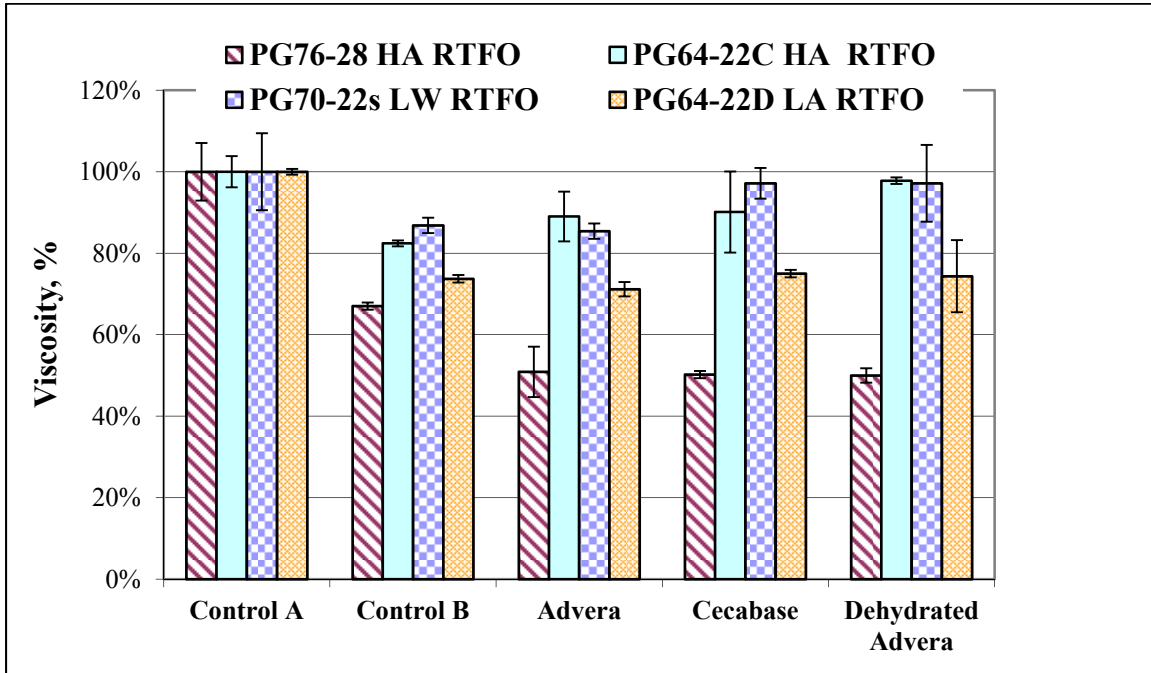

Figure 3.21: Influence of reduced short-term aging and WMA additives on viscosity of RTFO-aged binders measured at 135°C (results normalized by Control A)
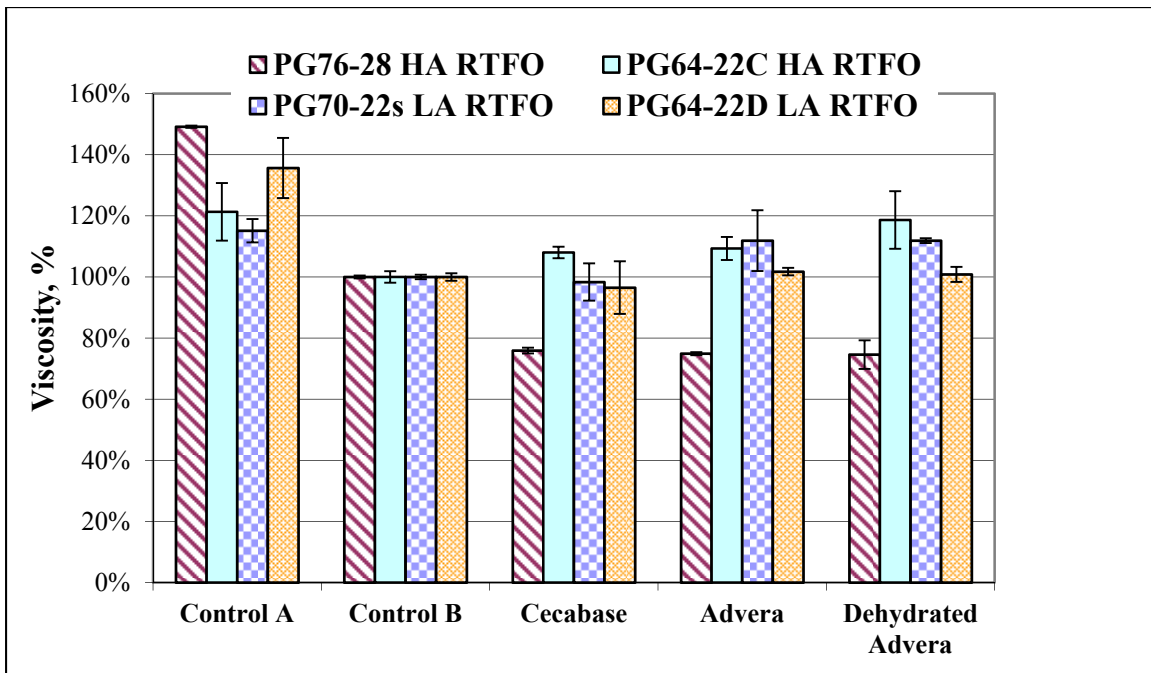

Figure 3.22: Influence of WMA additives on viscosity of RTFO-aged binders measured at 135°C (results normalized by Control B)

3.3.3 Resistance to Permanent Deformation

Stiffness of RTFO-Aged Binders

        Development of resistance to permanent deformation was evaluated using the Superpave G*/sinδ parameter at a strain of 10% and frequency of 10 rad/sec. The tests were conducted at the high PG temperature of the respective binder. Resistance to permanent deformation is characterized by an increase in stiffness and a decrease in phase angle by the asphalt binder. The test results normalized by Control A and Control B are summarized in Figures 3.23 and 3.24 respectively. When compared to Control A (equivalent HMA binder), all binder additive pairs, except PG64-22D LA, showed a reduction in the G*/sinδ parameter, indicating an increased susceptibility to rutting. However, this reduction in rutting resistance was not entirely due to reduced aging for Cecabase.

        When compared to Control B, results showed similar or reduced values of G*/sinδ for all binders (except PG64-22D LA) modified using Cecabase. This demonstrates that, in addition to reduced aging, Cecabase further reduced the stiffness of the binder. Moreover, lower values of G*/sinδ for binders modified with Advera compared to binders modified with dehydrated Advera indicate that the presence of water in the Advera zeolite does interact with the binder to reduce its stiffness (more specifically G*/sinδ). This reduction was most significant for the PG76-28 HA binder, which was not only high in acid but also high in wax content.
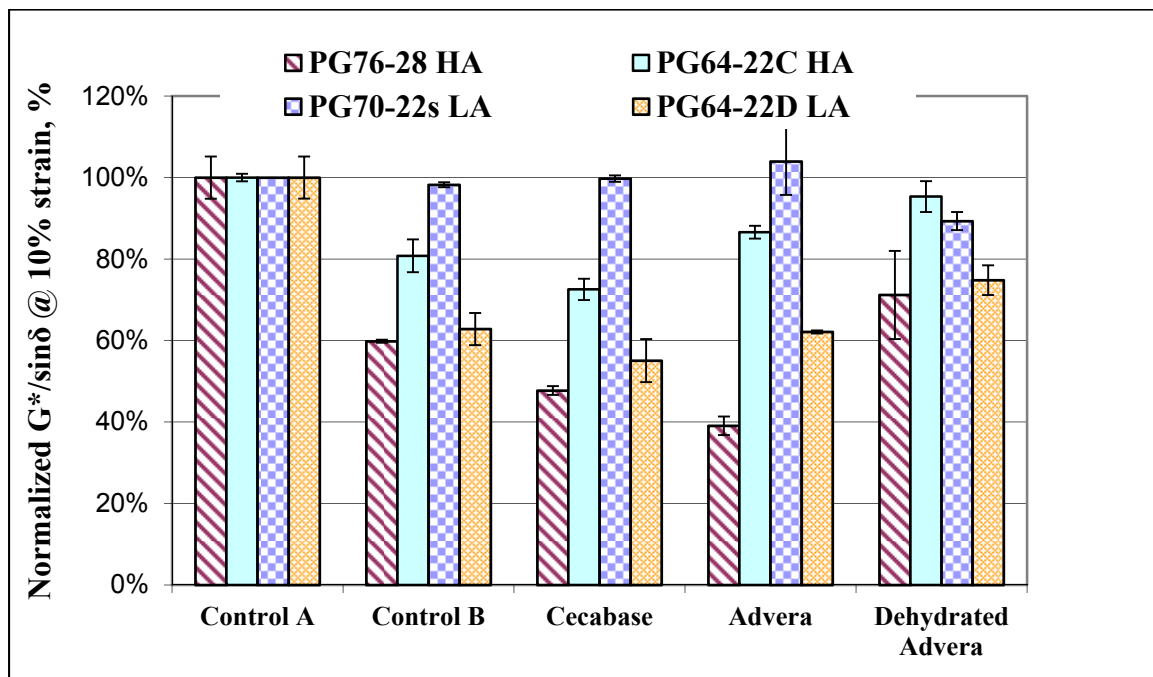

Figure 3.23: Influence of reduced short-term aging and WMA additives on resistance to permanent deformation (results normalized by Control A)
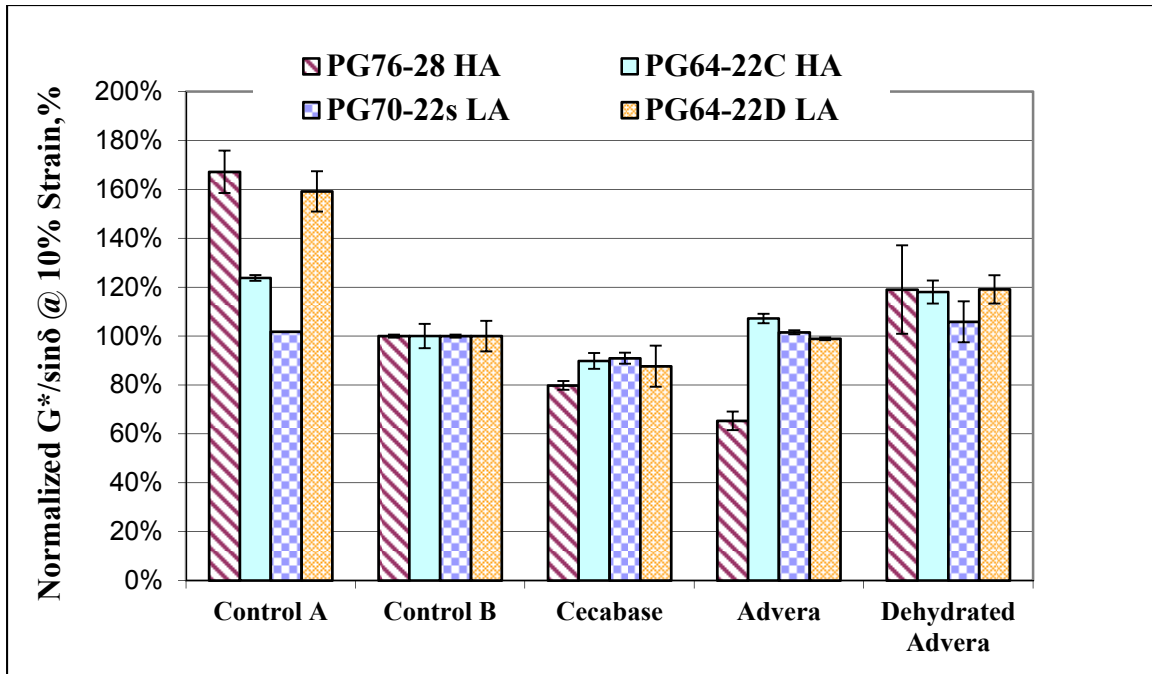
Figure 3.24: Influence of WMA additives on resistance to permanent deformation (results normalized by Control B)

Multiple Stress Creep and Recovery (MSCR) Test

Resistance to permanent deformation was further evaluated using the MSCR test at the high PG temperatures. The average non-recoverable creep compliance (J_{nr}) after 10 loading cycles was calculated from the results of testing. Non-recoverable creep compliance is the accumulated strain normalized by the applied stress, per ASTM D7405, and the J_{nr} after each loading/unloading cycle was averaged and reported. A lower value of the parameter corresponds to increased rutting resistance. Test results at the 3200 Pa stress level are presented in Figures 3.25 and 3.26, after being normalized by Control A and Control B respectively. The MSCR test was conducted at two stress levels: 100 Pa and 3200 Pa. However, tests results show that there was little difference in the *Jnr* values at the two stress levels, indicating that the binders were not sensitive to stress level.

The influence of reduced temperature short-term aging can be clearly seen by comparing results of Control A and Control B from the data presented in Figures 3.25 and 3.26. The negative impact of reduced short-term aging on non-recoverable creep compliance, *Jnr*, for PG76-28HA (a binder with high wax and acid content) is very high. Overall, qualitatively results are consistent with trends seen for the G*/sinδ measurements presented in Figures 3.23 and 3.24.
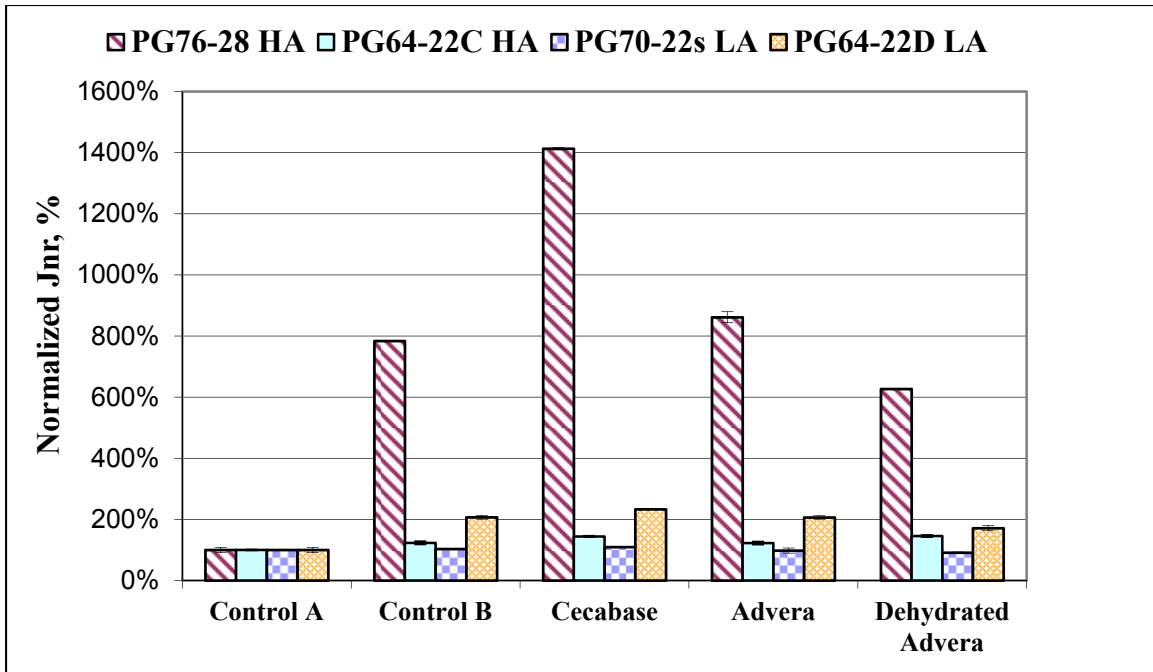
Figure 3.25: Non-recoverable compliance (Jnr) from MSCR testing of binders tested at the PG high temperature (results normalized by Control A)
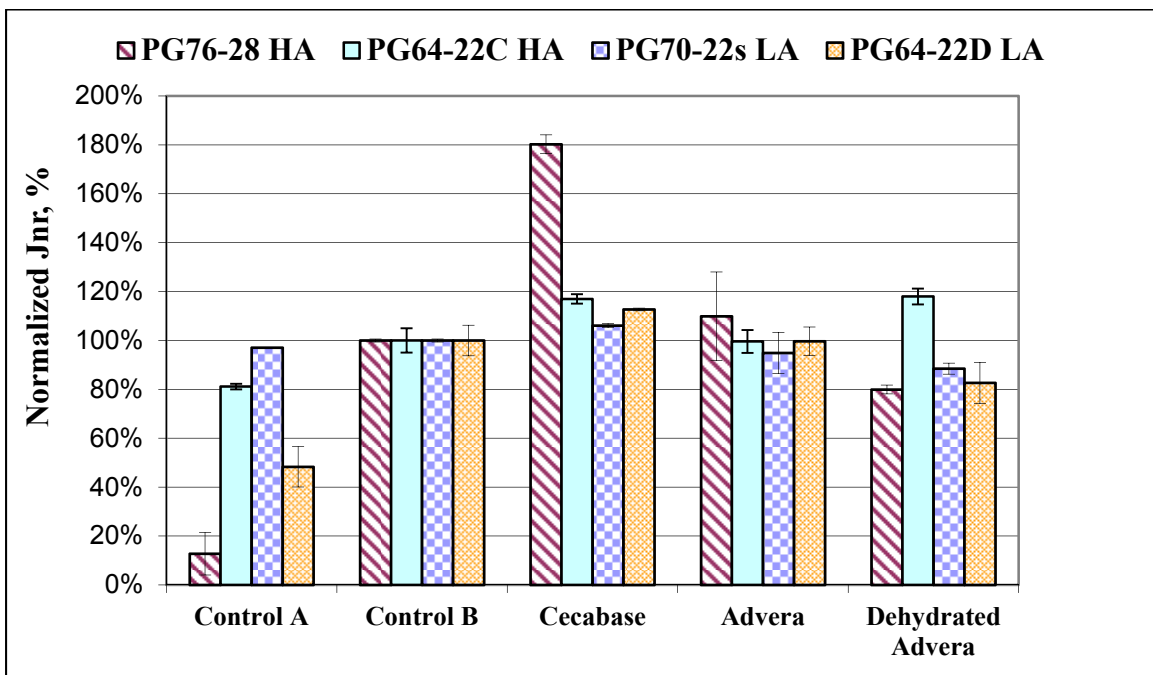

Figure 3.26: Non-recoverable compliance (Jnr) from MSCR testing of binders tested at the PG high temperature (results normalized by Control B)

3.3.4 Fracture

Stiffness of PAV-Aged Binders

      The Superpave G*sinδ parameter at 1% strain is again used in Phase II of this Task to investigate intermediate-temperature performance of the PAV-aged binders at 1% strain and 10 radians/second frequency. Figures 3.27 and 3.28 show the test results normalized by values of Control A and Control B respectively. Compared to Control B, G*sinδ values for Control A is significantly higher, except for PG70-22s LA, indicating that the reduced short-term aging temperature resulted in reduced G*sinδ values at intermediate temperature even after PAV aging. However, a comparison with Control B suggests that Cecabase further reduces the stiffness of the binder (in addition to reduced aging). Binders modified with dehydrated Advera showed higher values of G*sinδ compared to binders modified with dehydrated Advera. This demonstrates that the particle stiffening effect of dehydrated Advera may be offset by a small extent due to the presence of moisture in hydraulic zeolite (which may or may not have completely escaped during the mixing process). It is hypothesized that WMA additives (except Sasobit) and binder characteristics do not have a significant influence on the aging and stiffness relationships of the binder with age. It is speculated that the change in stiffness could be due to change in oxidation level of the binder after it is modified with the different WMA additives. The change in oxidation level could be attributed to chemical interactions between the additive and the asphalt binder. The influence of reduced temperature short-term aging and WMA additives will be further investigated using FT-IR tests to determine whether the change in G*sinδ is solely attributed to change in oxidation levels of the binders after WMA modification.

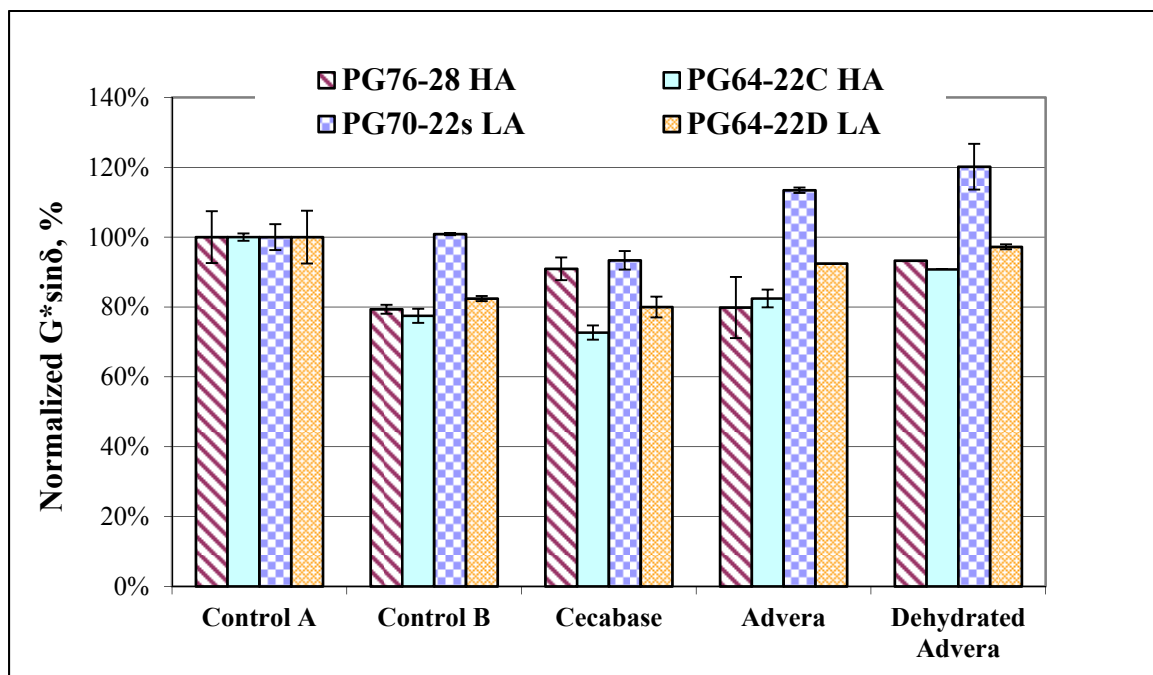*Figure 3.27: Influence of reduced short-term aging and WMA additives on G*sinδ of PAV-aged binders (results normalized by Control A)*
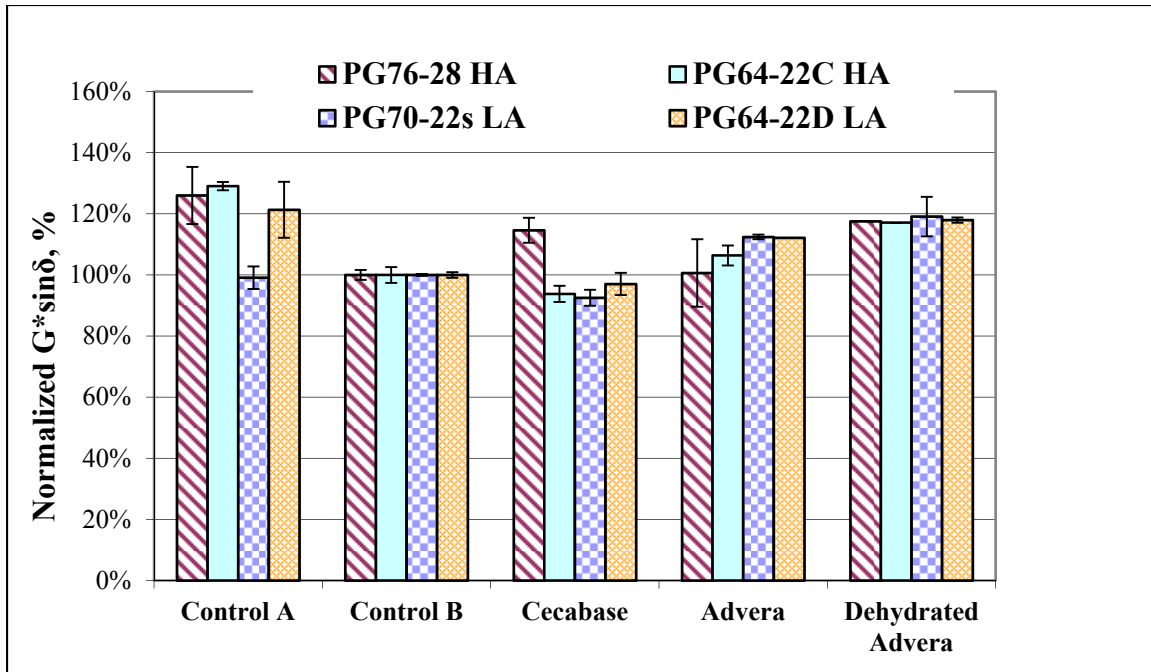
Figure 3.28: Influence of WMA additives on G*sinδ of PAV-aged binders (results normalized by Control B)

3.3.5 Low Temperature Cracking

The stiffness and m-value from the BBR testing were used to evaluate the effect of additives and reduced short-term aging temperature on the low temperature properties of the binder. The stiffness and m-value test results are presented in Figures 3.29 through 3.32 after being normalized by Control A and Control B. Test results shown in Figures 3.29 and 3.32 demonstrate that the stiffness of the PAV-aged binders at low temperatures were either similar to or slightly higher than the stiffness of Control A and Control B. Similarly, results presented in Figures 3.31 and 3.32 show that the m-value of the PAV-aged binders at low temperature is either similar to or slightly lower than the m-value of Control A and Control B. This leads to the conclusion that both reduced short-term aging temperature and WMA additives (Cecabase and Advera) do not seem to play a significant role in influencing the stiffness and m-value of the binder at low temperatures. The test results are consistent with the BBR test results obtained. Also, the slight increase in stiffness or slight decrease in m-value of binders with Advera can be attributed to the particle effect. This is based on comparison of results of Advera to dehydrated Advera.

Figure 3.29: Normalized creep stiffness of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control A)

Figure 3.30: Normalized creep stiffness of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control B)

Figure 3.31: Normalized m-value of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control A)
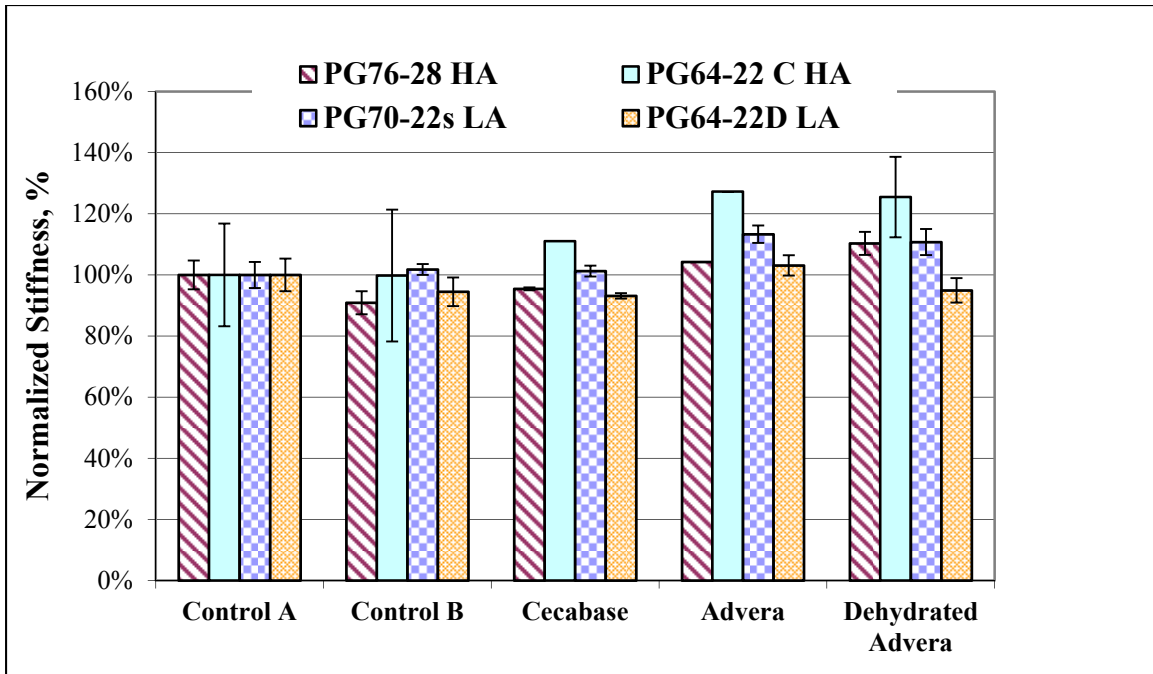

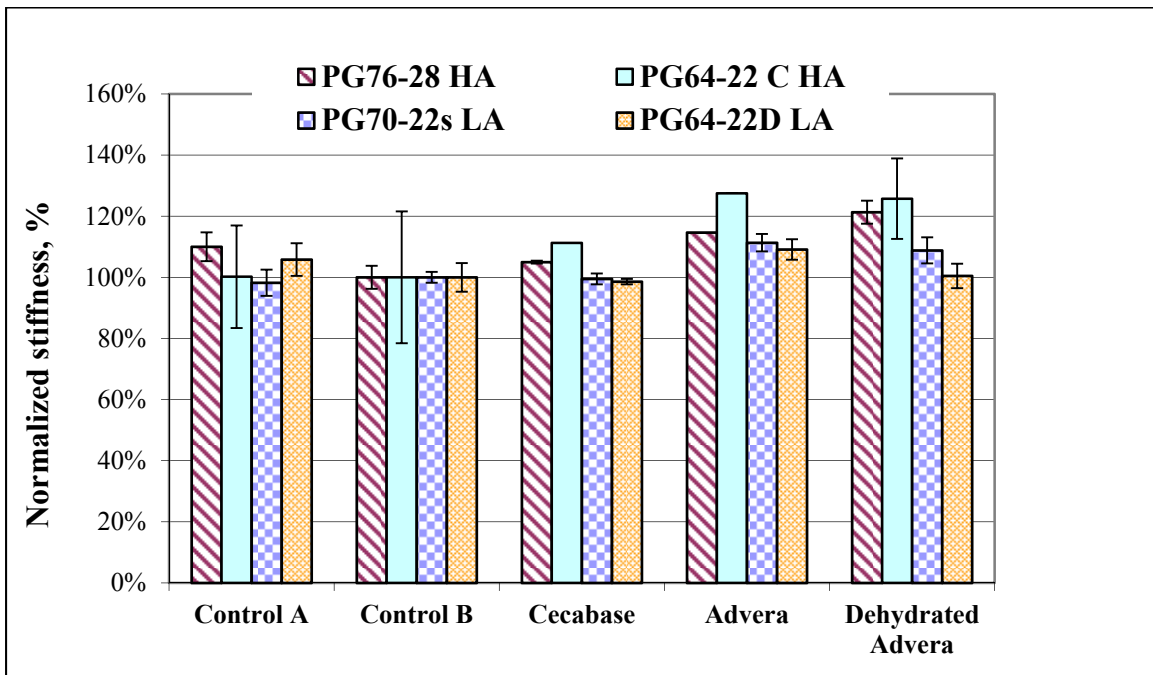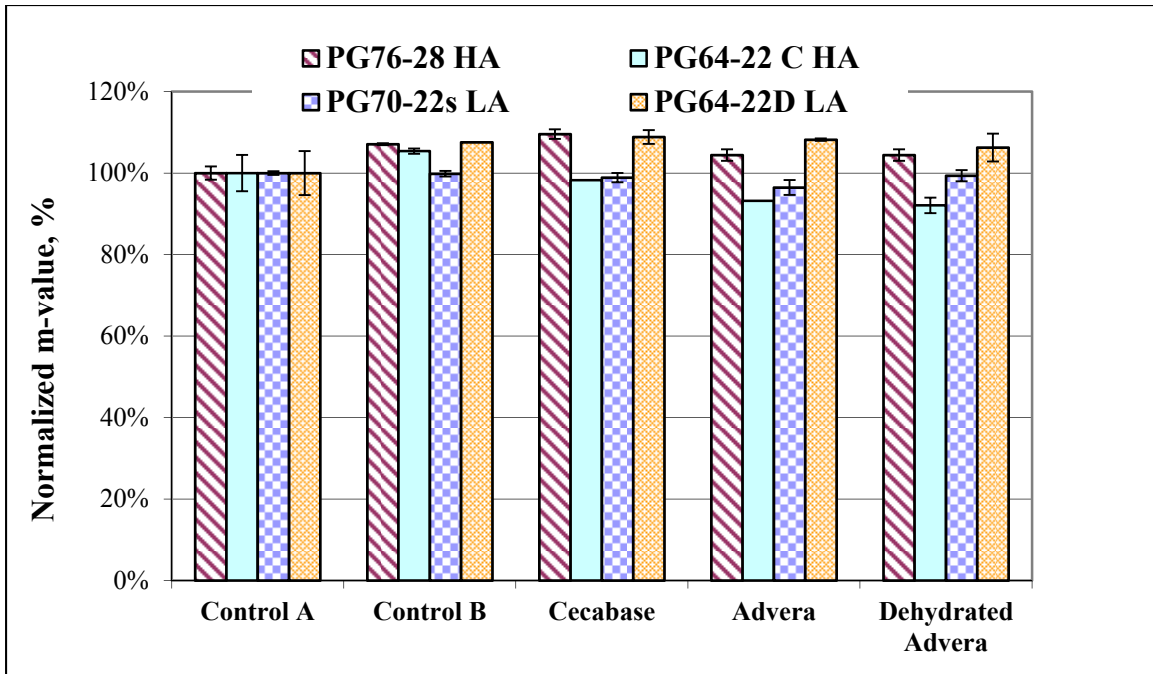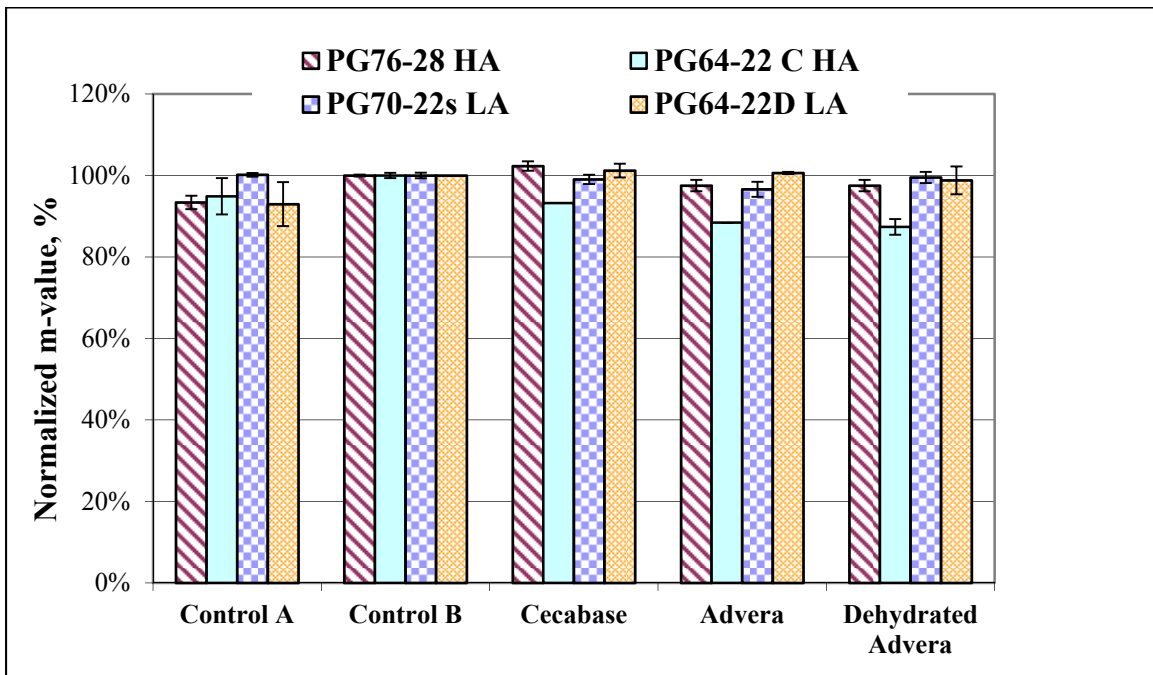*Figure 3.32: Normalized m-value of binders tested at -18°C for PG76-28 and at -12°C for the other three binders (results normalized by Control B)*

3.3.6 Summary of Results from Phase II

Analysis of the test results collected in Phase II of this Task led to the following findings. These findings are based on the viscosity at high temperatures and rheological properties at low, intermediate, and high temperatures.

- Except for one binder, reduction in viscosity of short-term aged binders was only due to reduced short-term aging. Most binders modified with Advera and Dry Advera have higher viscosities compared to Control B due to filler stiffening effect.

- As expected, *WMA binders are more susceptible to permanent deformation than their HMA counterpart due to reduced temperature short-term aging. Advera seemed to partially offset this effect due to the filler stiffening.*

- The influence of reduced short-term aging on fatigue cracking resistance of binders is found to be significant. After PAV aging, G*sinδ values for Control A was significantly higher than Control B for most binders, exhibiting the influence of reduced short-term aging temperature on the fatigue cracking resistance of asphalt binder. After PAV aging, the G*sinδ value for three of the four binders with Cecabase was only slightly lower than Control B. *Based on the G*sinδ parameter, Cecabase does not seem to play a significant role in influencing fatigue cracking performance.* However, three of the four binders modified with Advera had a G*sinδ that was only slightly higher than Control B. By comparing these results to those of dehydrated Advera, it appears that the presence of the zeolite particles may be compensating for the reduced stiffness to some extent. The influence of reduced temperature short-term aging and WMA additives will be further investigated using FT-IR tests to determine if the change in G*sinδ is only attributed to change in oxidation levels of the binders after WMA modification.

- BBR test results show that reduced RTFO aging temperature and additives do not seem to have a significant influence on either stiffness or m-value of binder after PAV aging. The result is consistent with the data presented in Phase I of this Task. The effect of RAP addition to WMA binders to mitigate rutting susceptibility will be investigated in Phase IV of this Task to determine if RAP is detrimental to fatigue cracking and thermal cracking resistance of binders.

- Acid content of binders does not seem to have any consistent interaction with these two additives as assessed, based on viscosity, resistance to permanent deformation, fatigue cracking, or thermal cracking resistance of binders.

3.4 Phase III: Investigating Oxidative Aging of Warm Mix Asphalt Binders

3.4.1 Objectives

Oxidative aging of asphalt binders plays an important role in dictating the durability of and long-term cracking resistance of asphalt pavements. Previous research indicates that chemical modifiers (such as those used to produce WMA) can influence the role of oxidative aging in asphalt binders. The main objective of Phase III of this Task was to evaluate aging characteristics and concomitant change in the stiffness of WMA modified binders.

3.4.2 Methodology and Experimental Plan

The overall methodology to achieve the aforementioned objective was as follows. The modified and control binders were artificially aged in a forced draft oven for up to a period of 132 days with a film thickness of 1 mm (0.04 inches) at 60°C (140°F). This artificial aging procedure was adopted based on previous studies by Glover et al. (2009).

Samples of the binders were obtained at periodic intervals during the aging process. These samples were tested to obtain the carbonyl area using the FT-IR spectroscopy, which is a measure of the extent of oxidative aging. The samples were also tested using the DSR. Stiffness results obtained from the DSR were correlated to carbonyl area measurements from the FT-IR results for each control and modified binder. Results obtained using the FT-IR measurements are compared with DSR frequency sweep results to investigate a correlation between carbonyl area and binder stiffness in terms of G*. Statistical tests were also conducted to examine the influence of WMA additive and binder characteristics on the aging and stiffness relationships of the binder with aging time.

This subtask involved use of one control binder and four WMA additives that were used to modify the binder. The binder selected for this study was PG64-22A LW. The PG64 low wax binder was selected to ensure that the high temperature properties of the binder could still be measured using the DSR even after extended aging. The four additives selected included Evotherm 3G, Sasobit, Cecabase RT 945, and Rediset WMX. The additives were blended with the asphalt binder using the procedure described in Chapter 2.

All asphalt binders with and without the WMA additives used in this study were aged in the RTFO to simulate short-term aging. All asphalt binders modified with the WMA additives were RTFO aged at 143°C (289°F). The control, which represents the unmodified binder, was RTFO aged at 163°C (325°F). The long-term aging of the binder was conducted by two methods. In the first method, the RTFO-aged binders were PAV-aged according to the procedure described in ASTM D6521. In the second method, 66 grams of RTFO-aged samples were poured into aluminum trays to achieve a film thickness of 1 mm (0.04 inches) and allowed to age in the environmental chamber at 60°C (140°F). Samples were obtained for testing after 0, 2, 5, 11, 22, 35, 67, and 132 days of aging time. The sample at 0 days represents an RTFO-aged sample that was not aged in the environmental chamber. Figure 3.33 illustrates a control sample being aged in the oven, and the specific experimental factors are presented in Table 3.1.
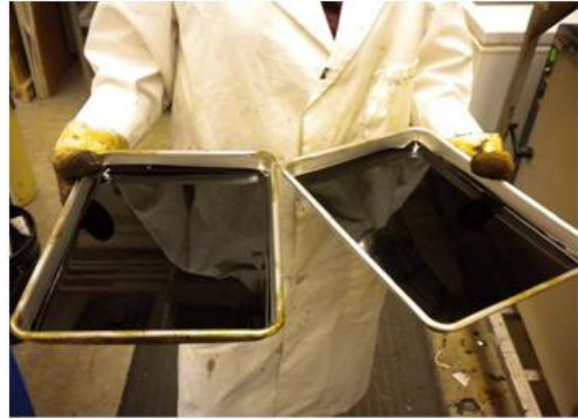
Figure 3.33: Aging of a PG64-22 Control Binder
Asphalt samples of 66 grams were poured into trays and allowed to age at the
predetermined aging time with a film thickness of 1 mm. Asphalt samples were
sampled from the tray for testing after the predetermined aging durations.

Table 3.1: Experimental Design for Binder Aging

Variables	Values	Levels
Binder source	PG64-22A LW.	1
WMA additives	Control	5
	Cecabase	
	Evotherm	
	Sasobit	
	Rediset	
RTFO aging temperature	163°C	1
	143°C	
Long-term aging	PAV	1
Long-term aging in the draft oven at 60°C, days	0	8
	2	
	5	
	11	
	22	
	35	
	67	
	132	

The extent of oxidative aging of these samples was quantified based on carbonyl area measured using FT-IR spectroscopy and the rheological properties were quantified by measuring the complex shear modulus at different frequencies (frequency sweep) using the DSR. Frequency sweep testing was conducted at 64°C (147°F) at a strain of 10% using a 25 mm (1 inch) diameter parallel plate geometry at frequencies between 0.1 and 25 Hz. Complex modulus of asphalt samples at a frequency of 0.1 Hz was selected to characterize material behavior as a function of aging time. Results from tests conducted at higher frequencies and longer aging time were not

included due to torque limitation of the DSR. Two replicates were tested at each condition. An example of the carbonyl peak and integration area is shown in Figures 3.34 and 3.35.
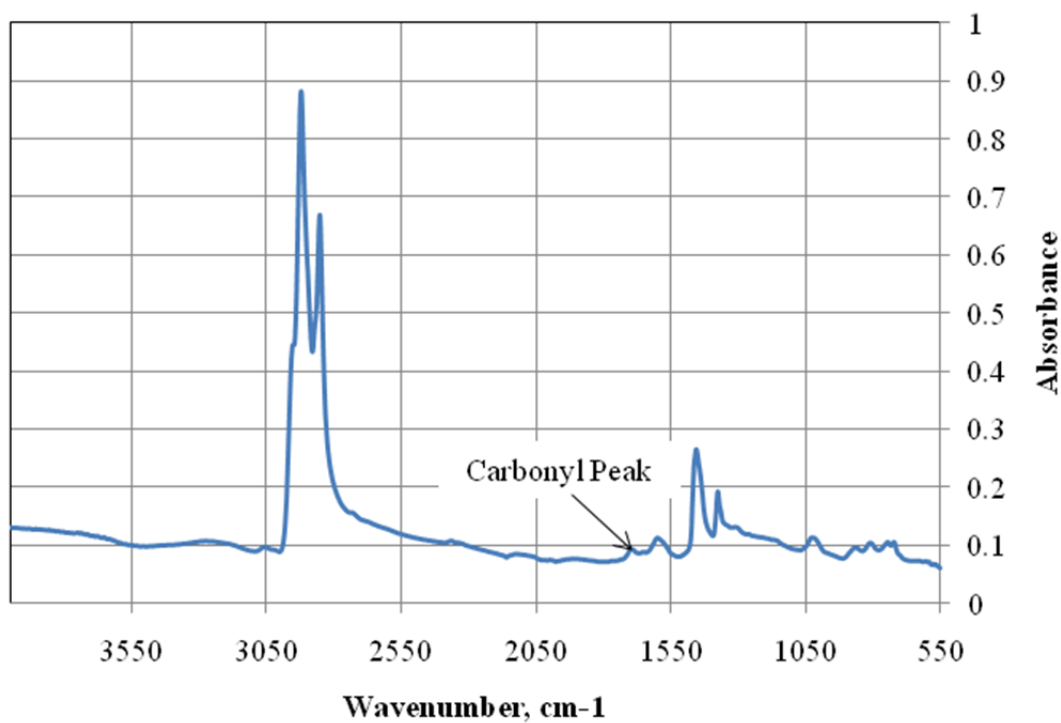

Figure 3.34: FT-IR carbonyl peak for the PG64-22 Control after 67 days aging
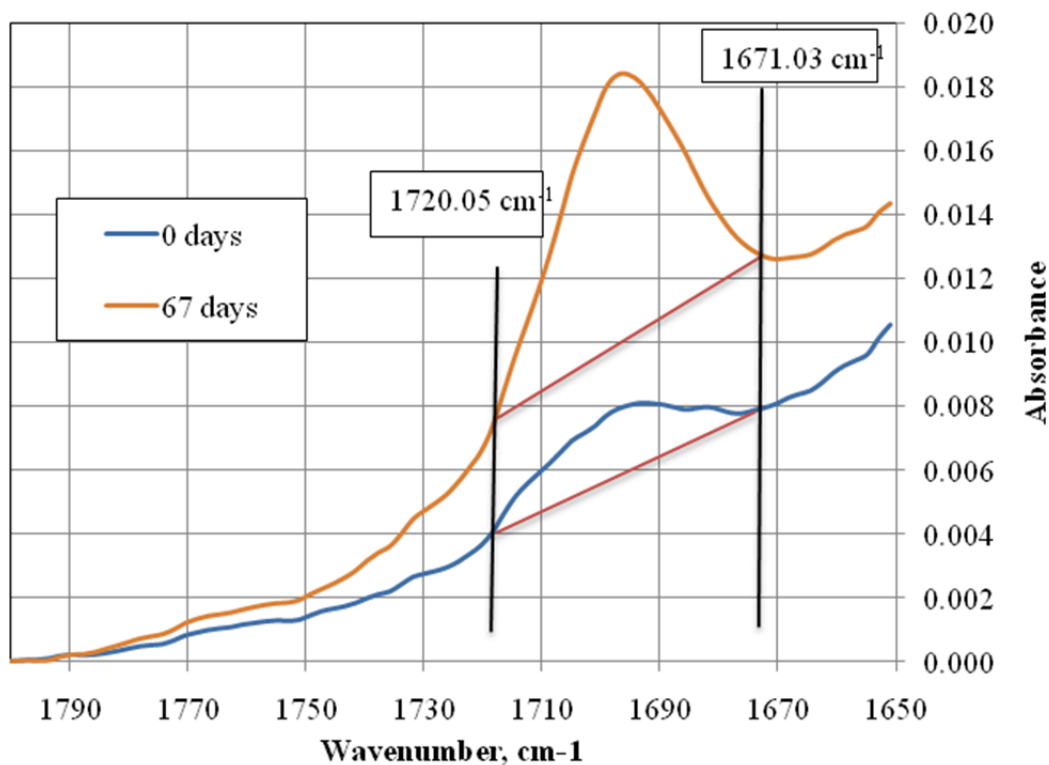
Figure 3.35: Carbonyl area integration for the PG64-22 Control after 0 and 67 days aging

3.4.3 Results and Analysis

Results are first presented separately for the frequency sweep and carbonyl area measurement results, and then collectively as further conclusions are drawn. The following analyses compares the stiffness and oxidation levels of a binder modified with various WMA additives to the Control binder subjected to various levels of aging. The stiffness is then correlated with the carbonyl area using similar samples prepared under identical conditions.

Results from the FT-IR Spectroscopy

The degree of oxidation in the binder was semi-quantitatively represented by measuring the carbonyl area using the FT-IR spectra. The carbonyl area, in arbitrary units, is the area under the absorption spectrum between wavelengths of 1671.03 and 1720.05 cm^{-1}. The OPUS software was used to run the tests and calculate the carbonyl areas. An example of the increase in carbonyl area over time due to oxidative aging is presented in Figure 3.36 for the Control binder. The carbonyl area test results presented in Figure 3.37 show that the binder modified with the additives has lower values of carbonyl area compared to the Control due to reduce short-term aging. Figure 3.37 also demonstrates significant differences in the effect of modification on binder oxidation, with the binder modified with Rediset experiencing the lowest oxidation levels throughout the aging period. A further point of reference is that results of the carbonyl area measurement for the Control binder indicate that the 35 days aging conditions produce levels of oxidative aging that is very similar to that for the PAV residue (the PAV-aged Control binder had a carbonyl area value of 0.17).
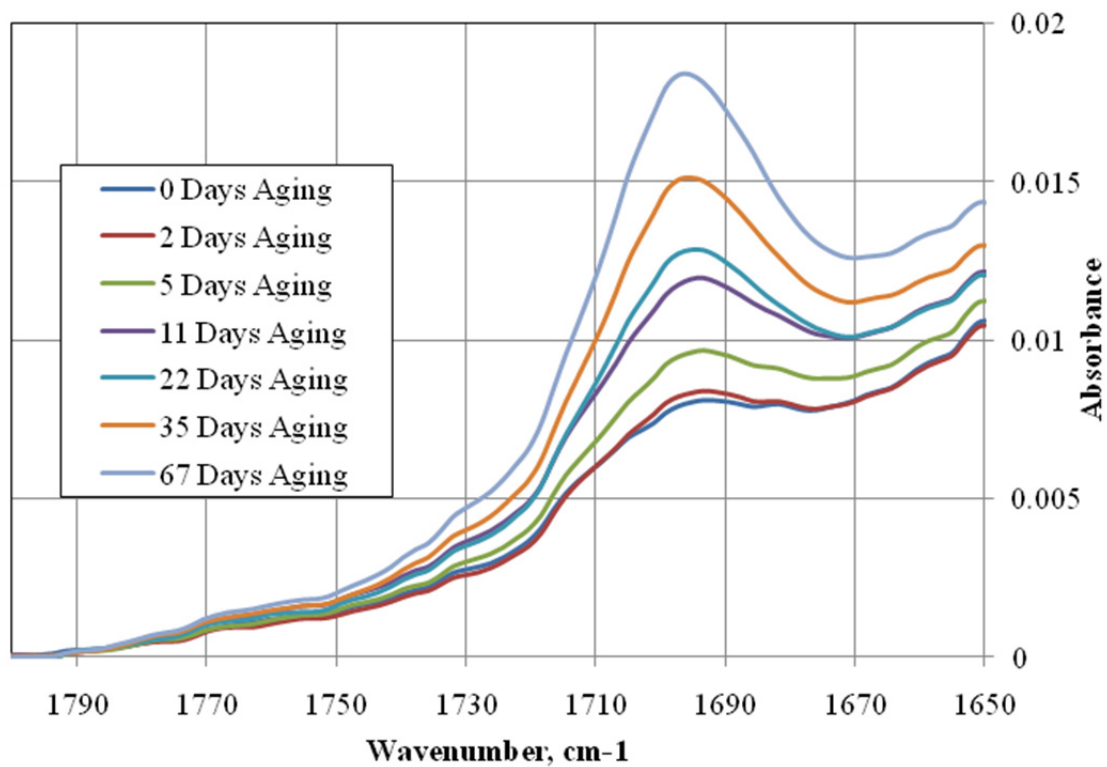
Figure 3.36: Development of carbonyl area over time for the PG64-22 Control binder
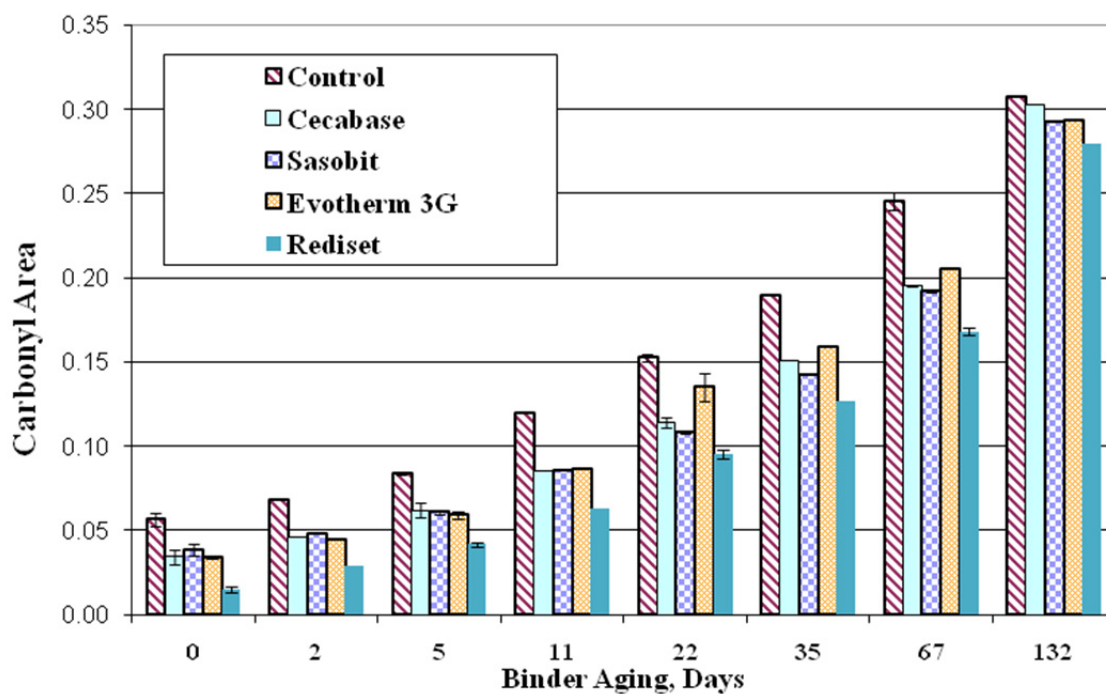

Figure 3.37: Carbonyl area at different aging periods

Results from the Frequency Sweep Tests

As previously mentioned, frequency sweep testing for the WMA-modified binders and the Control binder was conducted using the 25 mm (1 inch) diameter parallel plate geometry. Two replicates were tested for all the binders and the coefficient of variation for all the measurements was less than 10%. The complex shear modulus, G^*, of asphalt residues at a frequency of 0.1 Hz and a temperature of 64°C (147°F) at different aging times is presented in Figure 3.38. When compared to the Control, results showed similar values of G^* for the binder modified using Sasobit and reduced values of G^* for all binders modified with the other additives considered in this study throughout the aging period. This suggests that Sasobit compensated for the reduced stiffness due to reduced short-term aging, and the other additives either do not have a significant influence or further reduced the stiffness of the binder. Results also show that reduced short-term aging has a significant influence on the long-term performance of WMA binders, as shown by higher stiffness values of the Control after 132 days.
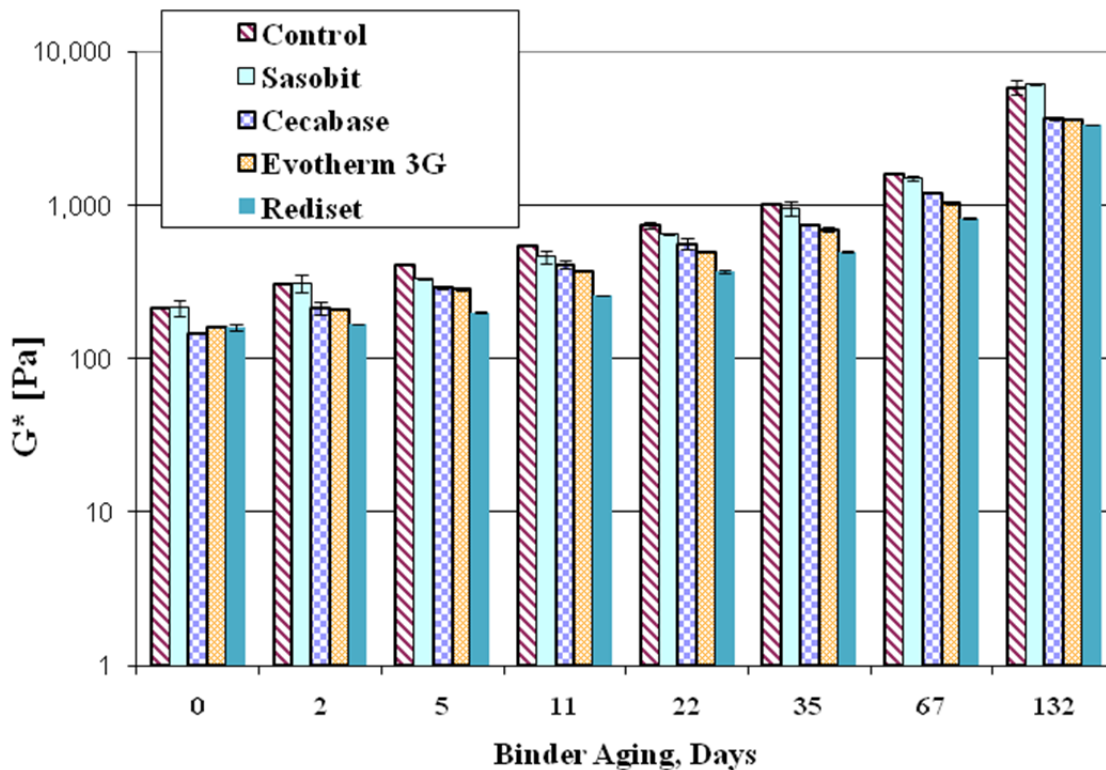
Figure 3.38: Complex modulus at different aging periods

Correlation between complex modulus and carbonyl area

To determine the significance of WMA additives and binder characteristics on the binder oxidation and stiffness relations, simple linear regression fitting and significance tests were conducted. Stiffness results were correlated to carbonyl area measurements for similar materials prepared under identical conditions in Figure 3.39. All the assumptions for the statistical analysis were verified for each assessment. Significance was determined using t-tests at a significance level, α, of 0.05. The developed regression equations followed the form of Equation 1, which related the stiffness, G^*, of the binder to the quantitative factor carbonyl area (CA) of the Control

binder and the qualitative factors: Sasobit, Cecabase, Evotherm, and Rediset. The regression analysis demonstrates a strong correlation between binder stiffness G* and carbonyl area of the WMA asphalt binder residue, which indicates that oxidative aging is a good indicator of WMA binder stiffness. CA can explain more than 98% of the change in stiffness. The binder modified with Sasobit showed a higher stiffness compared to other WMA additives at the same level of oxidation. The statistical analysis of the experiment identifies carbonyl area and Sasobit as significant factors, while Cecabase, Evotherm, and Rediset are identified as insignificant factors. Regression parameters optimized using Equation 1 are shown in Table 3.2. These results suggest that the change in stiffness of the binder modified with Cecabase, Evotherm, and Rediset was due to oxidation, and was the same as change in stiffness of the original binder. However, as shown in Figures 3.37 and 3.38, both the additives and reduced aging by themselves significantly alter the binder stiffness.

$$logG^* = \beta_0 + \beta_1 CA + \beta_2 Sasobit + \beta_3 Cecabase + \beta_4 Evotherm + \beta_5 Resdiset \qquad \text{[Eq. 1]}$$

Where:
CA   :   measured carbonyl area;
β_i :   regression coefficients, i = 0, 1,…, 5;

       Sasobit, Cecabase, Evotherm, and Rediset are categorical variables to compare the Control binder with the binder modified with Sasobit, Cecabase, Evotherm, and Rediset, respectively. They have a value of 1 or 0 depending on which additive is being compared with the Control binder.

Table 3.2: Regression Parameters Optimized Using Equation 1

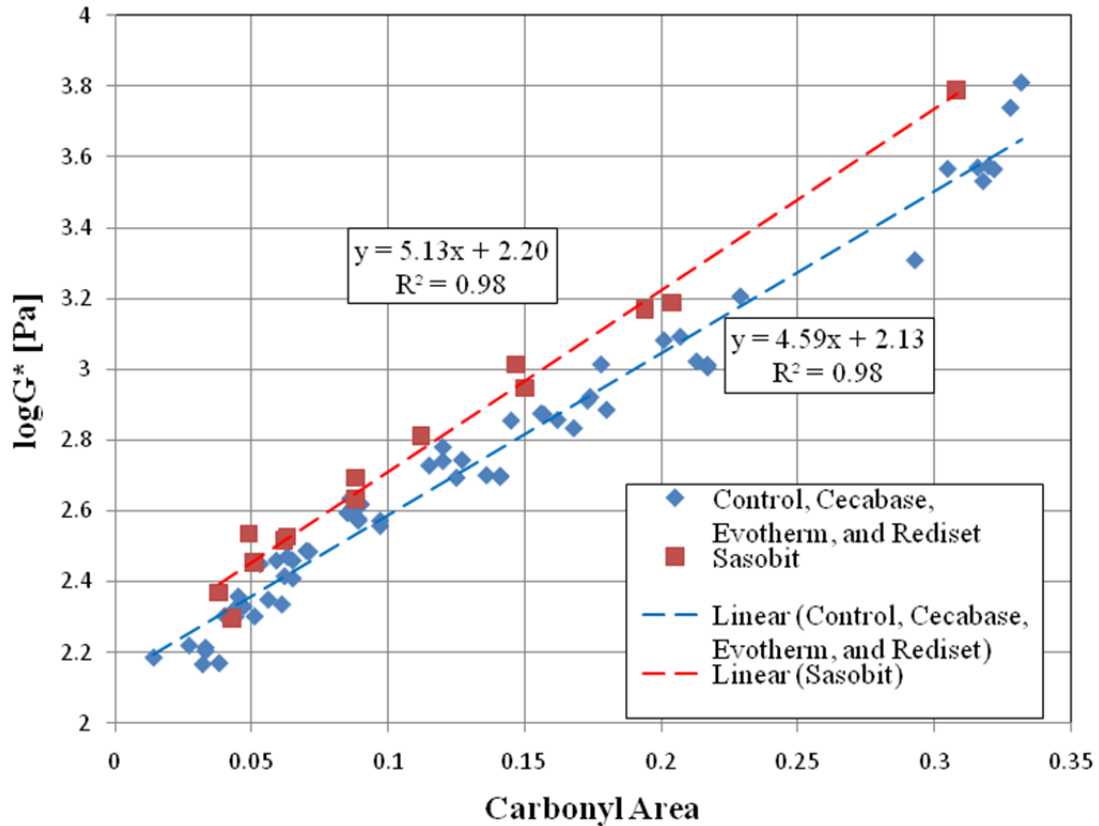| Predictor	Regression Parameter		t-stat	P-value	Significance
Constant	β_0	2.1	92.1	0.000	Significant
Carbonyl Area (CA)	β_1	-0.028	49.1	0.000	Significant
Sasobit	β_2	0.12	4.7	0.000	Significant
Cecabase	β_3	0.004	0.15	0.877	Not Significant
Evotherm	β_4	-0.034	-1.1	0.175	Not Significant
Rediset	β_5	4.6	-1.1	0.292	Not Significant

Figure 3.39: Correlation between G* and carbonyl area at different levels of aging

3.4.4 Summary of Results from Phase III

In this study, four WMA additives and one binder were used to evaluate aging characteristics and stiffness over time, to identify significant material properties and to evaluate the influence of WMA additive and binder characteristics on the aging and stiffness relationship. The unmodified asphalt binder was PG64-22A LW and the additives were selected based on their availability and most common use in the industry. The results suggest the following conclusions.

1) Carbonyl area measurements collected using the FT-IR demonstrated that the FT-IR can be effectively used to evaluate the influence of WMA additive and binder characteristics on aging rate of WMA binders over time, i.e., *change in oxidation levels explain more than 98% of the change in stiffness.*

2) *The analysis indicates that both reduced RTFO temperature and WMA additives significantly influence the long-term aging and stiffness of binders.* The WMA additives played a role in aging rate, as can be seen by the significant difference in carbonyl area measurements of the same binder modified with different WMA additives.

3) DSR test results show that reduced short-term aging has a significant influence on the long-term performance of WMA binders. Except for the binder modified with Sasobit, higher stiffness values are only associated with higher oxidation levels.

4) DSR G* test results and FT-IR carbonyl area measurements show strong correlations for similar materials under identical conditions. Cecabase, Evotherm, and Rediset did not have a significant influence on the oxidative aging versus stiffness relationship, although these additives do influence the level of oxidative aging in each binder. However, results of the binder modified with Sasobit demonstrated higher stiffness values compared to the other additives at the same levels of oxidation.

3.5 Phase IV: Characteristics of WMA Binders Containing Artificially Long-Term Aged Binders

3.5.1 Objectives and Test Plan

Results from the DSR test indicate that binders used in WMA are likely to have reduced stiffness and increased susceptibility to permanent deformation due to the reduced temperatures during short-term aging. BBR test results showed that binders with WMA additives that were subjected to reduced temperature short-term aging followed by PAV aging had similar resistance to thermal cracking. Moreover, reduced temperature short-term aging appeared to have a significant effect on fatigue cracking resistance of binders depending on the type of WMA additive and binder used. Based on the aforementioned results, a pertinent question then arises: Does the addition of reclaimed asphalt pavement (RAP) to binders, which is one of the most common strategies used to compensate for the initially reduced stiffness of the WMA, adversely influence the fatigue cracking and low temperature cracking resistance of asphalt binders?

The main objective of this study was to evaluate the effect of mixing artificially long-term aged binders (to simulate binder from the addition of RAP) with fresh asphalt binders and warm mix additives on fatigue cracking, and thermal cracking resistance of WMA binders. The experimental plan developed to accomplish these objectives included two binders and three WMA additives. The binders used were PG64-22B LW and PG76-28 HW, and the WMA additives were Sasobit, Evotherm 3G, and Rediset WMX. Samples of the PG64-22B LW and PG76-28 HW were PAV-aged twice to simulate long-term aged binder from RAP. The use of double PAV aging to produce a binder that simulates RAP was based on previous studies (Ma et al. 2010). In their study, Ma et al. (2010) compared the stiffness and m-value of mastics and mortars prepared using double PAV aged binder and RAP binder. The low temperature properties for the double PAV aged binder were reported to be similar. The artificial RAP binder was then blended with its respective binder such that the RAP binder was 25% by weight of the blend. Note that, in most cases, addition of 25% RAP by weight is allowed in asphalt mixtures without the need for any additional mixture design or binder grade adjustment procedure. Also, in this study the use of artificial RAP for each binder type avoids the influence of extraneous factors on the results (e.g., chemical compatibility of the binders, residue from solvent used for binder extraction, excessive aging of the binder from the extraction process). The blended binders were tested for creep stiffness and m-value using the BBR, and for G* and phase angle using the DSR. Lab procedure for preparation of WMA binders with 25% artificial RAP is shown in Figure 3.40.
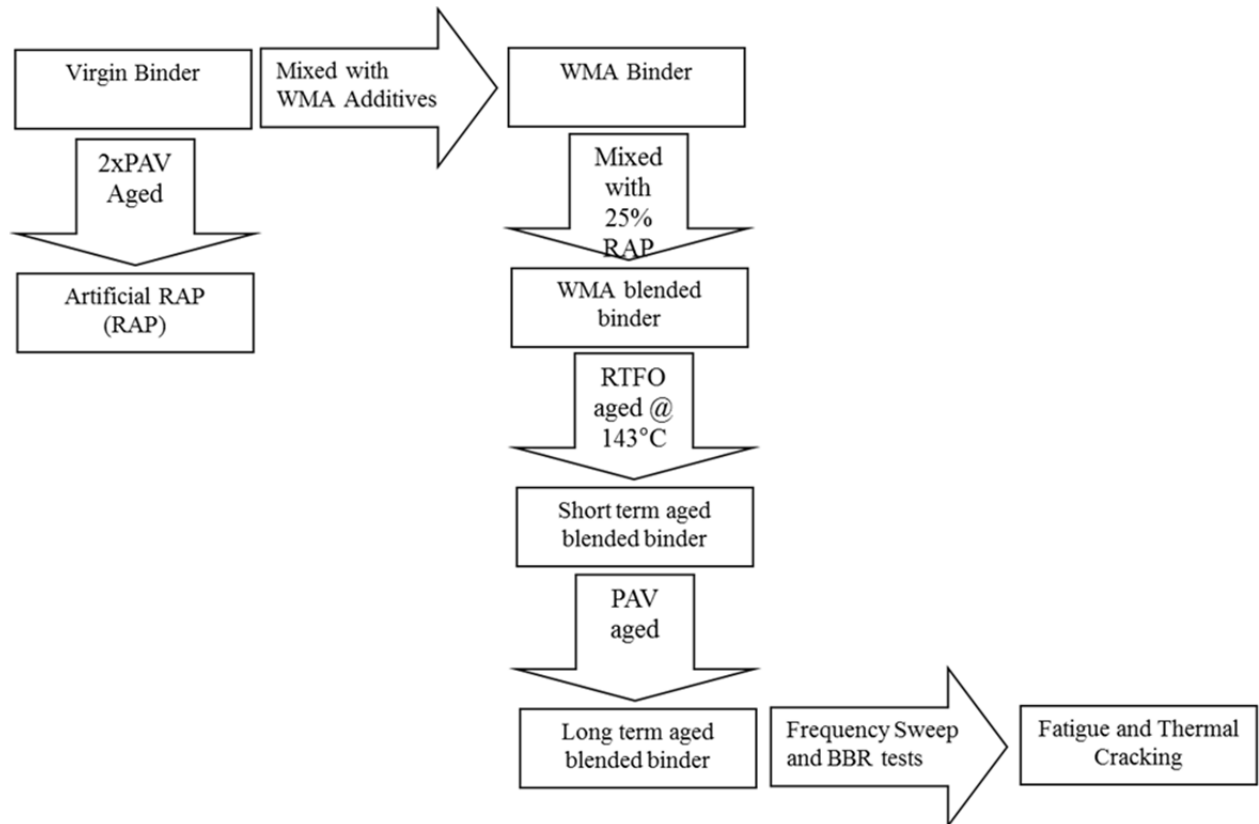
Figure 3.40: Lab procedure for preparation of WMA binders with 25% artificial RAP

3.5.2 Resistance to Fatigue Cracking

Resistance to fatigue cracking was evaluated using the parameter G*sinδ at 1% strain. Potential for reduction in fatigue cracking resistance of binders after blending with 25% artificial RAP was evaluated by comparison of blended binder properties to those of the Control binder. The Control binder represents the binder RTFO aged at 163°C (325°F) and the binder modified with WMA additive was aged at 143°C (289°F). Results are summarized in Figures 3.41 and 3.42. Most blended binders (HMA and WMA) exhibit G*sinδ values higher than a similar binder without the RAP. This behavior was more significant for the PG 64 binder and is expected due to the contribution of artificial RAP. The following two important observations can be drawn from the results presented in Figures 3.41 and 3.42:

- A WMA binder with RAP had similar G*sinδ compared to the HMA binder with RAP. Based on the current parameters for binder fatigue, this indicates that the fatigue cracking resistance of *WMA with RAP is likely to be similar to the fatigue cracking resistance of HMA with RAP. However, this interpretation is only as accurate as the use of G*sinδ to quantify the fatigue cracking resistance of the binders.*

- The PG 76 grade WMA binder with RAP had similar or lower G*sinδ compared to the HMA binder without RAP, indicating similar or better performance, whereas the PG 64 grade WMA binder with RAP had higher G*sinδ compared to the HMA binder without RAP, indicating reduced performance. In other words, *results*

suggest that addition of RAP to WMA in order to compensate the reduction in stiffness of binders due to reduced temperature short-term aging may reduce fatigue cracking resistance for softer binder grades when compared to a similar HMA.
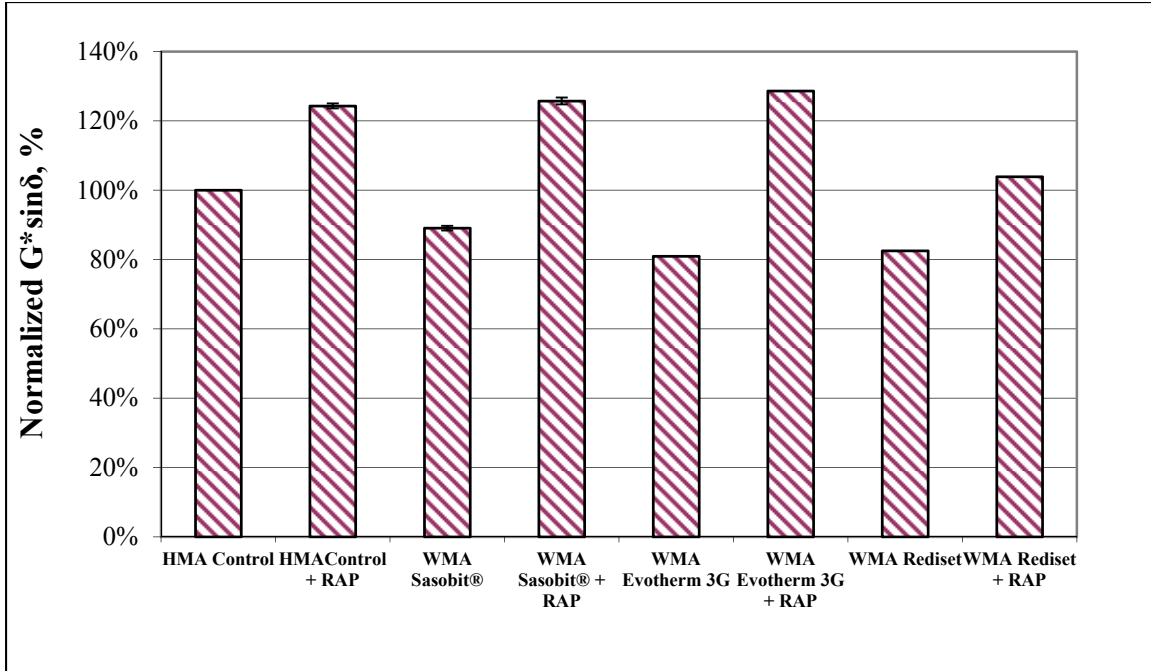

Figure 3.41: Normalized G*sinδ values of PG64-22B LW binder after PAV aging (values are normalized with respect to the Control binder without RAP)
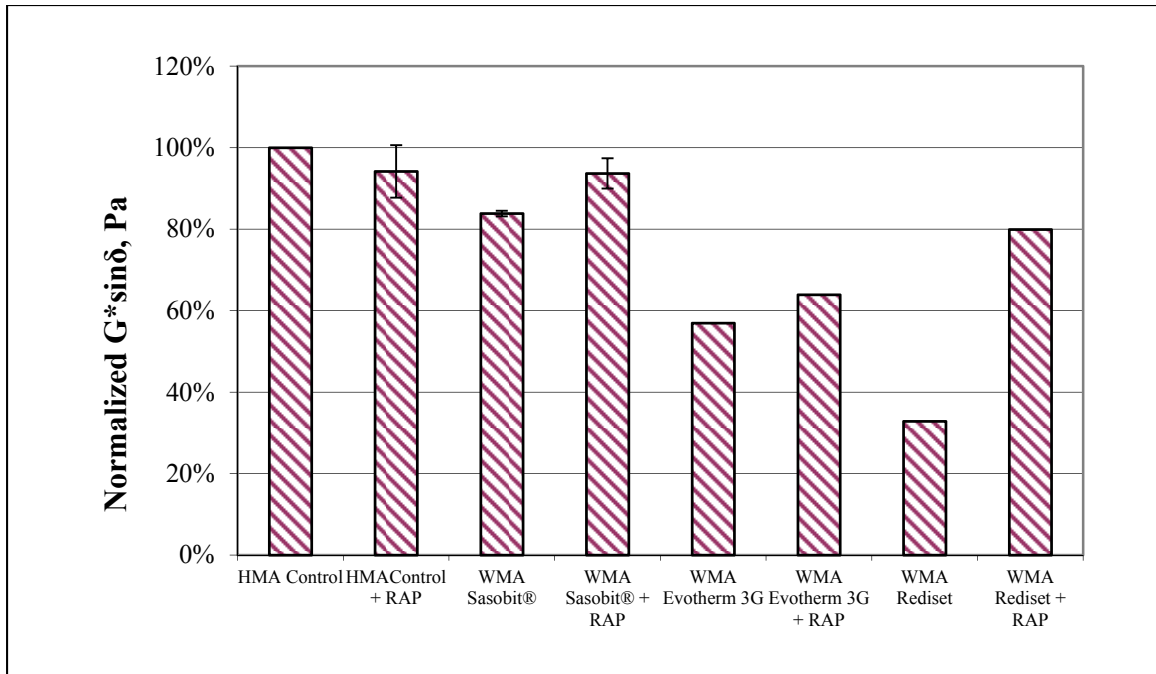
Figure 3.42: Normalized G*sinδ values of PG76-28 HW binder after PAV aging

3.5.3 Resistance to thermal cracking

The influence of using RAP with WMA on low temperature thermal cracking resistance was evaluated based on the stiffness and m-value measured using BBR at -12°C (10°F) for the PG64-22 binder and at -18°C (0°F) for the PG76-28 binder. Figures 3.43 through 3.46 present the stiffness and m-value of the asphalt binder after normalizing based on the values of the Control. Results clearly show that addition of RAP to WMA binder increased stiffness and decreased the m-value of the binder (when compared to a similar binder without RAP), suggesting blended binders have low resistance to thermal cracking. The following two important observations can be drawn from the results presented in Figures 3.43 through 3.46:

- The low temperature cracking resistance of *WMA binders with RAP was similar to* the low temperature cracking resistance of *HMA binders with RAP*.

- The *low temperature cracking resistance of WMA binders with RAP was slightly reduced when compared to the HMA binders without RAP*. However, the change in stiffness and m-value for the WMA binders with RAP was within 20% of the HMA binder without RAP.

In other words, *the risks of adding RAP to WMA binders, in terms of its susceptibility to fatigue and low temperature cracking, were similar or in some cases very slightly reduced when compared to the risks of adding RAP to regular HMA binders*.
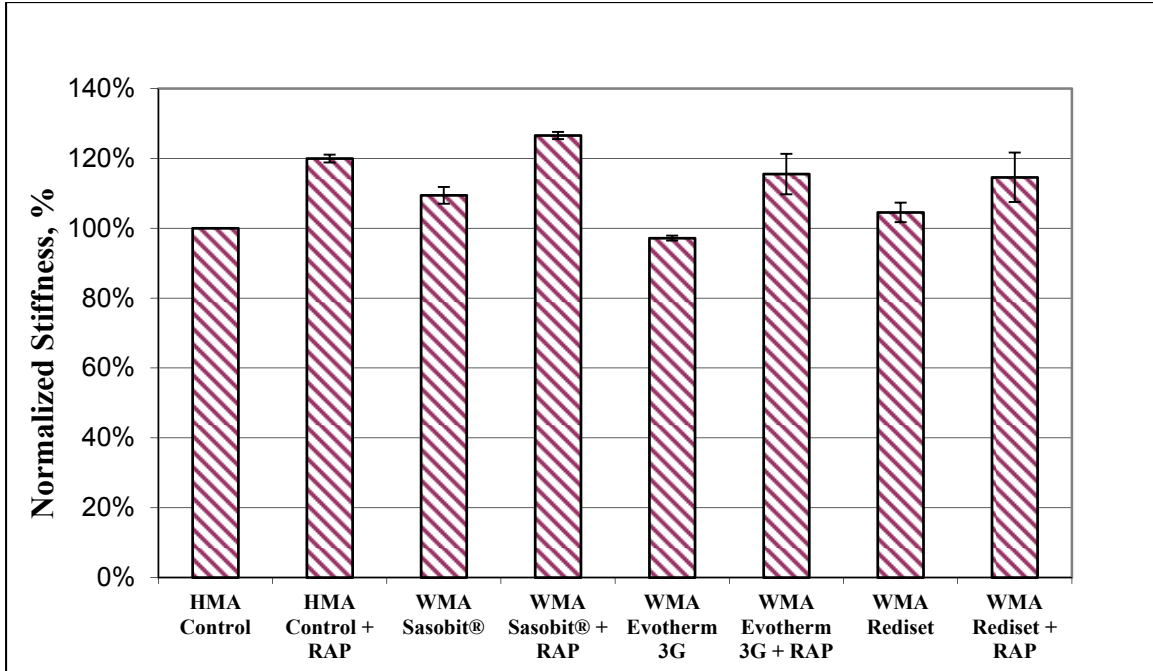
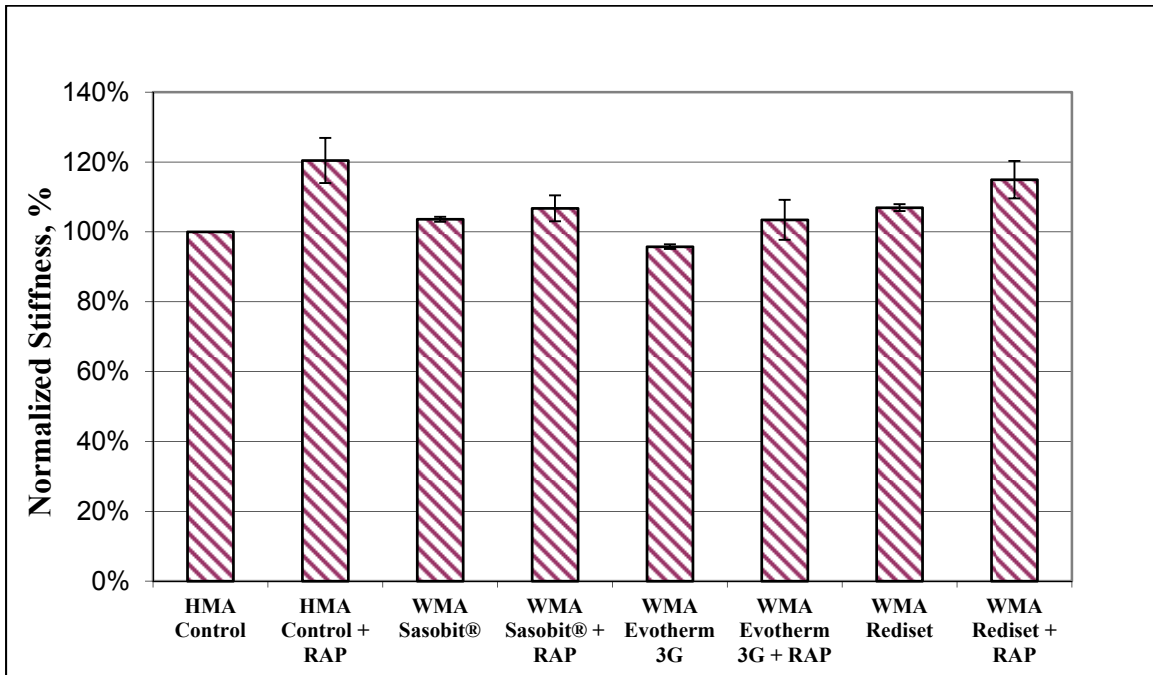*Figure 3.43: Normalized stiffness values of PG64-22B LW binder after PAV aging at -12°C*

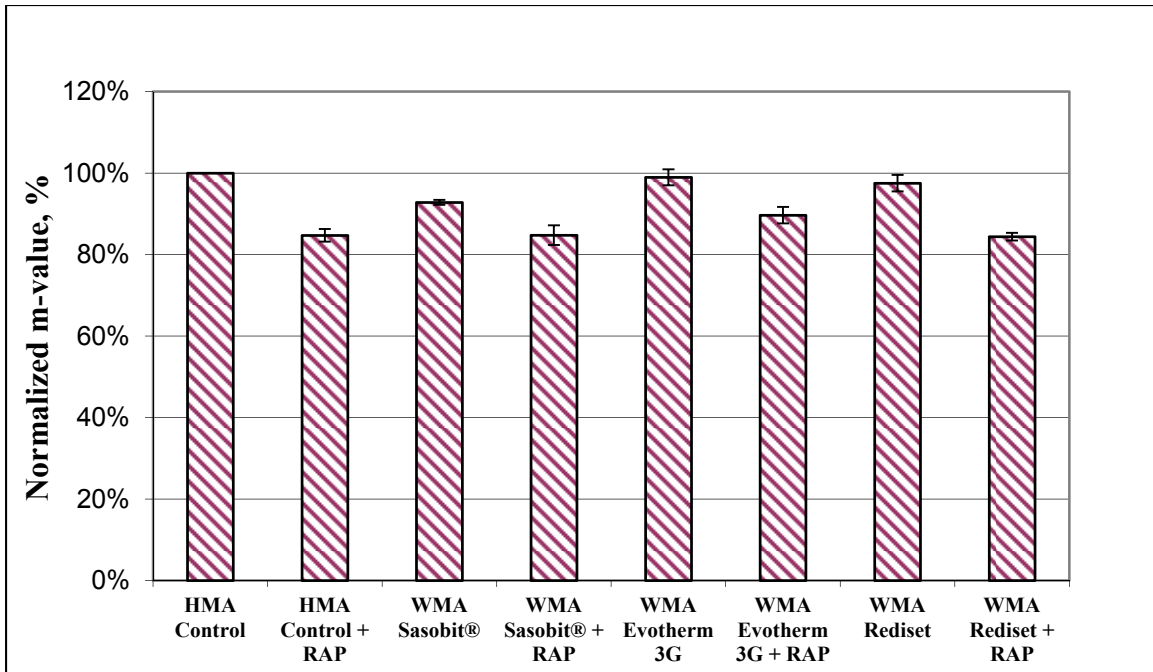*Figure 3.44: Normalized stiffness values of PG76-28 HW binder after PAV aging at -18°C*

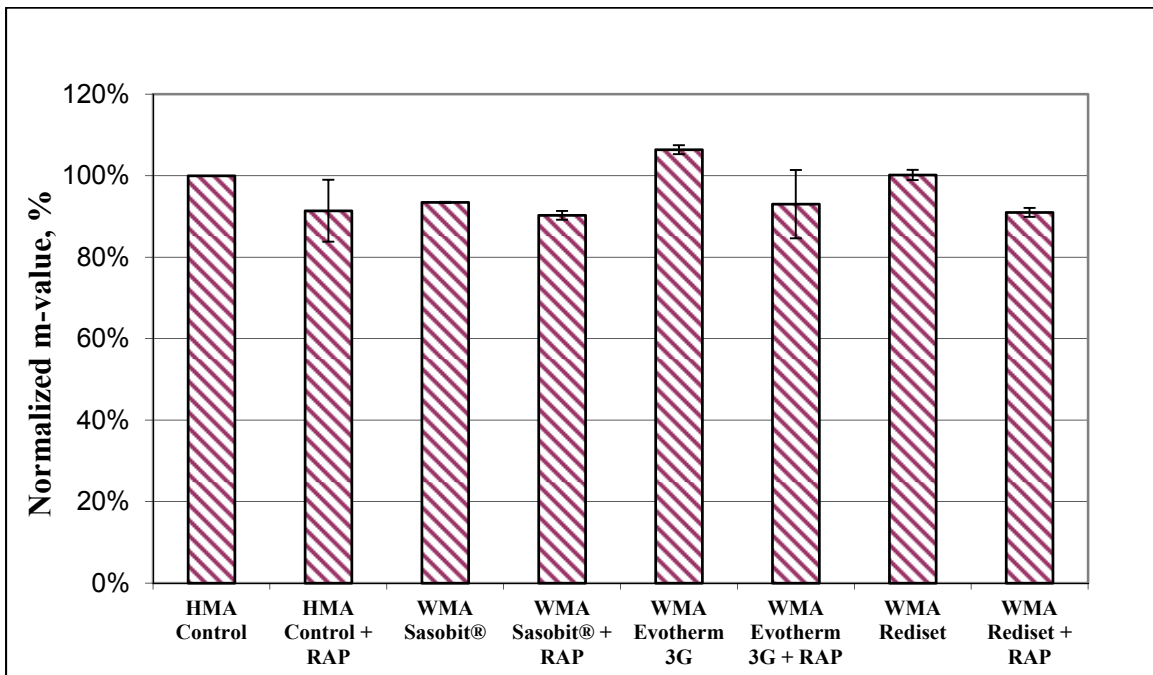*Figure 3.45: Normalized m-value of PG64-22B LW binder after PAV aging at -12°C*

Figure 3.46: Normalized m-value of PG76-28 HW binder after PAV aging at -18°C

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Chapter 4. Conclusions and Discussion

The main objectives of the tasks described in this report were to evaluate the influence of warm mix additives on the rheology and performance characteristics of asphalt binders. In order to achieve this objective the asphalt binders were first screened based on their chemical makeup. More specifically, 32 different asphalt binders used by TxDOT were screened to identify two groups of four binders each. The first group had two binders with relatively the highest natural wax content and two with relatively the lowest natural wax content. The second group had two binders with relative the highest levels of acid content and two with the relatively lowest level of acid content. The natural wax content and acid content were selected as the two attributes of interest because of the potential for the interaction of these two components with warm mix additives that were wax based or alkaline in nature. Six different warm mix additives were also selected for this study. Different combinations of the warm mix additives and the selected binders were evaluated to determine the effect of the reduced aging temperature and the warm mix additive on the viscosity of the binders at high temperatures; rheology at low, intermediate and high temperatures; rate of oxidative aging of asphalt binders modified using the warm mix additives; and the influence of RAP like binder on the rheology of warm mix binders.

Some of the findings based on the results from the tests conducted in this study are as follows:

4.1 Workability

- Viscosities of the unaged binder modified using the warm mix additives were similar to or, in most cases, less than the viscosities of the control binders. It should be noted that the viscosity measurements were made using the rotational viscometer at the same rotational speed for all binders, as prescribed in the current Superpave binder specification. Due to the non-Newtonian nature of asphalt binders, the differences in viscosities may be exaggerated at different rotational speeds. This needs to be investigated in future studies. It must also be noted that for certain additives, such as Advera, any reduction in viscosity may be confounded due to the presence of solid zeolite particles in the liquid asphalt binder.

- Warm mix additives were very beneficial in reducing the viscosity of binders that were aged for twice the duration of conventional RTFO aging. This was particularly significant for binders with high natural wax content (these are typically binders with a high temperature grade of 70 or 76). This indicates that the additives may be very useful in situations where it is expected that the asphalt mixture will be hauled for very long distances or stored in a heated silo for extended durations of time.

4.2 Early Age Stiffness and Rutting Resistance

- For three of the four binder modified using Sasobit, the $G^*/\sin\delta$ parameter was similar to the control. For 22 out of 24 combinations of binder and warm mix additives other than Sasobit that were tested, the $G^*/\sin\delta$ reduced by 80% or more when compared to the respective control binder.

- Comparison with the control binders with reduced aging revealed that Sasobit compensated for the effect of reduced aging on early age stiffness and rutting resistance. However, certain other warm mix additives further aggravated the effect of reduced aging on early age stiffness and rutting resistance. In other words, for about half the binder-warm mix additive combinations, there was stiffness loss that was due to the presence of the additive itself; this was in addition to the stiffness loss that was due to reduced aging.

- Results based on the non-recoverable compliance from the creep-recovery test (Jnr) were similar to those based on the G*/sinδ but with higher sensitivity. The <u>PG 76 binders (high wax) were very sensitive to reduced aging and additives</u> (other than Sasobit); for these binders Jnr increased by 2 to 8 times due to reduced aging and 5 to 50 times due to addition of additives and reduced aging.

4.3 Stiffness and Fatigue Cracking Resistance at Intermediate Temperatures

- Binders modified using the warm mix additive and long-term aged using the pressure aging vessel had similar or lower values of G*sinδ compared to their respective controls. It must be noted that this finding does not necessarily imply that the fatigue cracking resistance of warm mix binders is similar to or better than the control. This is in light of the recent research studies that have shown that the G*sinδ may not be an effective parameter to characterize binder fatigue.

- A subset of binders was evaluated using a thin film fracture test. For 10 out of 16 combinations of binder and warm mix additives that were tested, the fracture energy reduced by 80% or more when compared to the respective control binder.

- Although the fracture tests were conducted at one intermediate temperature and rate of loading, the discrepancy between the results from the fracture test and the G*sinδ parameter highlights the need for a better binder fracture test. These results also indicate that different asphalt binders react differently with each warm mix additives affecting their mechanical properties and fracture resistance. In other words, each binder and warm mix additive pair can be considered as a new modified binder.

4.4 Stiffness and Thermal Cracking Resistance at Low Temperatures

- Binders modified using the warm mix additive and long-term aged using the pressure aging vessel had similar stiffness (at 60 seconds) and "m" value as compared to their respective controls when tested at low temperatures. Binders modified using Sasobit showed a slight increase in susceptibility to low temperature cracking.

4.5 Rate of Aging

- Binders modified using the warm mix additive were RTFO aged at 143°C (control at 163°C) and subsequently aged in an environmental room at 60°C for a period of 132 days. Binder samples were analyzed and tested intermittently as they were being aged in the environmental room. Spectroscopic analysis of these binders

indicated that the binders modified using the warm mix additive and RTFO aged at lower temperatures had significantly lower oxidative aging compared to the control initially. However, after 132 days, the extent of oxidative aging in the modified binders was not very different from that of the control.

4.6 Influence of Recycled Asphalt

- One of the questions related to the use of WMA is whether a combination of the RAP binder and WMA can help mitigate the risk of fatigue and thermal cracking after long-term aging. To this end, warm mix additives were combined with virgin binder and laboratory produced recycled binder. This blend, along with a control, was further subjected to RTFO and PAV aging and evaluated (control was RTFO aged at 163°C and warm mix at 143°C). Results indicate that based on the stiffness at 60 seconds and the "m" parameter, the low temperature cracking resistance of a combination of warm mix binders with recycled asphalt was in most cases similar to that of the control hot mix binder with recycled asphalt.

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