Optimizing Laboratory Curing Conditions for Hot Mix Asphalt to Better Simulate Field Behavior

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16. Abstract

The engineering properties of asphalt mixtures change with time. Shortly after placement, asphalt concrete (AC) layers are more susceptible to rutting. As the pavement ages, the AC layer becomes stiffer, brittle, and thus more susceptible to cracking. Current protocols provide guidelines for the selection of materials, the determination of the material proportions (e.g., aggregates and binder content), and the evaluation of the engineering properties (e.g., cracking and rutting potentials) of any given asphalt mix design. However, these protocols do not provide any check on the impact of aging on the mixture. This study investigated existing and novel laboratory methods to determine protocols that simulate the two aging states needed to design an asphalt mixture to resist rutting and cracking and provide information on how curing affects the physical and engineering performance of binders and mixtures. This study leveraged existing research studies and available performance data along with a systematic test matrix to optimize the curing conditions. The wide range of tests conducted in this study indicated that a minimum time to achieve levels of long-term aging using an optimized laboratory protocol would be close to 24 hours. The steady state of aging for ranking different mixes may be adequately achieved after a period of 24 hours (including 2 hours of short-term aging) under Protocol 1 i.e., loose mixture aging in a conventional laboratory oven. The efficiency of other aging protocols using pressure and oxidated gases have been demonstrated. These methods can be used to more accurately characterize the performance of mixes under long-term aging within a time frame of 24 to 48 hours.

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Abstract

The engineering properties of asphalt mixtures change with time. Shortly after placement, asphalt concrete (AC) layers are more susceptible to rutting. As the pavement ages, the AC layer becomes stiffer, brittle, and thus more susceptible to cracking. Current protocols provide guidelines for the selection of materials, the determination of the material proportions (e.g., aggregates and binder content), and the evaluation of the engineering properties (e.g., cracking and rutting potentials) of any given asphalt mix design. However, these protocols do not provide any check on the impact of aging on the mixture. This study investigated existing and novel laboratory methods to determine protocols that simulate the two aging states needed to design an asphalt mixture to resist rutting and cracking and provide information on the influence of laboratory aging on the physical and engineering performance of binders and mixtures. This study leveraged existing research studies and available performance data along with the development of two new methods to accelerate oxidative aging of asphalt mixtures without compromising the chemical integrity of the reaction pathways. The steady state of aging for ranking different mixes may be adequately achieved after a period of 24 hours (including 2 hours of short-term aging) under Protocol 1 i.e., loose mixture aging in a conventional laboratory oven. The efficiency of other aging protocols using pressure and oxidated gases have been demonstrated. These methods can be used to more accurately characterize the performance of mixes under long-term aging within a time frame of 24 to 48 hours.

Implementation Statement

In this report, a number of recommendations have been made to develop aging protocols for mixtures to simulate short- and long-term aging in the field in an accelerated fashion. The recommendations are based on the test results of several materials from seven TxDOT sections.

At this time, the recommendations should be implemented on a number of new and ongoing projects to confirm their applicability and to adjust the limits and/or criteria recommended. As part of the implementation, a guide should be developed to disseminate to the TxDOT staff.

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Chapter 1. Introduction

Statement of Problem

The engineering properties of asphalt mixtures change with time. Shortly after placement, asphalt concrete (AC) layers become susceptible to rutting. As the pavement ages, the AC layer becomes stiffer, brittle, and thus more vulnerable to cracking. The Texas Department of Transportation (TxDOT) provides guidelines for the selection of materials, the determination of the material proportions (e.g., aggregates and binder content), and for the evaluation of the engineering properties (e.g., cracking and rutting potentials) of any given asphalt mix design. However, TxDOT specifications do not provide any check or guidance on the impact of aging on the mixture. This project seeks to investigate and propose protocols to simulate realistically the short- and long-term aging states required to evaluate the asphalt mixture resistance to rutting and cracking, respectively. Such conditioning protocols must be valid for all types of asphalt mixes including, but not limited to mixtures that incorporate reclaimed asphalt pavement (RAP), reclaimed asphalt shingles (RAS), recycling agents, and other additives.

This study focuses on developing and implementing optimized, representative, and practical laboratory aging protocols that improve the mix design process leading to the production and placement of long-lasting, stable, and durable asphalt mixtures. The findings from this research project are also expected to provide insight into the formation of distresses (rutting and cracking) associated with individual asphalt mixtures.

Objectives and Scope

The main goal of this project is to deliver implementable and optimized laboratory aging protocols that can be used to assess the engineering properties of asphalt binders and mixtures after short- and long-term aging. To achieve the goal of this project, the following activities had to be carried out:

1. Carry out a comprehensive literature search and gap analysis to identify weaknesses and strengths of current practices to simulate field aging in the laboratory.
2. Assess the effectiveness and practicality of current short- and long-term aging protocols for use with laboratory performance tests (e.g., Hamburg wheel-tracking device (HWTD), overlay tester (OT), and indirect tension (IDT) tests.
3. Formulate a robust and consistent analysis method, corresponding aging indices, and acceptance limits to determine the variation in the engineering properties of different asphalt mixtures and associated asphalt binders after simulated field aging.
4. Use either existing or novel laboratory methods to develop optimized aging protocols for laboratory- and plant-produced mixtures practically. These can be used to represent the early age rutting potential and cracking resistance after long-term aging of different mixtures and corresponding binders more accurately.

5. Generate laboratory and field performance data to verify the simulated field-aging conditions of laboratory-produced, plant-produced, and field-compacted asphalt mixtures and concomitant asphalt binders.
6. Revise current test methods and specifications to incorporate the proposed optimized laboratory aging protocols for asphalt materials (including asphalt binders and mixtures) into the balanced mix design process and specifications.

Organization of Report

To address the goal and objectives, the report is divided into nine chapters including the introduction. These are further categorized into three main phases:

- Phase I (*Documentation*) consists of documenting the current state of the practice aging protocols used by other agencies and recently developed methods related to this project. Several approaches were formulated, and the most promising approaches were selected. The primary outcome of this phase was identifying the candidate-aging protocols with an accompanying experiment design plan to address the technical objectives described in the problem statement. This phase consists of the following tasks:

  Chapter 2 - Comprehensive Documentation of Current Practices and Specifications
  Chapter 3 - Preliminary Assessment of Existing Laboratory Aging Protocols on Asphalt Performance Using Available Data
  Chapter 4 - Development of Experiment Design

- Phase II (*Evaluation*) involved conducting an initial experimental evaluation of the candidate laboratory aging protocols to assess their effectiveness in simulating the field-aging behavior of asphalt concrete and binders during the production, placement, and service life of a pavement. A laboratory-validated version of the aging protocol was selected to further evaluate the influence of mix design variables on the aging and performance of asphalt mixtures. This phase included the following tasks:

  Chapter 5 - Initial Evaluation of Aging Potential of Asphalt Concrete with Laboratory Aging Protocols
  Chapter 6 - Selection of Preliminary Optimized Laboratory Aging Protocols
  Chapter 7 - Extended Evaluation of Aging Potential of Asphalt Concrete with Refined Laboratory Aging Protocols

- Phase III (*Verification and Validation*) focused on limited validation of the recommended laboratory aging protocols based on additional laboratory and field performance data. The findings from this phase were used to make any adjustments or refinements to the recommendations from Phase II. This phase included:

  Chapter 7 - Verification of Preliminary Laboratory Aging Protocols
  Chapter 8 - Optimization of Laboratory Aging Protocols and Specifications

Chapter 2. Comprehensive Documentation of Current Practices and Specifications

Aging is considered a major environmental effect that drives changes in the engineering properties of asphalt mixtures during the design, production, placement, and service life of a flexible pavement. Aging of asphalt mixtures causes the material to stiffen over time and to become more brittle which contributes to durability problems such as cracking of the asphalt concrete (AC) layer. One of the main goals of evaluating engineering properties of an asphalt mixture is to screen for crack- and rut-susceptible mixtures. In order to do so accurately, one needs representative laboratory aging protocols that can produce specimens of asphalt mixtures simulating the two critical aging states, freshly placed and long-term aged asphalt mixture.

Current mix design processes usually require characterizing the engineering properties of an asphalt mixture using specimens conditioned with short-term oven aging (STOA) protocols. These specimens are typically oven cured for 2 hrs at a specified compaction temperature related to the performance grade (PG) of the binder. At the early aging state, asphalt mixtures are more susceptible to rutting. As the pavement ages, the asphalt concrete layer becomes stiffer and more prone to cracking. Thus, the current aging protocols to simulate field-aging behavior of asphalt mixtures do not accurately reflect the expected long-term cracking performance of asphalt mixtures.

Long-term oven aging (LTOA) protocols have been implemented mainly to predict the effects of aging on engineering properties of asphalt mixtures over time. In accordance with AASHTO R 30, the standard practice to simulate the field aging of an asphalt mixture consists of curing compacted laboratory specimens for 5 days at 185ºF (85ºC). Even though several studies have demonstrated LTOA protocols could produce relatively consistent results with the same degree of aging that would take in field practice, it is impractical to vary laboratory-curing conditions for asphalt mixtures several times before molding specimens for performance testing. The implementation of optimized laboratory curing protocols is paramount to prevent early age rutting and particularly long-term cracking susceptibility during the mix design process.

In the last several years, TxDOT has funded several studies to investigate the engineering properties of a wide range of asphalt mixtures, effectiveness of performance test methods, and implementation of sustainable measures such as incorporating recycled materials, warm-mix asphalt technologies, and recycling agents. these studies also focused on development of a performance measure to evaluate asphalt mixtures, which is necessary to develop a framework to implement the concept of "balanced mix design" or more accurately performance-based mix design. Some of these projects include:

- Research Project 0-5123, *"Implementation of Texas Asphalt Concrete Overlay Design"*
- Research Project 0-5597, *"Evaluation of Warm Mix Asphalt New Technologies"*
- Research Project 0-6009, *"Evaluation of Binder Aging and its Influence in Aging of Hot Mix Asphalt Concrete"*
- Research Project 0-6092, *"Performance Evaluation and Mix Design of High RAP Mixtures"*
- Research Project 0-6591, *"Developing a Fundamental Understanding of the Chemistry of Warm Mix Additives"*

- Research Project 0-6613, *"Evaluate Binder and Mixture Aging for Warm Mix Asphalts"*
- Research Project 0-6614, *"Use of Recycled Asphalt Shingles in HMA"*
- Research Project 0-6815, *"Improved Overlay Tester for Fatigue Cracking Resistance of Asphalt Mixtures"*
- Research Project 0-6682, *"Validation of the Maximum Allowable Amounts of Recycled Binder, RAP and RAS Using Accelerated Pavement Testing"*
- Research Project 0-6738, *"Performance Studies and Future Directions for Mixes Containing RAP and RAS"*
- Research Project 0-6744, *"New HMA Shear Resistance and Rutting Test for Texas Mixes"*
- Research Project 0-6925, *"Improving the Performance Graded Asphalt Binder Specification"*
- Research Project 0-6947, *"Revised Allowable Maximum Recycled Binder Ratio (RBR) Specification"*

Similarly, other State Highway Agencies (SHA) and national agencies have performed studies to develop and evaluate laboratory-aging methods to more accurately reflect aging that occurs in the field. The following is a list of some of these research projects:

- NCHRP Research Project 09-36, *"Investigation of Short-Term Laboratory Aging of Neat and Modified Asphalt Binders,"* documented the influence of short-term aging (STA) on the permanent deformation potential of asphalt mixtures.
- NCHRP Research Project 09-52, *"Short-Term Laboratory Conditioning of Asphalt Mixtures,"* evaluated the influence of long-term aging protocols on loose mixtures and compacted specimens.
- NCHRP Research Project 09-54, *"Long-Term Aging of Asphalt Mixtures for Performance Testing and Prediction,"* was funded to develop practical laboratory aging
- NCHRP Research Project 09-61 (Active), *"Short and Long-Term Binder Aging Methods to Accurately Reflect Aging in Asphalt Mixtures,"* investigates potential laboratory aging protocols to simulate the short- and long-term aging behavior of neat and modified binders.
- NCHRP Research Report 919, *"Field Verification of Proposed Changes to the AASHTO R 30 Procedures for Laboratory Conditioning of Asphalt Mixtures"* revised current protocols on LTOA and proposed changes to current standards.
- Virginia DOT Research Report 18-R26, *"Investigation of Binder Aging and Mixture Performance in Service: Reclaimed Asphalt Pavement Mixtures"* documented the effect of RAP in-service performance and the long-term binder aging properties.
- Illinois DOT Research Project R27-175, *"Development of Long-Term Aging Protocol for Implementation of Illinois Flexible Index Test (I-FIT)"* investigated the use of the I-FIT test with semi-circular specimens to analyze LTOA effect.
- CALTRANS CA13-2385A, *"Warm-Mix Asphalt Study - Evaluation of Hot and Warm Mix Asphalt with Respect to Binder Aging."* compared the result from WMA to HMA with respect to short- and long-term performance of the pavement.
- Minnesota DOT 2014-45, *"Optimal Timing of Preventive Maintenance for Addressing Environmental Aging in Hot-Mix Asphalt Pavements"* documented environmental aging effects on asphalt binders and mixtures properties on pavements.
- FHWA-IL-UI-277, *"Effects of Short-Term Oven Aging on Volumetric and Selection of N-Design"* analyzed the effect of conditioning time (STOA) on volumetric properties.

A common observation among many of these reports has to do with dissimilarities between the results of laboratory-mixed laboratory-compacted (LMLC) specimens and plant-mixed lab-compacted (PMLC) specimens. The difference is associated with the aging state of PMLC because specimens undergo a period of short-term aging in the plant. To prepare PMLC specimens, the mixture has to go through another aging process in the laboratory. On the other hand, LMLC specimens go through the mixing and compaction process in the laboratory without initial aging. A rigorous comparative analysis of the results between comparable PMLC and LMLC specimens is necessary to bridge the gap. This study will focus on the parameters that influence the oxidation of the asphalt binder and aging of mixtures.

Review of Previous Research

Laboratory Aging Protocols and Engineering Behavior of Asphalt Binders

Although extraneous factors such as temperature, pavement thickness, air void content and distribution dictate the spatial distribution and extent of aging in an asphalt mixture, it is ultimately the aging of the asphalt binder that dictates the change in the performance characteristics of the asphalt mixture. Consequently, any investigation on aging requires a focus on the aging characteristics of the asphalt binder.

The national standard specification to determine the performance grade (PG) of an asphalt binder, AASHTO M320, includes short- and long-term laboratory conditioning procedures to simulate the aging that occurs in asphalt binders during production and over the service life of the pavement. The conditioning procedures used in AASHTO M320 are (1) the Rolling Thin Film Oven Test (RTFO, AASHTO T240) for short-term aging and (2) the Pressure Aging Vessel (PAV, AASHTO R28) for long-term aging. These procedures were developed during the Strategic Highway Research Program (SHRP) based on previous experience and limited validation studies using mostly unmodified asphalt binders recovered from in-service pavements.

Bell (*1989*) documented several laboratory short-term protocols for asphalt binders and mixes, including the Thin-Film Oven Test (TFOT), the Rolling Thin-Film Oven Test (RTFO), the Stirred Air-Flow Test (SAFT), and the German Rolling-Flask Test (GRF). The RTFO exposes an asphalt binder film to continuous heat and airflow to induce oxidative aging and it is the most commonly used method to simulate short-term aging. Airey and Brown (*1998*) indicated that the RTFO conditions were not identical to the aging conditions induced during actual mixing but had shown good correlation to the aging conditions observed in the conventional batch mixes. In NCHRP Project 9-10, Bahia et al. (*2001*) concluded that RTFO did not adequately simulate the aging of modified asphalt binders, even though it might be satisfactory for neat binders. Under NCHRP Project 9-36, Anderson and Bonaquist (*2012*) evaluated the existing asphalt binder technologies to identify potential modifications for improving the short-term aging protocols. They recommended SAFT and the modified German rotating flask (MGRF) (*Glover et al., 2001; Robertson et al., 2001*).

Numerous studies have used pressurized air or oxygen to accelerate aging in the laboratory, specifically for performance characterization of asphalt binders. Bahia and Anderson (*1995*) indicated that the use of high pressure was desirable to simulate accelerated long-term aging in asphalt binders because: (1) volatile loss was minimized, (2) aging could be accomplished without high temperatures, (3) large sample sizes could be accommodated, (4) field climate conditions could be approximated, and (5) laboratory use was practical. The pressure-aging vessel (PAV, AASHTO R 28), which is believed to simulate about four to ten years of aging, is the long-term aging test currently specified in the Superpave specifications. Kandhal and Wenger (*1975*) concluded that stiffness of field cores reached an asymptotic value within 10 years of service. Mallick and Brown (*2004*) evaluated field cores extracted at different service lives. They stated that the PAV method could successfully simulate long-term aging of asphalt binder.

Short-Term Aging of Asphalt Concrete Mixtures

Asphalt mixture production temperatures are optimized to ensure complete drying of mineral aggregates, proper coating and bonding of the aggregate-binder system, adequate workability and compactability for handling and compaction (*Newcomb et al., 2015*). The production and placement of mixtures require conditioning temperatures that range from 220ºF (104ºC) to 325ºF (163ºC) (*Kuennen 2004; Newcomb 2005*). Most State highway agencies already have short-term aging procedures to simulate the aging and asphalt absorption of an asphalt mixture as it is produced in a plant and transported to the site. The effectiveness of short-term aging process to simulate the mixing, production and placement process for asphalt mixtures is important to estimate accurately the mixture's durability and stability.

The standard practice for asphalt mix design in the laboratory is to simulate the aging and binder absorption that occurs during production and construction by conditioning loose mix prior to compaction for a specified time and temperature. The recommended procedure in AASHTO R 30 for preparing specimens for volumetric analysis is 2 hrs at the compaction temperature, as described in the TxDOT mix design (Tex-205-F). One of the goals of NCHRP Project 09-52 was to investigate the short-term laboratory conditioning of asphalt mixtures. In that study, Newcomb et al. (*2015*) recommended the following basic changes to the AASHTO R30 short-term aging protocol: 1) fixing the compaction temperatures for warm mix asphalt (WMA) at 240ºF (116ºC) and hot mix asphalt (HMA) at 275ºF (135ºC) and 2) conditioning the sample for 2 hrs at the compaction temperature regardless of whether the specimens are being prepared for volumetric analyses or performance testing. These conclusions were drawn by comparing volumetric parameters (i.e., theoretical maximum specific gravity and binder absorption), dynamic modulus and resilient modulus, and HWT test results from lab-produced specimens to plant-produced specimens.

Table 2.1 presents a summary of some of the studies conducted to investigate the influence of short-term aging conditions (temperature and time), for HMA and WMA production. In general, lower curing temperatures are proposed for WMA mixtures than those normally specified for HMA. For HMA mixtures, 2 hrs of curing is the common recommendation.

Table 2.1 Summary of Laboratory Short-Term Aging Protocols for Asphalt Mixtures

Reference	STOA Conditions	Key Findings
Aschenbrener and Far (1994)	- 2 hrs at compaction temperature	- Reheating influenced HWT test results
Rashwan and Williams (2011)	- 2 hrs at 302°F (150°C) for HMA - 2 and 4 hrs at 230°F (110°C) for WMA	- Dynamic modulus and flow number tests results were higher for HMA with higher temperature and for mixtures with RAP
Bonaquist (2011)	- 2 and 4 hrs at compaction temperature	- Aggregate absorption and IDT test results comparable to field cores - Recommend 2 hrs at compaction temperature for WMA and longer aging period for rutting and moisture susceptibility
Clements et al. (2012)	- 0.5, 2, 4, and 8 hrs at 275°F (135°C) for HMA - 0.5, 2, 4, and 8 hrs at 237°F (114°C) for WMA	- Similar DCT test results for HMA and WMA - Reduced dynamic modulus and flow number and increased rutting for WMA and HMA
Estakhri (2012)	- 2 hrs at 275°F (135°C) - 4 hrs at 275°F (135°C)	- Equivalent HWT test results for WMA and HMA - Aging time and temperature effect on HWT and overlay tester results
Epps Martin et al. (2014)	- 2 and 4 hrs at compaction temperature at 275°F (135°C) - 2 hrs at compaction temperature + 16 hrs. at 140°F (60°C) + 2 hrs. at compaction temperature	- Recommend 2 hrs at 275°F (135°C) for HMA and 2 hrs at 240°F (116°C) for WMA

NCHRP Project 09-52: Short-Term Aging

As part of NCHRP 09-52, Newcomb et al. (*2015*) proposed changes to the AASHTO R30 protocol for conditioning of HMA and WMA. Newcomb et al. considered aging that occurred during production, construction, and in-service life of the pavement. Although NCHRP 09-52 focused on short-term laboratory aging to better simulate field behavior during mixing, transportation, and placement of asphalt mixtures, the long-term aging effect was also considered to reflect 1-3 years of in-service life of the pavement.

The study addressed the aging-patterns of LMLC specimens with raw materials, plant-mixed plant-compacted (PMPC) specimens and field cores. Three-performance tests, namely the resilient modulus, HWT and dynamic modulus tests were used to evaluate the effects of short- and long-term aging stages. They determined that the resilient modulus test was sensitive to assess aging progression. In addition, the resilient modulus test was used to compare stiffness of LMLC and PMPC cores. Binder was extracted and recovered from each field core to assess aging. Specimens were tested using the DSR and FTIR devices. Newcomb et al. (*2015*) validated the STOA protocol for LMLC and PMPC specimens indicating similar aging processes between the two.

Newcomb et al. (*2015*) focused on the impacts associated with climate, type of aggregate (including aggregate absorption), asphalt type and source, RAP/RAS content, type of plant and the temperature(s) volumetric and performance parameters of asphalt mixtures after the short-term conditioning. They found that the factors affecting performance of asphalt mixtures were the type and proportion of recycled material, aggregate absorption and asphalt source. They observed that the mixtures that included RAP and RAS in the asphalt mixture exhibited significantly higher stiffness. Conversely, they indicated that the stiffness of a material with high aggregate absorption would be lower. They determined that many more parameters affected the long-term aging.

Newcomb et al. (2015) carried out the study in two phases. Phase I consisted of verification of asphalt mixtures volumetric mix design, performance test, and analysis of factors affecting short-term aging. The volumetric mix design involved quantifying the theoretical maximum specific gravity, percent of absorbed binder, percent of effective binder, and effective binder film thickness of LMLC and PMPC specimens. They found good correlation between the volumetric properties extracted from LMLC and PMPC specimens except for one section with highly absorptive aggregates. They also reported that since the lab and plant mixtures from the same source did not have the same absorption rate, their theoretical maximum specific gravity and percent of absorbed binder differed.

The performance test results from comparable LMLC and PMPC specimens yielded reasonable correlations. However, the performance test results from LMLC specimens and field cores were poorly correlated. The field cores generally provided higher rutting resistance. Newcomb et al. (*2015*) explained the difficulty to fit the field cores into the HWTD molds as one possible factor for the lack of correlation. They reported good relationship between the level of binder oxidation and binder stiffness.

Newcomb et al. (*2015*) proposed an AASHTO practice for conducting plant-aging studies that provide detailed instructions on how to prepare LMLC or PMPC specimens. Figure 2.1 illustrates the recommended flowchart for conducting short-term aging on asphalt mixtures. From Phase I of this study, they determined that the sources of binder, aggregate absorption and the inclusion of recycled materials affected the stiffness and rutting potential of short-term aged AC mixtures. However, regardless of plant type or production temperature, no significant effect was observed on short-term aged AC mixtures.

Phase II consisted of quantifying field aging and the correlation of field aging to LTOA protocol. Newcomb et al. (*2015*) proposed cumulative-degree-days (CDD) as a field metric that captures field aging by considering climate and time. Field aging is quantified by the property ratio of an AC mixture from the sampling time to the original state (during construction). AC mixtures with a higher property ratio go through a higher rate of aging during the given period. They concluded that climatic zones that are warmer are more susceptible to aging and have higher CDD values. Conversely, cooler climatic zones experience lower levels of aging and CDD values. Newcomb et al. (*2015*) evaluated seven field sites; Equation 1 can be used to calculate CDD of field aging by accounting for different dates of construction and climatic zones.

$$CDD = \sum(T_{max} - T_{base}) \quad \textbf{(1)}$$

where T_{max} is the daily maximum temperature (ºF) and T_{base} is recommended as the base temperature of 32ºF (0ºC).

Newcomb et al. (*2015*) evaluated LTOA similar to STOA by analyzing production temperature, plant type, recycled material, and the aggregate absorption parameters. They determined that including recycled material and the aggregate absorption and the inclusion of recycled materials affected the stiffness of long-term aged AC mixtures. Similar to STOA, regardless of plant type or production temperature, no significant effect was observed on short-term aged AC mixtures. A significant increase in stiffness and rutting potential was reported for LTOA specimens with high absorption. They concluded that AASHTO R30 current long-term aging protocol reflected the first 1 to 3 years of in-service life of the pavement. Newcomb et al. (*2015*) validated the findings from Epps Martin et al. (*2014*) with similar behavior resulting from the LTOA protocols.

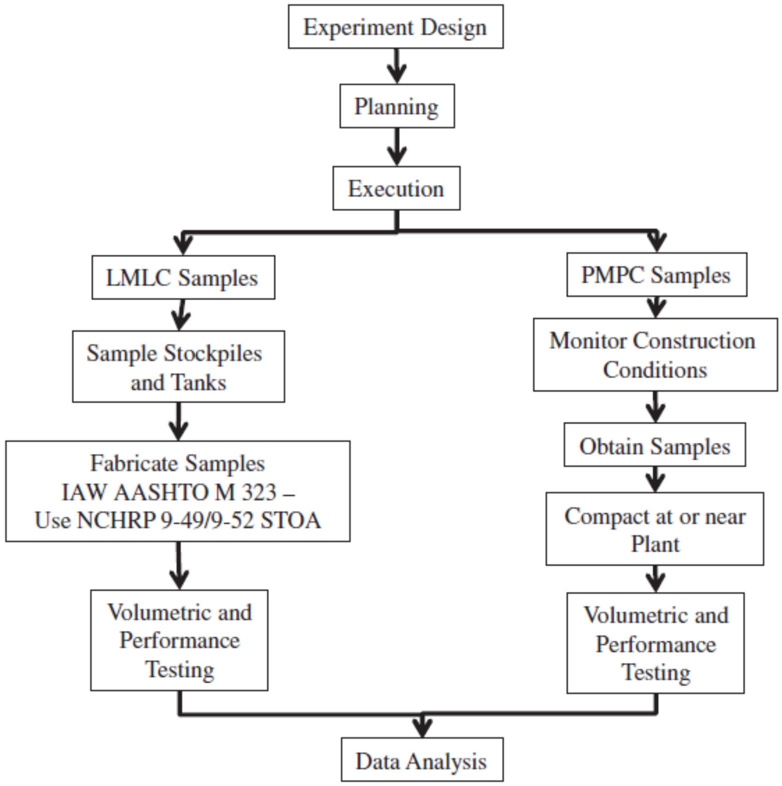


Figure 2.1 Short-Term Aging Flow Chart for Asphalt Mixtures (*Newcomb et al., 2015)*

Long-Term Aging of Asphalt Concrete Mixtures

The issue of aging of asphalt binder has been recognized and investigated for almost a century (*Hubbard and Reeve, 1913; Thurston and Knowles, 1936; and Van Oort, 1956)*. These previous studies confirmed that oxidation is responsible for changes in asphalt properties due to exposure to outdoor weathering. Subsequent studies have been conducted to develop aging protocols that can be implemented to condition laboratory specimens for performance characterization. Table 2.2 shows a summary of some of the relevant studies on the LTOA properties of asphalt mixtures. Two main aspects have been considered during the development and implementation of laboratory aging protocols: (1) state of material during aging (compacted specimen vs. loose mix) and (2) pressure level (oven aging vs. pressurized aging). These factors are discussed in more detail in the following sections.

Table 2.2. Summary of Laboratory Long-Term Aging Protocols for Asphalt Mixtures

Reference	LTOA Conditions	Key Findings
Morian et al. (2011)	- Lab aging (3, 6 and 9 months at 60ºC) - Compacted specimens	- Increased mixture dynamic complex modulus and binder carbonyl after LTOA - Binder source influenced aging rate - Aggregate source didn't have a major effect
Tarbox and Sias Daniel (2012)	- Lab aging of 2, 4 and 8 days at 85ºC - Compacted specimens from plant-produced mixtures	- Increased stiffness with LTOA regardless of mix characteristics - RAP mixes stiffens less than virgin mixes
Azari and Mohseni (2013)	- Lab aging of 2, 5 or/and 9 days at 85ºC - Compacted specimens	- Increased resistance to permanent deformation with LTOA - Relationship between results from STOA and LTOA protocols
Safaei et al. (2014)	- Lab aging of 2 and 8 days at 85ºC - Compacted specimens	- Good agreement between asphalt binder and mixture results - At extended LTOA, fatigue performance of WMA and HMA is negligible - HMA yields higher modulus than WMA mixtures and binders
Epps Martin et al. (2014)	- Lab aging of 1 to 16 weeks at 60ºC and 5 days for 85ºC - Compacted specimens	- Increased stiffness with higher temperatures and times for laboratory LTOA - After extended aging time, WMA and HMA exhibit similar performance
Newcomb et al. (2015)	- Lab aging of 5 days at 85ºC and 2 weeks at 60ºC - Compacted specimens	- Mixture aging is more sensitive to aging temperature than to aging time
Rad et al. (2017)	- Lab aging at 95°C and 135°C - Loose mixtures	- Unaffected relationship between binder chemistry and rheology with 95°C - Using 135°C decrease dynamic modulus and fatigue resistance
Kim et al. (2018)	- Lab aging at 95ºC and 135ºC - Compacted specimens and loose mixture - Draft oven and PAV	- Current AASHTO R 30 practices induce aging gradient - Loose mix state expedites aging - Loose mix aging at 95ºC is recommended for LTOA

NCHRP Project 09-54: Long-Term Aging

Kim et al. (*2018*), as part of NCHRP Project 09-54, deliberated on the long-term aging of asphalt mixtures in order to improve AASHTO R30 protocols. They indicated that one of the biggest shortcomings from AASHTO R30 was that only one temperature was used to simulate the various range of effects associated with different climatic zones.

Kim et al. (*2018) analyzed* the effect of aging between loose mixtures and compacted specimens, performed a sensitivity analysis on the effects of the curing temperature, compared results from the oven aging against pressure aging, and observed the effects associated with the depth of the HMA or WMA layers, to propose a replacement of current long-term aging protocols. The asphalt binders were evaluated using attenuated total reflectance ATR-FTIR spectroscopy, DSR after being aged using the PAV, RTFO and the universal simple aging (USAT) methods. Asphalt mixture performance testing included the dynamic modulus tests with the asphalt mixture performance tester (AMPT). All HMA asphalt mixtures were STOA for 4 hrs at 275ºF (135ºC) while WMA underwent STOA for 2 hrs at 243ºF (117ºC). They selected long-term aging curing temperatures of 203ºF (95ºC) and 275ºF (135ºC) for a wide range of curing times that were dependent on AIP of field cores to match aging levels.

Kim et al. (*2018*) reported that aging of loose mixture results in faster and more uniform aging when compared to compacted specimens. Increasing the curing temperature will reduce the time needed for oven curing the material. However, if the curing temperature exceeds 212ºF (100ºC) abnormal chemical reactions may occur that otherwise would not occur while the pavement is in service. A map of the US considering various climatic zones at specified depths (6 mm, 20mm and 50 mm) below the pavement surface and time of aging (4,8 and 16 years) was proposed as part of the new AASHTO R30 modification.

Similar to CDD proposed by Newcomb et al. (*2015*), Kim et al. (*2018*) developed the climatic aging index (CAI). It was reported that the advantages of CAI over CDD was that CAI used pavement temperature as opposed to using air temperature, and that CAI could estimate pavement temperature (hourly) at different depths. Equation 2 is used to determine the time required for oven curing to match field aging.

$$CAI = \sum_{i=1}^{N}[D\ x\ A\ x\ \exp\left(\frac{-E_a}{R\ x\ T_i}\right)/24] \qquad \textbf{(2)}$$

where CAI is the climate aging index or time required for oven curing (day), D is depth correction factor, A is the frequency factor, E_a is the activation energy (kJ/mol), R is the universal gas constant (kJ/mol-K) and T_i is the pavement temperature from the enhanced integrated climatic model (EICM) with respect to the target depth (K).

Kim et al. (*2018*) evaluated the influence of depth, frequency factor and the activation energy on obtaining the CAI value. Table 2.3 presents the correction factors for D, A and E_a parameters. The correction factors influence the scatter of the data points and the laboratory curing duration. With the use of Equation 2 and the correction factors from Table 2.3, Kim et al. was able to calculate the CAI values for different locations around the US.

Table 2.3. CAI Correction Factors (*Kim et al., 2018)*

MIX ID	Depth Correction Factor (*D*)	Arrhenius Equation, Pre-exponential Factor (*A*)	Arrhenius Equation, Activation Energy (E_a)
Surface Layer (6 mm)	1.0000	1.4096	13.3121
20 mm Depth	0.4565	1.4096	13.3121
Deeper Layer (below 20 mm)	0.2967	1.4096	13.3121

It was concluded that the long-term aging of loose mix material expedited the aging with more uniformity than specimens that are compacted. Similar to previous studies cited earlier (*Arega et al. 2013*), they also reported that the compaction of specimens after material has undergone LTOA did not affect the performance or compaction.

Kim et al. (*2018*) proposed a standalone procedure for long-term conditioning by separating AASHTO R30 short- and long-term condition of HMA. They recommended a curing temperature of 200ºF ± 5ºF (95ºC ± 3 ºC) to condition the asphalt material in its loose state. They indicated that the duration of curing was dependent on the age, climate, and depth using Equation 5. Their recommended specification will contain the US maps with time required to match either 4, 8 or 16 years of field aging for depth ranges of 6 mm, 20 mm or 50 mm.

Both NCHRP 09-52 and NCHRP 09-54 bring suitable information for STOA and LTOA, validation of these protocols was performed as preliminary assessment of current aging protocols.

Effect of STOA and LTOA on Stability and Durability of Asphalt Mixtures

Several researchers have studied the rutting potential of asphalt mixtures after subjecting asphalt mixtures to STOA and LTOA conditions. As a part of the NCHRP Project 9-23, *Houston et al. (2005*) used the dynamic modulus test to determine the stiffening of asphalt mixtures due to long-term aging. A clear increasing trend in the stiffness of the mixtures was not found in that study. Azari (*2011*) investigated the effect of STOA and LTOA on the flow number (FN) test results. She observed an increase in FN with respect to the aging condition of the mixture, but the trend was not enough to propose an appropriate aging duration. Azari and Mohseni (*2013*) determined the effect of STOA and LTOA conditioning on the permanent deformation of asphalt mixtures utilizing the incremental repeated load permanent deformation (iRLPD) test. They implemented the minimum strain rate (MSR) parameter to quantify the change in stiffness of the asphalt mixtures after short-term aging and various long-term aging conditions. They proposed conducting the iRLPD test on plant-produced mixtures at two stages after production and before the mixture was placed down to determine better the rutting potential of asphalt mixtures.

Unlike rutting, the cracking susceptibility of asphalt mixtures increases as the mixture loses ductility due to aging. In order to accurately assess the cracking potential of asphalt mixtures in the most critical state (i.e., after long-term aging), appropriate protocols must be used to simulate long-term aging in the asphalt mixture. However, if the goal is to screen different asphalt mixtures based on their cracking resistance, a more effective strategy would be to utilize laboratory aging protocols (time and temperature combinations) that allow asphalt mixtures to reach a steady-state condition in the shortest and most practical time possible.

A limited number of studies have been conducted to investigate the effect of aging on the cracking potential of asphalt mixtures subjected to long-term aging conditions. Kim et al. (*2012*) evaluated the fatigue performance of asphalt mixtures at four different aging conditions including 135ºC for 4 hrs and 85ºC for 2/4/8 days. Daniel (*2013*) investigated the aging potential of asphalt mixtures designed with different amounts of reclaimed asphalt pavement (RAP) under LTOA conditions. It was concluded that LTOA specimens yielded a higher stiffness value than STOA specimens. However, RAP content in the mixture did not increase mixture stiffness because RAP has pre-aged asphalt binder that oxidizes at a much lower rate compared to virgin asphalt binder. Blankenship et al. (*2018*) investigated the influence of various long-term aging conditions for mixtures using the disk-shaped compact tension (DCT) test. They commented that the LTOA procedure from AASHTO R 30 might not be harsh enough to simulate non-load associated aging that a pavement accumulates over its service life.

State of Material (Compacted vs. Loose) During Aging

In a laboratory setting, aging compacted specimens is a matter of practical convenience to determine the performance of asphalt mixtures. Following the standard method AASHTO R 30, the short-term aged mixture is compacted and cut to specimen dimensions before placing the finished specimens into a forced draft oven for long-term aging. The long-term aging simulation is carried out at 85ºC for 120 hrs to represent presumably five to ten years of aging in the field. To simulate long-term aging, Bell et al. (*1994*) conditioned asphalt specimens in the oven for different lengths of time before conducting performance tests on laboratory long-term aged specimens and their corresponding field cores. Their recommendation for the long-term aging of compacted specimens was to age specimens at 85°C for 2, 4, and 8 days in the oven. The compacted specimens conditioned for 2 days at 85°C simulate 1-3 years of field aging whereas the 4- and 8-day yield 9-10 years. Harrigan (*2007*) documented a few potential limitations with the current standard aging method such as 1) using only one aging temperature to represent the long-term aging spectra, 2) recommending too wide of a range for design purposes, and 3) not considering the effect of air void content.

The long-term oven aging of compacted specimens leads to both radial and vertical oxidation gradients, which is a concern for performance testing because the properties throughout the specimen can vary (*Elwardany et al., 2016*). Although less common than aging compacted specimens, the laboratory aging of loose (uncompacted) asphalt mixture has been recommended in recent years to eliminate the aging gradient within specimens (e.g., *Arega et al. 2013, Partl et al. 2013, Mollenhauer and Mouillet 2011, Van den Bergh 2011, Reed 2010, Braham et al. 2009, Dukatz 2015, Elwardany et al. 2016*). Von Quintus (*1988*) aged loose mixture at 135°C in a forced draft oven for 8, 16, 24, and 36 hrs to simulate long-term field oxidation. Van den Bergh (*2009*) conducted an experimental program for aging loose mixes in the laboratory with the goal of replicating RAP material. Mollenhauer and Mouillet (*2011*) also conducted a study on the aging of loose mixtures to produce RAP materials. The properties of the binders extracted from laboratory-aged mixtures were compared to the properties of binders extracted from 11 to 12-year-old sections. They concluded that oxygen pressure could significantly reduce the aging time.

Rad et al. (*2017*) investigated the implications of aging loose uncompacted asphalt mixtures at 135ºC. They performed Fourier transform infrared spectroscopy (FTIR) and dynamic shear rheometer (DSR) testing to document the influence of the selected curing temperature. They concluded that long-term aging a mixture at 135ºC negatively influenced the performance and determined aging temperatures at or below 95ºC as optimum.

Some advantages of aging loose mixtures over aging a compacted specimen are: (1) air and heat can easily circulate inside the loose asphalt mixture inducing uniform aging throughout the mix; (2) problems associated with compacted specimen integrity during laboratory aging may be reduced; and (3) the rate of oxidation may increase due to the larger area of the binder surface being exposed to oxygen (*Kim et al., 2018*) consequently reducing the time required to achieve a certain level of aging. On the other hand, it can be argued that compacting an aged loose mix results in different aggregate packing and internal structure, and consequently different performance as compared to aging a compacted test specimen. To address this concern, Arega et al. (2013) compared the internal structure of asphalt mixes compacted before and after long-term aging using X-ray tomography and reported that there was no significant difference in the internal structure of these mixes. They also pointed out that the rank order of fatigue cracking resistance of asphalt mixtures did not change significantly after long-term aging (*Arega et al., 2013*).

Lab Mixed Lab Compacted (LMLC) vs. Plant Mixed Lab Compacted (PMLC) Specimens

Different methods and approaches have been utilized to evaluate the performance of mixtures in a laboratory environment. Typically, testing of asphalt specimens in laboratories is conducted using laboratory fabricated specimens as it is implicitly assumed that these specimens simulate plant conditions. However, recent studies have highlighted the differences in the rheology of laboratory and plant produced specimens. To best implement performance and simulation-based approaches, it is critical to understand the salient variances between differently produced specimens. The commonly used laboratory compacted specimens that were considered in this study include:

- LMLC: Such specimens are produced in the laboratory using methods that are intended to simulate the plant mixing conditions *(Kim et al. 2003)*.
- PMLC: Such specimens are produced in the plant but compacted in the laboratory by reheating the loose mix.

These two methods employ different handling, mixing, and compaction techniques, which can impact the properties of the specimens. Past research efforts in this area have suggested mixed results and the likelihood that lab produced specimens could be stiffer than plant produced specimens. One study assessed mixtures containing different proportions of RAP and RAS (*Johnson et al. 2010*). It was seen that the dynamic modulus of PMLC specimens were lower than LMLC specimens. Xiao et al. (*2014)* tested the binders of recovered samples from different mixtures and showed that the failure temperature of lab-produced mixtures was higher than that of plant produced mixtures. However, McDaniel et al. *(2002)* performed frequency sweep tests on different specimens from various states and indicated that the stiffness of lab and plant produced mixtures were similar. Rahbar-Rastegar and Daniel *(2016)* compared the rheological characteristics of plant-produced and lab-produced specimens, and impact of different mixture variables. The results indicated that the mixtures produced in dissimilar conditions resulted in samples with different material properties. Additionally, there were no systematic differences between the properties in either of the production conditions. Rather, the differences were more strongly influenced by binder grade, gradation etc. A study by Yin et al. *(2015)* reported that current STOA criteria for HMA and WMA were sufficient to reproduce the changes seen in performance criteria and volumetric properties during plant production.

Influence of Pressure on Aging

The aging rate of asphalt mixtures can be accelerated by increasing the curing temperature during oven aging. This observation has led to proposals for the use of high curing temperatures such as 135ºC for loose mixture (e.g., *Braham et al., 2009; Blankenship, 2015; Dukatz, 2015*). However, increasing curing temperatures for LTOA condition may introduce inconsistencies on the chemical composition of the asphalt binder. The disruption of polar molecular association and subsequent sulfoxide decomposition become critical at temperatures that exceed 100ºC (e.g., *Herrington et al. 1994, Petersen 2009, Petersen and Glaser 2011, Glaser et al. 2013*). Rad et al. (*2017*) investigated the effect of two long-term aging temperatures, 95ºC and 135ºC, to age asphalt mixtures and determine their engineering properties based on dynamic modulus and damage resistance measured using the simplified viscoelastic continuum damage (S-VECD) model. They recommended an optimal aging temperature of 95ºC for loose mixtures. They indicated that the relationship between asphalt binder chemistry and rheology was unaffected if the aging temperature was below 95ºC.

As an alternative to the oven aging, pressure and forced air can be used to increase the rate of aging in asphalt mixtures. Several researchers have tried pressure aging of asphalt mixtures for both compacted and loose mixture specimens. Von Quintus et al. (*1988*) performed a study on LTOA and long-term pressure aging of compacted specimens. The study consisted of LTOA compacted specimens at 60°C for two days following five days of conditioning at 107°C. Pressure aging was conducted at 60°C and 100 psi (690 kPa) for durations of five and ten days. They concluded that higher aging levels were reached for oven-aged specimens compared to the pressure-aged samples.

Bell et al. (*1994*) evaluated several pressure-aging systems as part of the SHRP project. They tried 'pressure oxidation' of compacted specimens using several pressure/temperature combinations in a pressurized vessel. Compacted samples were exposed to air or oxygen for 0, 2, or 7 days at pressures of 690 kPa or 2070 kPa and at 25°C or 60°C. An unusual modulus trend was observed, i.e., as the oxidation level increased the modulus decreased.

Khalid and Walsh (*2002*) developed an accelerated pressure oven procedure to simulate long-term aging of porous asphalt mixtures. One of the main shortcomings of PAV aging compared to oven aging was the amount of material that could be aged simultaneously. In order to obtain uniform aging, a uniform thin layer of loose mix should be placed in the PAV, which reduced the capacity of the instrument to around 1 kg (*Partl et al., 2013*). Partl et al. (*2013*) study suggested that binder oxidation continued up to nine days of conditioning, but that the rate of oxidation decreased with an increase in the duration of the conditioning.

Influence of Ozone and Nitrogen oxide on Aging

More than 30 different laboratory procedures have been reported for simulating the aging of both loose and compacted asphalt mixes. Although some of those methods (including those previously mentioned) have been adopted widely, there are still some disadvantages associated with it. Firstly, high temperatures exceeding 100°C are often used which considerably exceeds the in-service field temperature. Such high temperatures can lead to chemical reactions that may not happen in-service, such as increased polymerization and oligomerization. In addition, conditions such as increased pressure and duration are often used which makes it challenging to adopt these methods for routine tests. A promising alternate method that negates some of these disadvantages is the Viennese aging protocol (VaPro) which employs highly oxidant gas to simulate long term aging (*Steiner et al. 2016*). Atmospheric chemistry reveals that the oxidation potential of oxygen (O_2) is below 200°C, but there are other gases in the atmosphere, which have a much higher oxidative potential. These gases, such as ozone (O_3) or nitrogen oxides (NO_x) are called reactive oxygen species (ROS) and are particularly important in the tropospheric oxidative cycle. VAPro uses traces of these gases to accelerate the simulated aging process. Through this procedure, it is possible to optimize mix design for short term performance and also take into consideration the impact of oxidative aging during its service life. Its design is based on a triaxial cell in which there is a regulated flow of an oxidizing agent enriched with ozone and nitric acid. The original setup of VaPro is presented in Figure 2.2. Promising results have been reported using this method wherein asphalt mixes have been shown to be long term aged at lower temperatures of around 60°C for 4 days. Based on the findings from other studies, it is possible to modify this process to be more efficient and shorter by aging loose asphalt mixtures prior to compaction and testing.

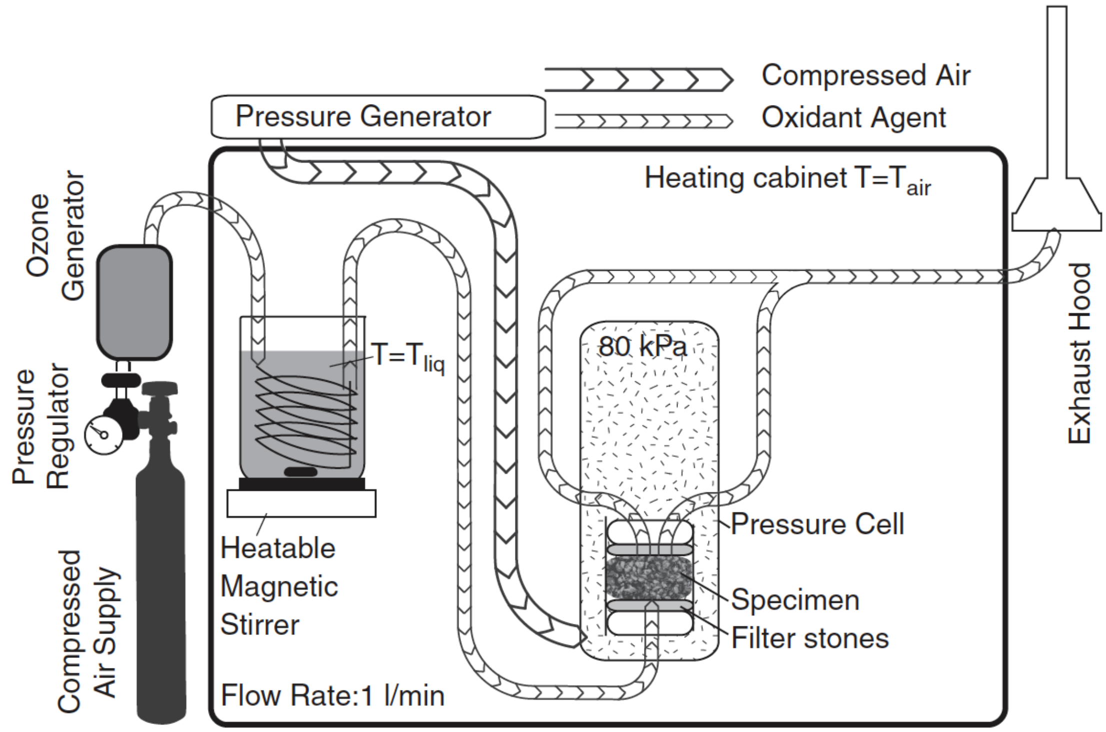


Figure 2.2. Overview of VAPro Procedure ***(Steiner et al. 2016*****)**

Laboratory Simulation of Binder Aging

Similar to the protocols used with asphalt mixtures, the aging of binders is broadly classified into short-term aging and long-term aging. However, unlike the methods of aging in mixtures the approaches used to simulate these stages in binders are well recognized and generally standardized. The most frequently used method to simulate STA and LTA include RTFO test and PAV test, respectively. In the RTFO test, bitumen is aged at 165°C in rotating glass bottles for 75 min. The mass of bitumen used and dimensions of bottles are kept consistent to ensure the sample thickness is the same. It has been reported that on average the RTFO test typically leads to a doubling of viscosity. However, it may vary generally between 1.5 and 4 for the viscosity at 60°C *(Christensen and Anderson, 1992)*. The test standard for this method is ASTM D2872. The simulation of long-term aging is much more complicated as there are many factors that influence it, such as location of the bitumen in the pavement, thickness of film, porosity etc. In the PAV test, binder samples are conditioned in 3.2 mm thick films at a temperature of 100°C for 20 hrs in a pressurized chamber at 2.07 MPa. For binders containing polymers, a reduced temperature of around 75°C is considered ideal. Studies have shown that PAV aging can satisfactorily simulate the aging of bitumen in surface courses for around 4-8 years *(Anderson et al. 1994*). The test standard for this method is ASTM D652. Ultraviolet (UV) aging is also another factor that contributes to the aging of binder in field. However, there is no standardized or well recognized method to simulate this satisfactorily in a laboratory environment.

Aging Characteristics of Asphalt Binders

Extraneous Factors that Influence Aging

The aging of asphalt binder is primarily a diffusion driven process that transpires as a result of thermal reactions and photo-oxidation between bitumen components and oxygen in the atmosphere

(Liu et al. 2014). Asphalt binder undergoes oxidation through the diffusion of oxygen which changes its chemistry and consequently its physical properties. Environmental conditions such as temperature, weather, extent of solar radiation can considerably affect the overall extent of oxidation. Most prominently, effect of temperature on asphalt oxidation has been studied by different researchers. It has been shown that the mechanism of oxidation is strongly correlated with temperature and that the oxidation products formed at different temperatures could be different *(Herrington and Ball, 1996*). The increase of aging, characterized by the changing viscosity at various temperatures has been correlated to obey a hyperbolic relationship with an asymptotic limiting viscosity which suggests that the oxidation reaction occurring at different temperatures could be dissimilar. The main reason for this has been ascribed to the variations in the temperature dependence of the rates of contending oxidation reactions and differences in the availability of reactive species as a result of temperature-dependent structural effects. Such effects are expected to give raise to alterations in the nature of oxidation products or the ratio of different products.

Factors related to the mixture and pavement structure, such as void percentage and compaction also affect the aging of asphalt mixtures. The extent of voids primarily dictates the extent to which oxygen can enter bitumen. Further, the percentage content of bitumen also affects the level of aging. The amount of bitumen in the mixture determines the thickness of the film around aggregates. Studies have reported that greater the thickness of film, the lower the aging seen. *(Goode and Lufsey, 1965) The* oxidation of mixtures proceeds from the surface down with time. Hence the hardening of binder is more severe in the upper part of the pavement and reduces with depth *(Petersen, 2009)*. UV radiation which occurs mostly in the upper layers of the pavement course also influence the extent of aging. It has been shown that viscosity and stiffness of binders can increase after UV radiation exposure which clearly illustrates the link between UV exposure and aging. Further higher rates of aging were found in samples below 150 mm *(Wu et al. 2010*).

Mechanism of Binder Aging

In order to develop accurate and efficient aging protocols, it is important to understand the fundamentals of aging in asphalt binders. These mechanisms can help guide the proposed research tasks. For example, an understanding of these mechanisms can help identify and establish steady-state oxidation rates, which is at the core of the proposed research approach; explain why certain methods to accelerate aging (e.g., use of temperatures higher than 100^{o}C) may not be appropriate; and facilitate exploration of other novel and efficient methods of aging that may be more accurate and faster (e.g., use of ozone instead of air).

The aging of asphalt binder is a complex process wherein the molecules may be irreversibly changed through oxidation reactions, polymerization, and the evaporation of lighter components. Rheologically, the aging of bitumen leads to hardening of the material which increases the potential for fatigue and low temperature cracking. Bitumen is a colloid composed of millions of organic molecules containing a variety of hydrocarbons: aliphatic, aromatic and a combination of both aliphatic and aromatic. The aliphatic molecules are saturated polycyclic structures, long-chain or branched paraffins or unsaturated olefins. The main constituents of the aromatics are the different polyaromatic structures with their fused rings. Moreover, various amalgamations of these structures are present with saturated hydrocarbon side chains and substitutions of heteroatoms like nitrogen, Sulphur, and oxygen *(Redelius and Soenen, 2015*). Naturally, the detailed identification of individual bitumen molecules is an unattainable task. However, groups of molecules exhibiting similar chemical behavior can be grouped or correlated together to identify the building blocks and properties of different bitumen.

The most frequently used practice is centered on the partition of bitumen into its polar fractions based on the increasing polarity and polarizability, and referred to as saturates, aromatics, resins and asphaltenes (SARA) fractionation. Among these, saturates and aromatics are the oily components that directly determine the workability and plasticity of the binder, resins are the sticky semi-solid components that dominate the ductility and cohesiveness, and asphaltenes exist as solid components that are dispersed in the oily component *(Xing et al, 2020)*. On this basis, the colloidal structure of bitumen is divided into three types:

- Sol structure, which includes lower amounts of asphaltenes that are fully dispersed into the oily components of saturates and aromatics;
- Gel structure, which includes large numbers of asphaltenes surrounded by resins, as well as less oily components; and
- Sol-gel structure, which belongs to an intermediate structure between sol- and gel-structures owning appropriate amounts of asphaltenes with more surrounded resins.

To quantify the different colloidal structures of bitumen, the colloidal index (CI) was introduced *(Zhang et al., 2020)*. It is calculated using Equation 3 and based on the respective relative proportion of SARA fractions in each bitumen.

$$\mathrm{CI} = \frac{\mathrm{m(asphaltenes)+m(saturates)}}{\mathrm{m(aromatics)+m(resins)}} \qquad \textbf{(3)}$$

For commonly used unmodified binders, the CI value ranges between 0.5 and 2.7, and the colloidal structure were determined in conformity with the rule that (1) when the CI value is lower than 0.7, the colloidal structure performs in sol-type state; and (2) when the CI value exceeds 1.2, it behaves in gel-type state *(Leseur, 2009)*.

Aging results in the decrease of aromatics and increase of both resins and asphaltene fractions *(Chen et al. 2018)*. There is a gradual shifting of the molecules from less polar fractions to more polar fractions. The extent of the percentage of asphaltene and resin is critical as the amount of asphaltenes influence concentration and accelerate ageing kinetics. When the maltene fractions are not in proportion to sufficiently solvate the asphaltene micelles, their solvating capacity is lessened and can further bond together. This may result in an irregular open packed structure of mixed composition. Such binders are closer to the “gel” structure in relation to the colloidal model of bitumen. The loss of maltenes also changes the colloidal stability of the binder causing the flocculation of asphaltenes, which can increase the stiffness. The changing fractions for unmodified binders with aging time is represented in Figure 2.3. Thus, aging brings about an increase of CI value and promotes the original colloidal structure approaching towards gel-type structure.

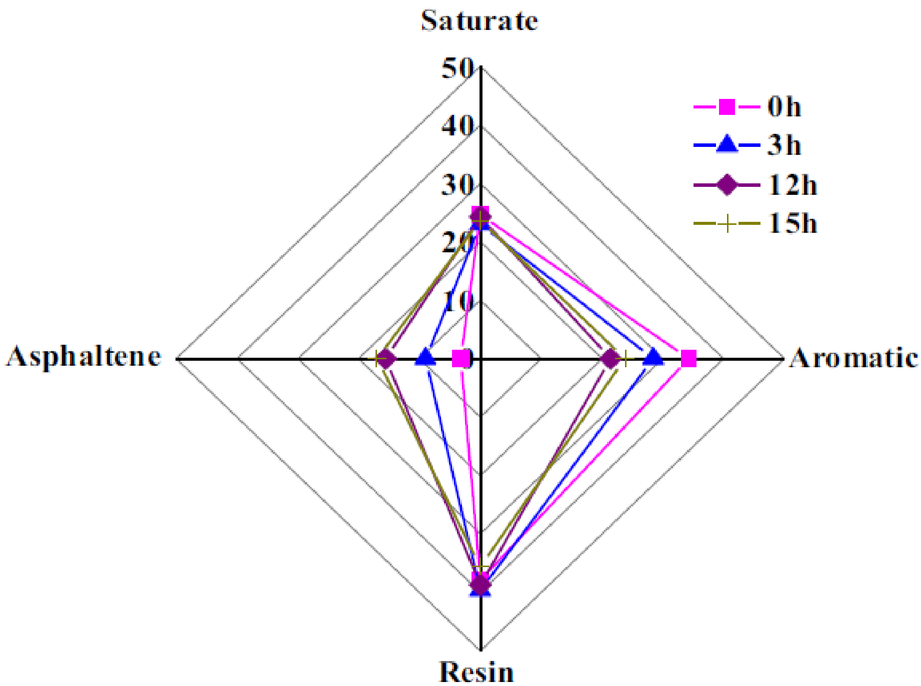


Figure 2.3. SARA changes of binders versus aging time *(Chen at al. 2018)*

Characterization of Aging

Different rheological, microscopic and chemical techniques have been reported to assess the aging features of asphalt binders. Other than the classical tests such as penetration, softening point and viscosity which have started to become outdated in recent times, rheological analysis is most frequently carried out using a DSR. Tests using the DSR are usually focused on the changes in complex modulus (G*) and phase angle (δ) under different conditions such as temperatures, and defined stress and strain levels. Using these data, parameters can be obtained in relation to the rheology of binders such as plastic deformation and cracking performance. Common indicators used in the PG grading specifications include G*/sinδ and G.sinδ which are used to indicate the rutting and fatigue susceptibility of binders. It has been widely reported that with ageing, the complex modulus of binders increases whereas phase angle decreases *(Fernandez-Gomez et al. 2013)*. The extent of these changes depending on the specific binder and testing conditions. In general, at low frequencies, an increased complex modulus is observed in aged binder in comparison to unaged binder. Conversely, at high frequencies ageing seems to have low influence on complex modulus. Other widely used tests conducted for rheological characterization using DSR include multiple stress creep recovery test (MSCR) and rheological ductility indicator test (Glover-Rowe).

Apart from DSR tests, another reported method is the poker chip test which has been proposed as in indicator of the true strength and fracture properties of an asphalt binder in the state of stress similar to what it actually experiences in a mix. Previous work *(Hajj et al. 2017)* indicated that this test can identify large differences in binder strength for binders assigned similar PG grades.
The low temperature creep response of binders is tested using the bending beam rheometer (BBR). Using this the stiffness (S) and rate of relaxation (m-value) of the asphalt binders at low temperatures (typically -6°C to -18°C) are obtained.

Other than rheological techniques, microscopic and chemical techniques have also been reported to evaluate the aging related behaviors of asphalt binders. Atomic force microscopy (AFM) has been used to study the topography and multi-phase characteristics of binders. Previous researches have been conducted to evaluate the how aging may affect the unique morphological features seen in binders. Many studies have reported the existence of ellipse-shaped structures with height indentations and commonly referred to as “bee structures” *(Loeber et al. 1996*). The two-phase morphology can be seen wherein domain bee structures are dispersed in a continuous matrix phase. Earlier studies attributed these structures to the asphaltene content, which increases with the onset of ageing, and correlated the changes to this *(Pauli et al. 2001)*. However, subsequent studies with the development of technology have indicated the structures observed could be caused by surface wrinkling as a result of the buckling of the bee laminate due to variations in phase stiffness with the continuous phase *(Lyne et al. 2013)*. So far there are no universally accepted conclusions on the origin of these structures and research efforts continue to identify the exact nature of these elusive entities

In terms of chemical techniques, the most commonly reported techniques include FTIR spectroscopy, gel permeation chromatography (GPC) and chromatographic based separation. The study of the natural oxidative ageing exposed by bituminous binders has been the prime focus of attention for asphalt researchers using FTIR. The variations in this level of oxidation have been used to correlate changes in the rheological and chemical properties of mixes in the past *(Ding et al. 2016)*. In asphalt binder chemistry, the carbonyl band (C=O) exhibited at around 1700 cm^{-1} and the sulphoxide band (S=O) exhibited around 1000 cm^{-1} of a bitumen spectrum are the major functional groups used to gauge the level of oxidation. However, the carbonyl band has been more commonly used and known to better correlate the level of long-term ageing *(Bowers et al. 2014)*. Binders extracted from aged materials exhibit significantly higher levels of C=O bonds due to the natural oxidation of asphalt binder during the producing process and service period on pavement whereas virgin binders exhibit little or no C=O bond at this wavelength. This difference in oxidation can be utilized to semi quantitatively estimate the extent of aging in binders *(Ding et al. 2016)*. GPC analysis has also been utilized in asphalt research in the past and found to be suitable to correlate ageing behavior of binders. GPC is based on size exclusion and differently sized molecules are separated on the basis of size. When a material with a combination of different molecules such as asphalt binder (dissolved in solvent) is applied to the top of the column, the smaller molecules are dispersed through a greater volume of gel than is accessible to the large molecules. Hence, the large molecules move more swiftly through the column and the mixture can be fractionated into different components. These components are usually clubbed into different size ranges based on a distinct series of sizes, such as large molecular sizes (LMS), small molecular size (SMS) and medium molecular size (MMS). Importantly, a nearly linear relationship exists between the percentage of LMS and levels of oxidation in binders *(Zhao et al. 2013)*. The LMS percentages obtained from the tests have also been successfully correlated with the extent of ageing and rheology by various studies in the past *(Kim et al. 2006)*. The chromatography-based tests are mainly based on the polarity-based constitution of binder and separation into generic fractions: saturates, aromatics, resins and asphaltenes, commonly referred to as “SARA” fractions. The aging of binders generally increases the proportion of asphaltenes and resins while saturates remain relatively unchanged. These polarity-based fractions of asphalt binder have been reported to have a strong connection with oxidation chemistry and is of significance in the study of aging and recycling of binders (*Sreeram and Leng, 2019*). However, there are constraints associated with such methods for fractionation that make it difficult to apply such techniques to understanding aging characteristics for a large and diverse set of binders. These constraints include difficult operation, long testing time, and large solvent consumption.

Influence of Mix Design Variables on Aging of Asphalt Mixtures

Several variables influence the aging and performance of asphalt mixtures including mix type, binder source, performance grade of the binder, aggregate source, aggregate absorption, aggregate gradation, and recycled material and its content within the mixture.

Elliot et al. (*1991*) studied the effects of gradation on the creep stiffness, split tensile strength, resilient modulus and air voids of asphalt mixtures. They concluded that the variations in gradation had the greatest effect, especially when the shape of the gradation curve changed. They indicated that the tensile strength was influenced more by the air void content and compaction than the variation in gradation. Abo-Quadis et al. (*2007*) tested different limits of ASTM specifications for aggregate gradation. The open-graded gradation had the least amount of stripping resistance mostly due to the amount of air voids in the mix as the interlocking of the particles was poor. Guler (*2008*) found that the gradation was the most influential design parameter for the mechanical properties (elastic modulus and yield stress) of HMA.

The type of aggregates utilized also affects the behavior of the mixture as different aggregates have different absorption levels. Abo-Quadis et al. (*2007*) found that unconditioned hot mix asphalt mixtures using limestone had better stripping resistance as opposed to basalt aggregates. However, they also found that when those hot mix asphalt samples were conditioned, the basalt mix showed better resistance than that of limestone. Braham et al. (*2007*) compared the performance of limestone and granite aggregates in HMA. They indicated that although granite provided higher fracture energy at low temperatures than limestone, a reverse trend was seen at higher temperatures. Benedetto et al. (*2014*) concluded that using basalt compared to limestone in the HMA with 20% RAP, yielded greater complex modulus and higher fatigue resistance.

Clyne et al. (*2008*) evaluated the effect of RAP in asphalt mixtures by varying the proportion and source of RAP in the mix. They determined that mixtures containing RAP had higher dynamic moduli compared to the mixes without RAP. Using the semicircular bend (SCB) test, they found that mixes containing 20% RAP demonstrated a higher fracture resistance at high and low temperatures compared to the mixes containing 40% RAP. Using single-sourced RAP and multi-sourced RAP had no effect on the dynamic moduli at low temperatures; however, they affected the dynamic moduli at high temperatures.

PG of binder used for AC affects the way in which asphalt mixtures react in different environmental conditions, as well as in the mix design in general. Abo-Quadis et al. (*2007*) found that higher adhesion was achieved when using 80/100 asphalt compared to 60/70 asphalt for HMA. Guler (*2008*) reported that the asphalt content of a HMA pavement was the second most critical variable in the design process when testing yield stress of specimens after compaction. Varying PG also affect dynamic modulus. Clyne et al. (*2008*), concluded that mixture with softer binder had higher dynamic moduli compared to stiffer binder at low temperatures. However, it was also found that stiffer binder resulted in higher dynamic moduli for mixes with and without RAP at high temperatures. Boriack et al. (*2014*) investigated the optimum binder content of an asphalt mixture with RAP. They found that adding 0.5% of binder to the mixtures containing 20% RAP, improved the fatigue and rutting resistance but slightly decreased the stiffness. Moreover, they concluded that adding different quantities of binder to mixes with 40% RAP led to a decrease in both rutting and fatigue resistance. They also observed that the number of gyrations had to be adjusted in order to obtain the same air void targets, and the resistance to moisture damage of HMA with high RAP content (as high as 50%) dealt more with the compatibility of PG of binder used with the RAP rather than the percentage of RAP in the mix. Benedetto et al. (*2014*) arrived to a similar conclusion that varying proportions of binder added to HMA mixtures with RAP, could potentially increase or decrease complex modulus and fatigue resistance depending on the performance grade of the binder used.

Assessment of TxDOT Current Practices and Specifications

The current TxDOT design, production and construction specifications are described in Items 340 through 346A for all mix types. Current TxDOT Test Procedure Tex-204-F provides general guidelines and steps to select the materials, determine the material proportions (e.g., aggregates and binder content) and evaluate the engineering properties (e.g., cracking and rutting potentials) of the asphalt mix design. TxDOT Test Procedure Tex-241-F is used to produce compacted specimens for performance tests. Table 2.4 illustrates the mixing temperatures based on the performance grade and type for lab-mixed specimens. Lab-mixed materials are prepared as per Test Procedure Tex-205-F for compaction, whereas plant-produced mixtures are handled following Test Procedure Tex-222-F.

Table 2.4. Mixing Temperatures by Grade and Type *(Tex-205-F, 2016)*

Type-Grade[1]	Asphalt Material Temp.	Mixing Temp.[2]
PG 70-28, PG 76-22	325ºF (163ºC)	325ºF (163ºC)
PG 64-28, PG 70-22	300ºF (149ºC)	300ºF (149ºC)
PG 64-22, PG 64-16	290ºF (143ºC)	290ºF (143ºC)
AC-3,5,10; PG 58-28, PG 58-22	275ºF (135ºC)	275ºF (135ºC)
RC-250	100ºF (38ºC)	165ºF (74ºC)
MC-250	100ºF (38ºC)	165ºF (74ºC)
MC-800	140ºF (60ºC)	190ºF (88ºC)
CMS-2	140ºF (60ºC)	235ºF (113ºC)
AES-300	140ºF (60ºC)	235ºF (113ºC)
Asphalt Rubber (A-R) Binder	325ºF (163ºC)	325ºF (163ºC)

1 *If using RAP or RAS and a substitute PG binder in lieu of the PG binder originally specified on the plans, defer to the originally specified binder grade when selecting the mixing temperature.*
2 *When using RAP or RAS, mixing temperature may be increased up to 325ºF to achieve adequate coating.*

Before compaction, the mixtures must be conditioned at a specified temperature and time. Table 2.5 presents the curing and compaction temperatures for asphalt mixtures. While the temperature is selected based on the asphalt binder performance grade (PG), a curing time of 2 hrs ± 5 min is specified for both lab-produced and plant-produced mixtures. On the other hand, WMA mixtures are cured for 4 hrs at a lower temperature (usually ranging from 215ºF and 275ºF). For PMLC that require reheating, a curing period of 1.5 hrs ± 5 min is recommended. The reheated materials should be mixed and split into specific specimen sample sizes where they are cured to reach their specified compaction temperature. As the use of RAP and RAS materials continues to increase, modifications to current procedures and protocols are being made to assure the proper short-term and long-term performance of the pavement is achieved.

Table 2.5. Curing and Compaction Temperatures *(Tex-241-F, 2019)*

Binder[1]	Temperature[2]
PG 58-28	250ºF (121ºC)
PG 64-22	250ºF (121ºC)
PG 64-28	275ºF (135ºC)
PG 70-22	275ºF (135ºC)
PG 70-28	300ºF (149ºC)
PG 76-22	300ºF (149ºC)
PG 76-28	300ºF (149ºC)
Asphalt Rubber (A-R) Binder	300ºF (149ºC)

Note: *Mixtures must be compacted at the selected compaction temperature within tolerance of ± 5ºF (±3ºC)*
1 *If using RAP or RAS and a substitute PG binder in lieu of the PG binder originally specified on the plans, defer to the originally specified binder grade when selecting the mixing temperature.*
2 *When using RAP or RAS, mixing temperature may be increased up to 325ºF to achieve adequate coating.*

Performance test are conducted on specimens that are short-term oven aged regardless of the engineering property of interest, cracking susceptibility, or rutting resistance. While the aging induced through the STOA protocol can positively influence the cracking resistance of a mixture, the rutting potential of an asphalt mixture were impacted due to the stiffening behavior of the mix. Therefore, laboratory-curing protocols must be developed and implemented to determine accurately the early-age rutting and long-term cracking potentials of asphalt mixtures. For the cracking susceptibility, the change in engineering properties after long-term performance is of great interest to avoid durability problems in the field during the service life of the pavement. The curing time and temperatures for assessing the rutting potential of an asphalt mixture must be representative and valid to prevent early age rutting right after placement activities.

The temperature and time specified for conditioning are assumed to simulate partially the aging and binder absorption that happens during the production and compaction of an asphalt mixture. During the design process, TxDOT does not require measuring the performance of asphalt mixes after being subjected to long-term aging. The current practice of conducting performance tests on specimens after short-term aging conditions implicitly assumes that the relative performance of asphalt mixtures does not change after long-term aging. Although mixtures that exhibit acceptable engineering properties after short-term aging may still underperform in service, it is impractical to vary laboratory-curing conditions many times before producing specimens that closely simulate field behavior of mixtures. Implementing optimized laboratory curing protocols in terms of long-term cracking and early age rutting is paramount to determine the engineering properties of underperforming mixtures during mix design process.

State of the Practice of DOTs for STOA and LTOA Curing Specifications

A number of DOTs, including Wyoming, Florida, Nebraska, Indiana and Virginia, currently follow the proposed STOA and LTOA curing as per AASHTO R 30. The key differences among various agencies are the mixing and compaction temperature ranges for aging of the asphalt mixtures. The performance grade of the binder is the controlling factor for each temperature, with special considerations for using modified binders. Various other state DOTs around the United States have specifications to simulate STOA as well as LTOA that differ from AASHTO R 30. The DOTs resources, environmental and loading conditions heavily influence their specifications.

Illinois DOT (IDOT)

The Illinois Department of Transportation has commissioned several studies aimed at verifying and modifying the current AASHTO standards. Their modifications to AASHTO R 30 include the short-term and long-term aging conditioning requirements for their volumetric mixture design. To assess the impact of short-term aging on performance, they require the Hamburg Wheel Tracking (HWT), Illinois Flexible Index (I-FIT, AASHTO TP-124), indirect tensile strength, and tensile strength ratio (TSR, AASHTO T-283) results. The effect of long-term aging is assessed by conducting I-FIT test. IDOT performance tests are only applicable to laboratory-prepared loose mixtures. Plant-produced mixtures are only evaluated for quality control (QC) or quality assurance (QA) purposes.

Table 2.6 illustrates the time required for curing of short-term aging for both HMA and WMA for IDOT's selected performance tests. The aging process can take place either immediately after mixing but before compaction or after the mixture has cooled to room temperature. The curing period is influenced by the absorption of the aggregates. Low-absorptive aggregates are cured for 1 hr ± 5 min prior to compaction. High-absorptive aggregate with a combined absorption greater than 2.5% are cured for 2 hrs ± 5 min. The conditioning time prior to mixing is not considered as time of mixing. IDOT specifications recommends compaction temperatures based on the performance grade of the binder identical to those temperatures provided in AASHTO R 30. The replicate I-FIT specimens are long-term aged for 3 days ± 1 hr at 200ºF ± 5ºF (95ºC ± 3ºC). The long-term aged specimens are cooled to room temperature, submerged into a water bath set at 77ºF ± 1ºF (25ºC ± 0.5ºC) for two hrs and tested in accordance with AASHTO TP 124.

Table 2.6. Short-Term Conditioning Requirements *(IDOT Central Bureau of Materials, 2019)*

Lab or Plant Produced Mix	Lab	Lab	Lab	Plant	Plant	Plant
Test Type	Volumetric	T 283	Hamburg / I-FIT	Volumetric	T 283	Hamburg / I-FIT
HMA	1 or 2 hrs	1 or 2	1 or 2	0	0	0
WMA	1 or 2	1 or 2	3 or 4	0	0	2

***1** When two different values are present within a single cell, the correct value is based on whether low or high absorption aggregates are used.*

Minnesota DOT (MnDOT)

The Minnesota Department of Transportation has funded several studies in an effort to address the environmental aging of asphalt mixtures. Some of those studies have focused on binder oxidation by using tests such as the PAV, RTFO, BBR and the DSR. MnDOT has modified AASHTO R 30 for STOA and LTOA of their asphalt mixtures. The mixing and compaction temperatures for the preparation of an asphalt mixture is based on the PG of the binder. MnDOT follows 4 hrs of conditioning to achieve short-term curing for LMLC specimens. MnDOT's curing temperature is specified at 290ºF ± 10ºF (143ºC ± 6ºC) to simulate the pre-compaction phase in the construction process. This curing temperature allows for proper absorption of the binder into the aggregates. For modified binders the minimum compaction temperature should be 290ºF. However, a higher compaction temperature should be used if included in the work.

Colorado DOT (CDOT)

The Colorado Department of Transportation follows CP-L 5112 and CP-L 5115, which are the procedures for performance testing with the HWT Test and preparing specimens with a Superpave gyratory compactor, respectively. Different sample preparation processes are provided for the LMLC and PMLC mixes. As shown in Table 2.7, CDOT recommends similar mixing and compaction temperatures as TxDOT. The performance grade of the binder is the controlling factor for the mixing and compaction temperature of the superpave mixture(s).

Table 2.7. Laboratory Mixing and Compaction Temperatures *(CP-L 5115, 2016)*

Superpave Binder Grade	Lab Mixing Temperature	Lab Compaction Temperature
PG 58-28	310ºF (154ºC)	280ºF (138ºC)
PG 58-34	310ºF (154ºC)	280ºF (138ºC)
PG 64-22	325ºF (163ºC)	300ºF (149ºC)
PG 64-28	325ºF (163ºC)	300ºF (149ºC)
PG 70-28	325ºF (163ºC)	300ºF (149ºC)
PG 76-28	325ºF (163ºC)	300ºF (149ºC)

Note: All Temperature in this table have a tolerance of ± 5ºF (± 3ºC)

Once material for laboratory-produced specimens has been mixed, the mixture must undergo short-term aging in an oven at its respective compaction temperature for 2 hrs. CDOT recommends an increase in duration if it is known that the mixture in the field were exposed to higher temperatures. PMLC mixtures on the other hand follow a 3 hrs ± 0.5 hr of reheating at its respective compaction temperature. A noticeable difference between CDOT and other DOTs is that they do not require stirring the mixture during the curing process to maintain uniform conditioning.

California DOT (CALTRANS)

The California Department of Transportation follows their Test Procedure 304, for short-term aging of mixes. CALTRANS, similar to TxDOT follow a range of mixing and compaction temperatures. However, CALTRANS allows the material to be kept in the oven until it reaches a workable temperature. Prior to compaction, the material must be mixed thoroughly at its respective temperature to allow for even conditioning. Typically, the material is oven cured for 2 hrs prior to mixing at a specified compaction temperature depending on the PG of the binder. They recommend short-term curing of 2 to 3 hrs at 295ºF ± 3ºF (146ºC ± 1.5ºC) for laboratory mixed specimens. Since plant mixes have endured the mixing process of short-term aging, thus 2 to 3 hrs of curing prior to compaction is required. The reheating of PMLC mixes should not exceed 3 hrs at a compaction temperature of 235ºF ± 3ºF (113ºC ± 1.5ºC). A compaction temperature of 305ºF ± 3ºF (152ºC ± 1.5ºC) is recommended for mixes with asphalt rubber binders.

Summary

This chapter presented a comprehensive documentation of current practices and specifications on previous and existing research projects. This comprehensive literature review was used to evaluate the most promising aging protocols. In addition, the information was used to formulate a two-tier approach for experimental design plan.

Chapter 3. Preliminary Assessment of Existing Laboratory Aging Protocols on Asphalt Performance Using Available Data

This chapter consists of a preliminary assessment of the impact of aging on the performance of asphalt mixture and binders using existing data. Relevant data from current and past TxDOT projects were used to quantify the impact of short- and long-term aging on the performance of AC pavements. For that purpose, relevant data were extracted from a comprehensive data repository, known as the Data Storage System (DSS), developed under TxDOT Research Project: 0-6658 "Collection of Materials and Performance Data for Texas Flexible Pavements and Overlays.

Preliminary Assessment of Data Storage System (DSS)

TxDOT Research Project 0-6658, a collaboration between The University of Texas at El Paso (UTEP) and Texas A&M Transportation Institute (TTI), resulted in a DSS that includes extensive pavement material properties and performance data for over 115 test sections located throughout Texas. Collected laboratory, field and performance data, traffic, and section details have been compiled and stored in the DSS. The DSS include new construction, overlay, rehabilitation and seal coats sections. Since the initial stage of construction, placed materials (hot mix asphalt, binder, base, subbase and subgrade) were collected and subjected to extensive laboratory testing and material characterization. These sections have been closely monitored (many continue to be monitored) for field performance and distress progression under in-service traffic conditions. The central purpose of assembling the DSS was to leverage the comprehensive data toward calibrating and validating mechanistic-empirical (M-E) design models. Such models include Flexible Pavement Design System (FPS), Texas M-E, Texas Overlay design system, and the AASHTO Pavement ME.

Figure 3.1 displays the Microsoft Access® DSS tool from TxDOT Project 0-6658. That relational database possesses a plethora of information for the test sections, including the test section details, construction and QA/QC data, and material testing data for the hot mix asphalt (HMA), binder, base/base, and subgrade. Field testing and time-based distress history is also documented. Traffic and climate information is also included in the repository. The DSS tool is updated semi-annually through performance monitoring, adding field testing data, photographs/video, field core information and visual distress survey information. The DSS tool can presently be used as a state-level database for pavement performance diagnostics. The comprehensiveness of the DSS allows for investigating short and long-term performance of pavement layers based on the laboratory and field-derived material properties, while also considering the routine monitoring for damage and the documented inventory of pavement system characteristics.

Figure 3.2 displays the distribution of test sections across Texas, as well as the number of test sections within each district. The selection of each section was based on the district location, climate zone, pavement structure configuration, and construction type (e.g., new construction, overlay, etc.). The DSS tool is also accompanied with global positioning system (GPS) coordinates and subsequent section mapping.

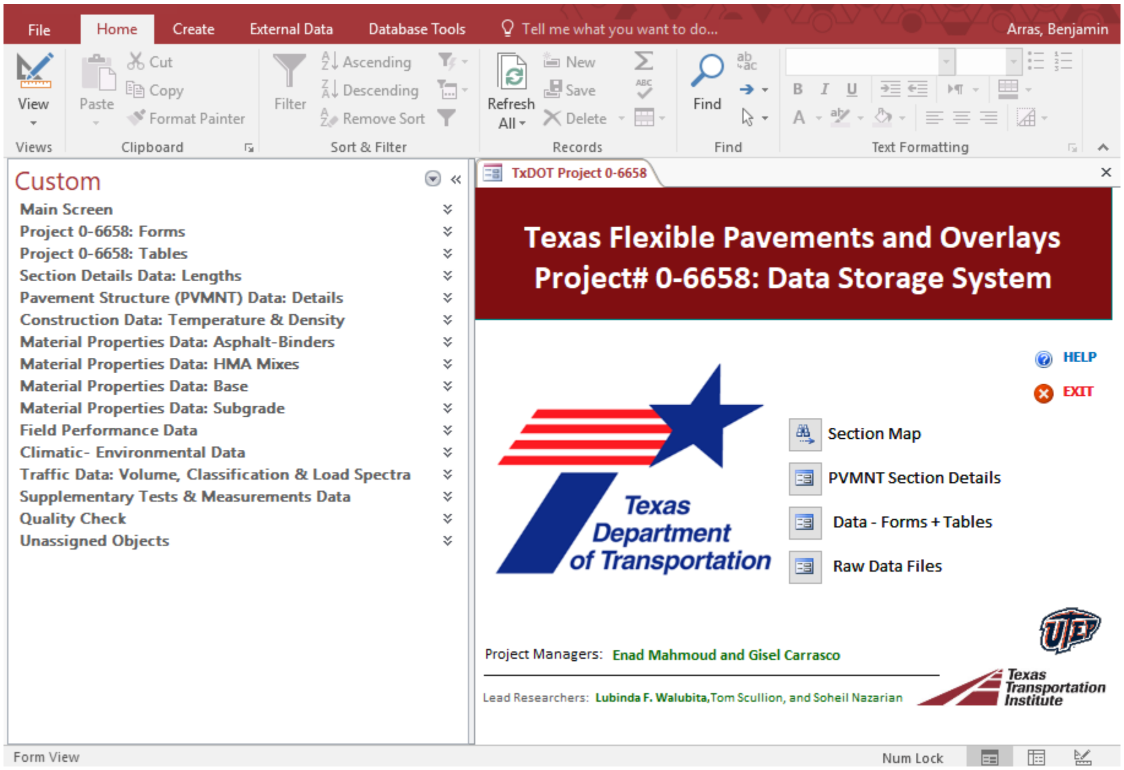


Figure 3.1. Database Storage System Graphical User Interface from TxDOT 0-6658

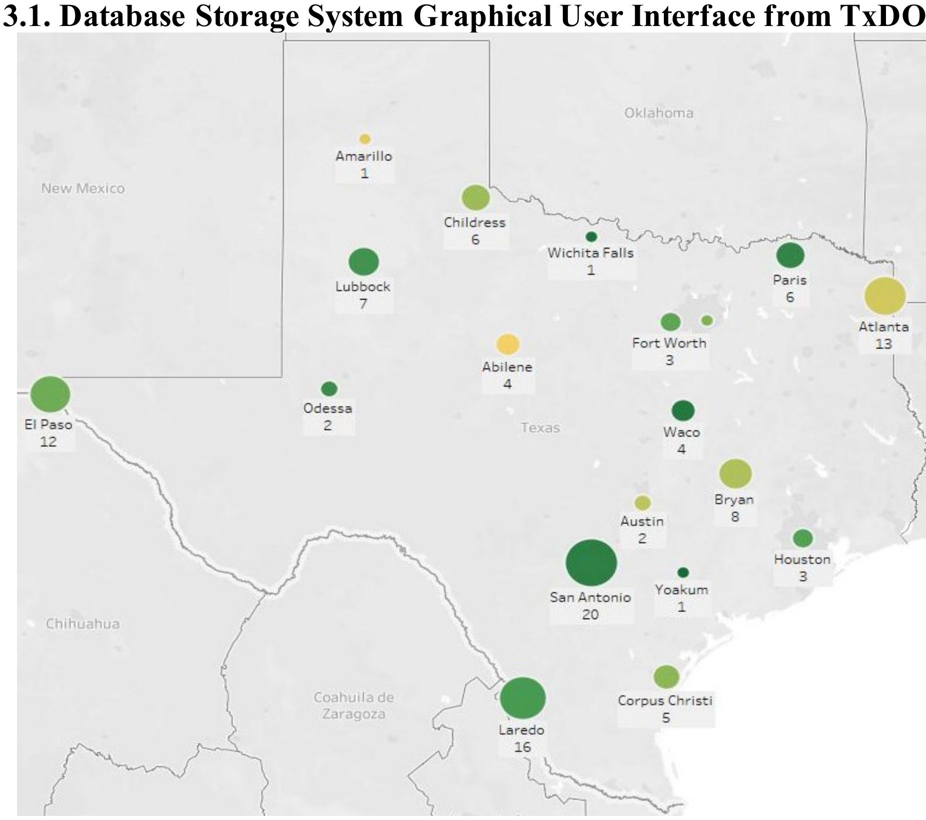


Figure 3.2. Test Locations in Texas from TxDOT 0-6658

Figure 3.3 displays a schematic exhibiting the information stored for each test section in the DSS. The database contains various HMA and binder parameters of the AC layer(s) of each test section.

The main relevant parameters to the scope of work of this study include: surface course mix type, job mix formula (JMF), RAP and RAS information, aggregate type, asphalt content, additives information, and the short and long-term field performance (i.e., with aging). The binder testing data includes results from the dynamic shear rheometer (DSR), bending beam rheometer (BBR), multi-stress creep recovery (MSCR), rolling thin-film oven (RTFO), specific gravity, viscosity and PG of the binder. The laboratory performance data include Hamburg wheel-tracking (HWT), overlay tester (OT), indirect tension (IDT), dynamic modulus (DM) and the repeated load permanent deformation (RLPD) tests. The database also includes nondestructive testing (NDT) data from the falling weight deflectometer (FWD) and the ground penetrating radar (GPR).

Figure 3.3. DSS Information based from Research Project TxDOT 0-6658

Figure 3.3. DSS Information based from Research Project TxDOT 0-6658

Characteristics of AC Mixtures

The main focus of this subtask was to analyze DSS information to assess the impact that field aging had on the performance and service life of the test sections. Table 3.1 displays the characterization of the test sections in the DSS by HMA item, surface course mix type, and sample size. The largest sample size was observed for dense graded mixtures (Item 341). The next largest sample size was for Stone Matrix Asphalt (SMA, Item 346). Surface courses comprising of Permeable Friction Course (PFC, Item 342), Performance Design mixes (Item 344), and Thin Overlay Mixes are also present for analysis.

Table 3.1. Hot Mix Asphalt Mixture Items and Types within TxDOT 0-6658 DSS

HMA Mix Group	HMA Mix Type	Total No. Mixes
341- Dense Graded HMA	Type B, Type C, Type D and Type F	76 Mixes
342- Permeable Friction Course	PFC	7 Mixes
344- Performance Design	SP-C and SP-D	6 Mixes
346- Stone Matrix Asphalt	SMA	14 Mixes
Thin Overlay Mixes	TOM	4 Mixes

The lowest asphalt contents (approximately 5%) were observed on the dense graded mixes. The highest asphalt contents (roughly 6.7%) were observed for the permeable friction coarse mixes. The Superpave (SP) and SMAs exhibited average asphalt contents of 5.5% and 6.2%, respectively. For the UTEP sections, the RAP and RAS content of the different HMA mixtures ranged from 0% to 23% for RAP and 0 to 5% for RAS content.

Figure 3.4 displays the sample size distributions of the test sections considering the facility type. The test sections within the DSS represent an array of facility types; consisting of U.S. highways, interstate highways (IH), farm-to-market roads (FM), state highways (SH), loops (LP) and spurs (SP). This bar chart is further delineated by those number of test sections managed by UTEP and those by TTI. The majority of the test sections within the DSS are on U.S. highways. Similar sample sizes are observed for test sections on IH, SH and FM roads.

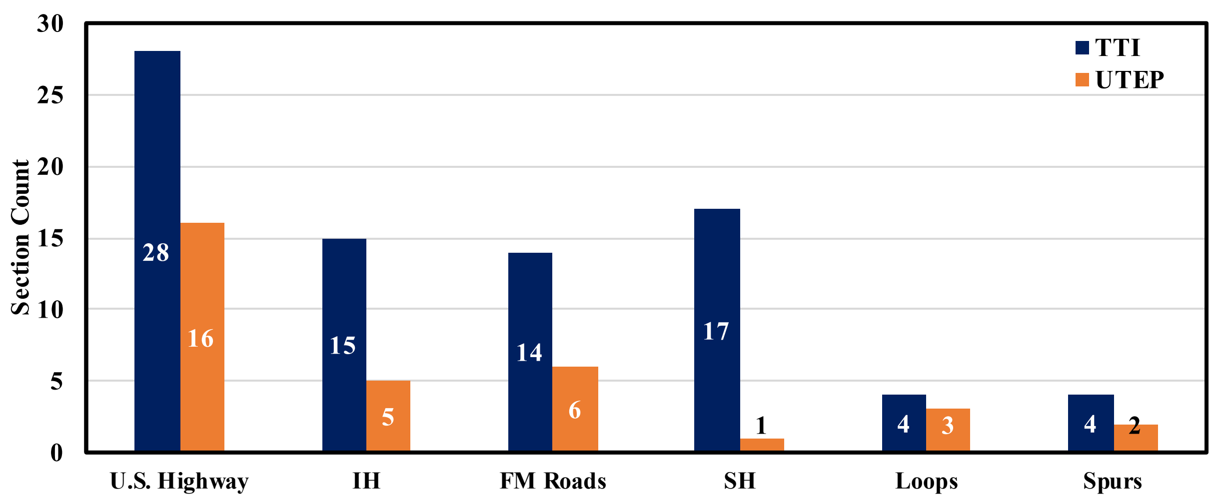


Figure 3.4. Section Distribution by Roadway Type from TxDOT 0-6658

Typical Performance and Aging Conditions of AC Layers and Pavements in Texas

The DSS tool was used to analyze performance of the field-aged AC mixtures. A preliminary data analysis using the DSS rut depths from the Hamburg Wheel Tracking Test (HWTT) and the number of cycles to failure from the Overlay Test (OT) were evaluated to estimate the performance of AC mixtures. For the sections that are monitored by UTEP, the raw data were reanalyzed to determine the crack progression rate (CPR) and the critical fracture energy (CFE) for each mix. CPR represents more rationally the rate of crack propagation and CFE relates to the measures of the crack initiation. The CPR and CFE have been shown to represent HMA cracking potential with much lower coefficient of variation compared to the number of cycles (Garcia et al., *2016*).

The *short-term aging*, which should represent the cumulative effects of aging due to plant mixing, storing, as well as transporting and placing the mix in the field, typically positively impacts the rutting performance of a mix. Figure 3.5 displays the average rutting resistance indices (RRI) from 17 pavement sections (monitored by UTEP) from laboratory-mixed laboratory-compacted (LMLC) specimens. The bar chart illustrates the average rut depth of dense graded (Type B and C), stone matrix asphalt (SMA), coarse matrix high binder (CMHB) and thin overlay mixes (TOM) while considering the different PG. Preliminarily, the SMA and TOM mixes performed well against rutting. A comprehensive study that incorporates more pavement sections were performed during the initial evaluation to establish preliminary threshold values for short-term aging or rutting.

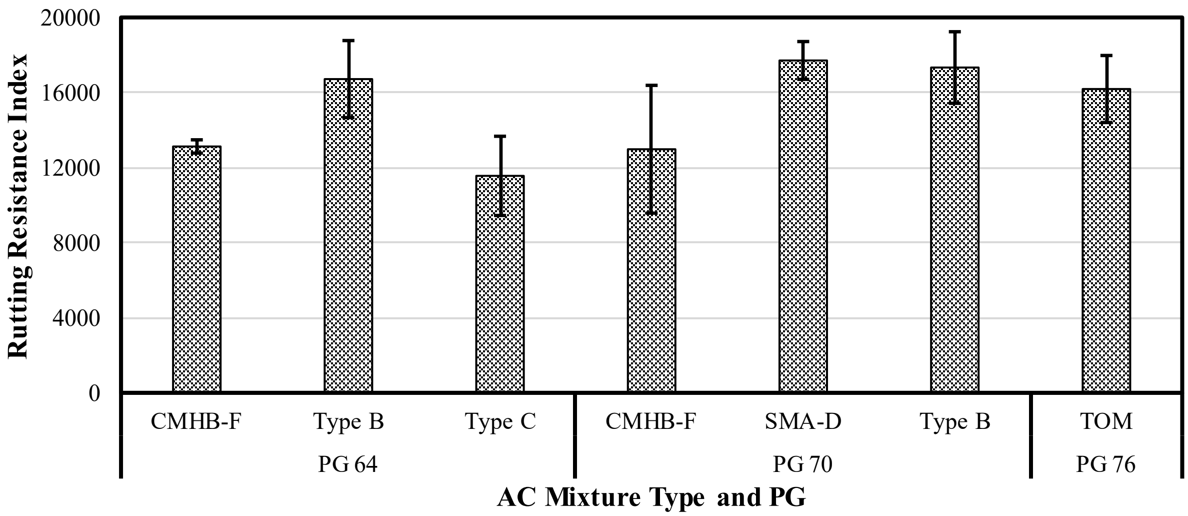


Figure 3.5. Hamburg Wheel-Tracking Test Comparison of AC Mixtures

The interaction plot shown in Figure 3.6, which is a cross plot of CPR and CFE, introduces the concept of how the performance of the AC mixtures with respect to cracking are assessed. The *acceptance limit for CPR* is provisionally set at a value of 0.45 (*SS 3074, 2019*). "Good" performing mixtures have showed to have a CPR value lower than the acceptance limit, while a "poor" performing mix exhibit values higher than the acceptance limit. CFE that assesses the resistance of the mixture to crack initiation during the first loading cycle has a provisional upper limit of 3 and a lower limit of 1.

Figure 3.6 illustrates examples of two pavement sections with "good" and "poor" crack performing AC mixtures. Based on the DSS, the FM pavement section does not exhibit significant cracking under a moderate traffic volume. The hollow circle that represents the initial evaluation of the mix exhibits a CPR of 0.34 (satisfactory performance). The shaded circle represents CPR from a field core 44-months post-mat placement. This data point shows a CPR of 0.42; still indicating a "good" performing mixture in terms of cracking. Figure 6 also shows the results from a pavement section located along a state highway that exhibited poor cracking performance in the field under low traffic volume. The hollow triangle that represents the initial evaluation yields a CPR of 0.68. This mix would not have been accepted, if cracking criterion had been incorporated in the mixture design process. The solid triangle, with a CPR of 1.47, represents the CPR obtained on a core extracted 58 months post-mat placement. Overall, as the pavement ages the cracking performance deteriorates.

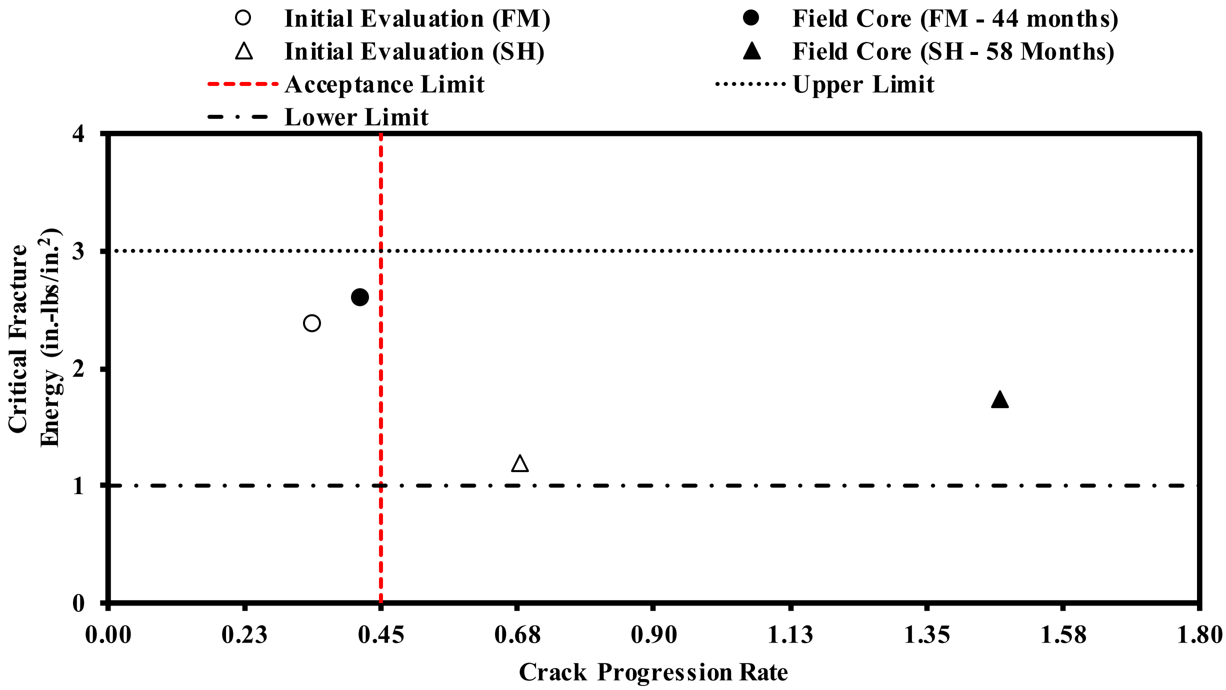


Figure 3.6. Interaction Diagram Plot for Cracking Performance

An evaluation was performed using data from the 17 sections from the DSS summarized in Table 2 to gain insight in the cracking performance of the AC mixtures as they aged. Based on the visual distress surveys three classes, namely, “good,” “marginal” and “poor,” were created to delineate the pavement performance with time. At that time, six of the sections were categorized as “good,” five as “marginal” and six as “poor.” Figure 3.7a shows the initial laboratory cracking performance of the pavement sections based on the OT data. Seven sections failed the OT cracking performance criteria, whereas the remaining ten sections were within the acceptance limits. The sections that were categorized as crack susceptible were the same sections that showed “marginal” and “poor” field performance as well. Hence, the field performance and initial OT test results display the effectiveness of the design interaction plot to predict the cracking potential of AC mixes.

To evaluate the effect of aging over time, two sets of field cores were extracted and assessed for their respective CPR values at different ages. Figure 3.7b shows the cracking performance of the first set of cores that were extracted 2-3 years after construction. Most of the initial “marginal” and “poor” sections exhibited higher CPR values (as compared to the initial results), indicating that they became more susceptible to cracking. Figure 3.7c show the cracking performance from some of the second sets of cores. These cores exhibited even higher CPR values, which reinforce that as AC mixtures age they become more brittle and prone to cracking. A tendency of increasing CPR can be concluded as most of the initial “marginal” and “poor” sections have gone beyond the acceptance limit.

Figure 3.8 illustrates how the CPR varies with time given different environmental conditions and pavement types. The numbers above each bar corresponds to the age of the pavement at the time of testing. A general trend of increase in CPR values with time can be observed for all pavement sections. In addition, the eight sections where a second sets of cores were extracted exhibited even higher CPR values as compared to the first set of cores.

Table 3.2. Field Section Details for Cracking Performance Analysis from TxDOT 0-6658

District	ID	Material and Design Information					Pavement Section Information			
		Mix Type	Binder Grade	Design AC, %	RAP , %	NMAS , mm	Constructed Year	First Core	Second Core	ADT
Lubbock	1	SMA-D	PG 70-28	6.3	20	12.5	2011	2014	-	3007
	2	SMA-D	PG 70-28	6.0	0	12.5	2011	2014	-	612
	3	SMA-D	PG 70-28	6.3	10	12.5	2011	2014	-	4837
	4	SMA-D	PG 70-28	6.3	0	12.5	2012	2015	-	2103
	5	SMA-D	PG 70-28	6.3	0	12.5	2011	2014	-	4600
	6	SMA-D	PG 70-28	6.3	20	12.5	2012	2015	-	337
El Paso	7	CMHB-F	PG 70-22	5.3	20	9.5	2013	2015	2020	579
	8	CMHB-F	PG 70-22	5.3	20	9.5	2013	2015	2020	372
	9	Type-C	PG 64-22	5.0	20	9.5	2013	2016	2020	343
	10	CMHB-F	PG 70-22	5.0	20	9.5	2013	2015	-	-
	11	Type-C	PG 64-22	5.0	20	19.0	2012	-	2020	3288
	12	Type-C	PG 64-22	4.6	20	19.0	2011	2015	2020	1545
	13	Type-C	PG 70-22	4.8	20	19.0	2011	2015	2020	4127
	14	Type-C	PG 70-22	4.8	20	19.0	2012	2016	-	4270
	15	TOM	PG 76-22	6.5	0	9.5	2012	2015	-	929
	16	TOM	PG 76-22	6.5	0	9.5	2013	2016	2020	3952
	17	TOM	PG 76-22	6.5	0	9.5	2013	2016	2020	2620

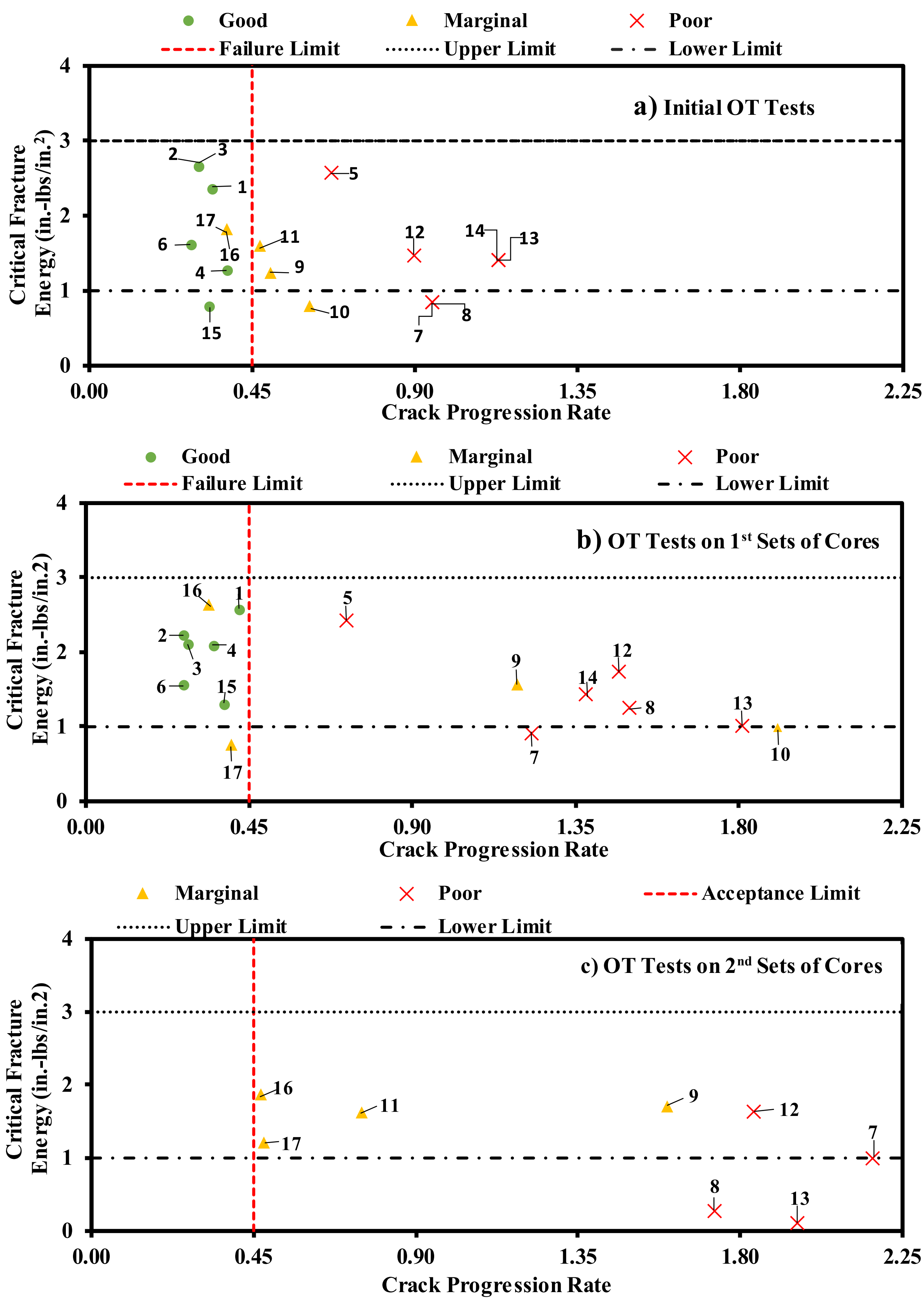


Figure 3.7. Mixture Cracking Performance Evaluation

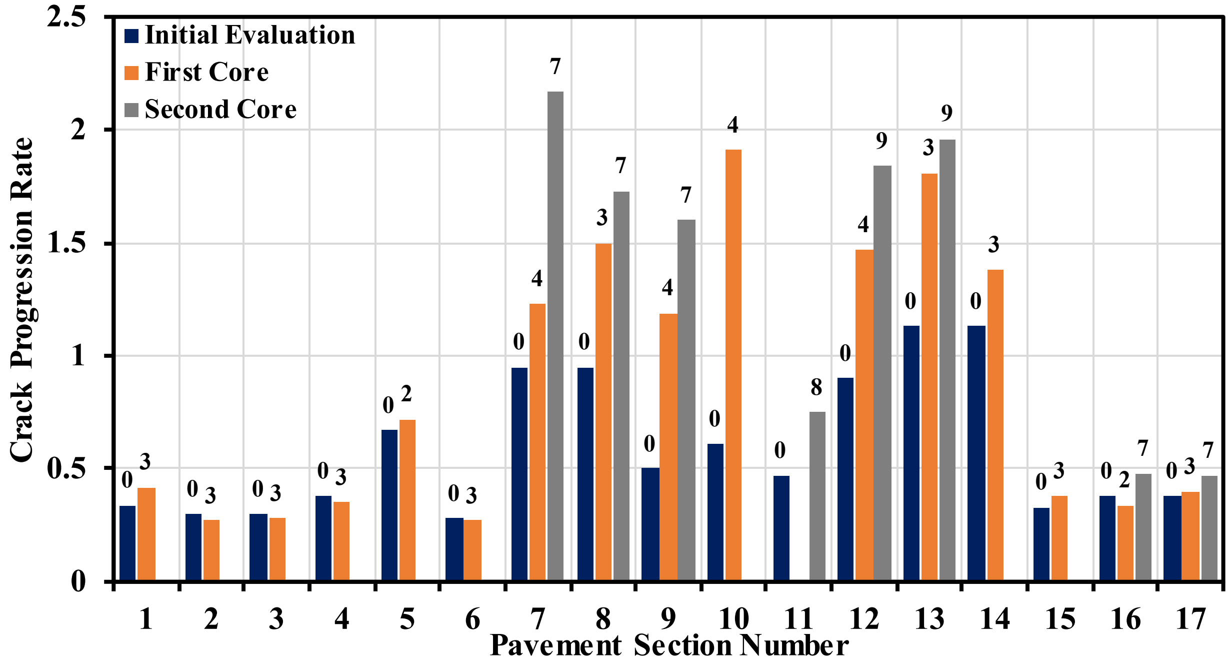


Figure 3.8. Mixture Cracking Performance Evaluation against Time

Influence of Short- and Long-Term Aging on Engineering Properties

Numerous studies have been conducted under the National Cooperative Highway Research Program (NCHRP) in-relation to the objective of this study. The following relevant research projects have been identified:

- NCHRP 09-36, *"Investigation of Short-Term Laboratory Aging of Neat and Modified Asphalt Binders."*
- NCHRP 09-52, *"Short-Term Laboratory Conditioning of Asphalt Mixtures."*
- NCHRP 09-54, *"Long-Term Aging of Asphalt Mixtures for Performance Testing and Prediction."*

Each of these reports have distinct objectives, but they include observations and information that can prove useful in preparation of the experimental design plan for this project. Table 3.3 highlights the different types of testing conducted in each study, as well as the aging conditions used for the specified tests and whether they were developed to simulate short- or long-term aging.

Table 3.3. Summary of Key Findings from NCHRP Studies Related to Aging

NCHRP Project No.	Test Type	Final Recommendations
09-36	Stirring Air Flow Test (SAFT) Modified German Rotating Flask (MGRF) Rotating Cylinder Aging Test (RCAT)	163°C for 45 minutes 165°C for 210 minutes 163°C for 235 minutes
09-52	Hamburg Wheel-Tracking Test (HWTT) Resilient Modulus	135°C and 116°C for 2 hours for HMA and WMA respectively
09-54	Dynamic Modulus (DM) Oven vs. Pressure Aging Cyclic Fatigue Test	95°C/135°C for 8.9 days/16.8 hrs 95°C and 135°C 85°C for 8 days

The following section aims to compare the results of the different tests used within each report and to focus on differences in aging and possible differences in various experimental factors that may impact replicability for future studies or other relevant procedures. Anderson and Bonaquist (*2012*) attempted to replace both Rolling Thin Film Oven Test (RTFOT) and the Pressure Aging Vessel (PAV) with a single device that would simulate short- and long-term aging. They examined the Stirring Air Flow Test (SAFT) and the Modified German Rotating Flask (MGRF) under varying operating conditions. They found MGRF to be an acceptable, less expensive replacement as they produced similar results with the same aging conditions. However, they also observed that MGRF averaged about 40% of the mass change that RTFOT caused for the specimens in their study. The SAFT showed little correlation to oven-aged mixtures and took twice the amount of time as the current PAV to show equivalent aging.

Newcomb et al. (*2015*) proposed changes to current AASHTO R 30 protocol and introduced the concept of cumulative degree-days as a metric to measure relative extent of field aging. Correlation between LMLC and PMPC specimens indicated that STOA protocols for this study were largely able to simulate plant aging and asphalt absorption that occurs during plant production. Critical in-service time when WMA equaled HMA was at 23,000 CDD; approximately equivalent to 17 months in service in warm climates, and 30 months in service in colder climates. Stiffness of WMA to equal initial stiffness of HMA was accomplished with field aging of 3,000 CDD; approximately equivalent to 2 months in warm climates and 3 months in colder climates.

Most recently, Kim et al. (*2018*) validated AASHTO R30 and proposed a standalone protocol for long-term aging. That study is vital as they report comparisons of the results among oven to pressure aging and loose mixtures to compacted specimens. The tests that included application of pressure in compacted specimen aging were found to expedite aging. The addition to aging of loose mixes also expedited oxidation but was found to be impractical as only 500g could be aged at a time. However, 7000-8000 g are needed for a super pave gyratory-compacted specimen. A clear difference between the fatigue performances of the asphalt mixture aged at 95°C to 135°C is presented. Long term aging at temperatures of 135°C should be avoided as they yield poor performance results.

Material aging

Identifying factors that contribute to the deterioration of the performance of asphalt mixtures is one of the more important objectives of this investigation. Yin et al. (*2017*) concluded that non-uniform field aging occurs to pavements in the field as the surface layer aged more rapidly when compared to the bottom. Azari and Mohseni (*2013*) studied the short- and long-term rutting performance of different AC mixtures. They concluded that the rutting performance is significantly affected by the mixture age and determined that different AC mixtures age differently. Radziszewski (*2007*) analyzed different components of AC mixtures and evaluated them on their relation to permanent deformation, which can be an indicator of aging through rutting and cracking. That study created three lists, each classifying the level of resistance to rutting and creeping after no aging, short-term aging, and long-term aging as shown in Table 3.4. Overall, it can be established that by judiciously selecting the pavement materials and mixture components will proactively result in high performing pavements.

Our research team performed a preliminary study to assess the short- and long-term aging cracking performance of varying AC mixtures across Texas using OT data[1]. The standard practices to

[1] This study was carried out as a preliminary investigation of aging cracking performance of mixtures.

simulate the field aging of an AC mixture was done by curing the loose-mixtures following (1) AASHTO R 30 for long-term oven aging (LTOA) and (2) Tex-206-F for short-term oven aging (STOA). The curing processes for LTOA comprised 5 days at 185ºF (85ºC). Curing for STOA is 2-hours at its respective compaction temperature (based on TxDOT standards). Table 3.5 displays the relevant mix design information of the different AC mix designs across Texas.

In accordance with AASHTO R 30, the loose mixture was cured at a maximum thickness of 2 in. to ensure even oven aging of the material. Figure 3.9 shows the CPR values obtained for LTOA and STOA specimens. A clear trend is observed where the LTOA materials exhibited higher CPR values as the material aged except for the three mixtures that showed marginal increase. The remaining nine sections had an increase in CPR ranging from 20% to 363% when compared to their respective STOA specimens. A new acceptance limit were considered and revised for future implementation considering that aging and the OT is sensitive to time. However, current acceptance limit of 0.45 for "good" mixtures was employed.

Table 3.4. Summary of Key Findings Related to Material Aging *(Radziszewski, 2007)*

Aging Condition	Most Resistance	Intermediate Resistance	Least Resistance
No aging	• Super pave • Porous asphalt mixture with 15% air voids	• Asphalt concrete with 17% of rubber modified binder • MNU with Modbit 30B binder • Asphalt concrete with 7% of plastomer modified binder	• Asphalt concrete with 3 and of plastomer modified binder • Asphalt concrete with 50/70 bitumen
Short-term aging	• SMA with 35/50 binder • Super pave	• MNU with Modbit 30B binder • Asphalt concrete with 5% of plastomer modified binder	• Asphalt concrete with 35/50 bitumen • Asphalt concrete with 50/70 bitumen
Long-term aging	• Asphalt concrete with 17% of rubber modified binder • SMA with 35/50 binders • Super pave	• Asphalt concrete with 5% of plastomer modified binder • MNU with 17% of rubber modified • Porous asphalt mixture with 20% content of air voids	• Asphalt concrete with 50/70 bitumen • Asphalt concrete with 3% of elastomer modified binder • Asphalt concrete with 35/50 bitumens

Table 3.5. Summary of Pavement Sections

Mix No.	District*	Mix Type	Original Binder	Asphalt Content (%)
1	Fort Worth	DG-C	70-28	4.6
2	Fort Worth	DG-C	70-28	4.6
3	Brownwood	SP-C	76-22	5.5
4	San Antonio	SP-C	76-22	5.0
5	Austin	DG-D	76-22	5.2
6	Fort Worth	DG-D	64-22	5.2
7	Fort Worth	DG-D	64-22	5.2
8	Atlanta	SP D	76-22	5.5
9	Corpus Christi	SP-D	70-22	5.3
10	Dallas	SP-D	70-22	5.3
11	Dallas	SP-D	64-22	5.4
12	Tyler	SP-D	70-22	5.4

**Districts that repeat have different mix designs (change in gradation/mix type/binder source).*

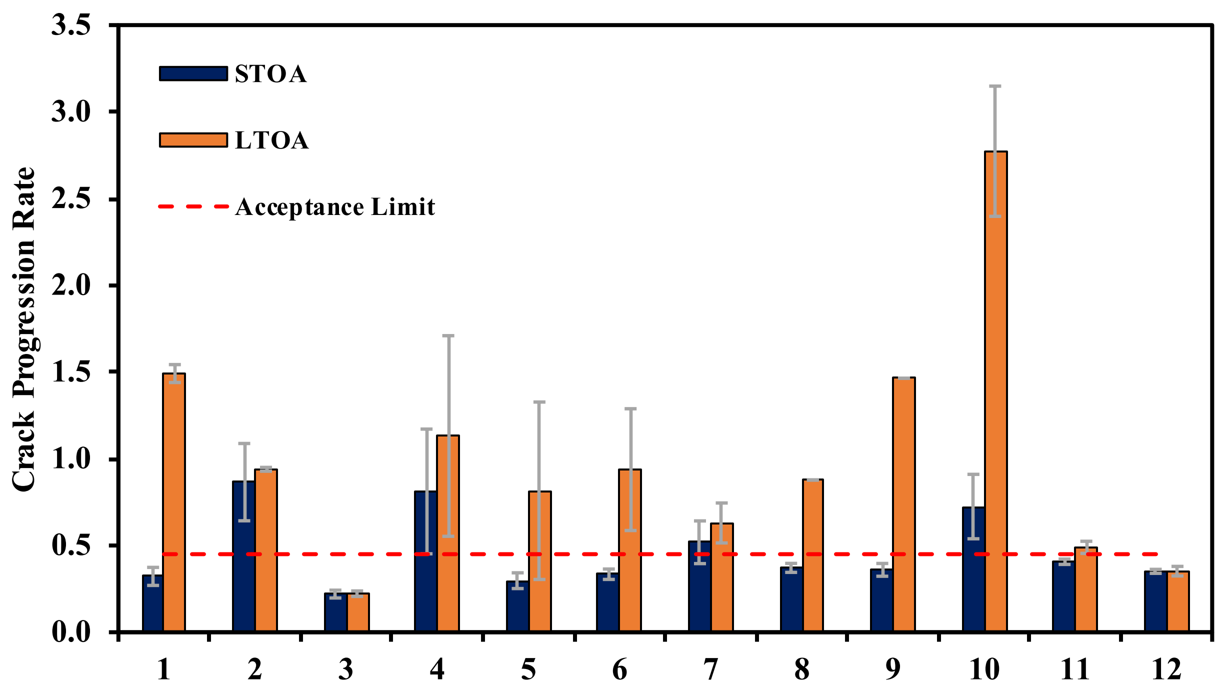


Figure 3.9. Crack Progression Rate Results for Aged Mixtures

Formulation of Laboratory Aging Protocol and Performance Indices

Existing AASHTO R 30 standard and NCHRP Project 9-54 attempt to simulate aging of the asphalt mixtures equivalent to a certain number of years in service. Given that the aging kinetics of binder reaches a steady-state condition after a certain amount of aging, it is hypothesized that there is a level of long-term aging after which the rank order of aging and consequently cracking characteristics of different binder-mixtures does not change. For a cracking test to distinguish accurately between crack-resistant and susceptible mixes, it is critical but adequate that mixes are aged to achieve this steady state rate even if the mixes do not reach a state that is equivalent to 10 or 20 years of field aging. Figure 3.10 illustrates an analysis of the aging kinetics for different binder-mixtures and their respective performance (e.g., cracking potential) rankings at different aging levels based on the steady-state aging condition of asphalt mixtures and binders. This project will utilize this concept to develop optimized laboratory aging protocols for mixtures.

Three main factors that dictate the aging rate of any given binder are:

- Temperature
- Duration
- Pressure

These factors were considered to select the best candidate aging protocols for further examination in the remainder of this study. In addition, an alternate method utilizing ozone gas was considered.

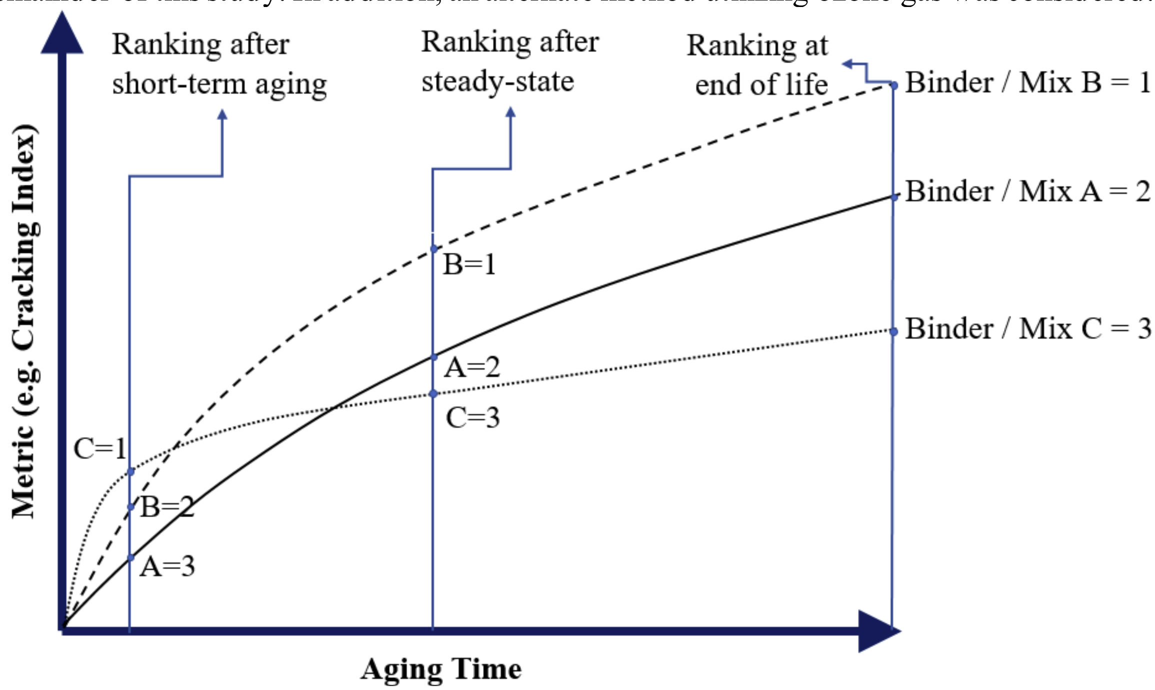


Figure 3.10. Representative Aging Kinetics of Asphalt Mixtures at Different Aging Levels

Temperature

AASHTO R 30 standard for traditional long-term aging conditions consist of curing compacted specimens at a temperature of 85ºC. NCHRP 09-54 recently compared the aging induced by oven-cured loose mixtures at 95ºC and 135ºC. They recommended a curing temperature of 95ºC for loose mixtures so that the chemical composition and integrity of the asphalt binder are not compromised. These studies clearly point out that for any aging procedure to be realistic, it is important that the procedure not utilize a temperature above 95ºC.

Duration

While AASHTO R 30 standard specifies a duration of five days for long-term oven aging, NCHRP Project 09-52 documented that the curing conditions from AASHTO R 30 approximately simulate the aging conditions of pavements with 11 and 22 months in service for warmer and colder climates, respectively. Based on the literature search, there is no consistent approximation for the long-term aging prediction between the laboratory aging protocols and field aging of asphalt concrete layers. NCHRP Project 09-54 recommended aging durations based on project-specific and climate-based determination. Although the laboratory aging conditions recommended may consistently predict the aging conditions of asphalt mixtures in the field, the practices from NCHRP Project 09-54 are not optimal for routine mix design applications. Therefore, it is hypothesized that there is a point during long-term aging after which different binders in different mixtures reach a steady-state aging rate.

As the first candidate protocol (Protocol 1), similar to NCHRP 09-52, the research team will attempt to age mixes continually at 95ºC while periodically withdrawing specimens and evaluating them for their binder and mixture performance characteristics to assess whether the mixes have achieved a steady-state aging condition. This information can then be used to identify the shortest and optimal long-term aging time that can distinguish between good and poor cracking characteristics of asphalt mixtures. As part of this, the research team will also potentially use a higher temperature around 150ºC following the same process as Protocol 1. While it is understood that this may not be a realistic approach to simulate the aging in terms of chemical mechanisms, it may be useful to set applicable temperature-based benchmarks for the aging of mixtures.

Pressure Aging Device

It is possible that the candidate protocols described above may still not result in laboratory aging protocols that achieves the steady-state aging condition for all mixes within a reasonable amount of time. In this case, the only other variable that can be manipulated to achieve steady-state aging in a reasonable amount of time is pressure. While use of pressure combined with high temperatures is a standard procedure for asphalt binders, it has also been explored as a potential method for asphalt mixtures.

As the second candidate protocol (Protocol 2), the research team will evaluate the use of a combination of high temperatures and pressures to achieve steady-state aging in a reasonable amount of time. This was conducted using a novel customized pressure chamber for asphalt mixture. For this candidate protocol, the temperature was held the same (95ºC or lower optimized temperature), and different combinations of pressure and aging duration were evaluated.

Alternate Method using Ozone (O_3)

Although some of these mentioned methods have been adopted widely, there are still some disadvantages associated with it. Firstly, high temperatures exceeding 90ºC are used which considerably exceeds the in-service field temperature. Such high temperatures can lead to chemical reactions that may not happen in-service, such as increased polymerization and oligomerization. In addition, conditions such as increased pressure and duration are often used which makes it challenging to adopt these methods for routine tests. A promising alternative that negates some of these disadvantages and can be considered is the Viennese aging protocol (VaPro). This protocol employs a highly oxidant gas to simulate long term aging (Steiner et al. 2016). Atmospheric chemistry reveals that the oxidation potential of oxygen (O2) is low below 200ºC, but there are other gases in the atmosphere, which have a much higher oxidative potential. These gases, such as ozone (O_3) or nitrogen oxides (NOx) are called reactive oxygen species (ROS) and are particularly important in the tropospheric oxidative cycle. VAPro uses traces of these gases to accelerate the simulated aging process. The design of VaPro is based on a triaxial cell in which there is a regulated flow of an oxidizing agent enriched with ozone and nitric acid.

In order to implement this candidate protocol (Protocol 3), the research team will design and set up the equipment required, and preliminary tests were conducted to test the effectiveness and optimize the conditions for this approach.

Analogous to the above, asphalt binders were subjected to aging using standard time-temperature-pressure combinations (e.g., RTFO and PAV aging) as well as potentially investigating newly developed protocols (e.g., using thin films of asphalt binders in the PAV to accelerate diffusion and aging as being investigated by the ongoing NCHRP project). Aging in asphalt binders were tracked in sync with aging in asphalt mixtures to get more precise and accurate measure of aging indices and further corroborate findings from the mixtures being investigated.

Metrics to Track and Quantify Aging and Impact of Aging on Performance

Based on prior results described above and technical experience of the research team, the following three approaches were used to assess the extent and impact of laboratory aging.

1. The extent of aging was quantified based on the relative extent of aging in the binder extracted from a mixture. In this study, actual field data and/or laboratory data obtained using the NCHRP 9-54 protocol were used as a benchmark for field aging behavior of asphalt mixtures and binders. Note that benchmarking using field data requires a longitudinal study that samples and measures aging for multiple years in field. While the research team are involved in several other studies that provide access to such materials in this study, the time duration (maximum field aging that can be accomplished) were somewhat limited.

   Specifically, the extent of aging was quantified using FTIR spectroscopy with the extracted asphalt binder.

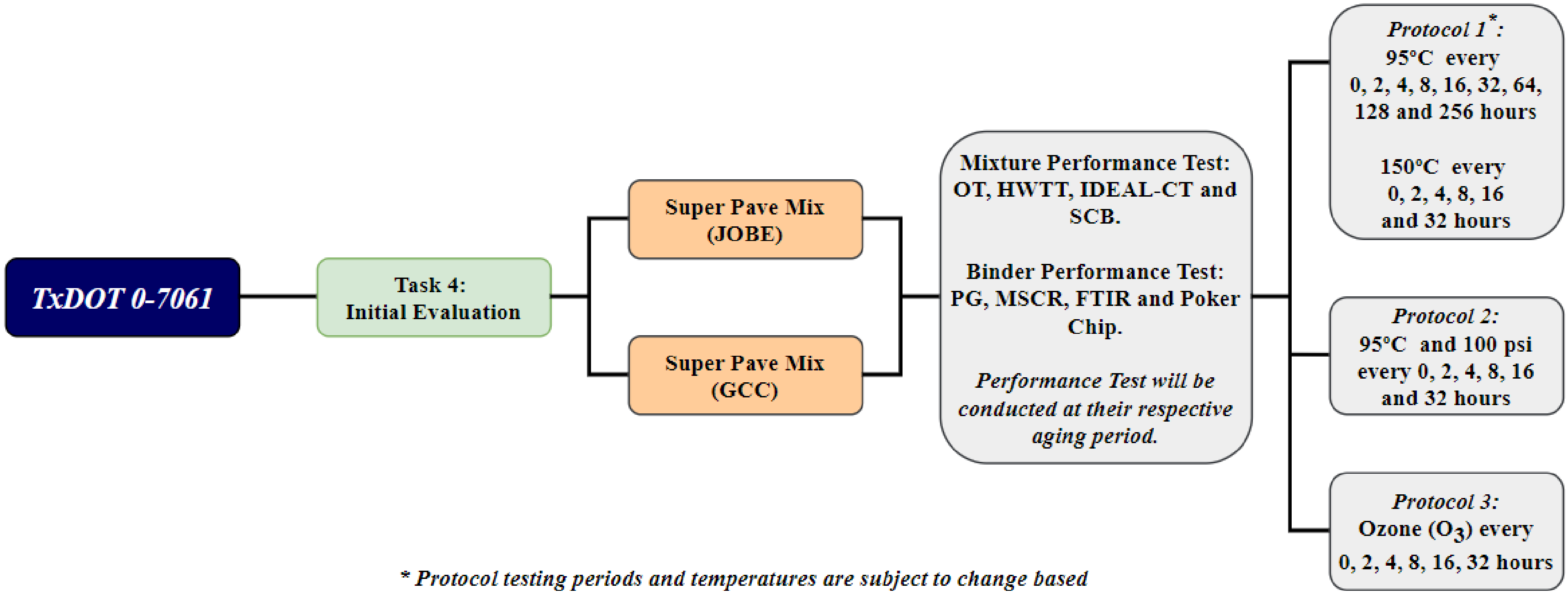
2. The second metric is the minimum extent of aging after which the binder and mixture reach a steady state condition and there is no change in the rate of change of rutting or cracking-resistance of the mixture. In other words, the stage after which the rank order of mixture performance does not change anymore, particularly in terms of cracking resistance. Specifically, the performance of the binder was evaluated using rheological indicators and the performance of the mixtures were evaluated using rutting and cracking tests after sampling materials at different stages of aging.
3. The aging sensitivity and performance of different mixtures were compared to the aging sensitivity of the binder from binder performance tests. For example, the ratio of $G^*/\sin\delta$ from unaged and RTFO aged binders is readily available for all binders and may reflect potentially problematic mixes that show anomalous behavior after long-term aging. The research team have conducted similar analysis using the LIMS binder database of TxDOT and identified relative trends in short-term and long-term aging. The gathered information was beneficial when setting appropriate benchmarks for the binders and mixtures being tested.

In order to establish the testing protocols for the remainder of the test matrix as a part of this project, the research team proposed to address these items by determining the aging level and engineering properties of two typical asphalt mixtures at each selected aging protocol. The research team aged mixes at different conditions and periodically withdraw specimens for evaluation as per the earlier described methods. Specimens were expected to be withdrawn and tested at intervals after 0, 2, 4, 8 and 16 hrs. Preliminary analyses were used to further refine and optimize the testing variables. The rank order of the cracking and rutting potentials of the candidate mixtures were tracked over time to identify when the steady-state aging condition is met. Figure 3.11 shows the initial evaluation flowchart based on the chosen approaches.

Summary

This chapter presented an extensive statistical analysis to study the impact of short- and long-term aging of materials in the AC mixtures. The following preliminary conclusions were drawn for further use in this study:

- The DSS from TxDOT 0-6658 provided useful information on the typical field performance of AC materials and binders over time that will help formulate the experimental design plan.
- Verification and validation of key findings from recent NCHRP reports were performed to meet the typical conditions exerted in Texas.
- The short-term rutting performance of AC mixtures needs further evaluation within the experimental design plan to better simulate field conditions.
- The cracking performance test (OT) provided good relationship between the anticipated long-term field performance of AC mixtures.
- The acceptance limits for the OT based on the crack progression rate provide an effective method of determining a mixture susceptibility to cracking. However, new criteria must be studied and implemented for short- and long-term aged AC mixtures.
- An initial evaluation of test methods based on the most promising aging protocols to evaluate aging extents have been determined for further testing.
- Specimens from mixtures were withdrawn periodically and tested using a suite of binder and mixture tests. The rank order of the cracking and rutting potentials of the candidate mixtures were tracked over time to identify when the steady-state aging condition is met.



** Protocol testing periods and temperatures are subject to change based on results obtained as we progress through the initial evaluation.*

Figure 3.11. Initial Evaluation Flow Chart based on Most Promising Aging Protocols

Chapter 4. Development of Experiment Design

This section comprises the gathering of data regarding the variations in testing protocols for both short- and long-term aging conditions. The more notable variations include the differences in the aging temperature ranges and durations for both asphalt mixtures and binders. To arrive at the most optimal aging protocol that will benefit TxDOT, various factors need to be evaluated ranging from mix types, aggregate types, binder sources, performance grade (PG) of the binders, recycled materials, and additives.

Initial Evaluation

To account for the requirements of this task, various asphalt mixtures were selected and sampled to be included in this study. The following parameters highlight the factors pertaining to selected asphalt mixtures that are recommended for consideration in the preliminary selection of aging processes:

- Frequency use of mix by TxDOT
- Pavement application (*preferably surface mixtures*)
- Historical performance of the AC mixes
- Geological composition (e.g., hardness and absorption) of aggregates
- Type of asphalt binder and source
- Use and type of additives
- Availability of recycled materials

The research team refined the experiment design plan with feedback from TxDOT during progress meetings.

As shown in Figure 4.1, the ***initial evaluation*** phase was designed for preliminary analysis to obtain relevant data from each of the selected aging protocols and compare results amongst the protocols. The data collected from the individual protocols and their respective curing conditions as well as limitations were used to pare down the number of aging protocols that were explored further in the extended evaluation during the extended evaluation. Two local sources of material to evaluate the aging protocols for lab-mixed lab-compacted (LMLC) and plant-mixed lab-compacted (PMLC) specimens were selected to analyze the most promising aging protocols.

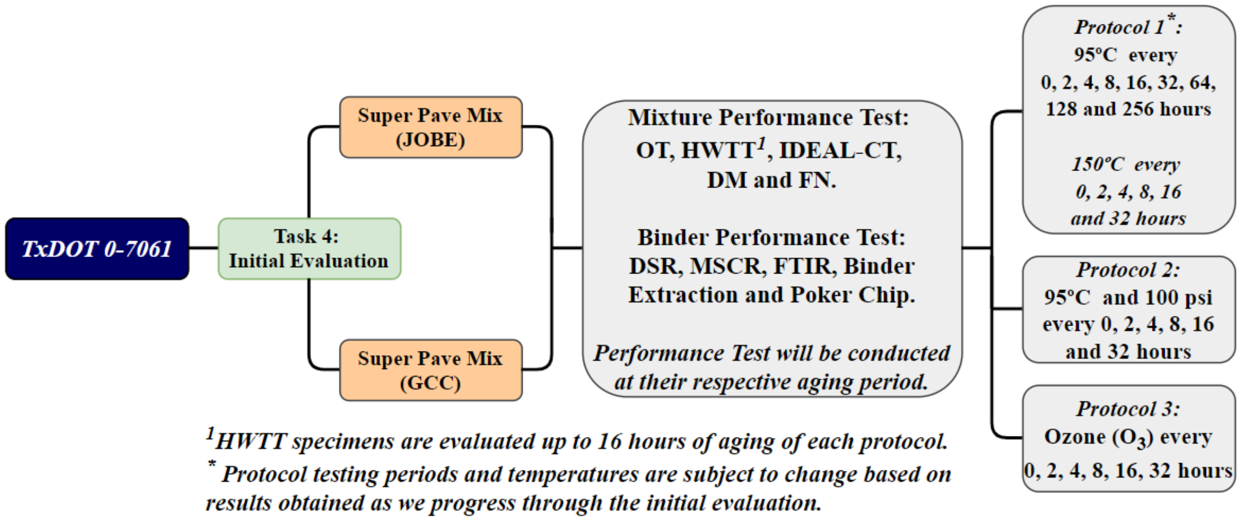


Figure 4.1. Initial Evaluation Design Plan

The concept of the initial evaluation is to capture the aging behavior of PMLC and LMLC specimens having varying mix types, mix source, binder sources, and content and recycled materials. The selected mixes were tested based on their response to different aggregate gradations, compaction methods, and compaction temperatures. Furthermore, to virtually target all possible aging factors, the binder sources were screened for aging sensitivities.

The selection of a mix design across Texas had to include at least four different bins of aggregates to evaluate the influence of aggregate type, gradation, mix type, asphalt content, and use of recycled material on the aging potential of the mix. The focus of the study was on surface mixes. Two Superpave[2] (SP) mixtures from El Paso were selected as part of the initial evaluation phase due to the travel constraints caused by the pandemic. Additional AC mixes and corresponding raw materials sources are presented in Appendix A. Figure 4.2 illustrates the typical SP mix design from El Paso. The influence of the gradation on the AC mix performance were further analyzed through alternative approaches such as the Bailey method, which determines the prime gradation of a mix design. Figure 4.3 illustrates the gradation of a typical SP mixture. "*S*" shape gradation curves have gained popularity. An investigation of the influence the gradation on the performance of the AC mixes were performed during the initial evaluation. The mix design gradation can greatly influence the amount of recycled material (RAP and RAS) that can be added to the mix, which in turn influences the mix performance.

Extended Evaluation

The ***extended evaluation*** was informed by the outcomes of the initial evaluation. The most promising protocol(s) were redefined and further evaluated by analyzing the following variables:

- Mix type
- Aggregate absorption
- Binder PG and source
- Recycled materials
- Recycled agents
- Additional additives

[2] Additional raw material evaluations were performed in collaboration with TxDOT IAC 49-20.

TEXAS DEPARTMENT OF TRANSPORTATION

2014 HMACP MIXTURE DESIGN : COMBINED GRADATION

Refresh Workbook — TX2MIXDE14 - File Version: 03/31/20 10:31:46

SAMPLE ID:	JMF-1	SAMPLE DATE:	4/20/20		
LOT NUMBER:		LETTING DATE:			
SAMPLE STATUS:		CONTROLLING CSJ:	0665-01-009		
COUNTY:	EL PASO	SPEC YEAR:	2014		
SAMPLED BY:	EDUARDO RASCON	SPEC ITEM:	344		
SAMPLE LOCATION:	MCKELLIGON CANYON	SPECIAL PROVISION:			
MATERIAL CODE:	ACTX-SPC-10R-VSA-70-22	MIX TYPE:	344-SP-C	WMA Additive in Design?	No
MATERIAL NAME:	SUPER PAVE C 10% RAP VADO SAC a 70-22			Target Discharge Temp., °F:	275
PRODUCER:	GCC SUN CITY MATERIALS			WMA TECHNOLOGY:	
AREA ENGINEER:	ALDO MADRID	PROJECT MANAGER:		WMA RATE: / UNITS:	% by weight of asphalt
COURSE\LIFT:	Surface	STATION: / DIST. FROM CL:		CONTRACTOR DESIGN # :	ASPH

Maximum Allowable, %	
Frac RAP:	20.0
Unfrac RAP:	10.0
RAS:	5.0
RB Ratio:	20.0

Recycled Binder, %	
Bin No.8 :	0.4
Bin No.9 :	0.0
Bin No.10 :	0.0
Total	**0.4**

Use this value in the QC/QA template>>

Ratio of Recycled to Total Binder, % (based on binder percent (%) entered below in this worksheet)
8.0

	AGGREGATE BIN FRACTIONS							"RECYCLED MATERIALS"			
Aggregate	Bin No.1	Bin No.2	Bin No.3	Bin No.4	Bin No.5	Bin No.6	Bin No.7	Bin No.8	Bin No.9	Bin No.10	
Source:	imestone_Dolomit	Igneous	imestone_Dolomit					Fractionated RAP			Material Type
Pit:	McKelligon-Dolomite	Vado	McKelligon-Dolomite								Material Source
Number:	2407201	0050310	2407201								RAS Type
Producer:	Cemex	Cemex	Cemex	Cemex				Cemex			RAP/RAS Producer
Sample ID:	McKelligon #67	Vado 3/8"	Fine Screenings	Section 10 Mortar Sand				Fine 1/2"			Sample ID
								Recycled Asphalt Binder (%)			
								4.3			
Hydrated Lime?:	Yes										

Individual Bin (%):	14.0 Percent		40.0 Percent		26.0 Percent		9.9 Percent		Percent		Percent		Percent	
Sieve Size:	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*
1"	100.0	14.0	100.0	40.0	100.0	26.0	100.0	9.9						
3/4"	92.0	12.9	100.0	40.0	100.0	26.0	100.0	9.9						
1/2"	33.0	4.6	100.0	40.0	100.0	26.0	100.0	9.9						
3/8"	17.0	2.4	97.0	38.8	100.0	26.0	100.0	9.9						
No. 4	2.0	0.3	37.0	14.8	100.0	26.0	100.0	9.9						
No. 8	1.0	0.1	4.0	1.6	63.0	16.4	99.0	9.8						
No. 16	1.0	0.1	2.0	0.8	37.0	9.6	98.0	9.7						
No. 30	1.0	0.1	1.0	0.4	24.0	6.2	91.0	9.0						
No. 50	1.0	0.1	1.0	0.4	17.0	4.4	42.0	4.2						
No. 200	0.3	0.0	0.4	0.2	10.6	2.8	0.5	0.0						

	Bin No.8		Bin No.9		Bin No.10		Combined Gradation									
	10.0	% of Tot. Mix		% of Tot. Mix		% of Tot. Mix	Total Bin	Lower & Upper Specification Limits			Restricted Zone			Individual % Retained	Cumulative % Retained	Sieve Size
	10.1	% of Aggreg		% of Aggreg		% of Aggreg	100.0%									
Sieve Size	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum.% Passing	*Wtd Cum. %*	Cum. % Passing	Lower	Upper	Within Spec's	Lower	Upper	Within Spec's			
1"	100.0	10.1					100.0	100.0	100.0	Yes				0.0	0.0	1"
3/4"	100.0	10.1					98.9	98.0	100.0	Yes				1.1	1.1	3/4"
1/2"	100.0	10.1					90.6	90.0	100.0	Yes				8.3	9.4	1/2"
3/8"	98.0	9.9					87.0	58.0	90.0	Yes				3.6	13.0	3/8"
No. 4	71.0	7.2					58.2	28.0	90.0	Yes				28.8	41.8	No. 4
No. 8	50.0	5.1					33.0	28.0	58.0	Yes	39.1	39.1	Yes	25.2	67.0	No. 8
No. 16	37.0	3.7					24.0	2.0	58.0	Yes	25.6	31.6	Yes	9.0	76.0	No. 16
No. 30	28.0	2.8					18.6	2.0	58.0	Yes	19.1	23.1	Yes	5.4	81.4	No. 30
No. 50	19.0	1.9					11.0	2.0	58.0	Yes	15.5	15.5	Yes	7.6	89.0	No. 50
No. 200	7.7	0.8					3.8	2.0	10.0	Yes				7.3	96.2	No. 200

(Bold Italic) Not within specifications ***(Bold Italic)*** Not within specificaitons- Restricted Zone *(Italic)* Not cumulative

Lift Thickness, in:	2.50	Binder Substitution?	No	Binder Originally Specified:	PG 70-22	Substitute Binder:	
Asphalt Source:	Western	Binder Percent, (%):	5.4	Asphalt Spec. Grav.:	0.996		
Antistripping Agent:	Lime	Percent, (%):	1				

Figure 4.2. Superpave-C Mix Design from El Paso District

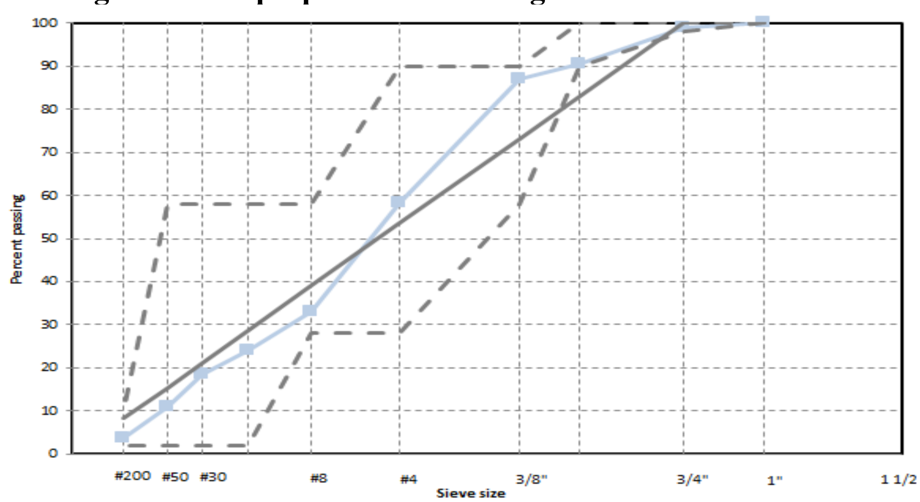


Figure 4.3. Gradation Curve for a Superpave C AC Mixture

Material Selection Overview

The mixtures and mixture elements selected in the initial evaluation were reviewed and additional mixtures or elements were added to the extended evaluation. This helped the research team compare once interaction had occurred, and newfound needs were possibly identified. During this phase, the research team obtained field cores to have another metric to determine the extent of aging achieved from the field compared to the prototype methods used in the laboratory. Additionally, the extended evaluation phase validated the findings from the initial evaluation and were subject to modifications based on the outcomes from Phase I (*Documentation*) and feedback from TxDOT for this study.

Selection of Mixture Types

The most common AC mixes in Texas are Type C and Type D dense-graded (DG), Superpave (SP), or stone-matrix asphalt (SMA) mixes. The research team suggested the following four mix types: DG-C, SP-C, SP-D, and SMA D for ***extended evaluation***. A WMA mix was also included in the experimental design plan for comparison with the HMA performance. The selection of the AC mixes and raw material were refined in collaboration with TxDOT. The rationale for these recommendations is the following items:

- Type C and Type D were compared to assess the impact of change in aggregate sizes and gradation.
- Type-D, SP-D, and SMA-D were compared further to delineate the influence of gradation and aggregate structure.
- Type C and SP-C were compared to evaluate the impact of different compaction methods.
- SMA-D mix is included to ensure that the process applies to different mix types.

Material Selection

The experiment design plan included a study to demarcate the influence of aggregate type and properties on the aging potential of asphalt mixtures and binders. The research efforts in TxDOT 0-6679 "*Performance Life of Various HMA Mixes in Texas*," identified three common types of aggregates used in Texas that were adapted as part of this study to properly document the influence of aggregate types and properties. Since the aging potential of asphalt mixtures is influenced by the absorption of the aggregates, an absorptive (primary limestone) and two non-absorptive (e.g., granite or sandstone) aggregates were considered in the development of the experiment design. For the non-absorptive aggregates, the research team sampled aggregates with different surface aggregate classification (SAC). Furthermore, selecting the candidate aggregates from the aggregate sources similar to those used in TxDOT Research Project 0-5268 "*Role of Coarse Aggregate Strength on Resistance to Load in HMA*" and ongoing TxDOT Research Project 0-6923 "*Develop Guidelines and Design Program for Hot-Mix Asphalts Containing RAP, RAS, and Other Additives through a Balanced Mix-Design Process*" were used, because of the wealth of data that already existed in terms of mix design and performance. These aggregates are summarized in Table 4.1, and the following rationale was considered in selecting the aggregate types:

- Absorptive and non-absorptive aggregates were used to compare their impact of the aging potential of AC mixes.
- Extraction of binder from absorptive and non-absorptive recycled material to compare the rate of extraction. The comparison of the interaction of binders and aggregates were assessed.
- Absorptive and non-absorptive aggregate types were compared to evaluate the influence of the aggregates' impact on the performance of the AC mix given different parameters.

Table 4.1. Proposed Pool of Aggregates for Consideration for Extended Evaluation

Sr. No.	Aggregate Type	Source Location
1	Hard-Limestone	Brownwood
2	Soft-Limestone	Marble Falls
3	Granite	El Paso
4	Sandstone	Brownlee
5	Gravel	Laredo

Asphalt Binder Selection

Four common asphalt binder grades used in Texas are PG 58-22, PG 64-22, PG 70-22, and PG 76-22. Using distinct sources of binders helped identify the influence of binders on aging. Based on the outcomes from the initial evaluation, pre-existing data available, and the interaction with TxDOT, additional binder grades with varying sensitivity to aging and varying grades, were selected for the extended evaluation phase. The research team included three asphalt binder grades: PG 64-22, PG 70-22, and PG 76-22. The rationale for these recommendations was the following items:

- Rutting of mixtures is most common for lower PG grades while higher PG grades yield a mixture more susceptible to cracking.
- Comparison of different binder grades to better understand and document the aging potential of AC mixes and binders.
- The use of different binder grades to analyze the aging effect of virgin binders when compared to modified/blended binders.

Recycled Material and Additive Selection

The use of recycled material in an AC mixture can negatively affect the performance and durability when the binder stiffens or consistency. Table 4.2 presents the possible scenarios to document the aging potential of asphalt mixtures containing recycled materials (RAP and RAS). The research team used a range from specified TxDOT limits. TxDOT Special Specifications 3074 and 3077, allow for a maximum RAP of 35% and 20%, respectively. The maximum RAS amount is specified as 5% and 3%. To account for the variability of RAP and RAS materials, the asphalt contents and aggregate gradations were evaluated at least three times for each source. The following TxDOT test methods were followed to verify the asphalt content and aggregate gradation of the recycled materials:

- Tex-236-F: *Determining Asphalt Content from Asphalt Paving Mixtures by the Ignition Method*
- Tex-200-F: *Sieve Analysis of Fine and Coarse Aggregates*

Table 4.2. Possible Scenarios to Delineate Influence of Recycled Material Content

RAP/RAS Levels	0%	10%	15%	20%	30%	40%
0%	✓	✓	✓	✓	✓	✓
2.5%	✓	✓	✓	✓		
5.0%	✓	✓	✓			

Several additives were used to improve the performance of an AC mixture. Rejuvenating agents can potentially reduce the viscosity of aged binders, thus improving the adhesion and cohesion properties as well as the flexibility of the asphalt binder that is of interest to this study. The effect of rejuvenators may be of value to determine the influence it imposes on the aging of AC mixtures. For this study, rejuvenator agents were evaluated to document the effectiveness and feasibility as sustainable preservation measures. The performance of the rejuvenated mix was compared to the performance of a mix without additives to delineate the influence of additives. The selection of the rejuvenator agents as well as additive levels and mixes were defined in close interaction with TxDOT.

Performance Test Methods

Asphalt Concrete Mixture Tests

Figure 4.4 shows a list of promising performance test methods for asphalt mixtures and binders that were included in the experiment design plan. The main distresses associated with short- and long-term aging are early-age rutting and long-term cracking, respectively. The rutting susceptibility of an AC mixture were evaluated through the Hamburg Wheel-Tracking (HWT). The cracking potential of an AC mix were determined through the Overlay Tester (OT) and IDEAL-Cracking Tests (IDEAL-CT). For the extended evaluation, with the consultation of TxDOT and the outcome of the initial evaluation, the research team will decide if the identified test methods are the most appropriate for this study.

The following test methods were used for an initial evaluation to determine the aging potential of AC mixtures (see Appendix B):

- Overlay Tester (Tex-248-F): The OT will assess AC mixture susceptibility to fatigue or reflective cracking. There are two major contributing factors to the mixture's cracking resistance, critical fracture energy (*Gc*), and the crack progression rate (CPR). The critical fracture energy is known as the energy that is necessary to start a crack from the bottom of the specimen at the first loading cycle. The CPR is known as the process in which the specimen will undergo loading cycles in the OT that allows for propagation of the crack.
- IDEAL-Cracking Test (Tex-250-F): The IDEAL-CT will estimate the stiffness properties, tensile strength, and the cracking tolerance index (CT_{index}) of AC mixture specimens of the HMA mixtures.
- Hamburg Wheel-Tracking Test (Tex-242-F): The HWT test were adapted in this study to evaluate the rutting susceptibility and moisture damage of AC mixtures.
-

Asphalt Concrete Binder Tests

In addition to the mixture tests, various binder tests were conducted to evaluate the rheology and chemistry of binders. The tests described below and in Figure 4 were conducted on asphalt binder samples recovered from mixtures subjected to different aging conditions.

- PG: The high, intermediate, and low-temperature grades were evaluated using a TA AR2000 DSR instrument as per Superpave specifications which is incorporated in Item 300 of the TxDOT specification.
- Ductility Indicator: The ductility of a binder indicates its potential resistance to low temperature and fatigue cracking. A rheological parameter that correlates to this can be measured using the DSR and referred to as the Glower Rowe parameter.

- ΔTc: Although this parameter was intended to serve as an indicator for certain types of potentially deleterious binder extenders, it can identify the potential for age-related cracking of different binders.
- Multiple Stress and Creep Recovery (MSCR) Test: The MSCR test were conducted for the binders as per AASHTO TP 70 to determine the non-recoverable compliance, elastic recovery, and stress sensitivity.
- Poker Chip Test: Poker chip tests were conducted to measure the fracture resistance of the asphalt binders using a thin film. The test geometry simulates the confined stress state experienced by a binder in an asphalt mixture while also ensuring that the entire test specimen is subjected to a uniform stress state.
- Fourier Transform Infrared (FTIR) Spectroscopy: FTIR tests were conducted to evaluate the functional groups in different asphalt binders and to correlate the different levels of aging.

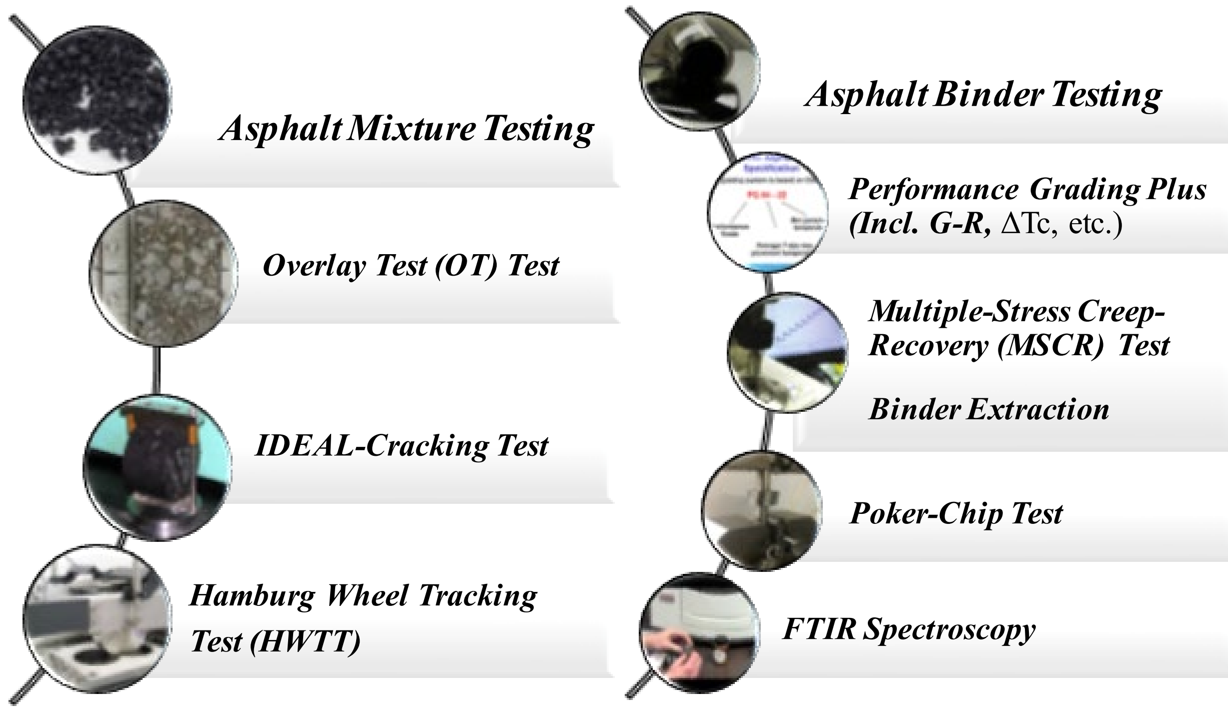


Figure 4.4. Experimental Design Plan for Performance Test Methods

Criteria for Aging Performance of AC Mixtures

Existing AASHTO R 30 standard and NCHRP Project 9-54 attempt to simulate the aging of the asphalt mixtures equivalent to a certain number of years in service. Given that the aging kinetics of binder reaches a steady-state condition after a certain amount of aging, it is hypothesized that there is a level of long-term aging after which the rank order of aging and consequently cracking characteristics of different binder-mixtures does not change. For a cracking test to distinguish accurately between crack-resistant and susceptible mixes, it is critical but adequate that mixes are aged to achieve this steady-state rate even if the mixes do not reach a state that is equivalent to 10 or 20 years of field aging. This project will utilize this concept to develop optimized laboratory aging protocols for mixtures.

Three main factors that dictate the aging rate of any given binder are:

- Temperature: AASHTO R 30 standard for traditional long-term aging conditions consists of curing compacted specimens at a temperature of 85ºC. NCHRP 09-54 recently compared the aging-induced by oven-cured loose mixtures at 95ºC and 135ºC. They recommended a curing temperature of 95ºC for loose mixtures so that the chemical composition and integrity of the asphalt binder are not compromised. This study points out that for any aging procedure to be realistic, it is important that the procedure does not utilize a temperature above 95ºC.
- Duration: NCHRP 09-54 recommended a curing temperature of 95ºC for loose mixtures so that the chemical composition of the asphalt binder is not compromised. While AASHTO R 30 standard specifies five days for long-term oven aging, NCHRP Project 09-52 documented that the curing conditions from AASHTO R 30 only simulate the aging conditions of pavements with 11 and 22 months in service for warmer and colder climates, respectively. Based on the literature search, there is no consistent approximation for the long-term aging prediction between the laboratory aging protocols and field aging of asphalt concrete layers. NCHRP Project 09-54 recommended aging durations based on project-specific and climate-based determination. Although the laboratory aging conditions recommended may consistently predict the aging conditions of asphalt mixtures in the field, the practices from NCHRP Project 09-54 are not optimal for routine mix design applications.
- Pressure: It is possible that the candidate protocols described above will still not result in laboratory aging protocols that achieve the steady-state aging condition for all mixes within a reasonable amount of time. In this case, the only other variable that can be manipulated to achieve steady-state aging in a reasonable amount of time is pressure. While the use of pressure combined with high temperatures is a standard procedure for asphalt binders, it has also been explored as a potential method for asphalt mixtures.

These factors were considered to select the best candidate aging protocols for further examination in the remainder of this study. Additionally, an alternate method utilizing ozone gas was considered.

Methodology: Protocols (1 through 4)

Using the concept of steady-state aging, the research team will develop optimized laboratory procedures to simulate the aging of asphalt mixtures. The main factors that can dictate the aging rate of mixtures include temperature, duration, pressure, and aging environment. These factors were considered to establish the four different aging protocols for examination in the remainder of the study. These protocols are indicated in Table 4.3 and summarized below.

Table 4.3. Optimized Laboratory Aging Protocols

Protocol Number	Temperature	Pressure	Curing Times
1	Cured at 95°C	**-**	0, 2, 4, 8, 16, 32, 64, 128, 258 hours
2	Cured at 150°C	**-**	0, 2, 4, 8, 16, 32 hours
3	Cured at 95°C	50 to 60 psi	0, 2, 4, 8, 16, 32 hours
4	Ozone (O_3)	Ozone (O_3)	0, 2, 4, 8, 16, 32 hours

Protocol 1: Conventional Oven Aging

As the first candidate protocol (Protocol 1), similar to NCHRP 09-52, the research team attempted to age mixes continually at 95ºC while periodically withdrawing specimens and evaluating them for their binder and mixture performance characteristics to assess whether the mixes have achieved a steady-state aging condition described earlier. This information was used to identify the shortest and optimal long-term aging time that can distinguish between good and poor cracking characteristics of asphalt mixtures.

Protocol 2: Elevated Temperature Oven Aging

Similar to Protocol 1, the research team explored a higher temperature of approximately 150ºC following the same process to achieve steady state. While it is understood that this may not be a realistic approach to simulate the aging in terms of chemical mechanisms, it may be useful to set applicable temperature-based benchmarks for the aging of mixtures. Figure 4.5 shows a sample of loose specimens in an oven used to conduct Protocol 1 and Protocol 2.

Figure 4.5. Loose Mixture Aging in Oven (*Protocol 1 and Protocol 2*)

Protocol 3: Pressure Aging

As Protocol 3, the research team evaluated the use of a combination of medium to high temperatures and pressures to achieve steady-state aging in a reasonable amount of time. This were conducted using a novel customized pressure chamber placed inside a heating oven for loose asphalt mixtures as shown in Figure 4.6. For this candidate protocol, the temperature used were around 95ºC or lower, and different combinations of pressure and aging duration were evaluated.

Figure 4.6. Pressure Aging Chamber for Loose Mix (*Protocol 3*)

Protocol 4: Ozone Enriched

The fourth candidate protocol using Ozone gas aimed to negate some of the disadvantages associated with the other methods. In the previous methods, high temperatures exceeding 90ºC are used which considerably exceeds the in-service field temperature. Such temperatures may lead to chemical reactions that may not happen in-service, such as increased polymerization and oligomerization. In addition, conditions such as increased pressure and duration employed make it potentially challenging to adopt these methods for routine tests. This protocol employs a highly oxidant gas to simulate long-term aging of asphalt mixtures. Atmospheric chemistry reveals that the oxidation potential of oxygen (O_2) is low below 200°C, but there are other gases in the atmosphere, which have a much higher oxidative potential. These gases, such as ozone (O_3) or nitrogen oxides (NOx) are called reactive oxygen species (ROS) and are particularly important in the tropospheric oxidative cycle. This method uses traces of these gases to accelerate the simulated aging process of loose asphalt mixtures. The flowchart indicating the outline of the method involved is shown in Figure 4.7. Firstly, O_3 and NOx are generated by passing filtered air through an ozone generator. The ROS generated are used to oxidize loose asphalt mixture in an enclosed tank at a temperature of around 70ºC. Subsequently, the ROS is degraded for safe exhausting using appropriate fluids. The preliminary setup of the device as assembled by the research team is shown in Figure 4.8.

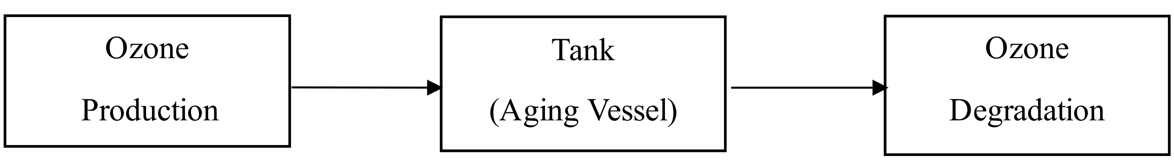


Figure 4.7. Simple Flowchart of the Ozone Aging Method (*Protocol 4*)

Figure 4.8. Setup of the Ozone Based Aging Device (*Protocol 4*)

Summary

This chapter developed the rationale for the experimental design plan. The experimental design plan process contained the selection of the AC mixes, material, PG of the binder, as well as the selection of performance test for binders and mixes. The most promising aging protocols were proposed considering three main factors: temperature, duration, and pressure. An alternative approach using ozone was also proposed was a potential aging method.

Chapter 5. Initial Evaluation of Aging Potential of Asphalt Concrete with Laboratory Aging Protocols

This chapter documents the findings from the evaluation of different aging protocols and investigation into the hypothesis of steady-state aging. As part of the initial evaluation, two typical Superpave mixes were evaluated. Both the lab-mixed lab-compacted (LMLC) and plant-mixed lab-compacted (PMLC) specimens were considered for the first mixture, while only LMLC specimens were used for the second mixture. The results from both mixes are presented in this chapter.

Asphalt Concrete Mixture

The particle size distribution of Mix 1 (blue line) and Mix 2 (orange line) are shown in Figure 5.1. Overall, Mix 1 had finer gradation and contained 1.5% more RAP asphalt binder than Mix 2. The same gradation distribution was used to prepare all specimens. The asphalt content of the RAP material was estimated following Tex-236-F "Determining Asphalt Content from Asphalt Paving Mixtures by the Ignition Method." This process, along with the volumetric analysis (Tex-241-F), is crucial to assure the optimum asphalt content and the target density requirements were met for all mixtures before conducting performance testing.

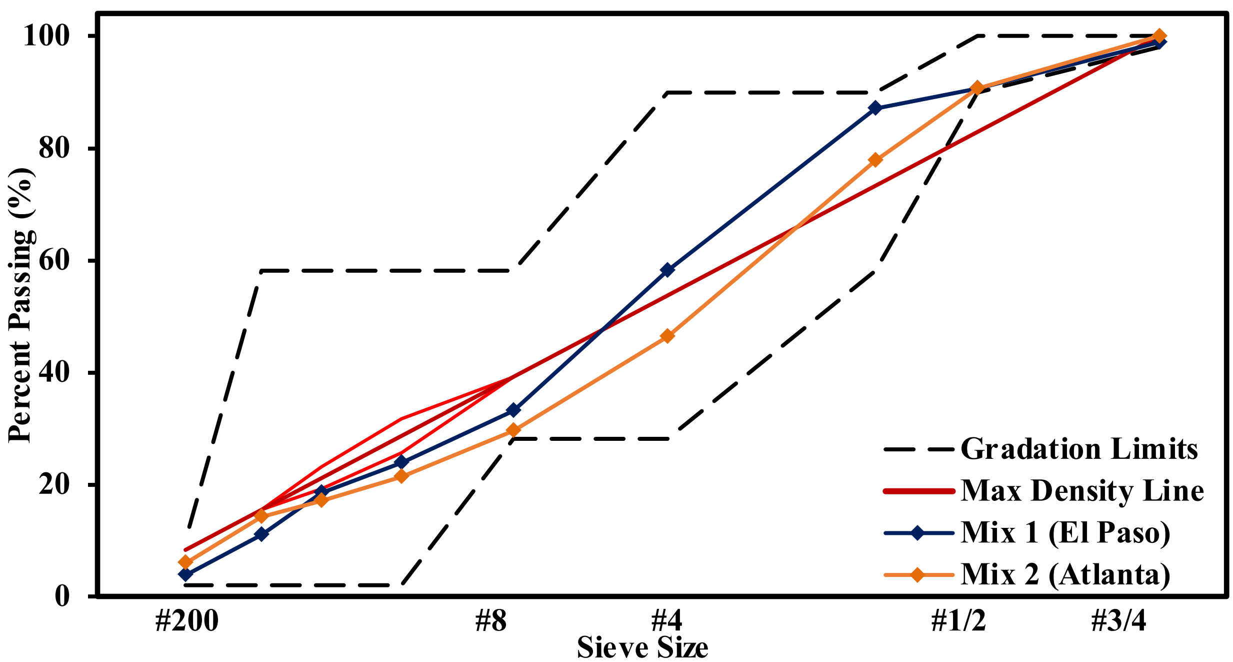


Figure 5.1. SP-C Mixture Aggregate Gradations

The AC mixes are both Superpave C mixtures designed with a Superpave gyratory compactor to meet a 96% target density according to Tex-241-F. The asphalt binders used for each mixture were PG 70-22 for Mix 1 and PG 76-22 for Mix 2. The volumetric properties such as optimum asphalt content (OAC), voids in mineral aggregate (VMA), RAP asphalt content, and dust/binder ratio of the AC mixes are summarized in Table 5.1. The current requirement of 15% minimum VMA for Superpave C mixtures was met for both mixes. The average asphalt content from four replicate samples matched the values reported for both mix designs. The requirement for dust/binder ratio ranges from 0.6 to 1.6, and both mixes yielded values within the required limits. The RAP content for both mixes was 10% of the total mix.

Table 5.1. Volumetric Properties of AC Mixes

Mix No.	OAC (%)	VMA (%)	RAP Asphalt Content (%)	Dust/Asphalt Ratio
1	5.4	16.5	4.3	0.7
2	5.4	16.6	5.8	1.1

Asphalt Concrete Mixture Tests

The results from the following test methods are presented in this report:

- *Hamburg Wheel-Tracking (HWT) Test (Tex-242-F):* The HWT test evaluated AC mixtures' rutting susceptibility and moisture damage.
- *Overlay Tester (Tex-248-F):* The OT evaluated the susceptibility of an AC mixture to fatigue or reflective cracking. There are two major contributing factors to the mixture's cracking resistance, critical fracture energy and the crack progression rate (CPR). The critical fracture energy is known as the energy necessary to start a crack from the bottom of the specimen during the first loading cycle. The CPR represents the rate at which a crack propagates during the cyclic loading of the test specimen.
- *IDEAL-Cracking Test (Tex-250-F):* The IDEAL-CT is essentially an indirect tensile strength test. The cracking tolerance index (CT-index) is a parameter based on the failure stress and post-peak failure rate used to evaluate the cracking resistance of the AC mixtures.

Asphalt Concrete Binder Tests

The following tests were conducted on asphalt binder specimens recovered from corresponding mixtures subjected to different aging conditions. The Tex-210-F procedure was followed to extract and recover the asphalt binder samples from the aged asphalt mixture.

- *Performance Grade (PG):* The high-temperature grades were evaluated using a Dynamic Shear Rheometer (DSR) as per Superpave specification incorporated in TxDOT Item 300. Since the tests were conducted using small samples of recovered asphalt binder from loose aged mixes, the low-temperature properties were evaluated using a DSR with a 4mm parallel plate geometry.
- *Fourier Transform Infrared (FTIR) Spectroscopy:* FTIR tests were conducted to evaluate the oxidation-related chemical functional groups in different asphalt binders and correlate the different levels of oxidation products formed in the asphalt binder.

The results from the LMLC and PMLC specimens for Mix 1 and LMLC specimens for Mix 2 are compared in the following sections. Nine-time intervals (0 hrs, 2 hrs, 4 hrs, 8 hrs, 16 hrs, 32 hrs, 64 hrs, 128 hrs, and 256 hrs) were chosen for Mix 1 to illustrate the gradual increase in aging metrics for the initial evaluation. The time intervals for Mix 2 were shortened for the binder testing based on the initial results from Mix 1. An additional time interval of 24 hrs was added as preliminary data showed a steady-state trend on the AC tests.

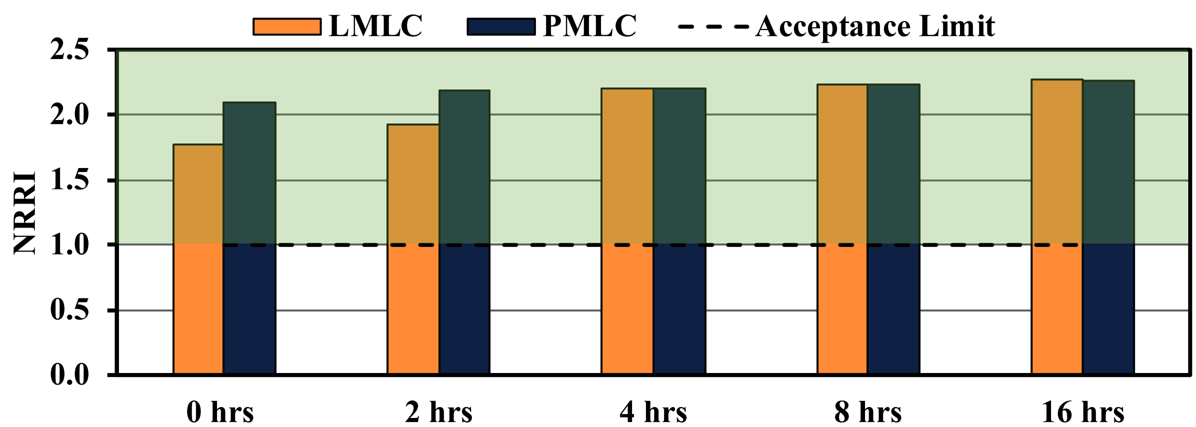
Protocol 1: AC Mixture Results

The normalized rutting resistance index (NRRI) from the HWT tests as a function of aging duration for Mix 1 (LMLC and PMLC) and Mix 2 (LMLC) specimens are presented in Figures 5.2 and 5.3. As the aging duration increased, the mixes become stiffer and more brittle. However, the rutting resistance improved with the increase in the aging period regardless of mixture type (LMLC or PMLC). Since aging the material for more than 2 hrs does not result in a significant change in the NRRI values, future work will focus on early age (between 0 to 2 hours) rutting with different binder PGs, aggregates, mix types, RAP, and additive contents. The difference between the PMLC and LMLC mixture is believed to come from human error coming from how long the mix sits in silos or the mixing temperature.

The OT Critical Fracture Energy (CFE) variations with aging time are presented in Figures 5.4 and 5.5 for Mixes 1 and 2, respectively. Triplicate specimens were tested to account for the repeatability of the test. The current upper and lower CFE limits of 1 and 3 lb.-in./in.^{2} were considered for the preliminary evaluation. However, the acceptance values were refined during Phase II. The standard deviations of the results that are shown using error bars were within ±0.4 for both LMLC mixes and ±0.6 for the PMLC specimens. The highest coefficient of variation (COV) was 16%. The CFE values for both the LMLC and PMLC specimens steadily increased with the aging of the mixes and were within the acceptance limit for up to 32 hrs of oven aging for Mix 1. Similarly, Mix 2 had a steady increase in CFE value until 8 hours. The increase in CFE value from 8 to 64 hours shows the mixture reached a steady-state that were further evaluated in the extended evaluation.

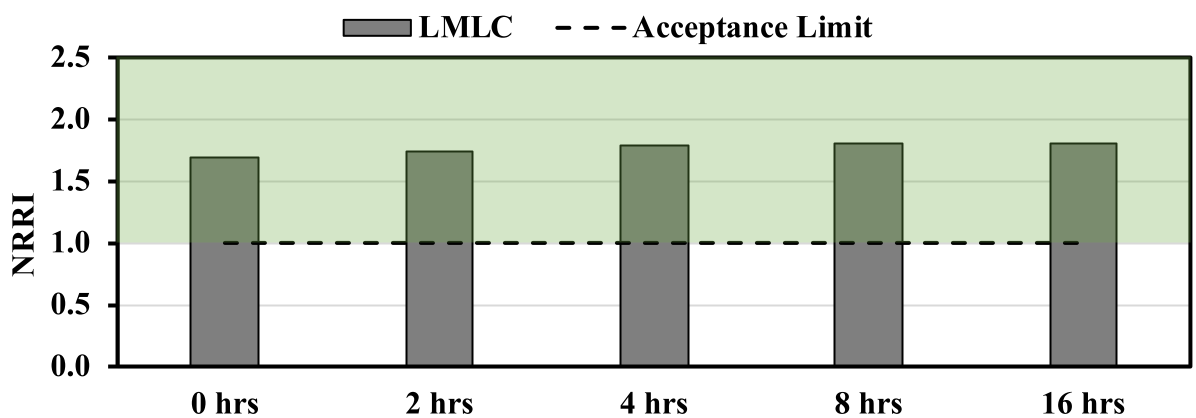
The variations in the OT crack progression rate (CPR) values with the aging period are presented in Figures 5.6 and 5.7. The COV values ranged from 2% to 10% for both LMLC mixes and from 3% to 15% for the PMLC specimens. Similar to the CFE values, the CPR values increase steadily and exceed the acceptance limit of 0.45 after 32 to 64 hours of oven aging.

The variations in the IDEAL-CT indices obtained from triplicate specimens with the aging period are presented in Figures 5.8 and 5.9. The COV values ranged from 7% to 19% for both LMLC mixes and 6% to 19% for the PMLC specimens. The current acceptance criterion for the CT-index is 80 or greater. The CT-indices from both the LMLC and PMLC specimens fall below the acceptance limit after 4 to 8 hrs of oven aging for Mix 1. However, Mix 2 saw the values fall below the acceptance criteria after 24 hrs of aging. For Mix 1, the LMLC specimens had lower CT-index values compared to the PMLC specimens. The difference between the mixes can be attributed to the original PG of the binder used in the mixes. Mixes with excessive aging could not maintain their solid structure under the compressive load and showed fair tensile strength.



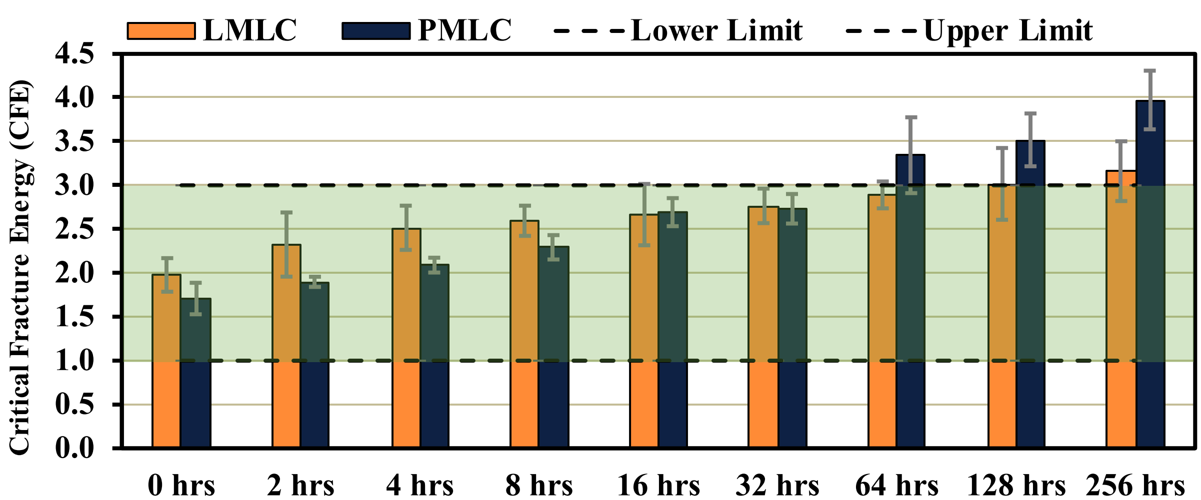
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.2. HWTT Results for Mix 1 Using Protocol 1



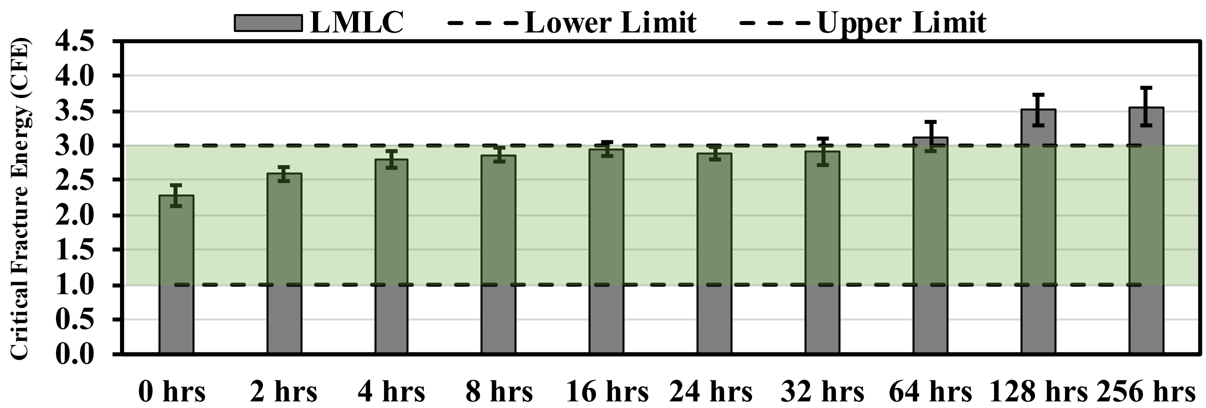
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.3. HWTT Results for Mix 2 Using Protocol 1



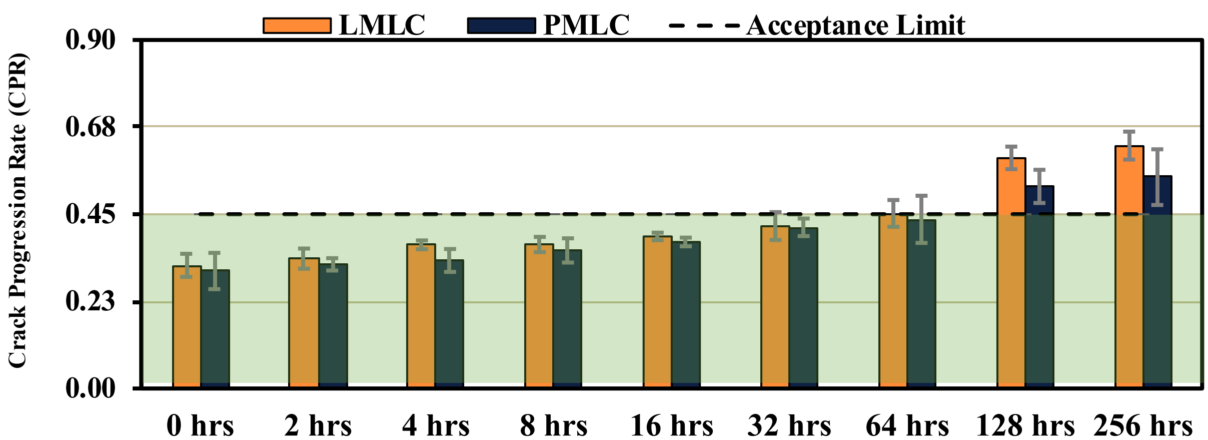
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.4. OT Critical Fracture Energy Results for Mix 1 Using Protocol 1



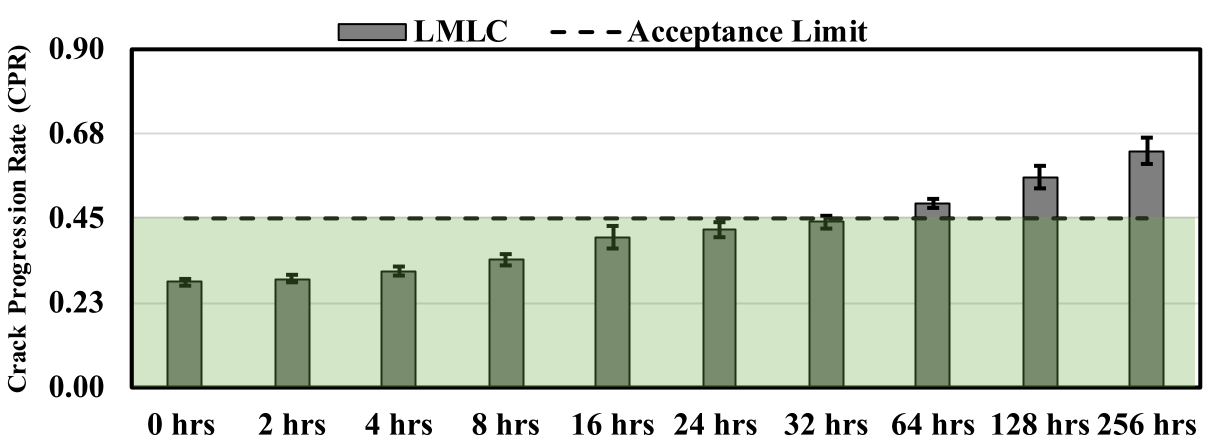
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.5. OT Critical Fracture Energy Results for Mix 2 Using Protocol 1



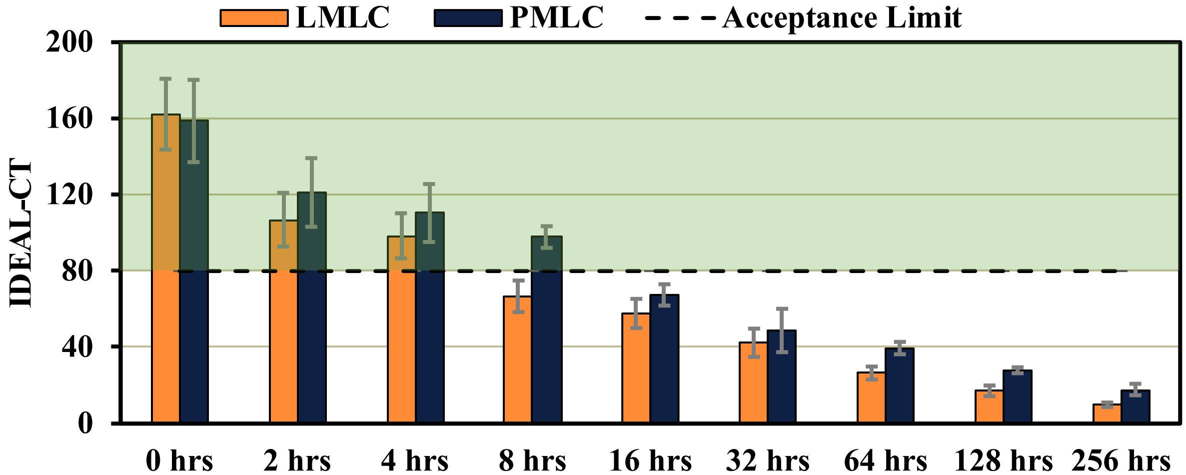
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.6. OT Crack Progression Results for Mix 1 Using Protocol 1



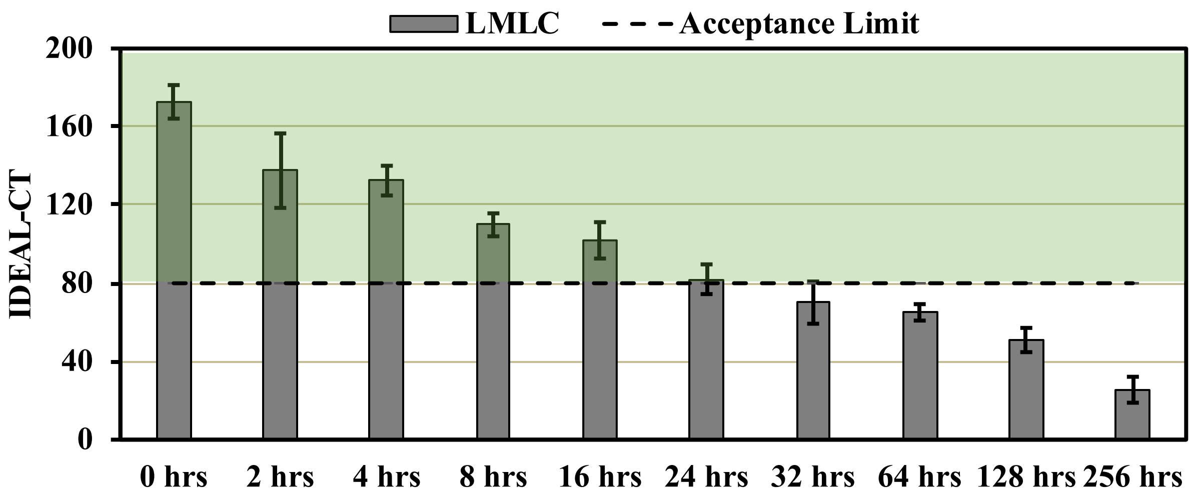
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.7. OT Crack Progression Results for Mix 2 Using Protocol 1



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

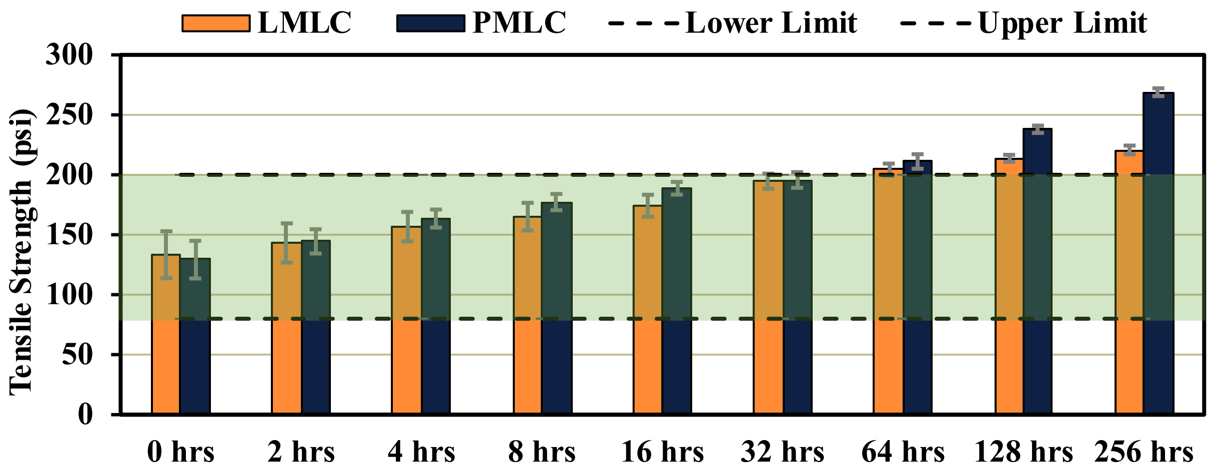
Figure 5.8. IDEAL-CT Results for Mix 1 Using Protocol 1



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

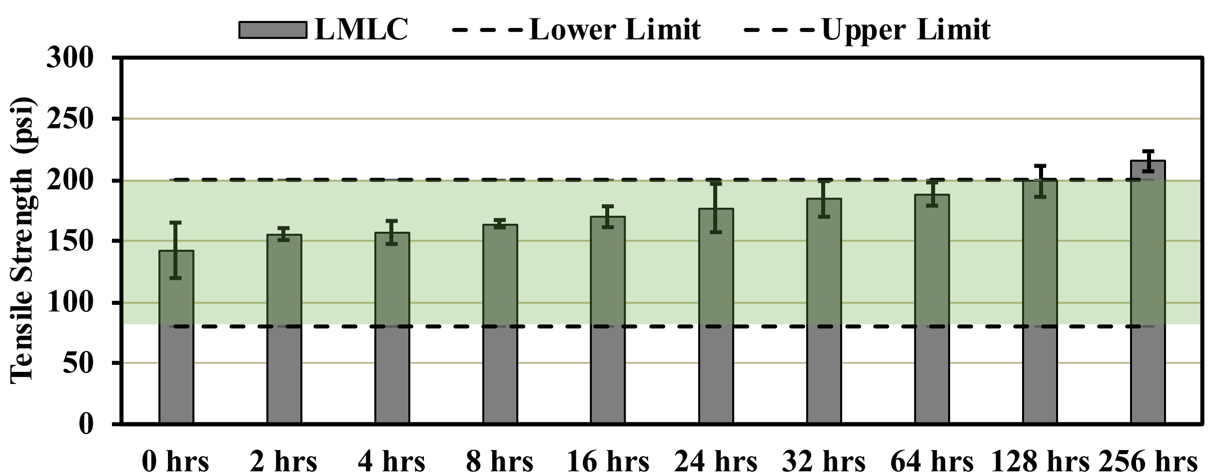
Figure 5.9. IDEAL-CT Results for Mix 2 Using Protocol 1

The variations in the IDT tensile strength with the aging period are presented in Figures 5.10 and 5.11. The COV values ranged 3% to 10% for both LMLC mixes and from 3% to 9% for the PMLC specimens. Considering the current lower and upper acceptance limits of 80 and 200, the mixtures exhibit a pattern similar to the OT results where the IDT tensile strength steadily increases and exceeds the limit after 32 to 64 hrs of aging.



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.10. Indirect Tensile Strength Results for Mix 1 Using Protocol 1



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.11. Indirect Tensile Strength Results for Mix 2 Using Protocol 1

Asphalt Concrete Binder Tests

The binder tests were performed on extracted and recovered samples of asphalt binders from loose asphalt mixtures. This process inevitably blends and reflects the average aging characteristics of the entire asphalt binder film that coats aggregate particles, even though these films have an aging gradient as one moves away from the surface of the binder coating an aggregate particle and deeper towards the surface of the aggregate particle. The rheological and chemical binder testing results presented in this report use an average of two test samples.

To evaluate the high-temperature stiffness of the various binders, a DSR device was used to measure the complex shear modulus of the recovered binders at a static frequency of 10 rad/s and a parallel plate geometry at various temperatures using a TA Instruments Rheometer. A 25 mm plate was used with the gap between the two circular plates set at 1 mm. Similarly, to determine the low-temperature PG and stiffness of the binders, a 4 mm plate was used with a gap distance of

2.2 mm. The method used to obtain the low-temperature grade of binders using the 4 mm diameter geometry with a DSR is described in detail by Hajj et al. (2019) and is briefly summarized below:

$$\log|G^*(\omega)| = \delta + \frac{\alpha}{1 + e^{\beta+\gamma\log(\omega)}} \qquad \textbf{(5.1)}$$

The two focal steps to obtain the low-temperature grade using a DSR were: (i) switching the domain response of frequency to the domain response of time and (ii) switching the response of shear to uniaxial response. The asphalt binders were subjected to a temperature-frequency sweep using frequencies ranging from 100 rad/s to 0.2 rad/sec and temperatures of -6, -12, and -18°C. The resulting data were fit to a master curve using the model described in Equation 3. Where $|G^*(\omega)|$ is the dynamic shear modulus in the frequency domain, ω is the angular frequency, δ is the minimum value of $|G^*|$, $\delta + \alpha$ is the maximum value of $|G^*|$ and β, γ are parameters denoting to the shape of the sigmoidal function. The dynamic shear modulus $|G^*(\omega)|$ was converted to the dynamic shear compliance $|J^*(\omega)|$ at any given frequency. The data from the frequency domain was converted to the time domain using Equation 5.2, where J'' and J' are the loss and storage compliance, respectively.

$$J(t) = J'(\omega) + 0.4J''(0.4\,\omega) - 0.014J''(10\omega) \qquad \textbf{(5.2)}$$

The shear compliance $J(t)$ was then transformed to tensile compliance $D(t)$ using Equation 3 where v signifies a Poisson`s ratio of the binder which is time-dependent. A constant value of 0.35 was used for v. The Bending Beam Rheometer (BBR) equivalent stiffness, $S(t)$, was calculated by reversing the data on the creep compliance curve. Using this, the BBR equivalent m-value and stiffness (S) were obtained to estimate the low-temperature grade of the binder.

$$D(t) = \frac{J(t)}{2(1+v)} \qquad \textbf{(5.3)}$$

Binder Results

The binders from the corresponding mixtures shown above were extracted and characterized using the rheological and chemical tests. The high-temperature PG of the mixtures at various times are shown in Figures 5.12 and 5.13. The results were calculated based on the value of G*/sinδ parameter and using the rolling thin-film oven aged binder criterion in AASHTO T316 (i.e., a temperature that results in a G*/sinδ value of 2.2kPa). A higher value of high-temperature PG indicates a stiffer binder, which in this case can be attributed to the effect of oxidative aging.

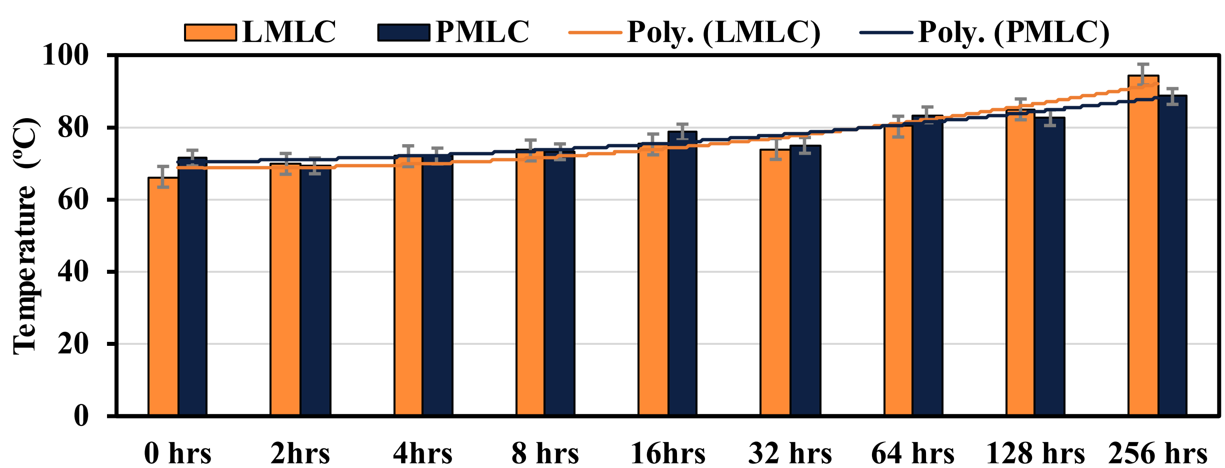


Figure 5.12. High-Temperature PG for Mix 1 Using Protocol 1

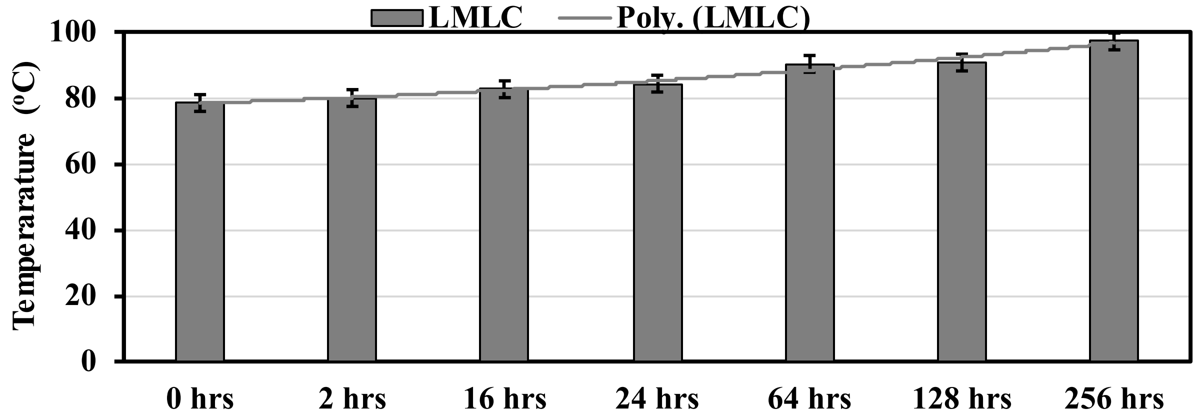


Figure 5.13. High-Temperature PG for Mix 2 Using Protocol 1

Literature indicates that with higher levels of aging, the complex modulus of binders increases and phase angle decreases, rendering more solid-like binder (*Xu and Isacsson, 2002*). An increase in high-temperature PG that is indicative of an increase in stiffness was seen for both sets of mixes. Trend lines are included in some of the figures to better illustrate the relative increase with aging time. For Material 1, at time intervals between 0 hrs and 32 hrs, the increase in stiffness was gradual but the upward trend was not entirely consistent due to limited time between sample collection. However, from 64 hrs onwards, the increase in stiffness was more pronounced. For Material 2, the trend of increase in high-temperature PG was more apparent due to fewer time intervals and more significant time gaps. The stiffness true grade and the m-value true grade of the binders extracted from Mix 1 aged for different durations are presented in Figures 5.14 and 5.15, respectively. Mix 2 illustrates the same extracted values in Figure 5.16. As per the requirements in AASHTO T313, the stiffness true grade is 10ºC below the lowest temperature at which the stiffness of the binders does not exceed 300 MPa. Similarly, the m-value true grade is 10ºC below the lowest temperature where the m-value exceeds a value of 0.3. When comparing the same binders exposed to different aging methods, a lower value of the stiffness and m-value true grade indicates the presence of a softer binder that has undergone comparatively less oxidation. A steady decrease in stiffness and m-value true grades with time could be seen for all sets of mixtures, which is the expected trend, following the high-temperature PG results.

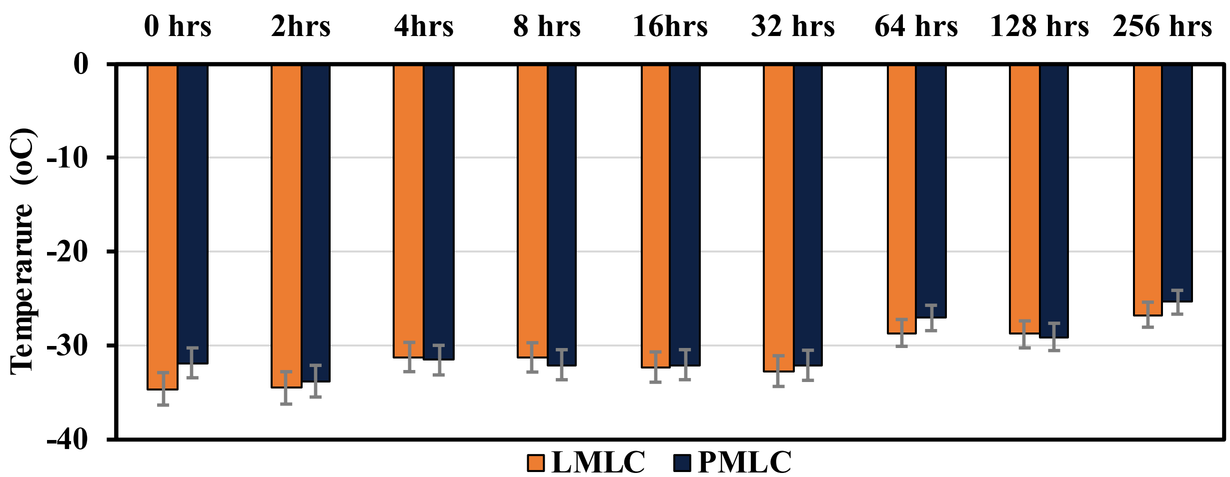


Figure 5.14. Stiffness-Based True Low-Temperature Grade for Mix 1 Using Protocol 1

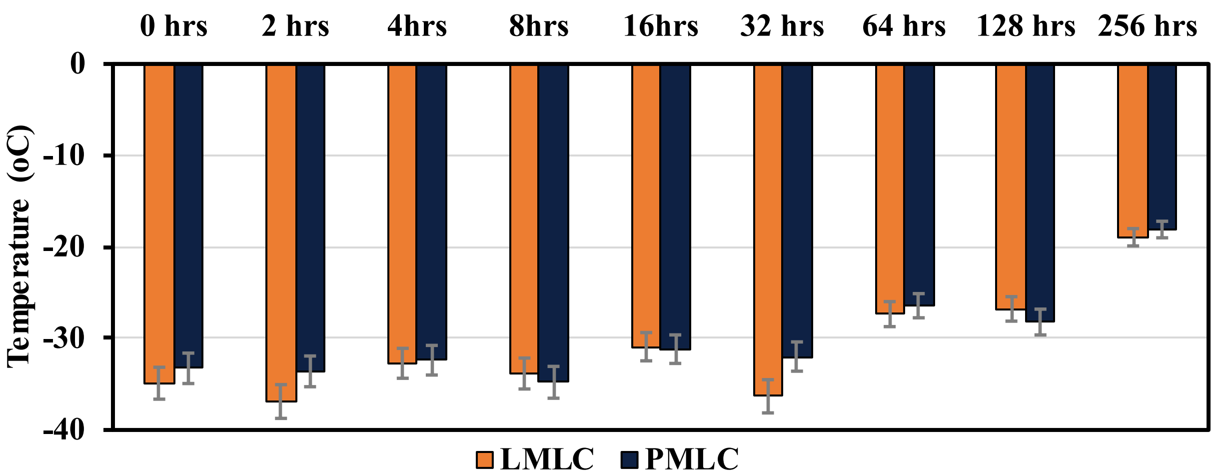


Figure 5.15. m-Value-Based True Low-Temperature Grade for Mix 1 Using Protocol 1

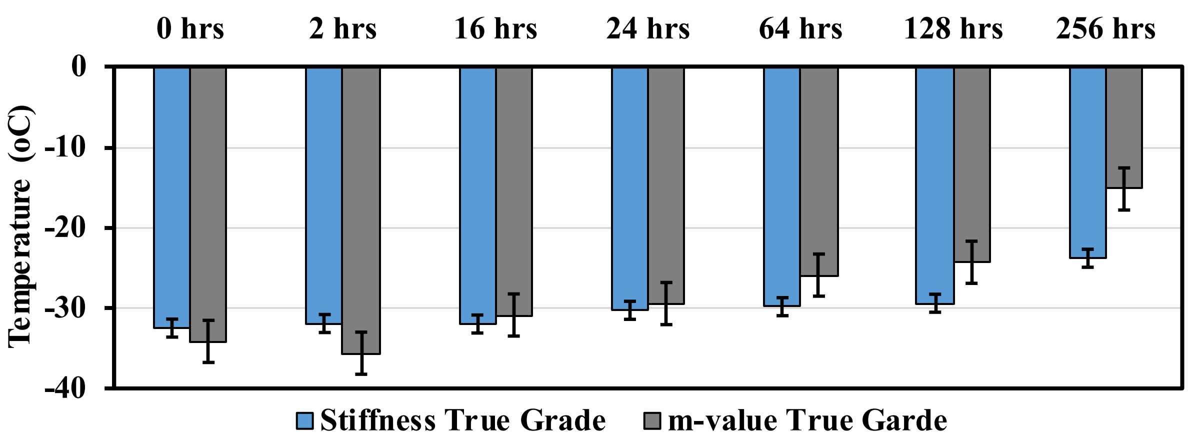


Figure 5.16. Low-Temperature PG for Mix 2 Using Protocol 1

The oxidation levels of the various binders were examined using FTIR spectroscopy. Two bands, comprising the carbonyl group and sulfoxide group were used to determine the extent of oxidation of the binders. The quantification of the oxidation bands was conducted as per Hofko et al. (2017). The analysis of FTIR spectra involved an absolute baseline, normalized spectra, and integration of areas. The following equation was used to calculate the respective area of analysis for a single spectrum:

$$A=\int_{N_{l,oa}}^{N_{u,oa}} VA_{norm}(N)\, dw \qquad \textbf{(5.4)}$$

In Equation 4, A is the normalized integrated area; $N_{u,oa}$ is the limit for the upper wavenumber of the structural group; $N_{l,oa}$ is the limit for the lower wavenumber of the structural group and $VA_{norm}(N)$ is the normalized absorbance at wavenumber *N*. The carbonyl and sulfoxide bands were stipulated from 1666 to 1746 cm^{-1} and 944 to 1066 cm^{-1} respectively (*Hofko et al., 2017*). Figure 5.17 shows a characteristic FTIR spectrum of a binder in unaged and aged conditions, which exhibits the heightened oxidation peak at the wavelength of roughly 1700 cm^{-1} and 1000 cm^{-1} for the aged binder. The various spectra were collected using a Thermo Scientific Nicolet iS5 spectrometer with a zinc selenide (ZnSe) ATR module. A small piece of the sample was placed on the ZnSe crystal, and the spectra were recorded from 4000 to 600 cm^{-1} using a resolution of 4 cm^{-1}.

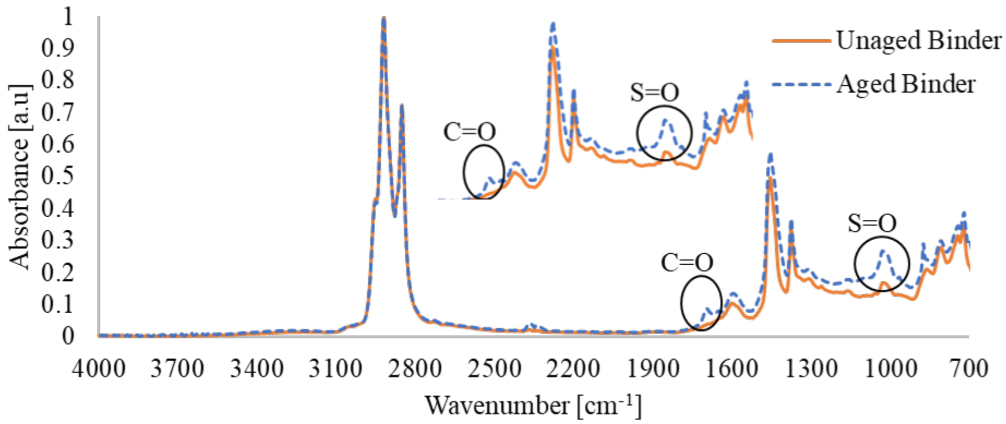


Figure 5.17. Sample FTIR Spectra of an Aged and Unaged Binder

The results for the C=O and S=O indices for Mix 1 are presented in Figure 5.18 as well as Figure 5.19, and the same is presented for Mix 2 in Figures 5.20 and 5.21 correspondingly. The general increase of those oxidation indices with aging time was visible for binders from both sets of mixtures. Out of the two indices calculated, the C=O index seems to be a more reliable indicator of aging with the linear increase. This is also an established concept in literature wherein studies have shown that the C=O index is better correlated with long-term aging as opposed to the S=O index (Sreeram and Leng., 2019). However, although FTIR indices are helpful to indicate the relative increase in oxidation, the parameters are semi-quantitative in nature and the relative increase of this parameter would be more pronounced when considering longer time intervals between tests as seen with Mix 2.

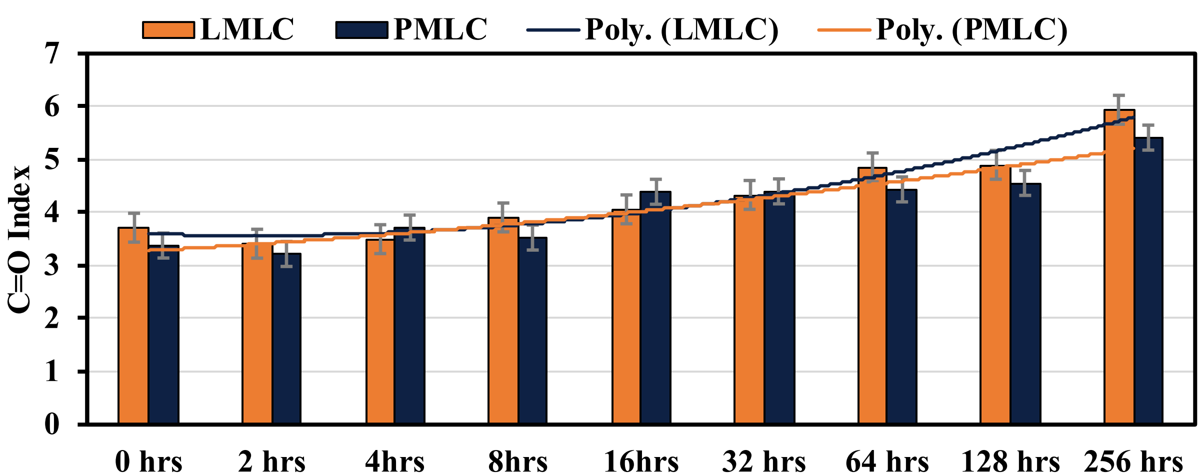


Figure 5.18. C=O Index for Mix 1 Using Protocol 1

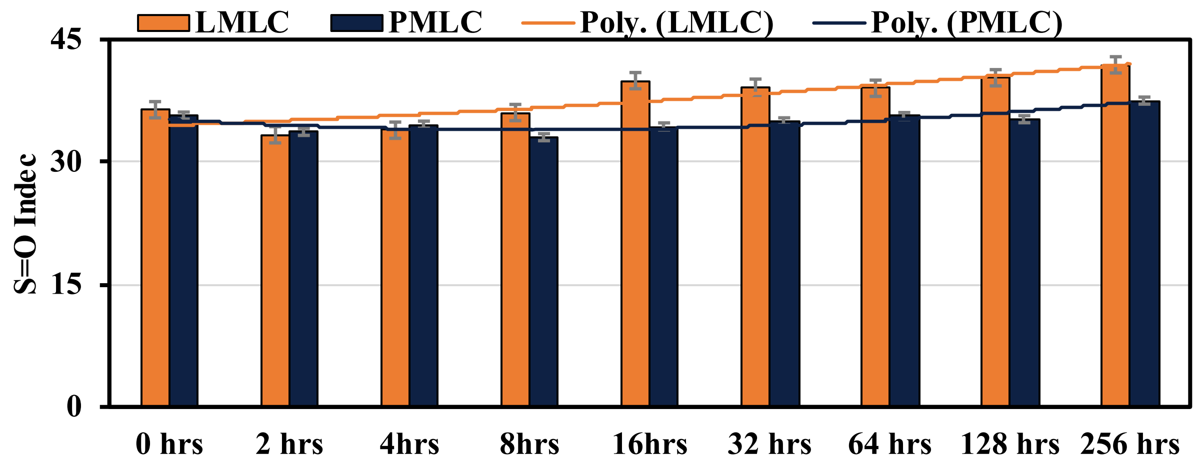


Figure 5.19. S=O Index for Mix 1 Using Protocol 1

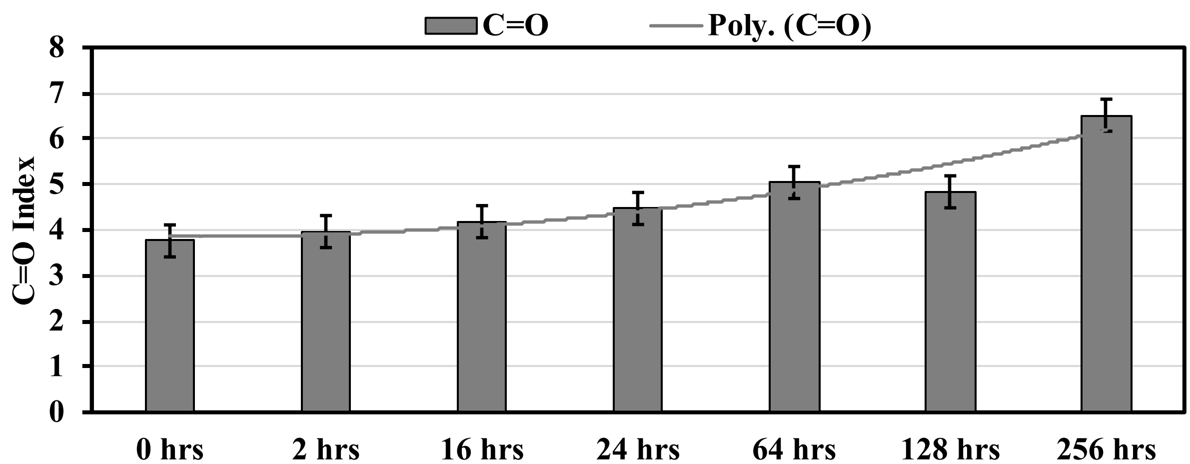


Figure 5.20. C=O Index for Mix 2 Using Protocol 1

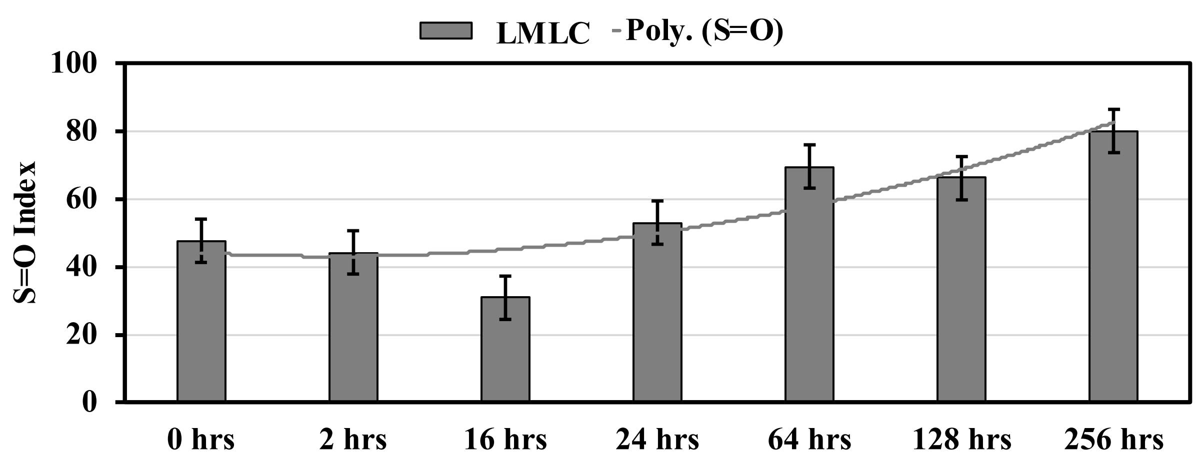
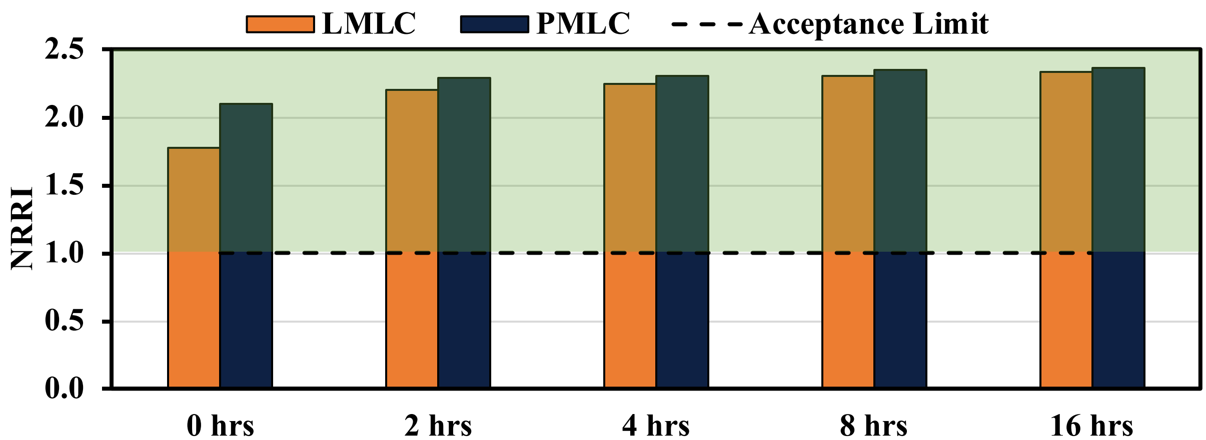


Figure 5.21. S=O Index for Mix 2 Using Protocol 1

Protocol 2: AC Mixture Results

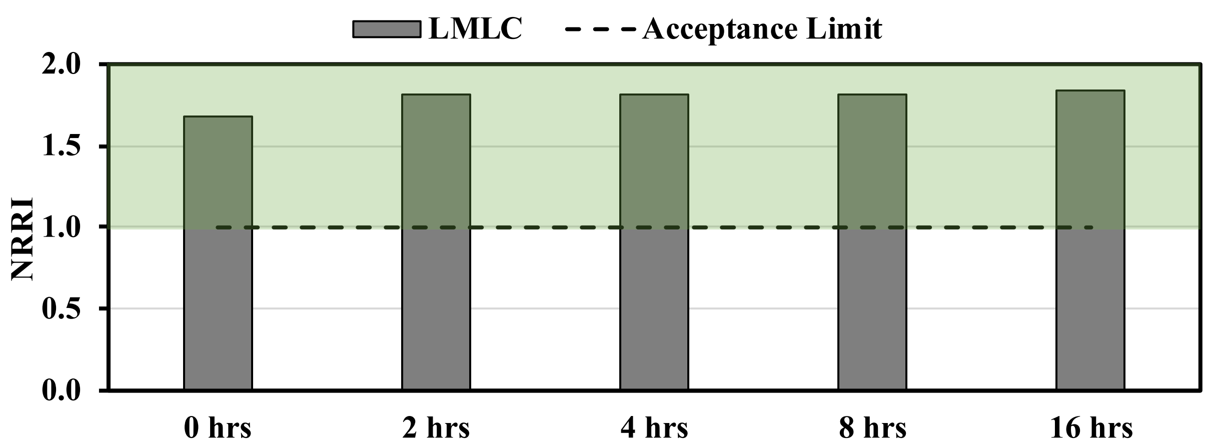
The following section shows the results from the mixture and binder tests of mixtures aged at an elevated temperature of 150ºC. Given the increase in temperature, the aging durations were reduced to a maximum of 32 hrs. Even though it is known the oxidation of the binder could be compromised at higher temperatures, the results are useful to set oxidation benchmarks and evaluate the impact an increase in temperature would have on AC mixtures.

The variations in NRRI from the HWT tests with the aging period are illustrated in Figures 5.22 and 5.23. Similar to Protocol 1, the mixture performance improved with the increase in the aging period. After the recommended 2 hours of oven aging, the NRRI values are reasonably constant.



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

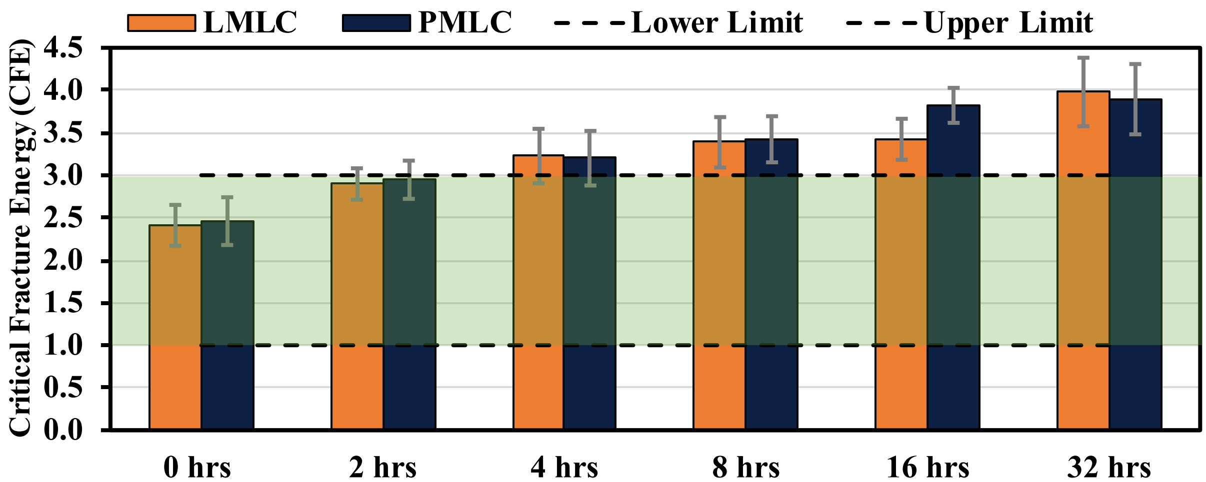
Figure 5.22. HWTT Results for Mix 1 Using Protocol 2



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

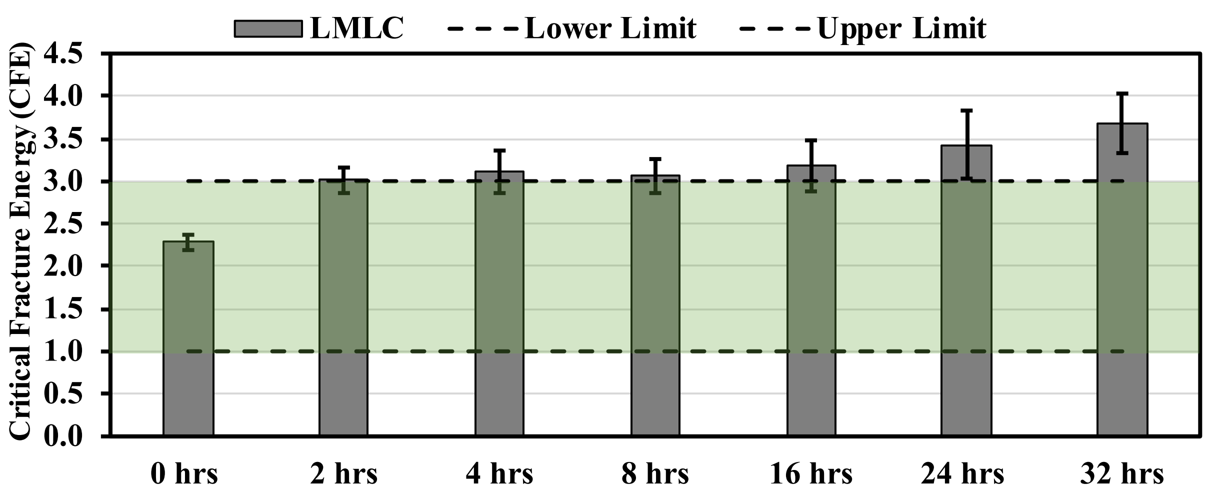
Figure 5.23. HWTT Results for Mix 2 Using Protocol 2

Figures 5.24 and 5.25 show the CFE values for Mix 1 and Mix 2, respectively. The upper acceptance limit is exceeded after 2 hrs of oven aging, much faster than 64 hours observed for Protocol 1.



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.24. OT Critical Fracture Energy Results for Mix 1 Using Protocol 2

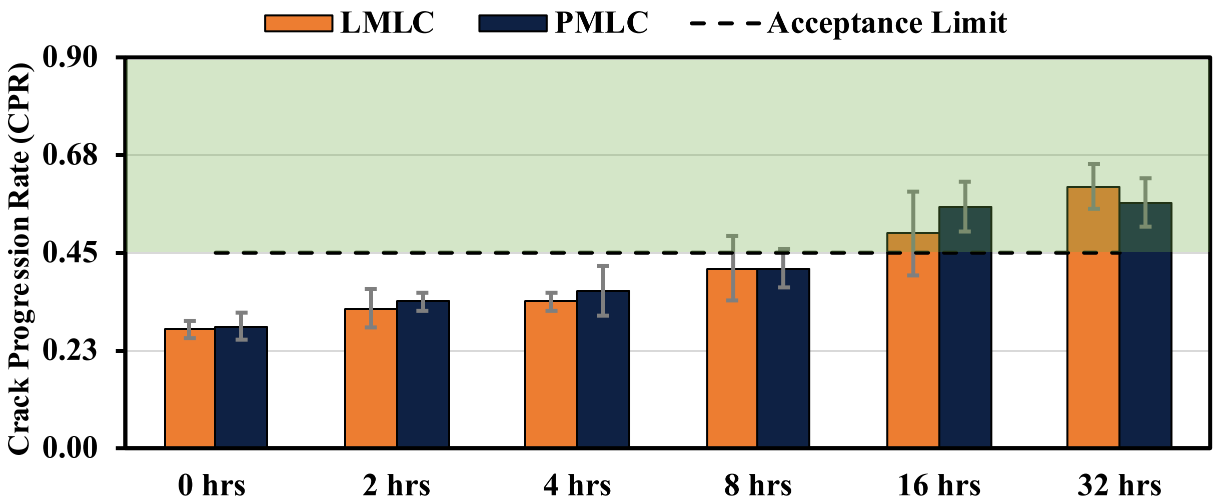


Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.25. OT Critical Fracture Energy Results for Mix 2 Using Protocol 2

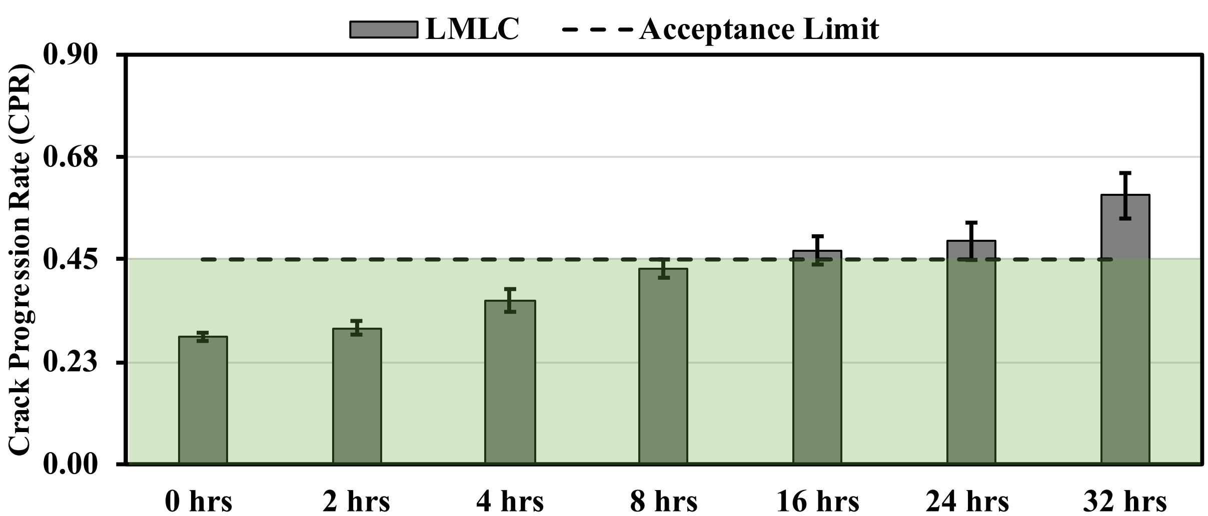
Similarly, as shown in Figures 5.26 and 5.27, the CPR value from the OT exceeds the acceptance limit of 0.45 after 8 hrs of oven aging, as opposed to 32 hrs as per Protocol 1. In addition, the range for the variability of the replicate samples was much higher compared to the lower temperature from Protocol 1.

The IDEAL-CT index is less than the acceptance limit of 80 after 2 hrs of oven aging (as opposed to 8 hrs for Protocol 1) as evident in Figure 5.28 for Mix 1. In contrast, the CT-index is less than the acceptance threshold after 4 hrs of oven aging for Mix 2 as shown in Figure 5.29.



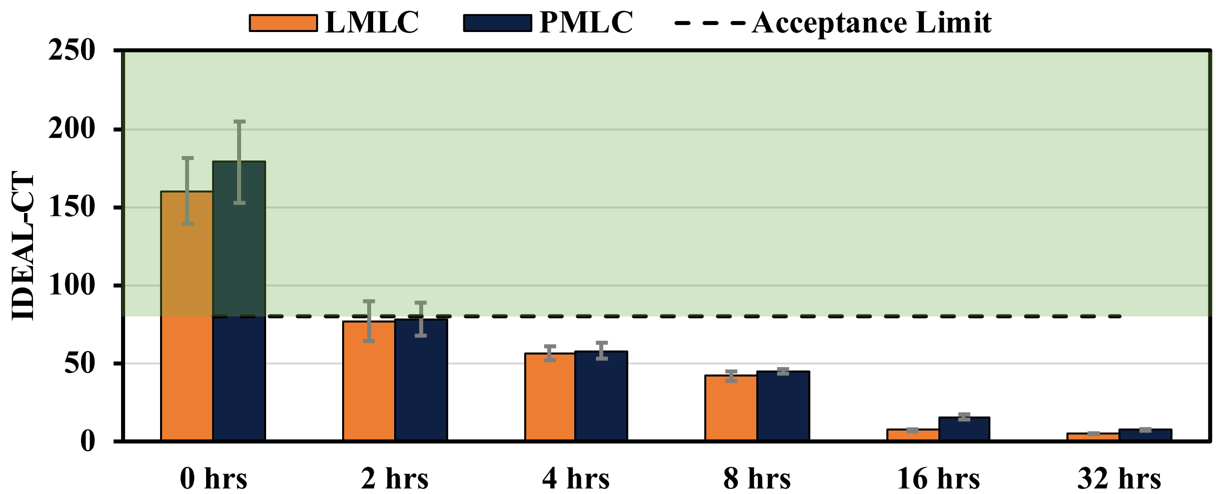
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.26. OT Crack Progression Results for Mix 1 Using Protocol 2



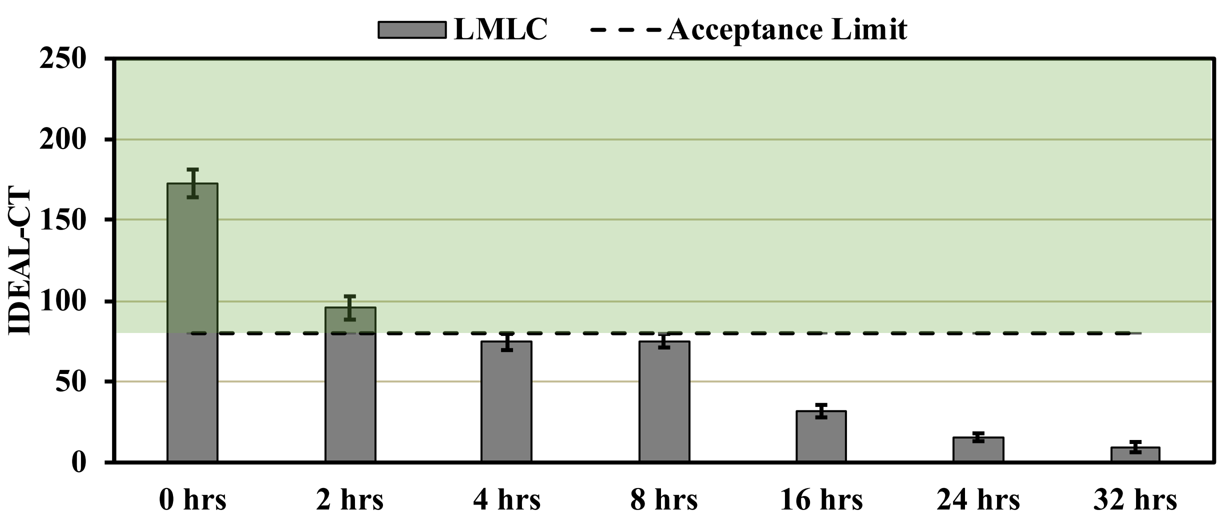
Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.27. OT Crack Progression Results for Mix 2 Using Protocol 2



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

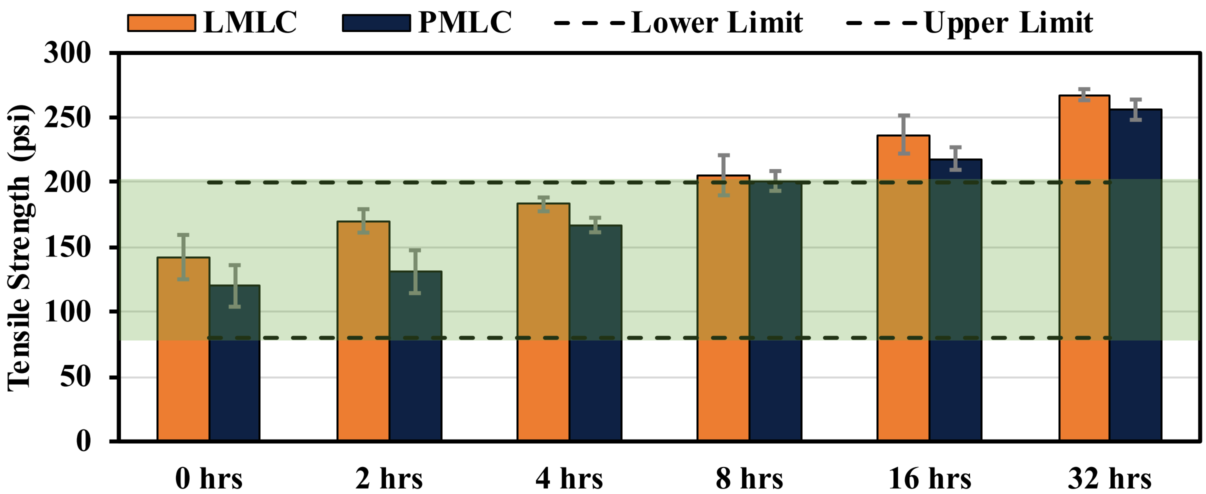
Figure 5.28. IDEAL-CT Value Results for Mix 1 Using Protocol 2



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

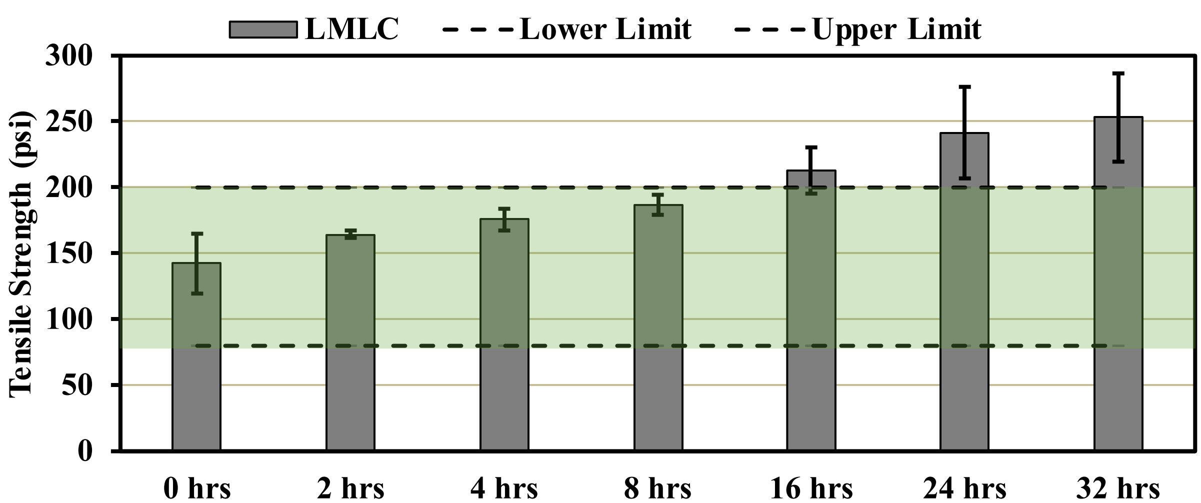
Figure 5.29. IDEAL-CT Value Results for Mix 2 Using Protocol 2

Once again, the IDT tensile strengths presented in Figures 5.30 and 5.31 exceeded the upper limit of 200 psi after 8 hrs for Mix 1. On the other hand, Mix 2 exceeded the limit after 4 hrs of oven aging. The IDT strengths increase much more rapidly as compared to the results from Protocol 1.



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.30. Tensile Strength Results for Mix 1 Using Protocol 2



Note: Acceptable limits shown are based on properties measured after 2 hours of aging and should likely be relaxed for longer aging durations.

Figure 5.31. Tensile Strength Results for Mix 2 Using Protocol 2

Rheological Testing Results

The high- and low-temperature PG of the extracted binders are presented in Figure 5.31, Figure 5.32, and Figure 5.33, respectively. Comparable results were seen for the high and low PG results as in Protocol 1. Parameters corresponding to an increase in stiffness were observed at increased times. However, the rate of increase is considerably higher and more pronounced as the temperature of aging used is higher when compared to Protocol 1.

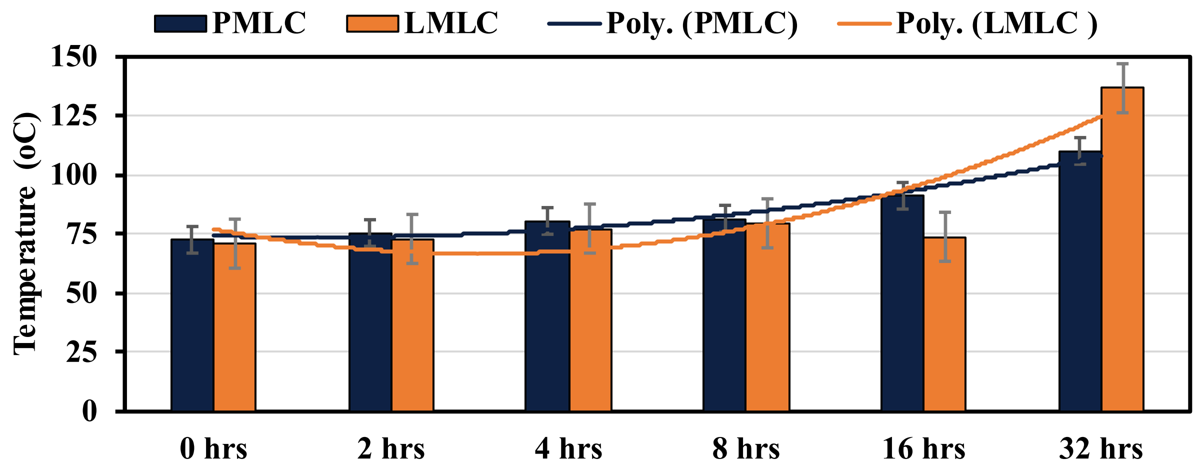


Figure 5.31. High-Temperature PG for Mix 1 Using Protocol 2

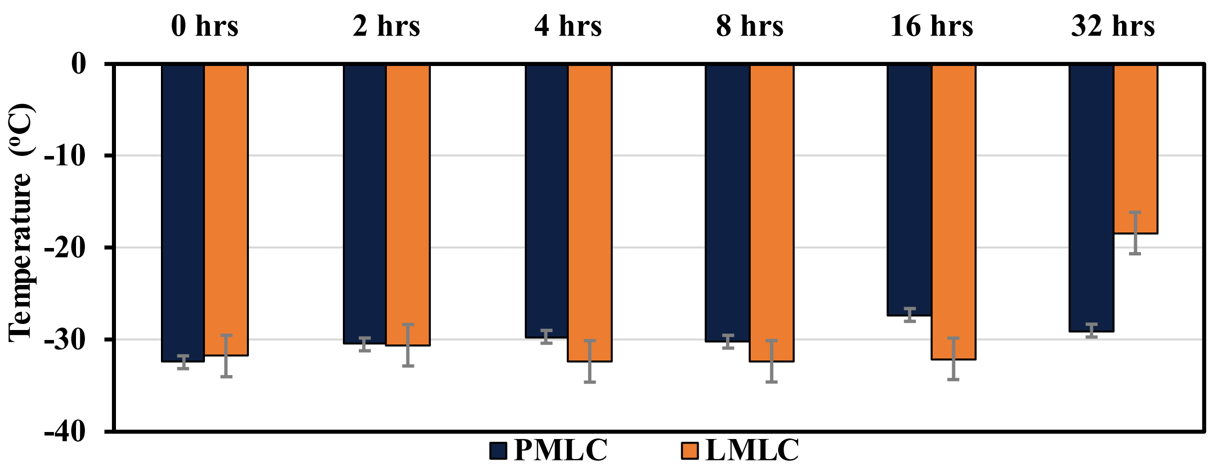


Figure 5.32. Stiffness Based True Low-Temperature PG for Mix 1 Using Protocol 2

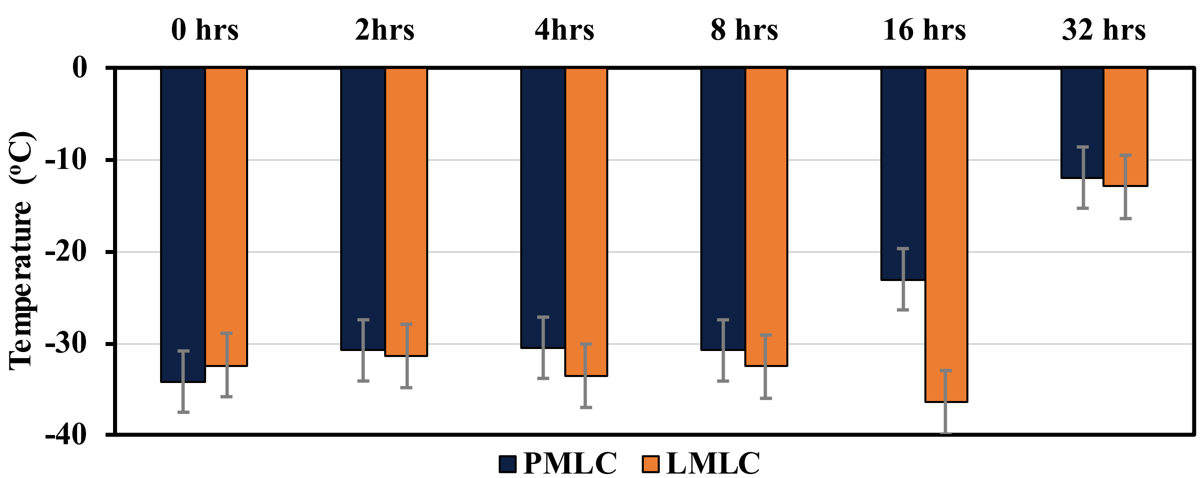


Figure 5.33. m-Value-Based True Low-Temperature Grade for Mix 1 Using Protocol 2

The results for the C=O and S=O indices are presented in Figure 5.34 and Figure 5.35, respectively. Akin to the high PG results, a distinct change in the C=O index was observed at increased time intervals. For the S=O index, a general increase is seen but the results are more variable. Although Protocol 2 is a good method to accelerate oxidation, it may not be a representative method of oxidation that occurs in the field. Studies have shown that using a temperature greater than 100^{o}C could critically disrupt the association of polar molecules and decomposition of sulfoxides, which may lead to a fundamentally differently aged binder than what occurs in the field (*Kim et al., 2018*).

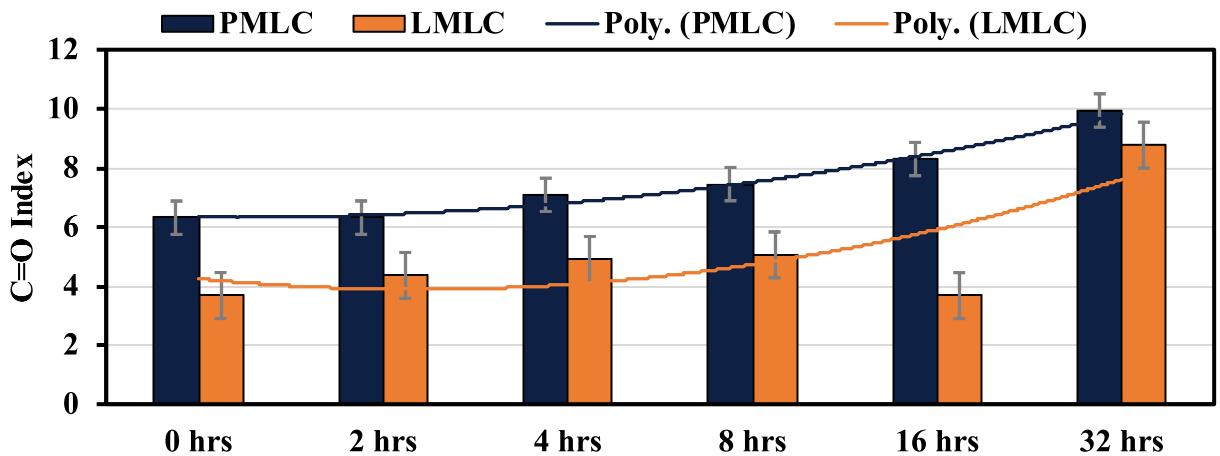


Figure 5.34. C=O Index for Mix 1 Using Protocol 2

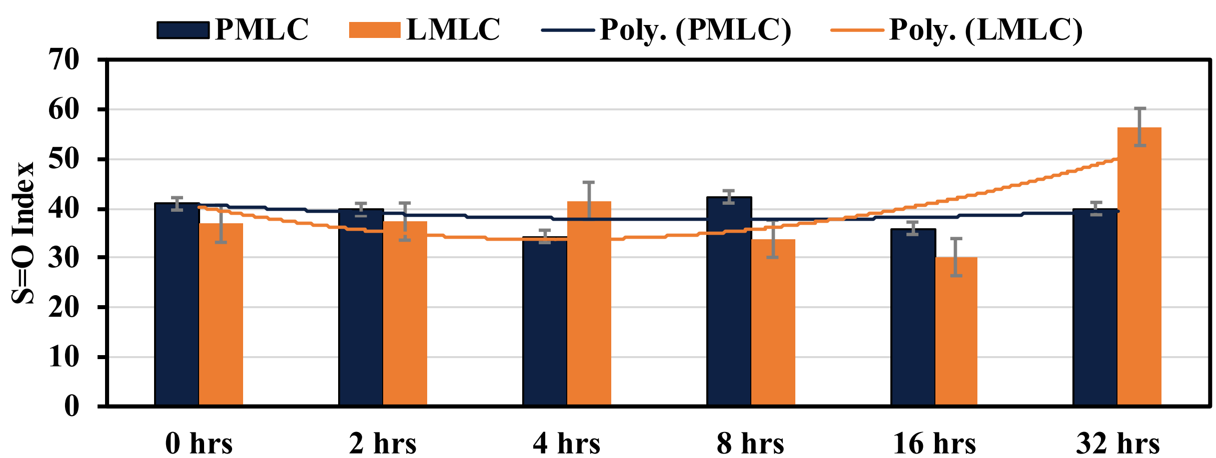


Figure 5.35. S=O Index for Mix 1 Using Protocol 2

Protocol 3: Pressure Aging Device Results

Methodology

One way to accelerate aging would be to increase temperature and thereby increase the rate of oxidation. However, as mentioned earlier using a temperature greater than 100°C could critically disrupt the association of polar molecules and increase the effective concentration of mobile reactive species that may lead to a fundamentally differently aged binder than what occurs in field. Another influencing factor would be to pressurize the specimen that can similarly increase the rate of oxidation. Hence, the research team also proposed an alternative aging method that uses both temperature and pressure to accelerate the aging process. The research team also proposed an alternative aging method that uses temperature and pressure to accelerate the aging process. Figure 5.36 illustrates the pressure device along with the current setup. To assure the mixture is evenly aged, a stainless-steel stand with 7/32 in. holes on each plate was used to allow airflow. The loose mixture was then put into each level to seal the tank with constant pressure and temperature.

Figure 5.36. Stainless Steel Stand and Pressure Device Setup

Aging Process

This protocol follows the aging process from NCHRP 09-54 to satisfy short- and long-term aging requirements. For the NCHRP protocol, the specimen is exposed to 4 hrs of short-term aging at 135ºC, while maintaining the thickness of the material being aged to less than 2 in. Next, the AC mixture is put into a pressurized tank that ages the loose mixture at the predefined curing conditions. Three sets of IDT specimens using Mix 1 were made to determine the uniformity of aging between the top and bottom tiers of the steel stand. Figure 5.37 illustrates that the tensile strengths from the top and bottom tiers are comparable and the aging process is repeatable since the IDT strengths vary between109 psi and 116 psi for the top tier specimens and between 106 psi and 115 psi for the bottom tiers.

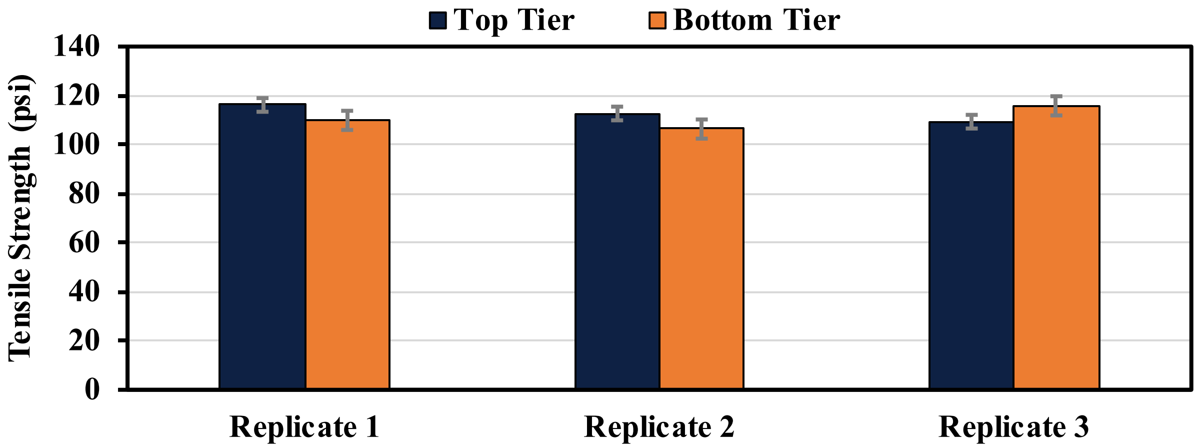


Figure 5.37. Tensile Strength Comparison Using Protocol 3

Once the pressure device demonstrated consistent aging throughout the device a separate study was performed to determine the optimum pressure level. The pressure device is able to withstand pressure levels up to 120 psi. Mix 1 (El Paso) PMLC samples were used for this purpose. Figure 5.38 shows the IDEAL-CT indices of a few of the iterations. The NCHRP 09-54 method requiring 5 days of aging at 95ºC was used as a benchmark. The results show the specimens aged for 24 hours was not adequately aged, while those aged for 48 hours met or exceeded the benchmark IDEAL-CT index.

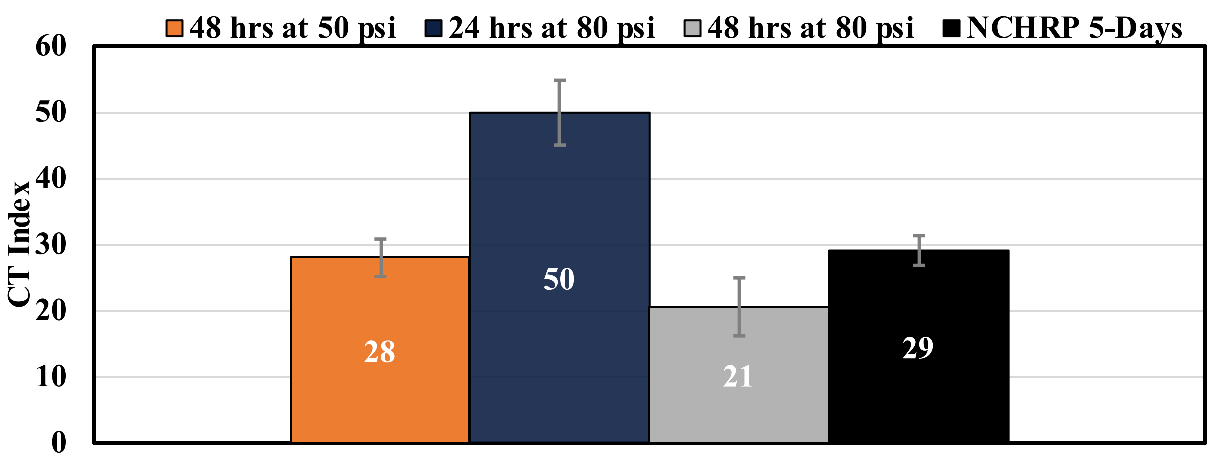


Figure 5.38. IDEAL-CT Index Value Comparison Using Protocol 3

The IDT tensile strength comparison between different aging processes is presented in Figure 5.39. These initial results indicate that Protocol 3 could surpass the benchmark tensile strength within 48 hrs. Following the recommended curing conditions for Protocol 3, 95°C with 80 psi of pressure, the aging time is reduced to two days.

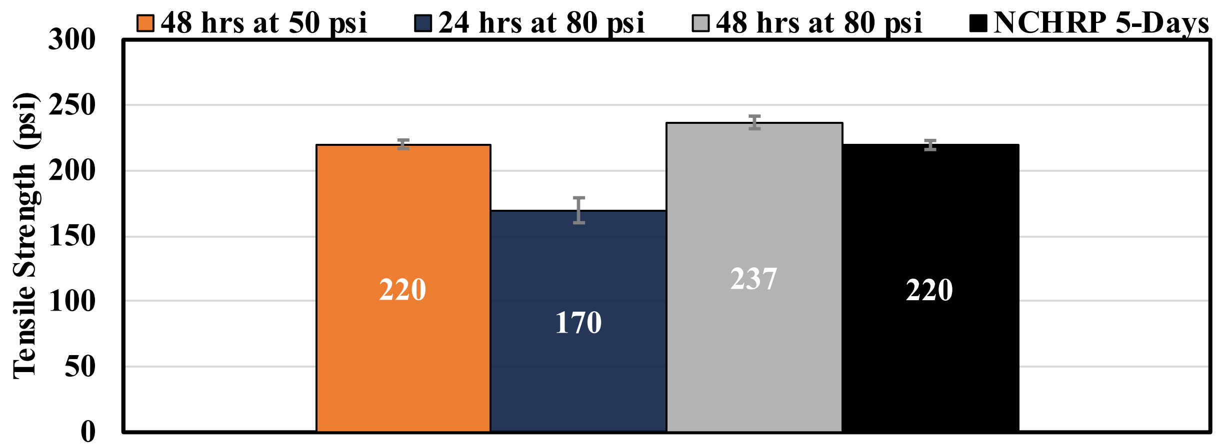


Figure 5.39. IDT Strength Comparison Using Protocol 3

Materials

An additional five mixtures were evaluated with different binder PG, aggregates sources, and admixtures to assure the feasibility of this method. Two of the Five mixtures (No. 1 and No. 5) have the same characteristics and gradation from Mix 1 and Mix 2, respectively. Table 5.2 presents the details related to the constituents of these five mixtures. Each mix was subjected to aging regimes of NCHRP 09-54 (5 days of aging at 95ºC) as a benchmark, and 24 hrs and 48 hrs of aging at 80 psi.

Table 5.2. AC Mixtures for Validation Using Protocol 3

No.	Aggregate Type	Binder Grade	Mix Design Characteristics
1	Limestone	PG 70-22	SP-C: 10% RAP, HydroFoam IEQ, 5.4% AC
2	Limestone	PG 70-22	SP-C: 20% RAP, HydroFoam IEQ, 5.1% AC
3	Igneous	PG 70-22	SP-C: 15% RAP, N/A, 5.3% AC
4	Igneous	PG 76-22	SP-C: 10% RAP, N/A, 5.4% AC
5	Limestone/Igneous	PG 70-22	SP-C: 15% RAP, Evotherm, 5.5% AC

The IDEAL-CT indices and IDT tensile strengths at different aging conditions are compared in Figures 5.40 and 5.41, respectively. Four of the five mixes met or exceeded the benchmark values in 48 hours or less. The abnormal behavior from Mix 4 could be from the increase in the binder's high PG.

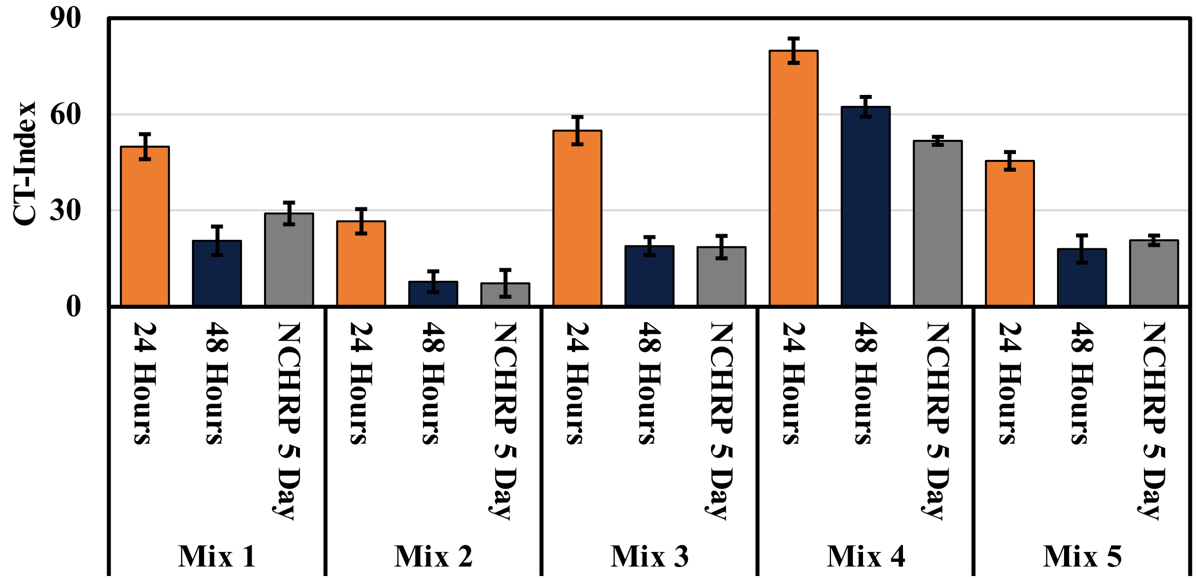


Figure 5.40. IDEAL-CT Index Value Comparison Using Protocol 3

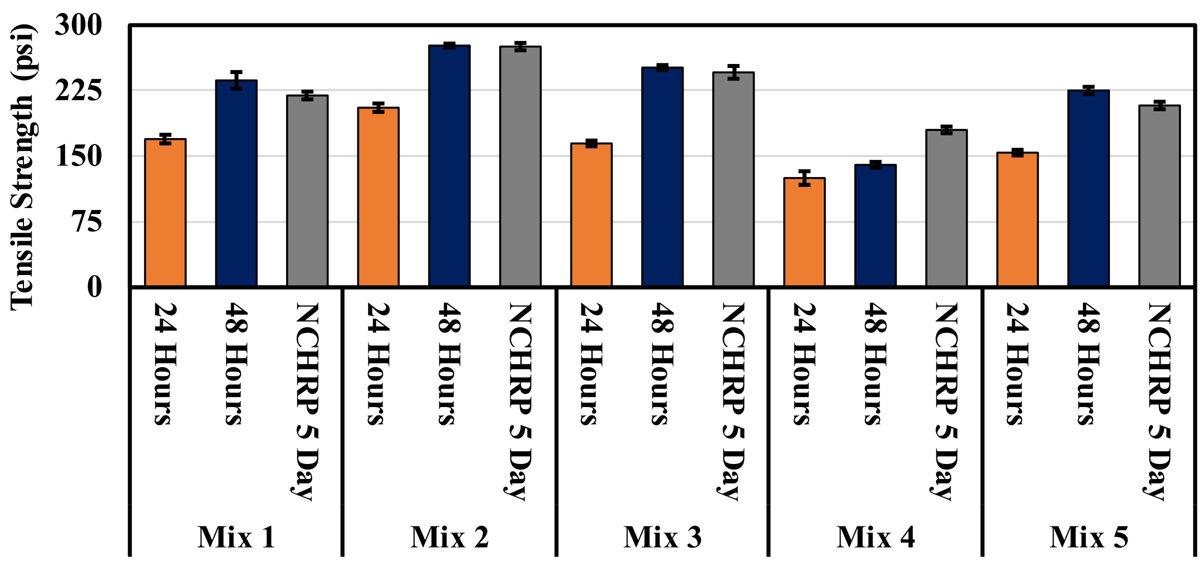


Figure 5.41. IDT Strength Comparison Using Protocol 3

Protocol 4: Ozone Enriched Device

For this protocol, an experimental method was developed for the accelerated simulation of long-term aging of loose asphalt mixtures using highly oxidative gas within one day (24 hrs). The feasibility of this method was verified through mixtures that were prepared using five different binders. The chemical and rheological properties of the binders were evaluated post aging and compared to corresponding binders extracted from mixtures aged using the protocol recommended by NCHRP 09-54, i.e., oven aging of loose mixtures at 95ºC for five days. Some preliminary information regarding the setup of this device and results are presented in the section below.

Materials

Five different binders were used to prepare the asphalt mixtures in this preliminary evaluation. Table 5.3 presents the details related to the constitution of these five binders. Binder-4 and Binder-5 were prepared by blending the indicated amount of SBS modifier with an unaged binder using a high shear mixer for two hours at around 170ºC. The asphalt mixtures were prepared using locally available non-absorptive igneous aggregates. The binder content used for the mixtures was 5.3% by weight of the mix. Figure 5.42 presents the gradation curve of the mixture, with the maximum nominal aggregate size of 0.5 in. (12.5 mm).

Table 5.3. Binder Names and Constituents

Binder Name	Binder Grade	Comments
Binder-1	PG 64-22	The binders were from different producers and very likely unmodified
Binder-2	PG 64-22	The binders were from different producers and very likely unmodified
Binder-3	PG 70-22	This binder was from a third producer and was likely polymer modified
Binder-4	PG 64-22+ 1.5 % SBS	These two binders were polymer-modified in the lab
Binder-5	PG 64-22 +3% SBS	These two binders were polymer-modified in the lab

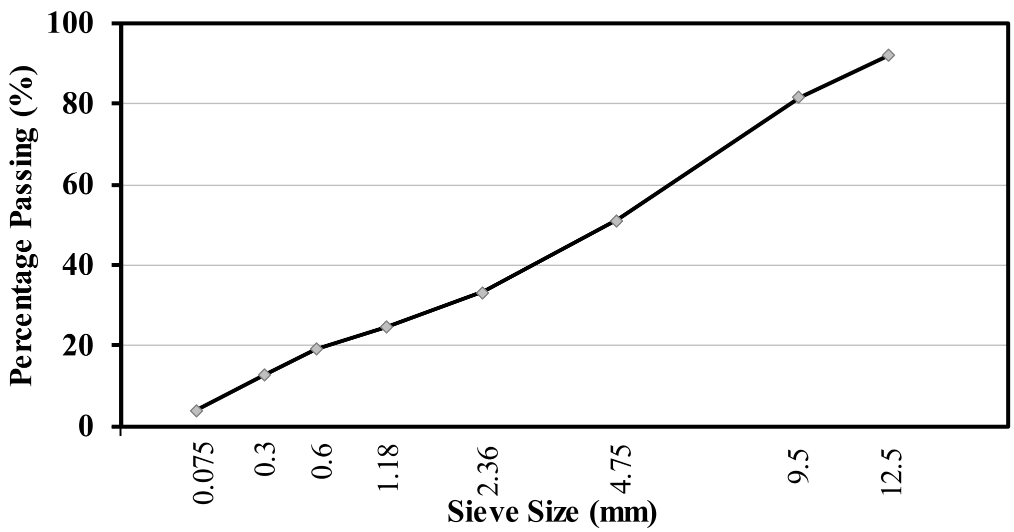


Figure 5.42. Grading Curve of Mixtures Using Protocol 4

Experimental Methods: Accelerated Aging Using ROS

Figure 5.43 illustrates the setup and equipment used for the accelerated aging method used in this study. The three main stages involved in the process were: (i) the production of ROS, (ii) the aging of loose mixtures in an enclosed and heated tank, and (iii) the degradation of oxidative gases for release into an exhaust hood. The tubing and fittings used in the setup were chosen to be compatible with ROS, in other words, resistant to corrosion.

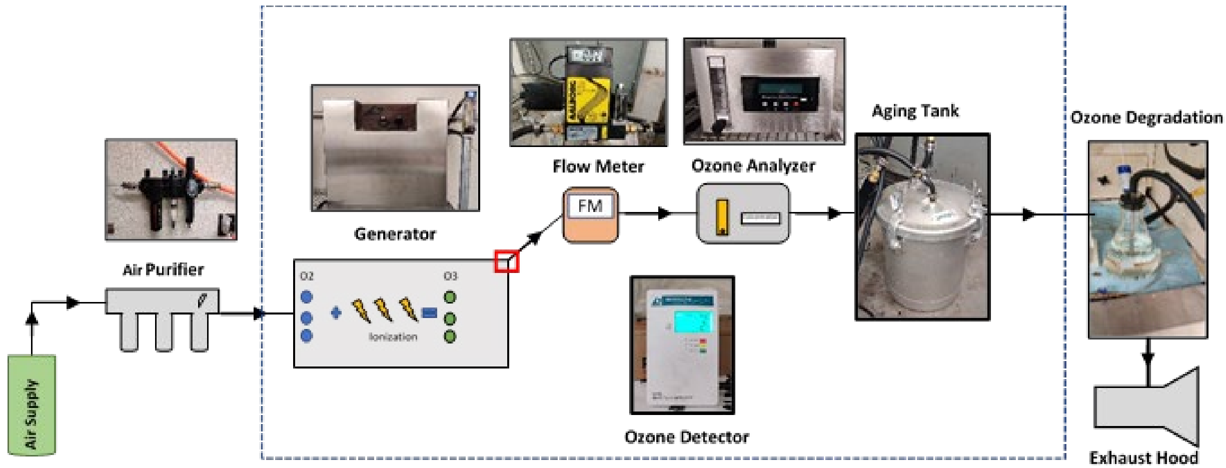


Figure 5.43. Setup of the Accelerated Aging Procedure

The ROS for the accelerated oxidation of the loose mixtures were produced using an ozone generator. The generator used in this study was purchased from Absolute Ozone® (Model Atlas 30) which has a maximum ozone production capacity of up to 50 g/h. Compressed air at a pressure of 20 psi was passed through the ozone generator which produces two types of ROS comprising O_3 and NO_x (Mirwald et al., 2020). Following this, the oxidative species were led to a mass flow controller that regulates the flow of gas into the aging tank to 100 ml/min. The flow controller used was purchased from AALBORG Instruments and Controls, Inc. (Model GFCS-020923) with a maximum flow rate of 500 ml/min. The ozone concentration in the system was measured using an ozone analyzer purchased from Guangzhou Zeao Ozone Equipment Co. Ltd (Model ZA-UV 300B). The equilibrium ozone concentration at the generator output and flow rate specified above was measured to be 1.15 mg/L. Note that the ozone analyzer was used to record the concentration of ozone for calibration purposes. Although, the use of this is analyzer in the aging manifold is not operationally necessary.

Loose asphalt mixture samples of 1.2 kg were placed in a galvanized steel tank with a volume of 2.5 gallons (9500 cm^3). The tank was customized to allow for continuous flow of oxidant species from an inlet to an outlet. The tank was heated using a heating blanket and the temperature inside the tank was maintained at 95ºC. As the temperature is a vital design component in the setup, standalone mini data loggers manufactured by iButton® were used to monitor and validate the temperature inside the tank. The data logger was placed among the loose asphalt mixture during multiple experiments. It was confirmed that the temperature of 95˚C was maintained inside the tank throughout the testing period.

The oxidative species that pass the outlet of the aging tank were degraded before being led to an exhaust. This was accomplished using a washing flask with diesel engine fluid as previously shown in Figure 44. An ozone detector manufactured by Absolute Ozone® (Model AOM3000) with a measuring range between 0-5000 ppb was placed near the vicinity of the manifold as a safety precaution and to ensure that there was no ozone being detected in the air.

Samples of 1.2 kg of loose mixtures were also long-term aged using the loose mixture aging protocol recommended in the NCHRP 09-54 report. In this method, the loose asphalt mixtures were oven aged for five days at 95°C. The period chosen was based on the climatic region of this study and it is known to simulate aging of around eight years at a depth of 20 mm beneath the pavement.

Aging Procedures

All loose mixtures were initially short-term aged by placing them in an oven for 2 hrs depending on the PG of the binder as per local specification Tex-206-F. A loose mix sample of 1.2 kg for each mixture type was then long-term aged using the accelerated aging method for 24 hrs. The period of 24 hrs included approximately 2 hrs that were required to reach thermal equilibrium. Similarly, the same quantity from each mixture was also long-term aged using the loose mixture aging protocol as per NCHRP 09-54 for five days at 95ºC. Following long-term aging, the binders from the various mixtures were extracted as per Tex-210-F.

The binders aged using the two different long-term aging protocols were evaluated per the techniques described earlier to determine the extent of carbonyl and sulfoxide groups, the results are presented in Figures 5.44 and 5.45, respectively. The effectiveness of the ROS aging method for accelerated oxidative aging can be seen when comparing the indices from the two long-term aging methods. For the different binders tested, the binders aged using the ROS-based method (labeled ROS Aging-24 hrs) showed a similar or higher carbonyl index as compared to those aged using the five-day aging protocol (labeled 5-day Aging). When comparing the sulfoxide index, three of the five binders tested showed a higher S=O index and two of the binders showed similar or slightly lower S=O index as compared to the five-day aging method. It must be mentioned that the level of oxidation and kinetics of aging exhibited by the binders are a function of different factors such as sulfur content, availability of oxygen, and diffusion. Hence, the same binder can show different quantitative levels of these indices when exposed to different experimental conditions.

Figure 5.46 illustrates the general Aging Index (AI) values of the various binders. This parameter serves as a combined indicator for the extent of cumulative aging of the binders when combining both oxidation indices. The carbonyl and sulfoxide indices obtained were then used to calculate an overall AI, which is an indication of the total oxidation exhibited by the binder using Equation 5.5:

$$AI = \frac{IA_{C=O} + IA_{S=O}}{IA_{CH_3}} \tag{5.5}$$

From Equation 5, CH_3 is the aliphatic vibration ranging from 1350 to 1525 cm^{-1}. The various testing (chemical and rheological) results presented in this study represent a mean result of two tests performed.

Comparable levels were obtained for both sets of binders, which confirms that the accelerated aging method using ROS can oxidize mixtures to comparable or higher aging levels as the five-day oven aging method. The effect of this oxidation on the rheology is discussed in the next section.

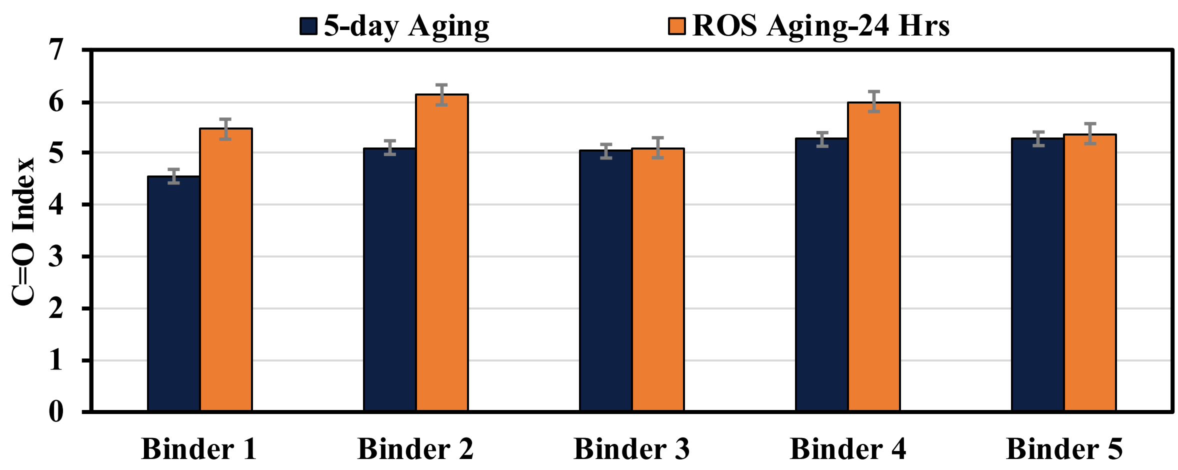


Figure 5.44. Carbonyl Index of AC Binders Using Protocol 4

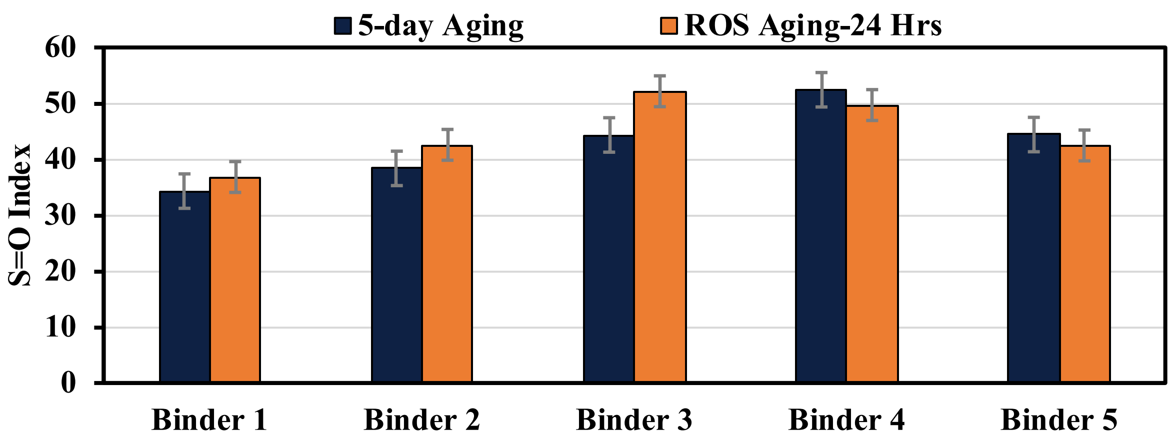


Figure 5.45. Sulfoxide Index of AC Binders Using Protocol 4

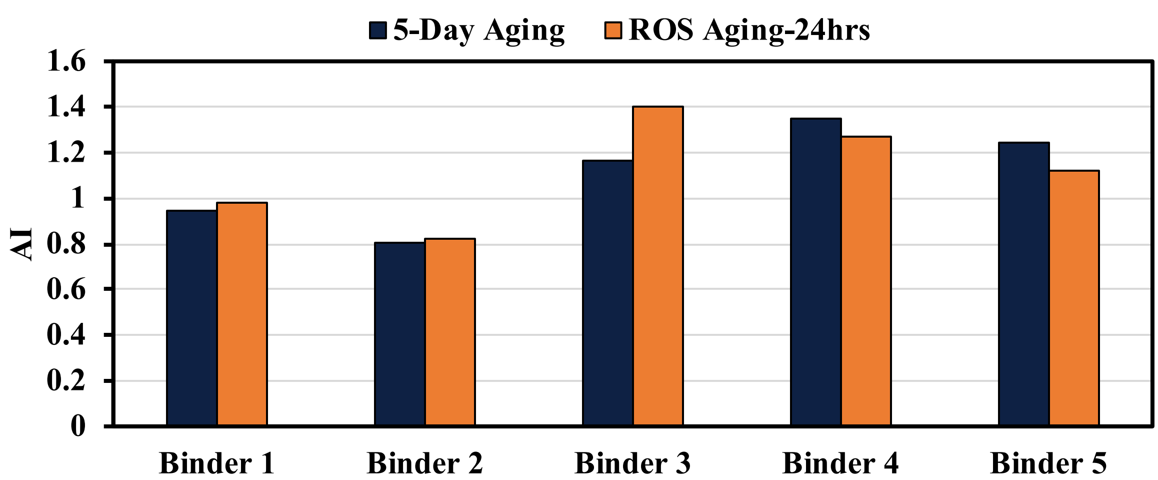


Figure 5.46. Average Aging Index of Binders

Rheological Properties

The rheological properties of the extracted binders along with the short-term aged binder were measured using a DSR. When comparing the extracted binders from the aging protocols, it was seen that in most instances, the binders aged using the ROS method showed a greater high-temperature PG compared to the binders from the five-day aging method, as shown in Figure 5.47. For Binder 1, Binder 2, and Binder 4 the high-temperature PG measured was higher after ROS aging, while Binders 3 and 5, the high-temperature PG was comparable when using both methods. The largest difference was seen for Binder 4 where the high-temperature PG evaluated using the ROS aging method was 96ºC as compared to 85ºC for the five-day aging method.

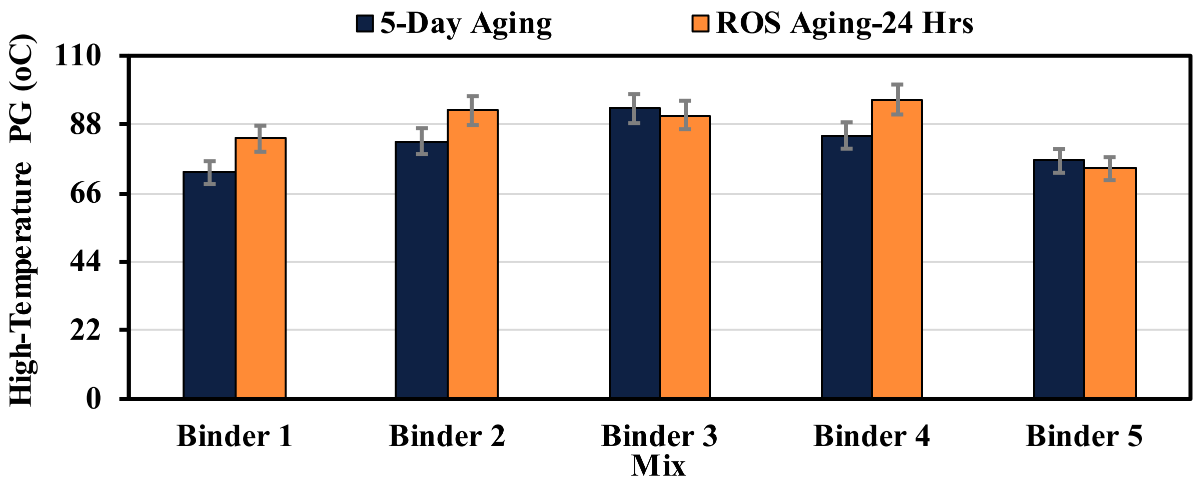


Figure 5.48. High-Temperature PG of AC Binders Using Protocol 4

The low-temperature properties of the extracted binders were also evaluated. The stiffness and m-value true grades of the binder are presented in Table 5.4. For Binder 1, Binder 2, Binder 3, and Binder 4, the stiffness and m-value true grades were seen to be higher using the ROS aging method as compared to the five-day aging method. For Binder 5, the results were the opposite.

Table 5.4. Low Temperature PG of AC Binders Using Protocol 4

Binder Property	Stiffness true grade (ºC)	m-value true grade (ºC)	Stiffness true grade (ºC)	m-value true Grade (ºC)
Aging	*5-Day*	*5-Day*	*ROS-24 Hrs.*	*ROS-24 Hrs.*
Binder 1	-28.1	-27.9	-25.4	-15.1
Binder 2	-36.7	-32.8	-30.2	-23.4
Binder 3	-30.4	-24.9	-29.9	-25.2
Binder 4	-27.1	-23.6	-26.6	-16.6
Binder 5	-28.4	-23.6	-30.1	-28.1

Steady State of AC Mixture

More relevant to Protocol 1 and Protocol 2, it is hypothesized that the aging kinetics of binder reaches a steady-state condition after a certain amount of aging, and hence there could be a level of long-term aging after which the rank order of aging of different binder-mixtures does not change. For a cracking test to distinguish accurately between crack-resistant and -susceptible mixes, it is critical but adequate that the mixes are aged to achieve this steady-state rate even if the mixes do not reach a state that is equivalent to 10 or 20 years of field aging. Figure 5.49 illustrates an analysis of the aging kinetics for different binder-mixtures and their respective performance (e.g., cracking potential) rankings at different aging levels to develop optimized laboratory aging protocols based on the steady-state aging condition of asphalt mixtures and binders.

From the results obtained, Protocol 2 proved largely inadequate to capture the realistic effects of aging on mixtures. Protocol 1 provided satisfactory results and it seems likely that a steady state of asphalt mixture could exist after 24 hours of oven aging. Moreover, Protocol 3 and Protocol 4 provide a useful benchmark for the accelerated aging of mixtures within a short period that is more representative of the long-term aging. Discussions regarding the optimization of these different techniques were continued in subsequent tasks.

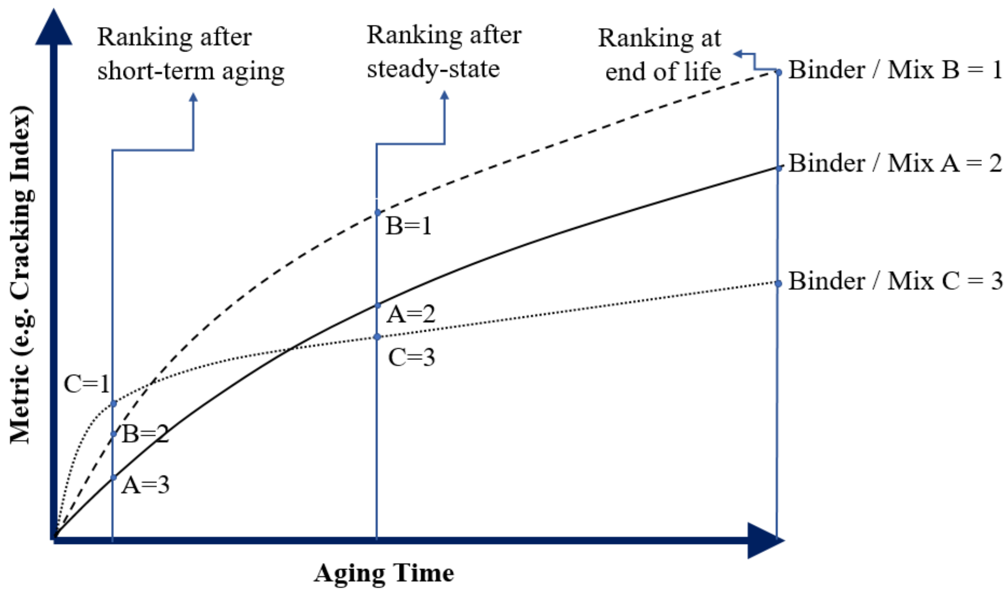


Figure 5.49. Steady-State Aging of Asphalt Mixtures

Summary

This chapter documented the findings from the initial evaluation. Four different aging protocols to simulate the aging characteristics of AC mixtures were deliberated. These protocols differed in terms of the temperature, duration, pressure, and aging environment. From the range of materials tested in this study, the main findings were as follows:

- Protocol 1 appears to be a promising protocol for routine rank ordering and extended evaluation of this approach were conducted further.
- Mixture and corresponding binder tests showed a consistent increase in aging-related metrics with increased testing time. A preliminary analysis indicated that a minimum time for mixing to achieve steady state would be close to 24 hours. The research team believes that a steady state of aging may be adequately reflected after a period of 24 to 32 hours during Protocol 1 for relative comparison of different mixes. This was further investigated using a diverse set of mixes in the extended evaluation.
- Protocol 3 and Protocol 4, using pressurized aging vessel and ozone gas respectively, also showed promise as a technique that can accelerate the actual aging and was further considered.

- The research team does not recommend Protocol 2 to be considered further in this project as the results were unsatisfactory and the technique might not be scientifically sound for routine testing.

Chapter 6. Selection of Preliminary Optimized Laboratory Aging Protocols

This chapter documents the findings from activities to propose optimized aging methods to assess the short- and long-term aging behavior of mixtures and binders. This chapter also documents the preliminary guidelines and specifications for implementing optimized laboratory aging protocols with more specific parameters.

To assess the early-age rutting and long-term cracking potential of mixtures, the research team recommends evaluating various mixtures with different mixture variables as shown in Figure 6.1.

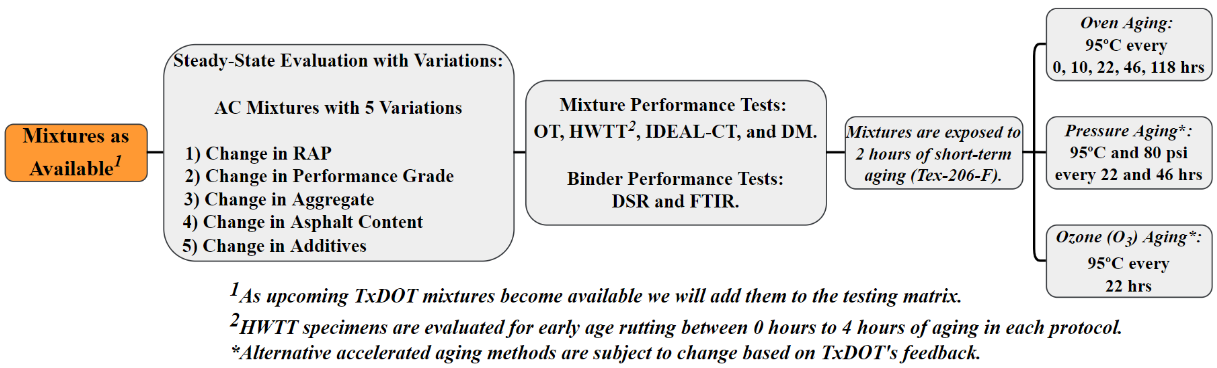


Figure 6.1. Extended Evaluation Overview

The research team evaluated mixtures by varying several mixture variables including:

- *RAP Contents:* To delineate the aging potential of asphalt mixtures containing recycled components, mixtures with different sources of RAP and varying RAP contents ranging from 0% to 30% were used.
- *Performance Grades:* To include binders with differing sensitivity to aging and grades. PG 64-22, PG 70-22, and PG 76-22 binders were selected. Changes in the sources from binder grades were evaluated to understand the behavior of both mixtures and binders.
- *Aggregate Sources:* Since the aging potential of asphalt mixtures is significantly influenced by the absorption of the aggregates, absorptive (primary limestone) and nonabsorptive (e.g., granite or sandstone) aggregates were considered in the experiment design protocol. Additionally, using aggregates with different surface aggregate classification (SAC) were considered.
- *Asphalt Contents:* The aging behavior of binders can be influenced by the thickness of the binder film in the mixtures. The research team also changed the asphalt content of mixtures to evaluate their aging potential.
- *Additives:* The aging behavior of mixtures and binders can be influenced by additives such as rejuvenators or recycling agents in the mix. The modified mixtures with commercially available rejuvenators were evaluated to understand their aging characteristics.

Methodology: Protocols

The research team evaluated AC mixtures using the three different protocols summarized in Table 6.1. Protocol 2 and Protocol 3 were mainly used to provide the long-term accelerated aging of mixtures within a short period.

Table 6.1. Optimized Laboratory Aging Protocol

Protocol No.	*Temperature*	*Pressure*	**Curing Times**
1	Cured at 95°C	**-**	0, 10, 22, 46, 118 hours
2	Cured at 95°C	80 psi	22 & 46 hours
3	Ozone and ROS (O_3)	Ozone and ROS (O_3)	22 hours

Protocol 1: Conventional Oven Aging

In Protocol 1, similar to the NCHRP 09-52 project, loose mixtures were continuously aged at 95℃ in a draft oven as shown in Figure 6.2. The materials were periodically withdrawn and evaluated to assess the early-age behavior of mixes and the probability that the mixes have achieved steady-state aging. This information was then used to identify the optimal aging time to delineate asphalt mixtures with good and poor cracking characteristics.

Figure 6.2. Loose Mixture Aging in Oven (*Protocol 1*)

Protocol 2 (Protocol 3 in previous chapter): Pressure Aging Device

A customized pressure chamber with loose asphalt mixtures were placed inside an oven, as shown in Figure 6.3. The temperature was maintained at 95ºC and at the constant pressure of 80 psi.

Figure 6.3. Pressure Aging Device (*Protocol 2*)

Protocol 3 (Protocol 4 in previous chapter): Ozone Enriched

As shown in Figure 6.4, this protocol employed highly oxidant gas to simulate the long-term aging of asphalt mixtures. Atmospheric chemistry reveals that the oxidation potential of oxygen (O_2) is very low at temperatures 200ºC, but there are other gases with much higher oxidative potential. These ROS's, such as ozone (O_3) or nitrogen oxides (NOx) are particularly important in the tropospheric oxidative cycle.

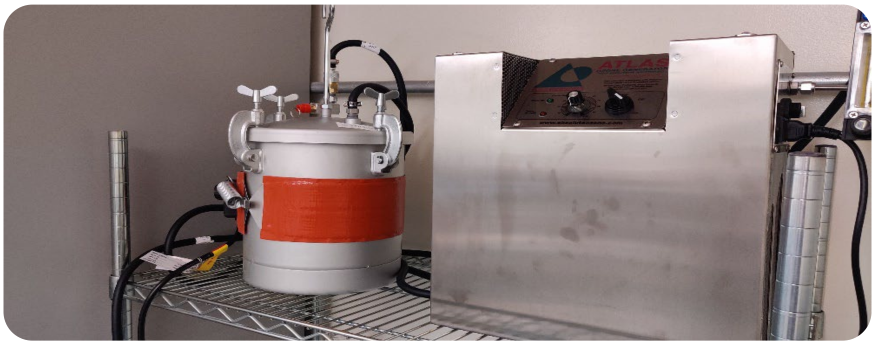


Figure 6.4. Ozone Aging Device Setup (*Protocol 3*)

Performance Test Methods

The following test methods were used for the aging-related testing of AC mixtures

- *Hamburg Wheel-Tracking (HWT) Test (Tex-242-F)* to evaluate AC mixtures' rutting susceptibility and moisture damage.
- *Overlay Tester (Tex-248-F)* to evaluate the susceptibility of an AC mixture to fatigue or reflective cracking.
- *IDEAL-Cracking Test (Tex-250-F* to evaluate the cracking resistance of the AC mixtures.

The following tests were conducted on asphalt binder specimens recovered from corresponding mixtures subjected to different aging conditions. The Tex-210-F procedure were followed to extract and recover the asphalt binder samples from the aged asphalt mixture.

- *Performance Grade (PG):* The high-temperature grades were evaluated using a Dynamic Shear Rheometer (DSR) as per Superpave specification incorporated in TxDOT Item 300. Since the tests were conducted using small samples of recovered asphalt binder from loose aged mixes, the low-temperature properties were evaluated using a DSR with a 4mm parallel plate geometry.
- *Fourier Transform Infrared (FTIR) Spectroscopy:* FTIR tests were conducted to evaluate the oxidation-related chemical functional groups in different asphalt binders and correlate the different levels of oxidation products formed in the asphalt binder.

Further Investigation into the Steady State of AC Mixture

Relevant to Protocol 1, it is hypothesized that the aging kinetics of binder reaches a steady-state condition after a certain amount of aging, and hence there could be a level of long-term aging after which the rank order of aging and consequently cracking characteristics of different binder-mixtures does not change. For a cracking test to distinguish accurately and rank order crack-susceptibility of mixes, it is adequate that the mixes are aged to achieve this steady-state rate even if the mixes do not reach a state equivalent to 10 or 20 years of field aging. Figure 6.5 illustrates an analysis of the aging kinetics for different binder-mixtures and their performance (e.g., cracking potential) rankings at different aging levels to develop optimized laboratory aging protocols based on the steady-state aging condition of asphalt mixtures and binders. The outcomes from the preliminary investigation indicated that a steady state of asphalt mixture could exist after 24 hours of oven aging. The research team explored this hypothesis in this section to formulate more robust and generalizable conclusions.

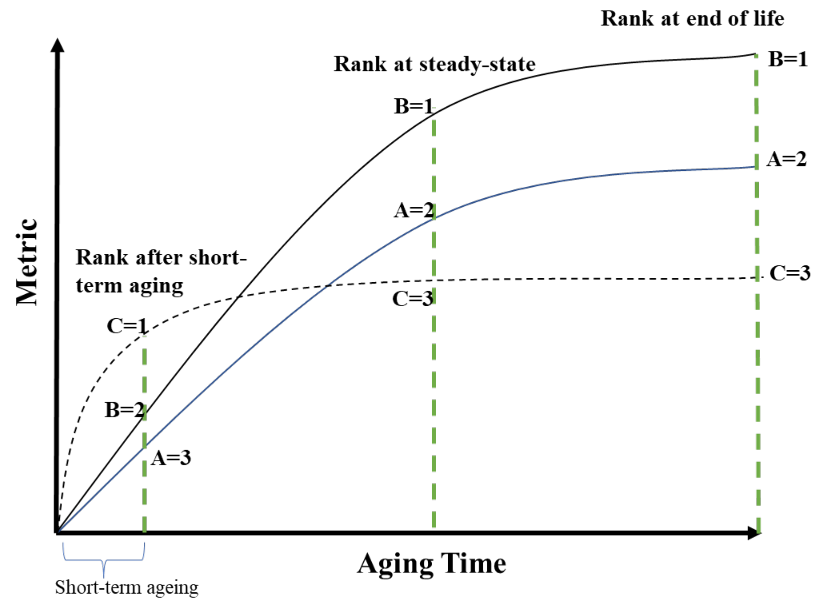


Figure 6.5. Steady-State Aging of Asphalt Mixtures

Evaluation of Steady-State: Mixture Performance Results

Asphalt mixtures with four different RAP contents of a mix (0%, 10%, 20%, and 30%) with a PG 70-22 binder were oven aged following Protocol 1 for different periods. The IDT tensile strength comparison on triplicate specimens between different RAP content is presented in Figure 6.6. The variations in the OT's CPR and CFE values with the RAP contents are shown in Figures 6.7 and 6.8. As hypothesized, the rank order of samples at 24 hours was identical to the rank order at around 120 hours for both IDT and OT results.

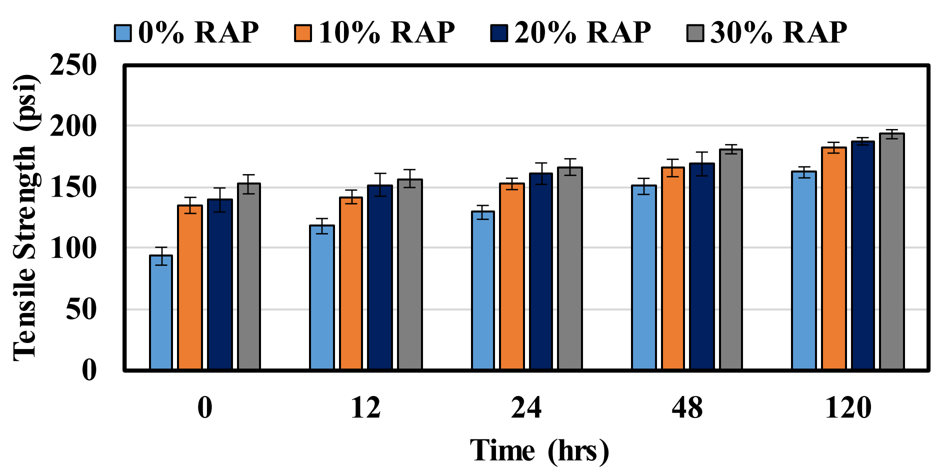


Figure 6.6. Rank Order Comparison for IDT Tensile Strength

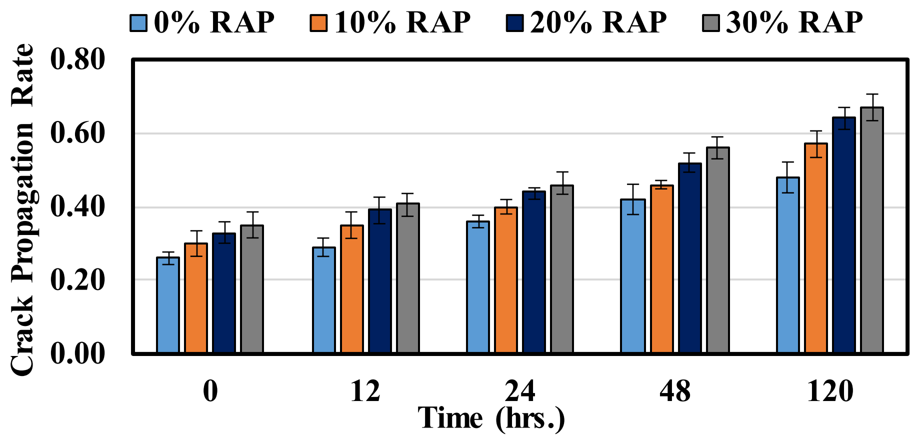


Figure 6.7. Rank Order Comparison for OT Crack Progression Rate

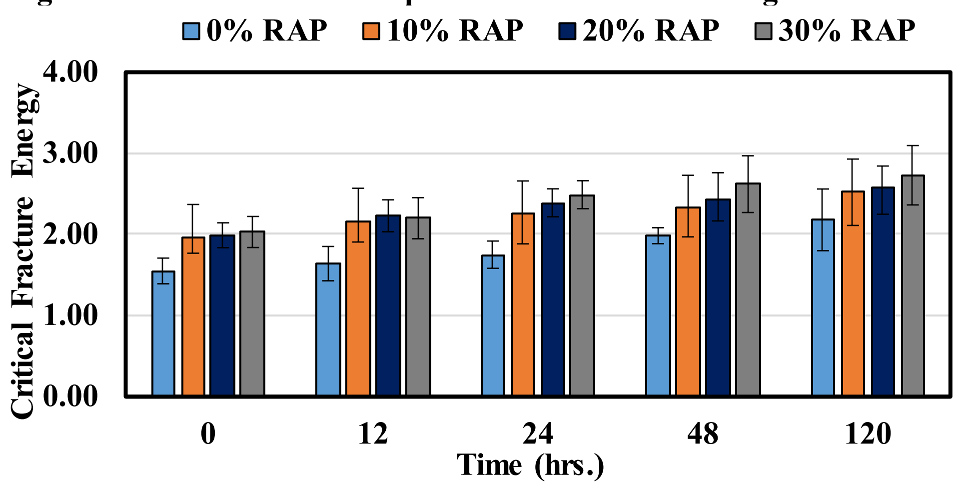


Figure 6.8. Rank Order Comparison for OT Critical Fracture Energy

Binder Performance Results

Five different binders (Sample Q, T, V, E, V) of PG 64-22 binders were oven aged (post short-term aging) using Protocol 1 (i.e., oven aging at 95°C) for different periods. For this, 1.5 grams of binder was placed in a small silicon mold to fabricate different samples with a thickness of about 3 mm and placed in a laboratory oven, as shown in Figure 6.9. These samples were periodically withdrawn to test their rheological and chemical properties. For example, the samples' high-temperature PG grade at different times was measured, and the results are presented in Figure 6.10. Interestingly, the rank order of samples at around 24 hours was identical to the rank order at around 168 hours, although the sensitivity of the difference was higher after 168 hours. These results indicate the likelihood of a "steady-state" of aging and provide encouraging results for further testing. The research team further explored this hypothesis with the upcoming tests considering more mixture variables to formulate more robust and generalizable conclusions at both the mixture and binder level. Considering that all five binders were of the same PG, a few more important observations from these tests are:

- After 168 hours, the high grade for Sample V was nearly two full grades (12°C) higher compared to Sample Q,

- Sample V showed a very high value for the high grade after just 6 hours of aging,
- Although samples Q, J, and E had similar high grades after 6 hours of aging, samples J and E had an aging rate that was almost twice that of sample Q,
- Binders of the same grade from different sources can show extremely different aging trajectories, thereby resulting in very different expected serviceable life of the pavement.

Figure 6.9. Binder Aging in Oven

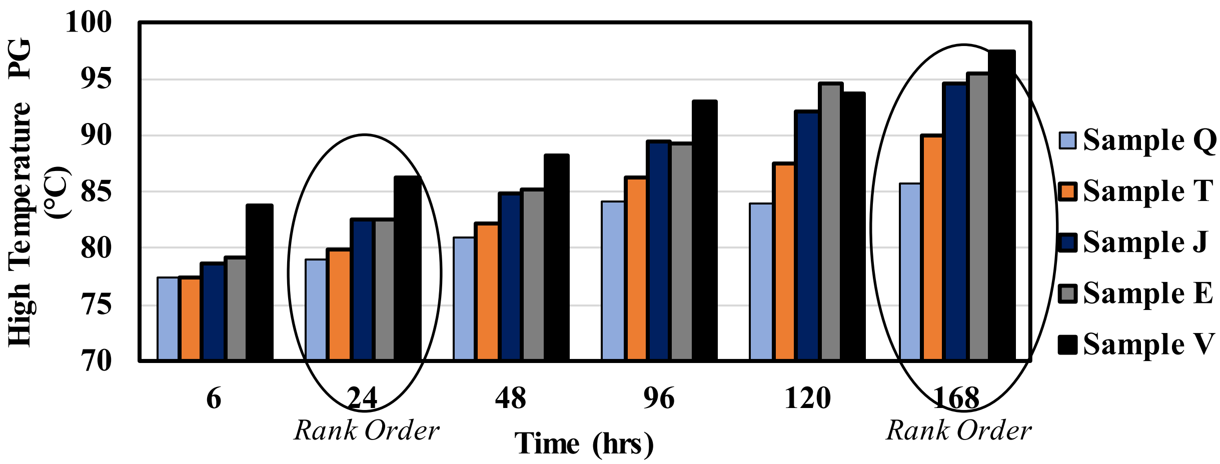


Figure 6.10. Rank Order of Binders (High PG)

Chapter 7. Extended Evaluation of Aging Potential of AC with Refined Laboratory Aging Protocols

This chapter documents the extensive parametric study of mix design variables on aging behavior. The chapter also documents the updated methodology for developing optimized aging protocols to assess the short- and long-term aging behavior of mixtures and binders. The methodology captures the minimum aging duration required that results in no significant change to engineering properties or the rank-order of binder and mixture susceptibility to aging. Additionally, a comprehensive evaluation of two accelerated aging protocols is also presented as alternative aging methods.

Extended Evaluation

Mixture Design Variables

The research team considered the selection of five variables to study the influence of mix design variables on aging behavior including change in RAP (content and source), PG (source and grade), aggregate (type and surface aggregate classification), asphalt content, and additives as recommended by the manufacturer. Table 7.1 documents the different variations proposed for each of the mixtures.

Table 7.1. Mix Design Breakdown and Variations

	5 Recommended Variations						
Mix No.	1: RAP	2a: PG Source	2b: PG Grade	3a:Aggregate Gradation	3b:Aggregate Source	4:Asphalt Content	5:Additive
1	10/20/30						
2	10/20/30		76/70/64				1.5x & 2.0x Dose
3		3 Sources			SAC A to B		
4				MDL		-0.5 & 1.0	
5	0 to 30						

Asphalt Concrete Mixture Characteristics

As part of the extended evaluation, one Dense Graded and four Superpave mixtures were evaluated. Each had different variations within the control mix, as seen in Table 7.1. All the mixtures were lab-mixed lab-compacted (LMLC) specimens. Table 7.2 shows the details related to the constituents of these five control mixtures and the targeted variations. Each loose mixture was subjected to long-aging regimes of NCHRP 09-54 (5 days of aging at 95ºC) following the short-term aging condition from TxDOT. Appendix A presents all the information pertaining to the mixture's combined gradation.

Table 7.2 – Mixtures Constituents for Protocol 1

Mix No.	Aggregate Type	Binder Grade	Mix Design Characteristics
1	Limestone Dolomite	PG 70-22	SP-D: 20% RAP, Evotherm, 6.1% AC
2	Limestone Dolomite	PG 64-22	SP-D: 30% RAP, Evotherm, 6.0% AC
3	Gravel	PG 70-22	SP-C: 10% RAP, ZycoTherm, 5.3% AC
4	Igneous	PG 70-28	DG-D: 8% RAP, N/A, 5.6% AC
5	Gravel	PG 70-22	SP-C: 20% RAP, N/A, 5.8% AC

Mixture Performance Results

Hamburg Wheel-Tracking (HWT) Test (Tex-242-F) Results

During the initial evaluation performed, it was determined that AC mixtures are most susceptible to rutting during the early stages. Oven aging beyond 4 hours showed no significant loss in the mixture's rutting resistance and the rutting characteristics of the mix improved. In this task, the research team focused on understanding the rutting resistance of mixtures for five variations, as presented in Table 7.2. The mixture variations are different in nature, making it difficult to generalize the results. However, Figure 7.1 suggests that with the increase in RAP content in the mixture NRRI consistently increased with oven aging times. Figure 7.2 illustrates the change in additives and indicates a drop in rutting performance as the aging hours increase, regardless of binder type and additive dosage. This is somewhat counter-intuitive because generally rutting resistance is expected to increase with an increase in aging duration. It is speculated that this anomalous behavior could be due to volatilization or degradation of the additive. Figure 7.3 confirms that changes in the binder's performance grade impact rutting results. The combined effects of binder source and SAC are illustrated in Figure 7.4. The rate of change of NRRI values beyond 2 hours of short-term oven aging for the given variations are minimal. Figure 7.5 shows the impact a coarser or finer mix will have on the rutting performance while Figure 7.6 illustrates the results for change in asphalt content, again, with minimal change.

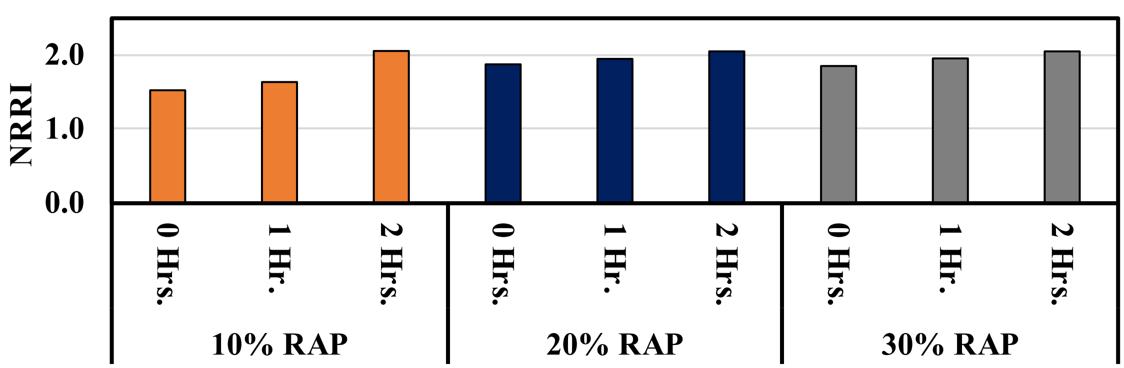


Figure 7.1. NRRI Results for Change in RAP (Mix 1)

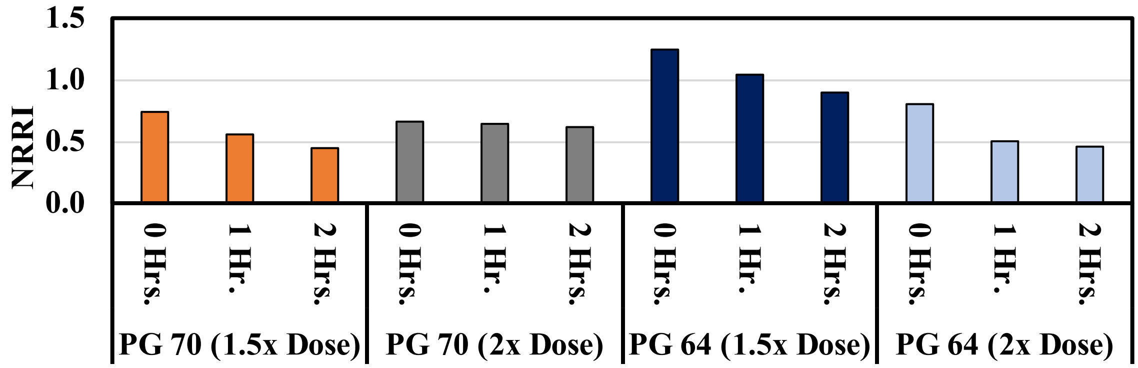


Figure 7.2. NRRI Results for Change in Additives (Mix 2)

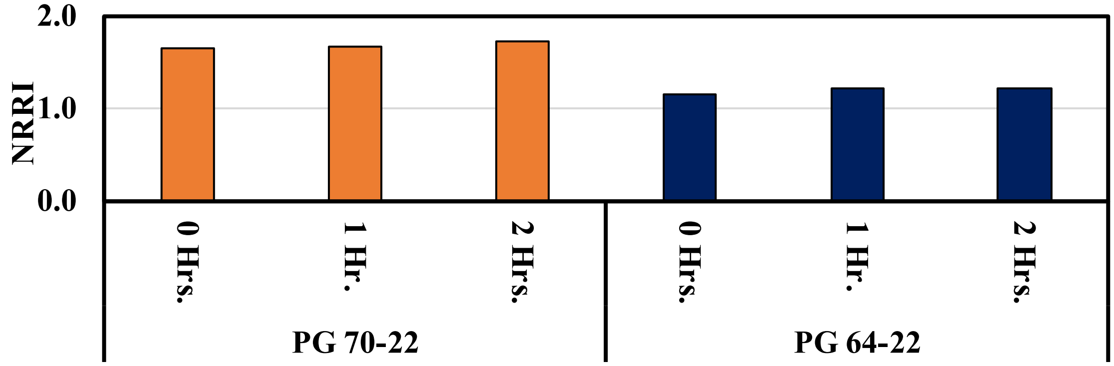


Figure 7.3. NRRI Results for Change in Performance Grade (Mix 2)

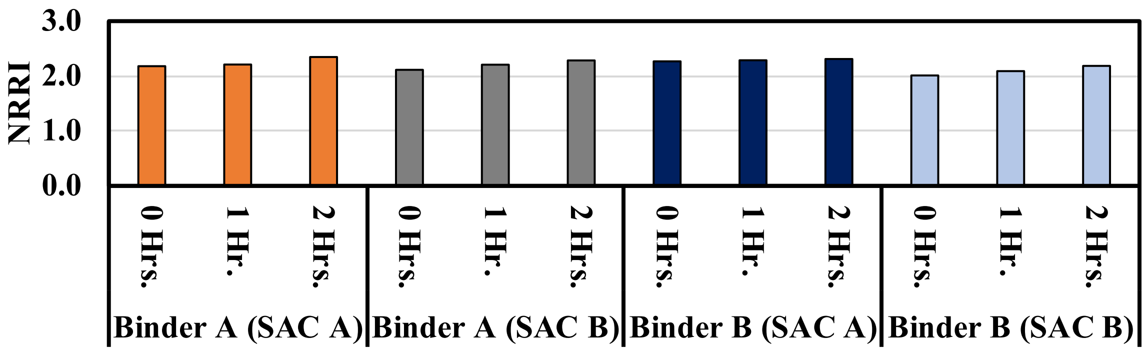


Figure 7.4. NRRI Results for Change in Binder Source and SAC (Mix 3)

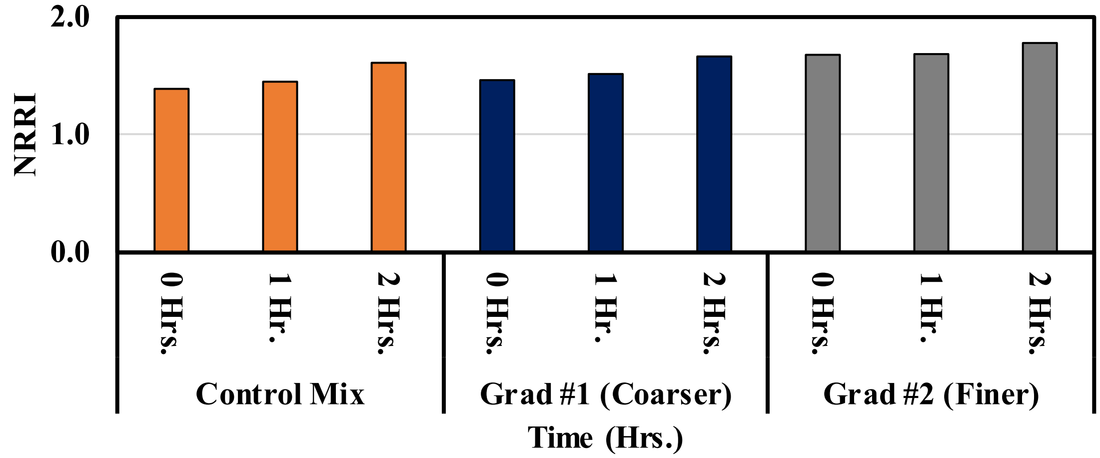


Figure 7.5. NRRI Results for Change in Gradation (Mix 4)

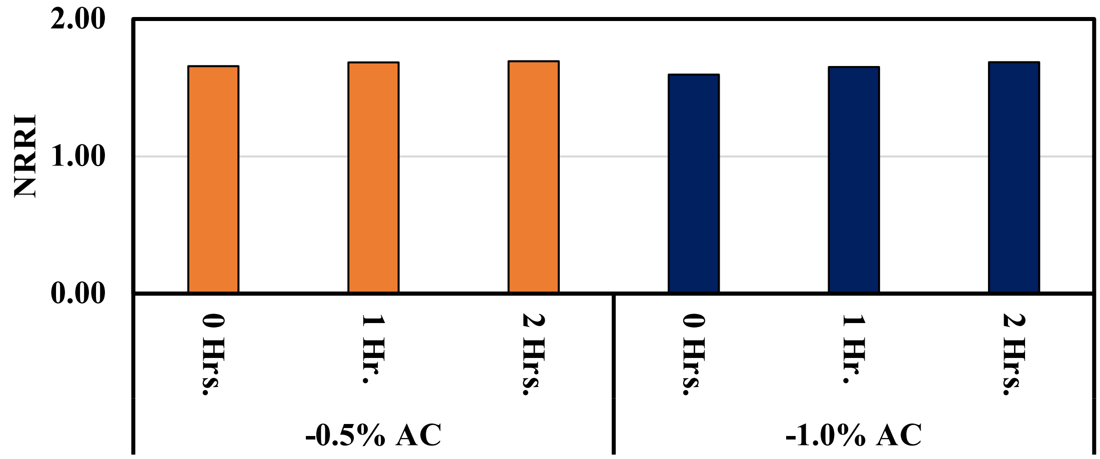


Figure 7.6. NRRI Results for Change in Asphalt Content (Mix 4)

IDEAL Cracking Test (Tex-250-F) Results

The changes in tensile strength and CT-Index of mixtures for four variations, as summarized in Table 7.2 are presented in Figures 7.7 to 7.12. The dashed line in the figures below are the respective 5-day benchmark value for each test. The results suggest that with the increase in oven aging duration the mixtures become more crack susceptible, regardless of the variation. Most asphalt mixture combinations evaluated do not meet the minimum required CT-Index criteria. Figure 7.7 shows that increasing the RAP content in the mixtures yields an increase in the tensile strength whereas the CT-Index decreases. In the case of PG grade type, PG 70-22 shows a higher tensile strength and CT-Index compared to PG 64-22. Figure 7.10 illustrates the effect of SAC A and SAC B aggregates with different binder sources on tensile strength and lower CT-Index. Overall, the results appear conclusive across all mixture variations.

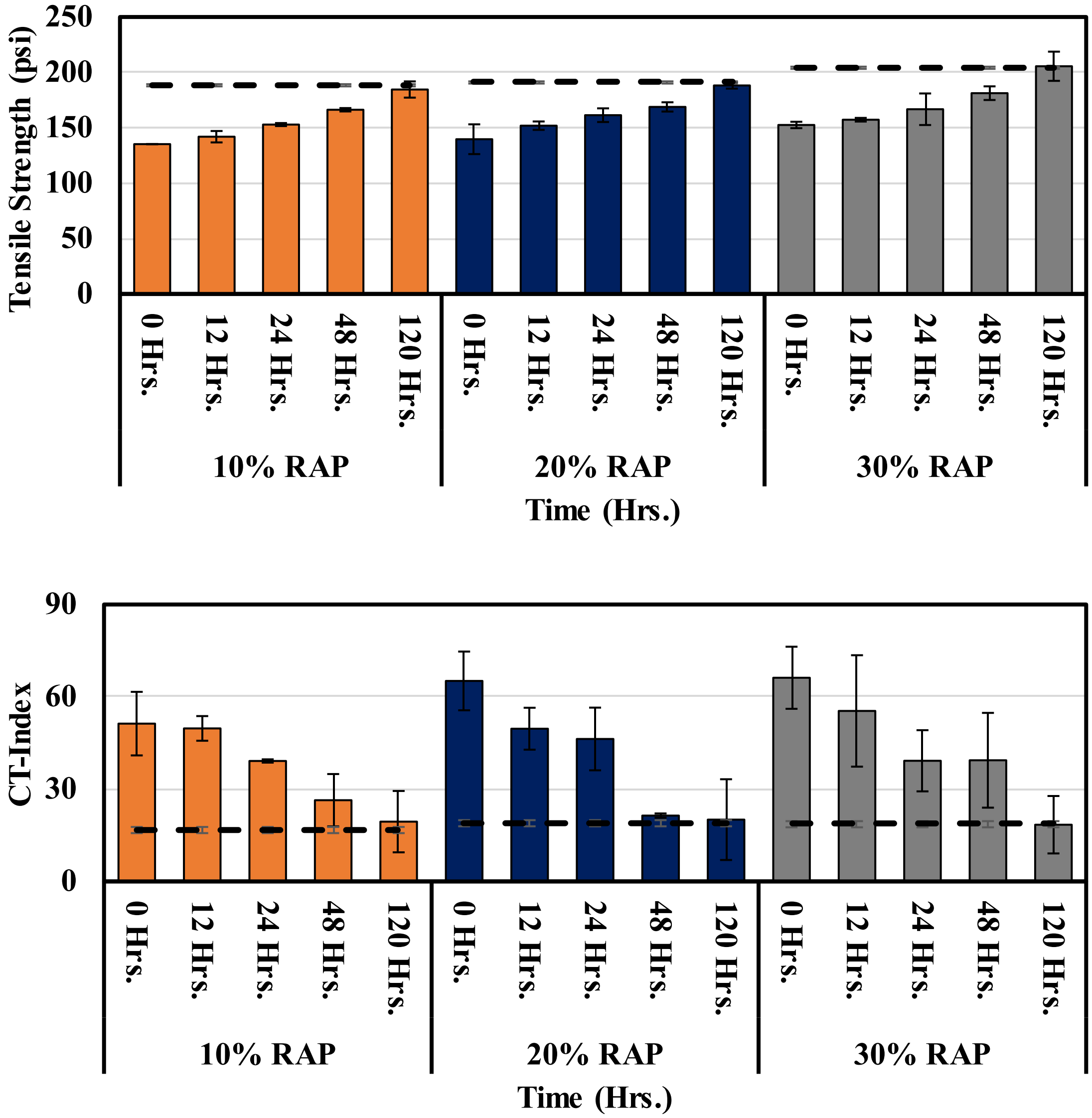


Figure 7.7. Tensile Strength and CT-Index Results for Change in RAP (Mix 1)

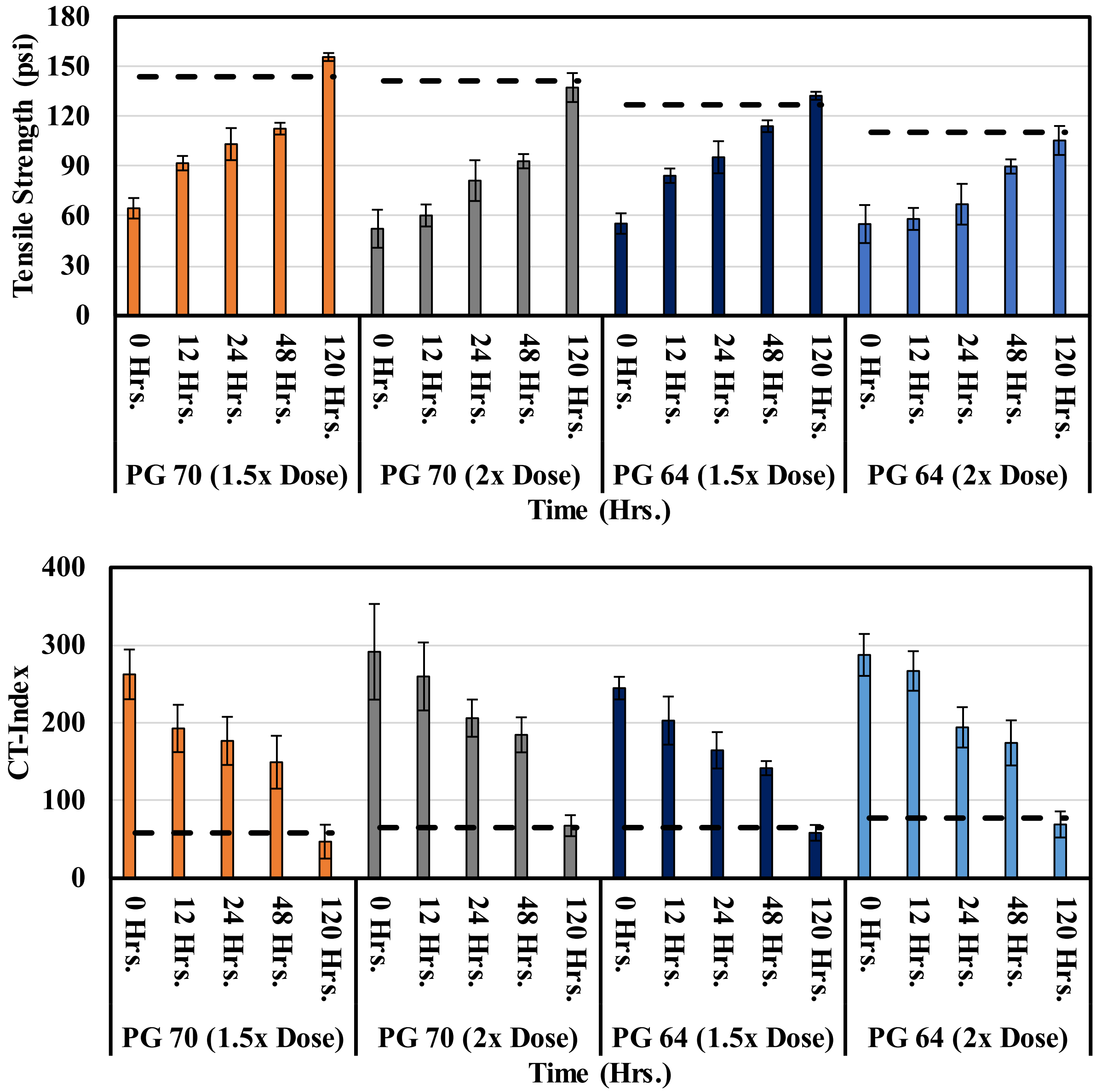


Figure 7.8. Tensile Strength and CT-Index Results for Change in Additives (Mix 2)

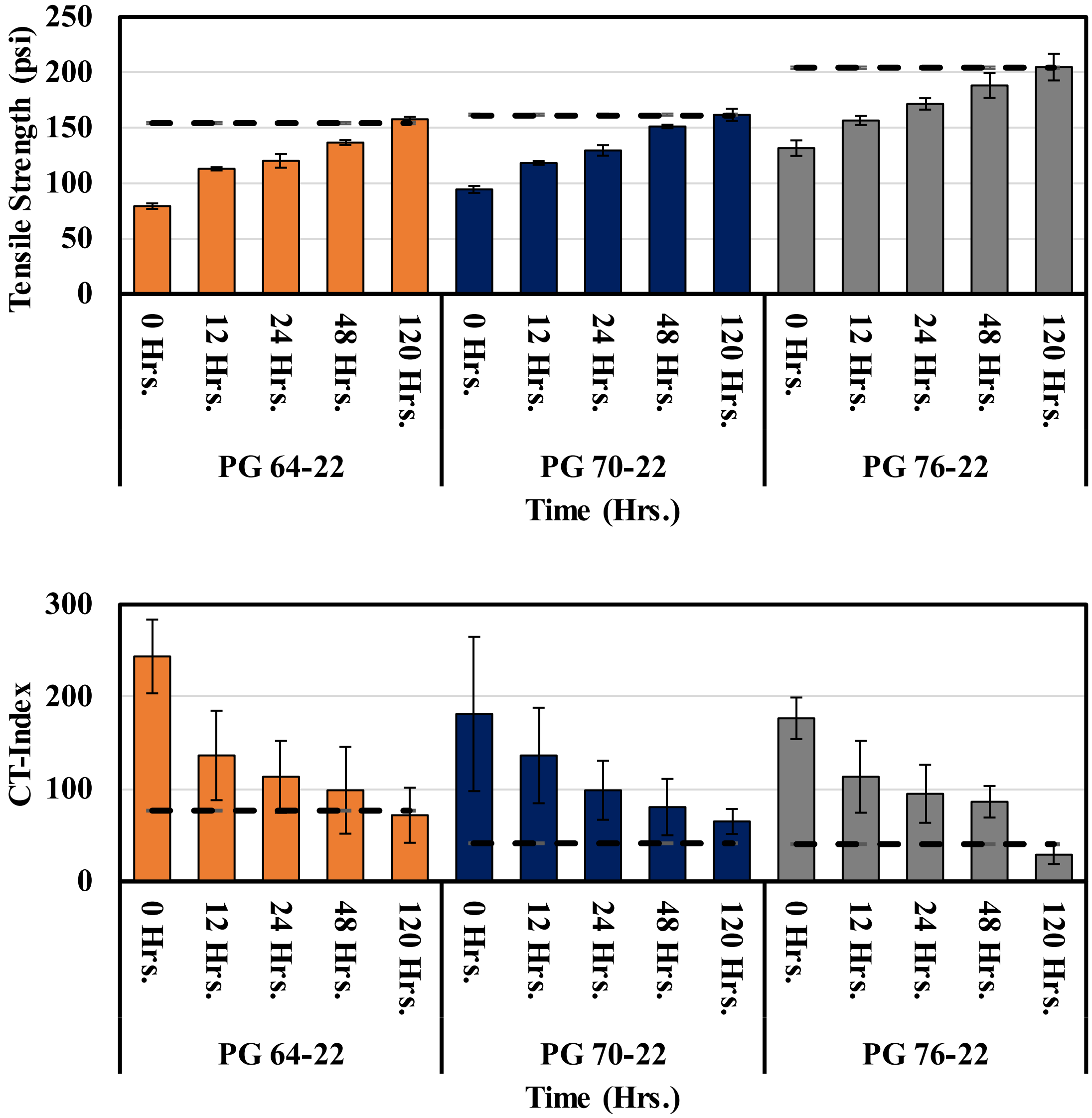


Figure 7.9. Tensile Strength and CT-Index Results for Change in PG (Mix 2)

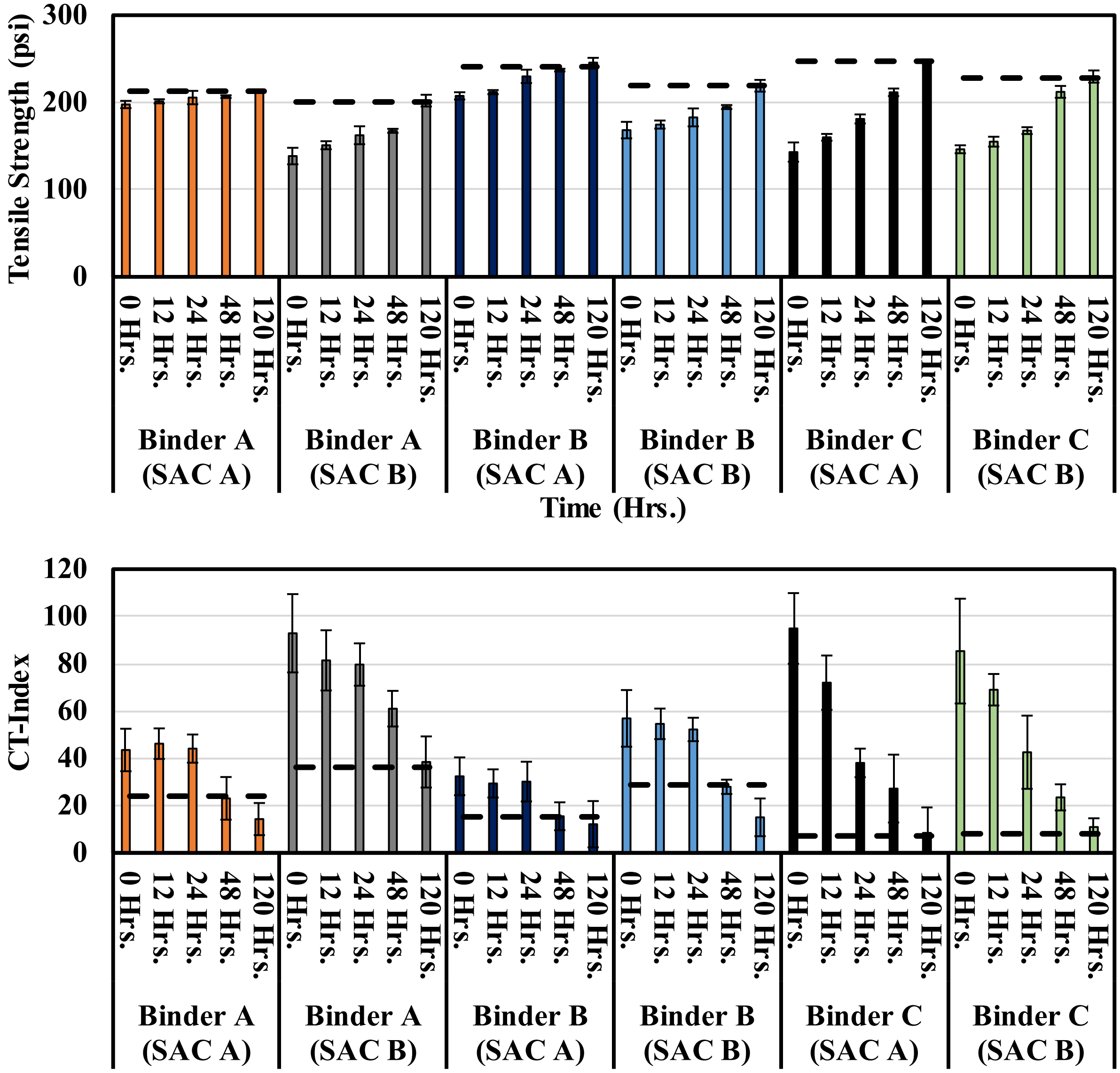


Figure 7.10. Tensile Strength and CT-Index Results for Change in Binder Source and SAC (Mix 3)

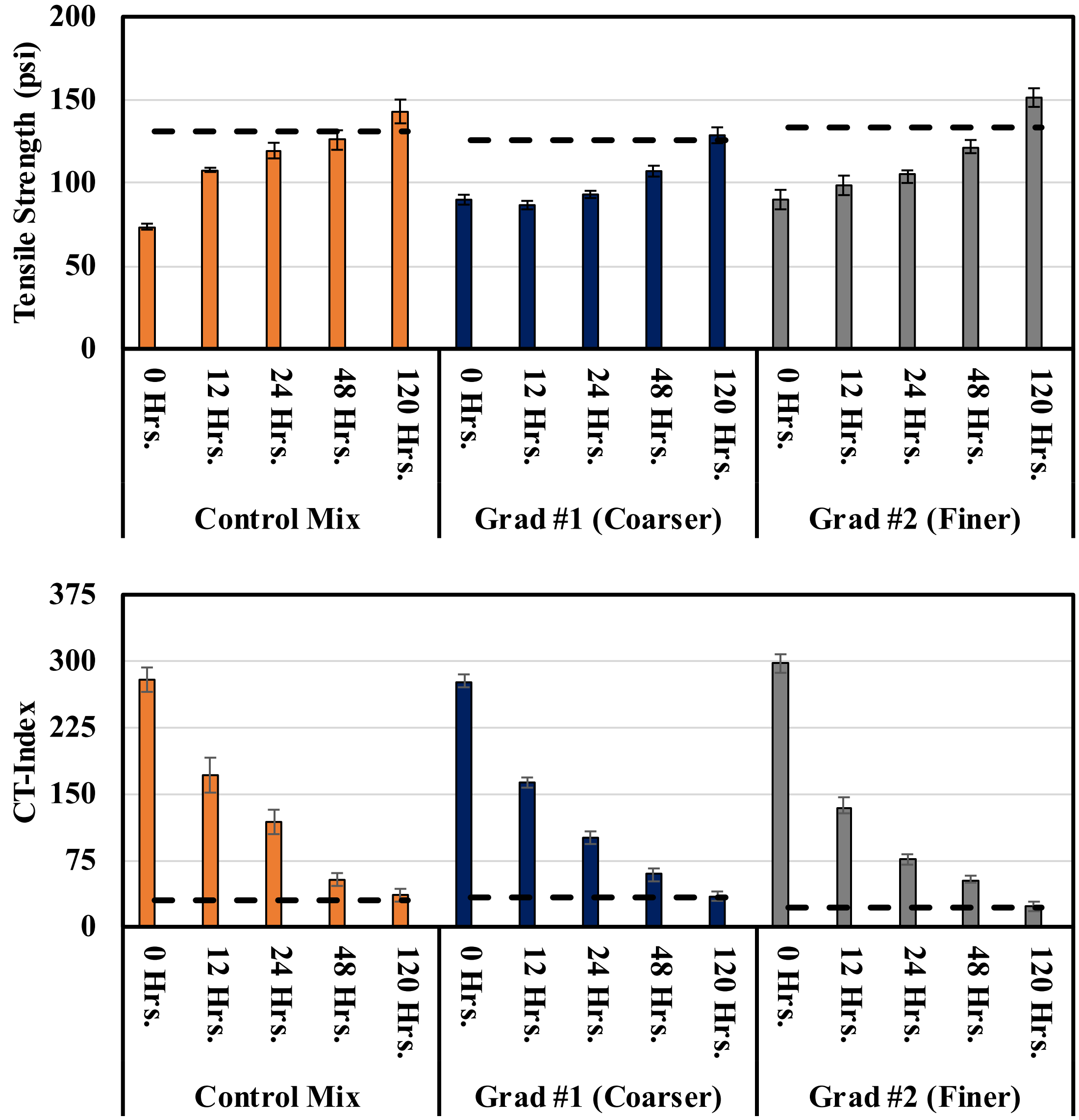


Figure 7.11. Tensile Strength and CT-Index Results for Change in Gradation (Mix 4)

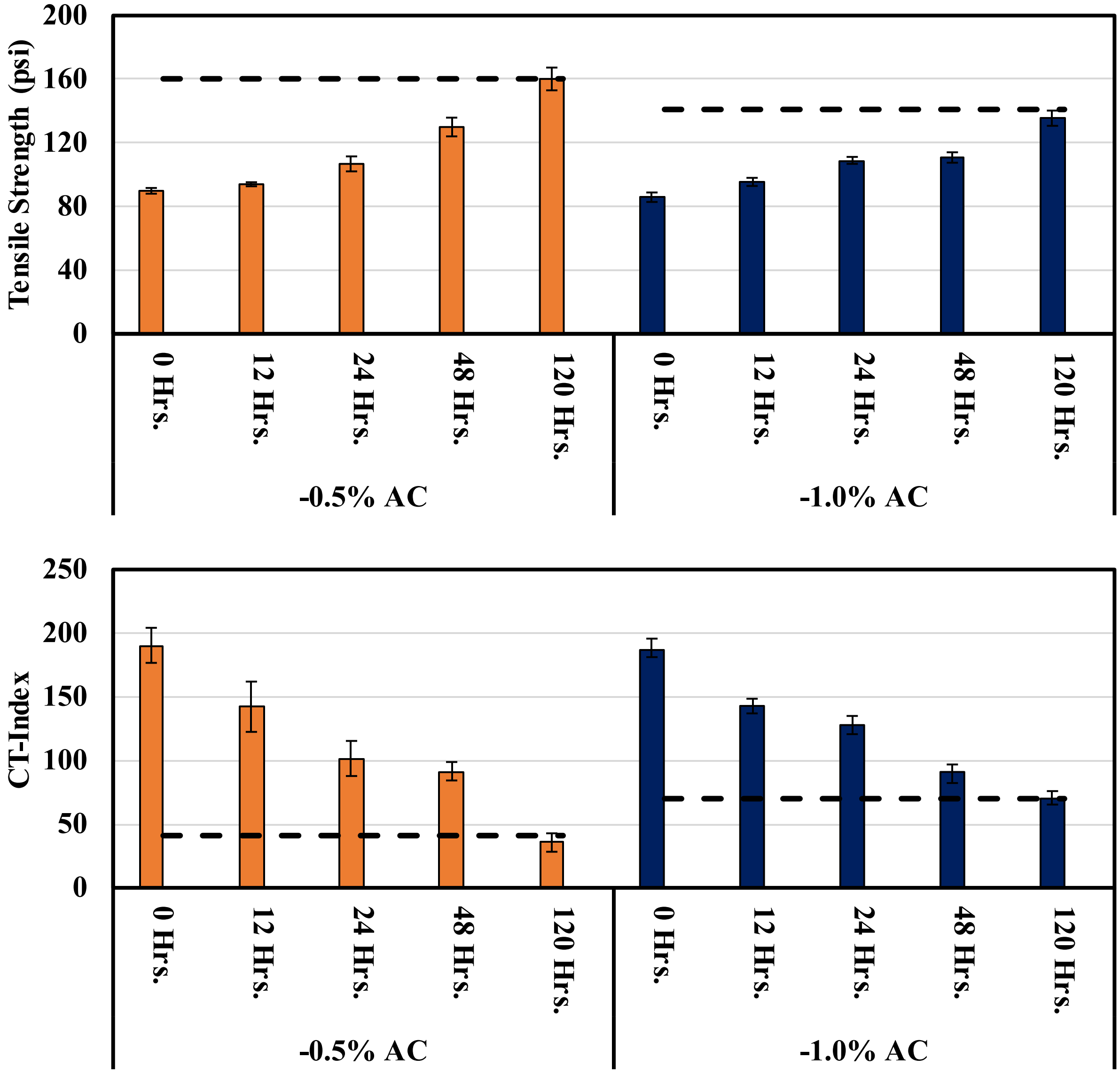


Figure 7.12. Tensile Strength and CT-Index Results for Change in Asphalt Content (Mix 4)

Overlay Tester Results (Tex-248-F)

The CPR and CFE values of mixtures are presented in Figures 7.13 to 7.15. The CPR and CFE increased with the increase in the mixtures' aging time regardless of the mixture's RAP content. From Figure 7.14, adding a 2.0x dose of additive yields a lower CPR and CFE than a 1.5x dose. Nevertheless, increments in aging time intensified the CPR and CFE. On the other hand, mixtures prepared with a high PG grade binder (PG 70-22) show higher CFE and CPR than a lower PG grade binder (PG 64-22). The observed trends for both cracking parameters are reasonable and in good agreement with the IDEAL cracking test results. As the aging hours increase, the asphalt mixtures are more susceptible to cracking. All the asphalt mixture variations evaluated seem to experience the same decrease in performance caused by the aging protocol applied. An aspect that might require a more careful analysis is that in some cases, the asphalt mixture combinations fail using CPR criteria and pass based on CFE requirements, or vice versa. The extent of this behavior should be further evaluated, especially at 120 hours.

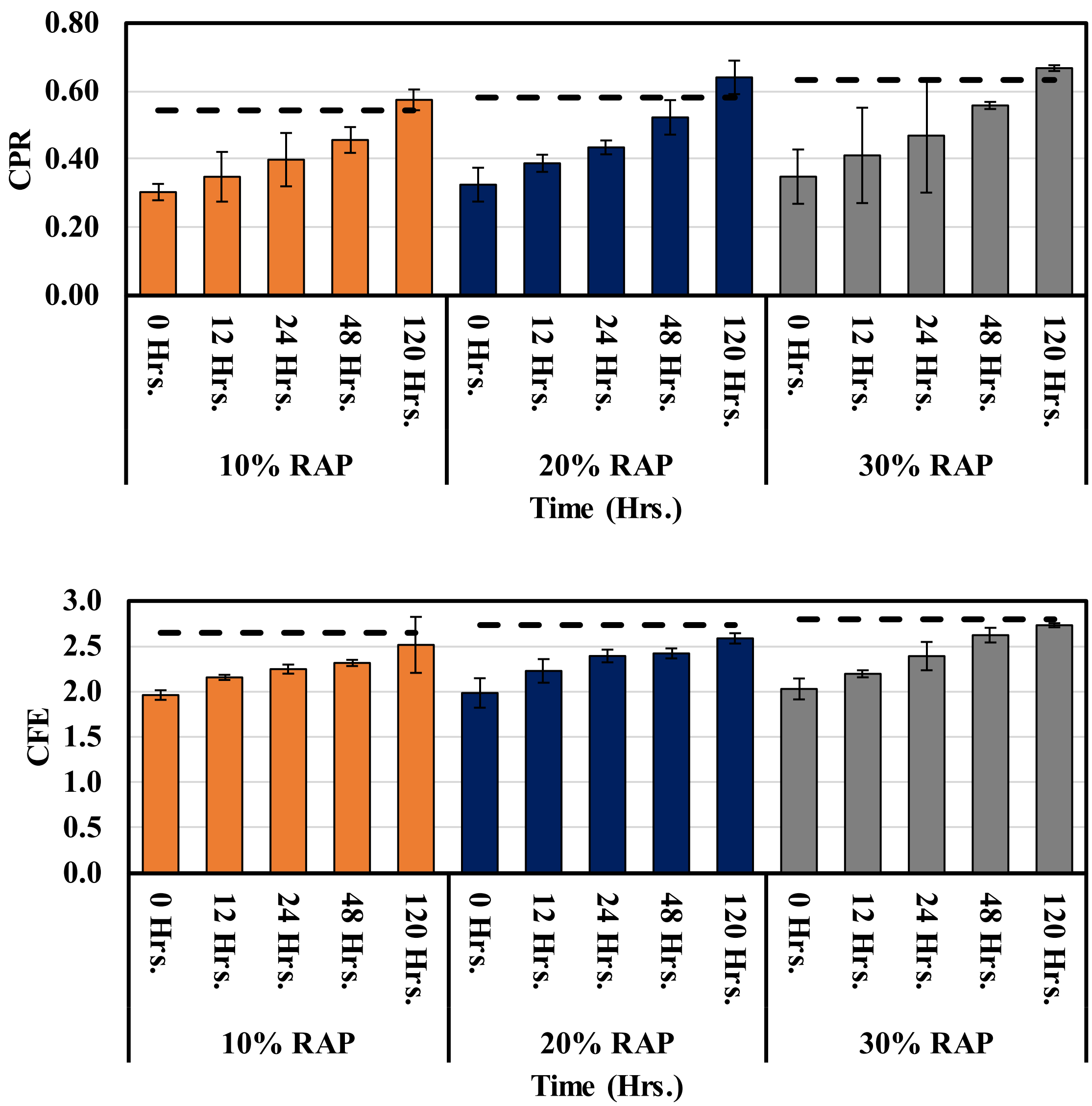


Figure 7.13. CPR and CFE Results for Change in RAP (Mix 1)

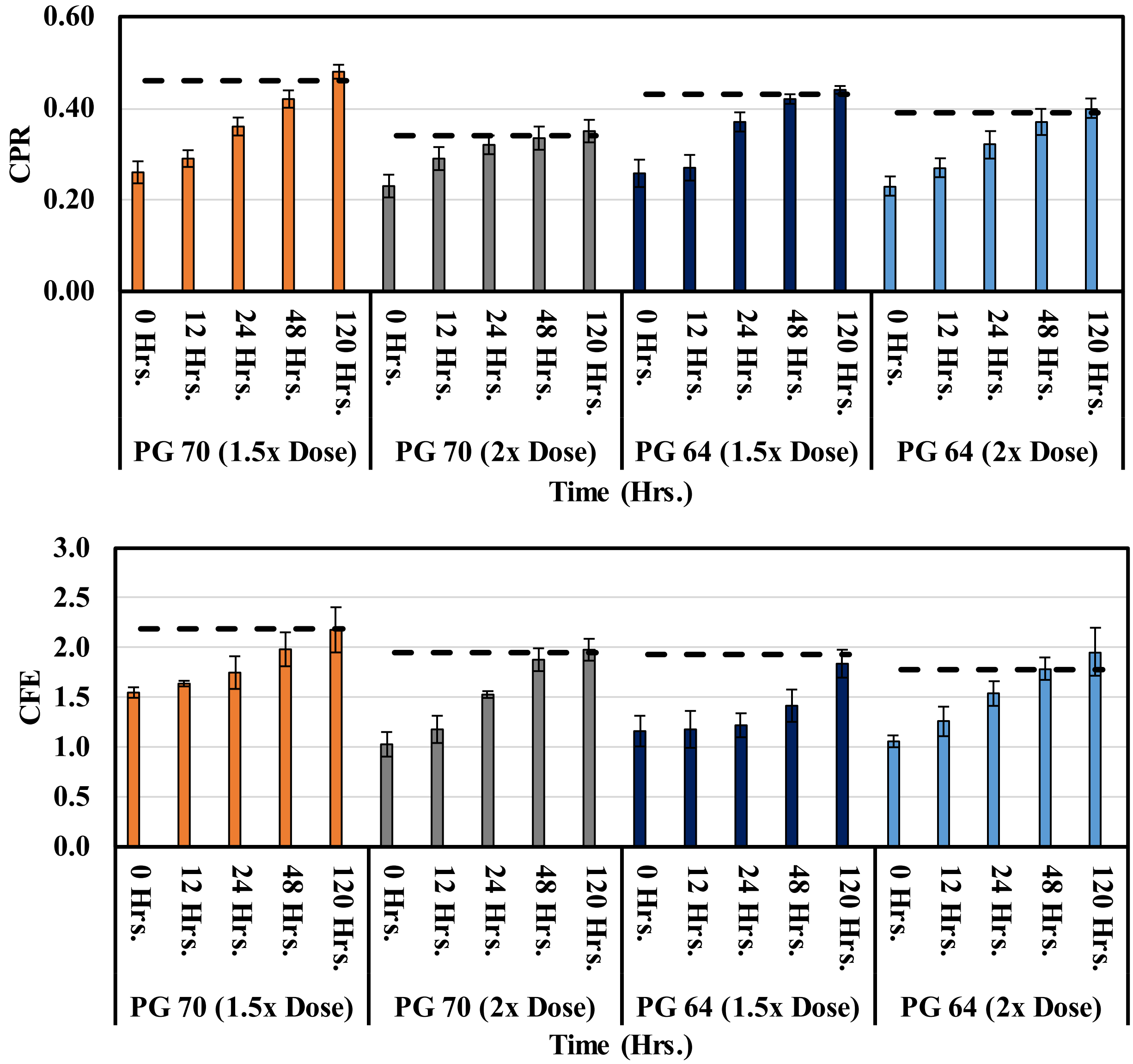


Figure 7.14. CPR and CFE Results for Change in Additives (Mix 2)

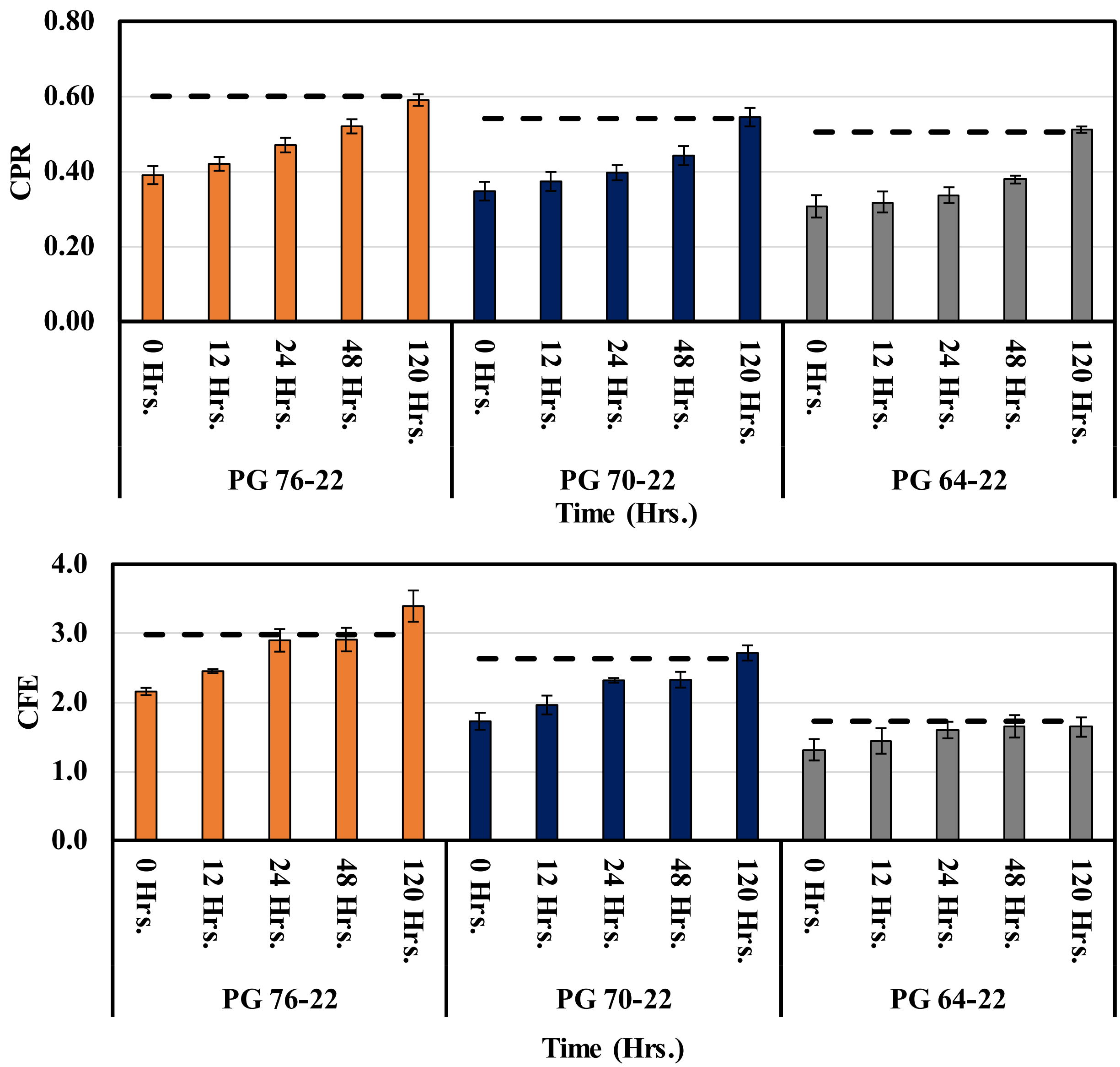


Figure 7.15. CPR and CFE Results for Change in PG (Mix 2)

Binder Results

The high and low temperature PG of the extracted binders was calculated using a DSR. In term of chemical properties, the oxidation levels of the various binders were examined using FTIR spectroscopy. Two bands, comprising the carbonyl group and sulfoxide group were used to determine the extent of oxidation of the binders.

Change in RAP Content

The binders from the corresponding mixtures shown above were extracted and characterized using the rheological and chemical tests. The high temperature PG of the mixtures for the change in RAP variation at various times are shown in Figure 7.16. The results were calculated based on the value of $G^*/\sin\delta$ parameter and using the RTFO aged binder criterion in AASHTO T316 (i.e., temperature that results in a $G^*/\sin\delta$ value of 2.2kPa). A higher value of high-temperature PG indicates a stiffer binder, which in this case can be attributed to effect of oxidative aging. Literature indicates that with higher levels of aging, the complex modulus of binders increases and phase angle decreases, rendering the binder more solid like. An increase in high-temperature PG with time, that is indicative of an increase in stiffness was seen for all sets of mixes. Interestingly, the relative trend between mixtures at 0 hours, 24 hours, and 120 hours was seen to be the same. The stiffness true grade of the binders extracted mixes aged for different durations of times are presented in Figure 7.17 respectively. The m-value true grades followed the same trend and are not presented here for the sake of brevity. As per the requirements in AASHTO T313, the stiffness true grade is 10ºC below the lowest temperature at which the stiffness of the binders does not exceed 300 MPa. When comparing the binders exposed to different aging methods, a lower value of the stiffness and m-value true grade indicates the presence of a softer binder that has undergone comparatively less oxidation. A steady decrease in stiffness with time could be seen for all sets of mixtures, which is the expected trend, following the high-temperature PG results.

The results for the C=O indices are presented in Figure 7.18. The general increase of these oxidation indices with aging time was visible for binders from all set of mixtures. Out of the two indices calculated, the C=O index presented here as seems to be a more reliable indicator of aging with a generally linear increase. However, although FTIR indices are helpful to indicate the relative increase in oxidation, the parameters are semi-quantitative in nature and the relative increase of this parameter would be more profound when considering longer time intervals between tests.

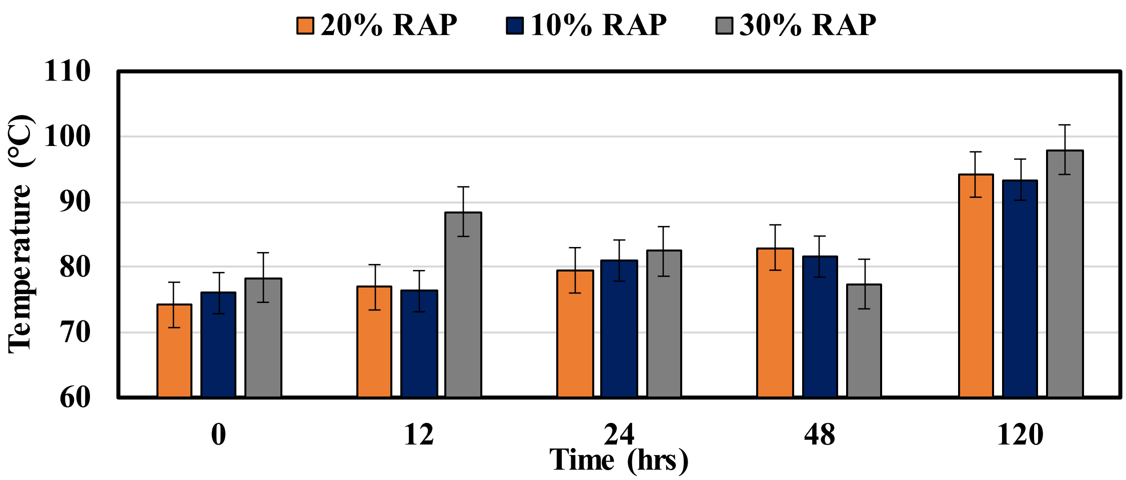


Figure 7.16. High PG results for change in RAP

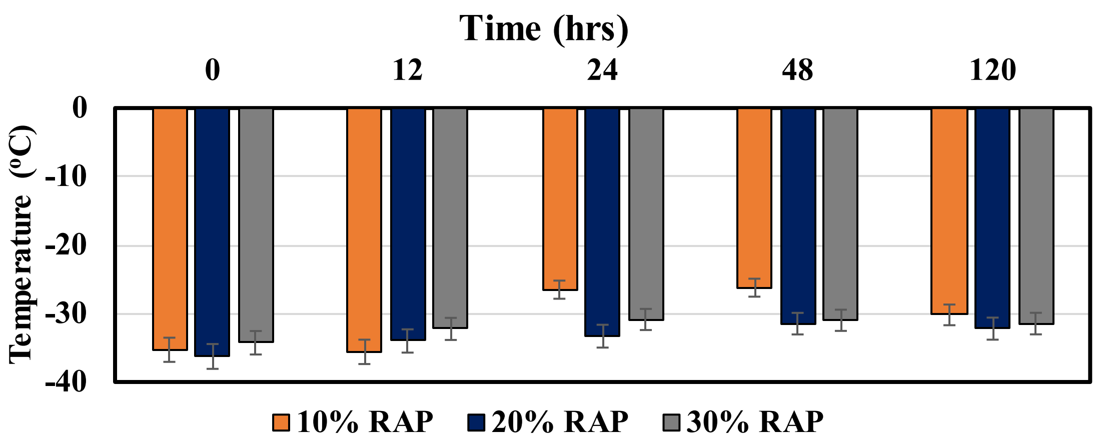


Figure 7.17. Low PG results for change in RAP

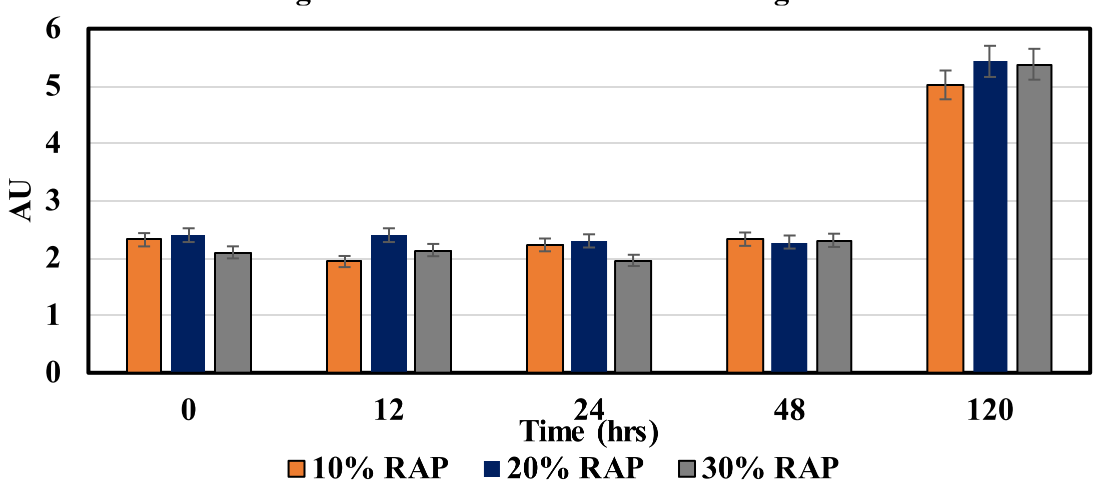


Figure 7.18. C=O Index results for change in RAP

Change of PG Grade

The rheological and chemical results for the change in PG grade variation are presented in Figure 7.19, Figure 7.20, and Figure 7.21 respectively. Similar results were obtained as the previous variations with an increase of aging metric (both rheological and chemical) with time.

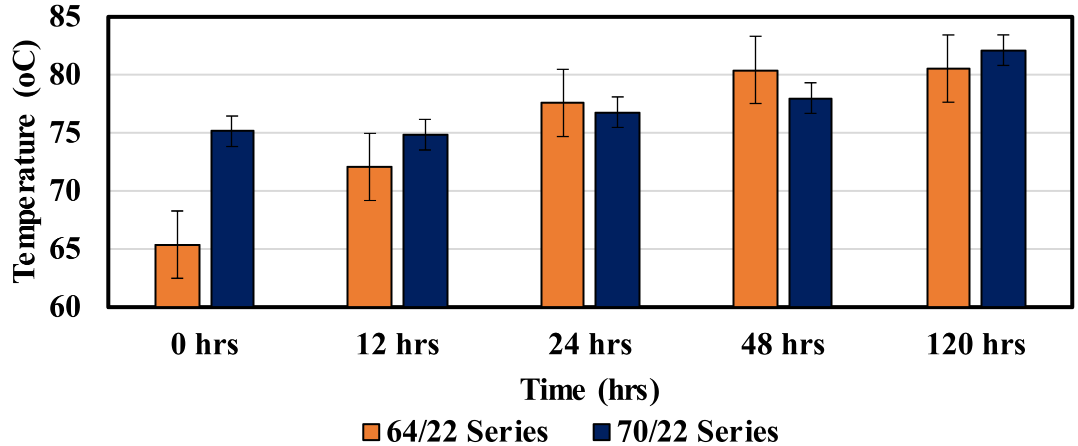


Figure 7.19. High PG results for change in PG grade

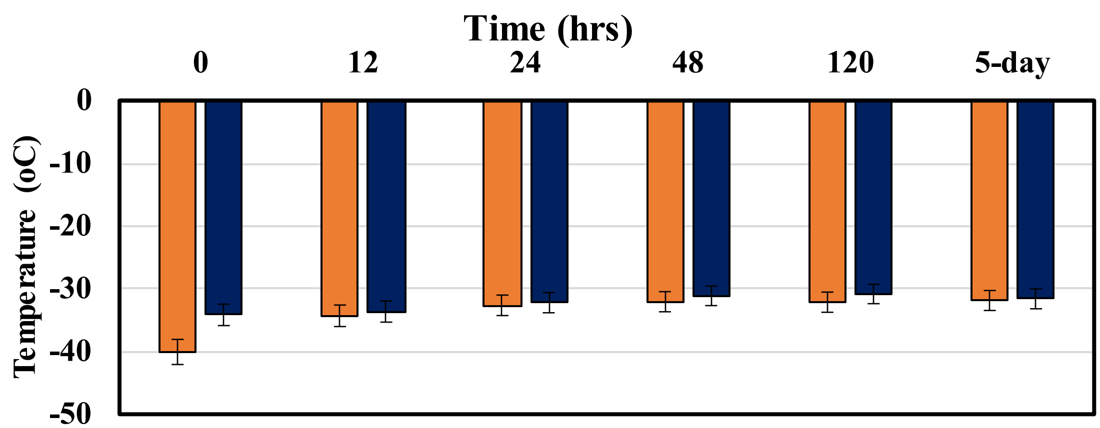


Figure 7.20. Low PG results for change in PG Grade

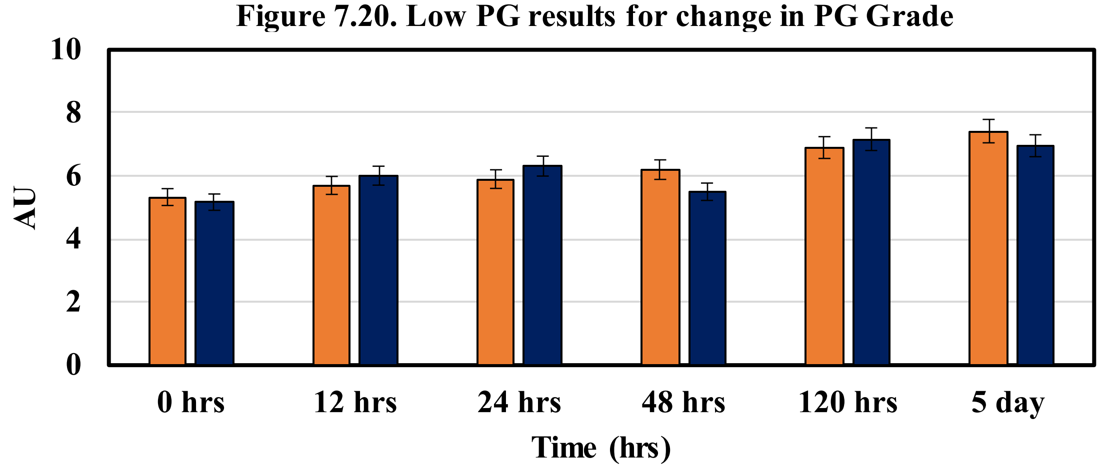


Figure 7.21. C=O Index results for change in PG Grade

Change of Additive

The rheological and chemical properties for the change in additive variation was determined for two different base binder, one with PG grade 70-22 and another with PG grade 64-22. The different results for the PG 70-22 binder are presented in Figure 7.22, Figure 7.23, and Figure 7.24 respectively. Similarly, the results for the PG 64-22 binder are presented in Figure 7.25, Figure 7.26, and Figure 7.27 respectively. Firstly, it could be noticed that the addition of the additive significantly softens the base binder, and this effect is attenuated at higher dosages. In terms of aging characteristics identical results were obtained as previous variations with an increase of aging metrics with time.

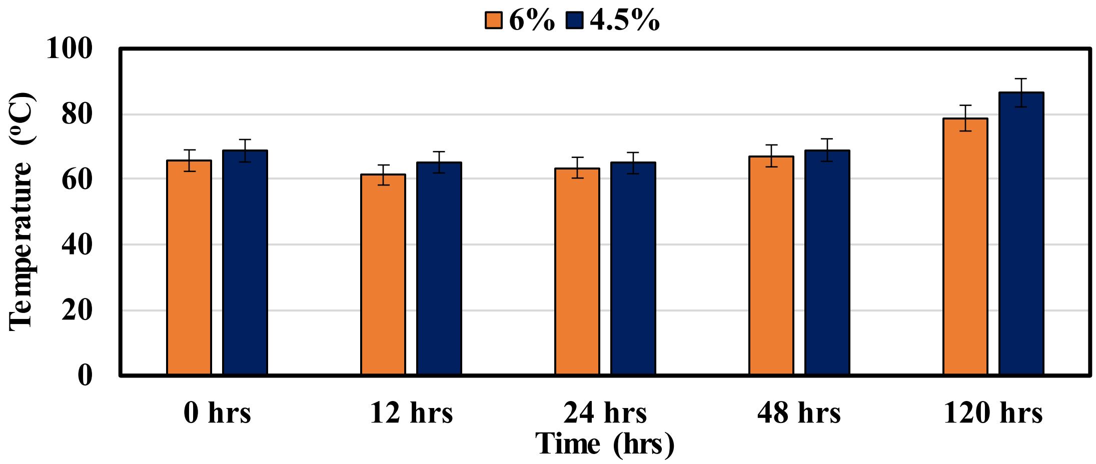


Figure 7.22. High PG Results for Change in Additive (70-22)

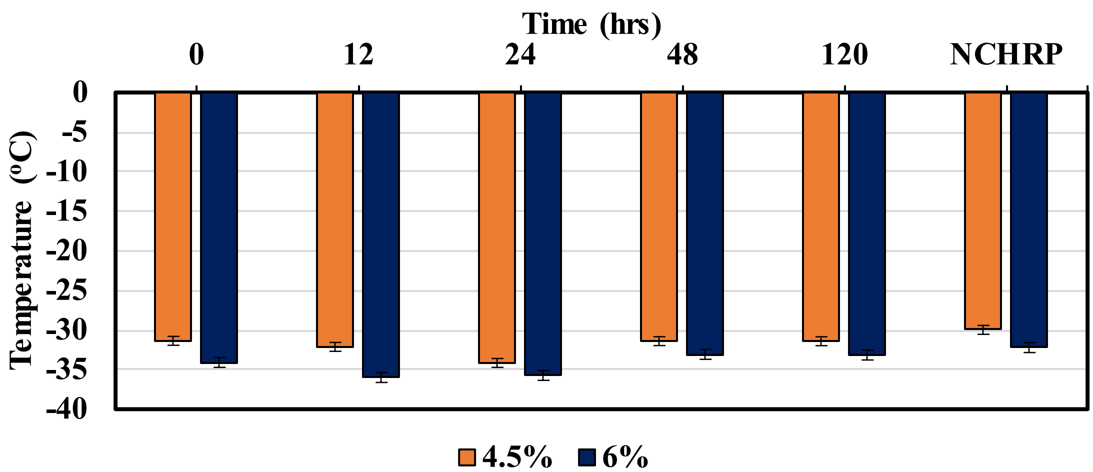


Figure 7.23. Low PG Results for Change in Additive (70-22)

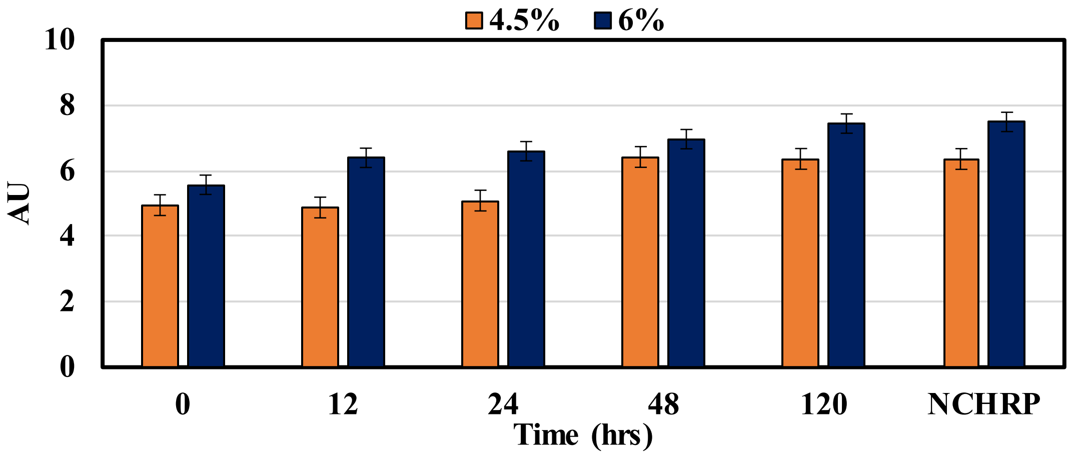


Figure 7.24. C=O Index Results for Change in Additive (70-22)

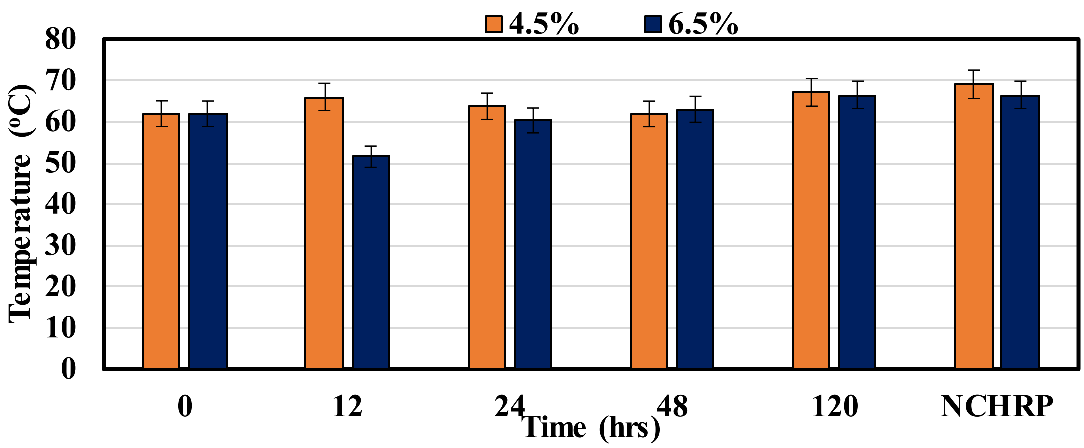


Figure 7.25. High PG Results for Change in Additive (64-22)

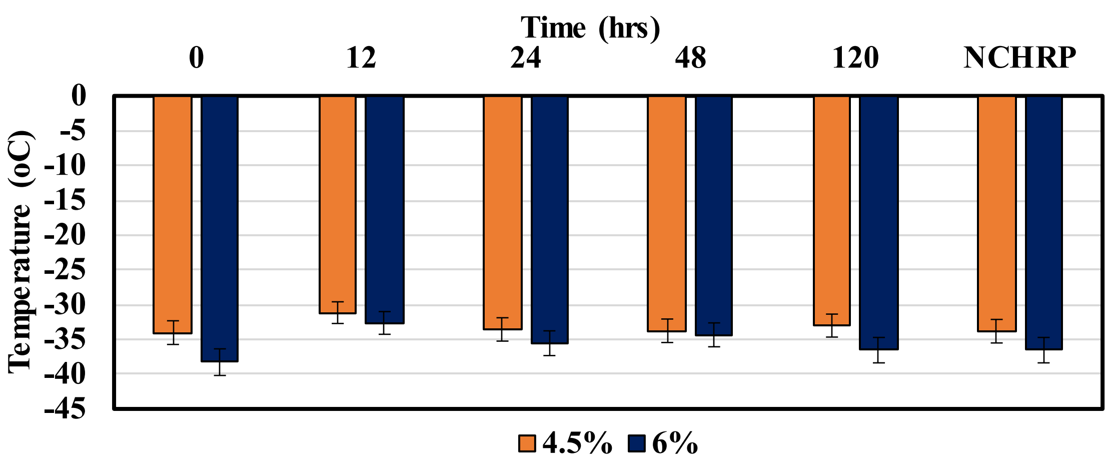


Figure 7.26. Low PG Results for Change in Additive (64-22)

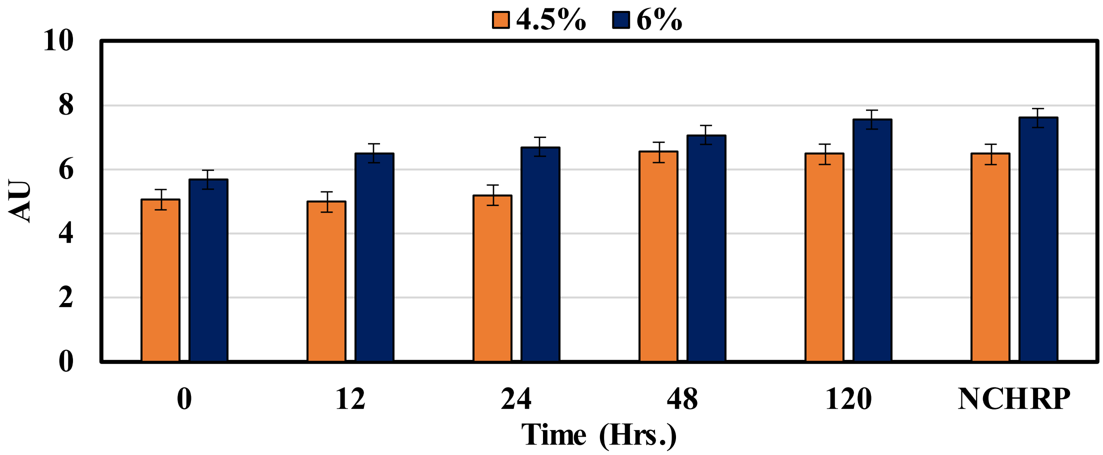


Figure 7.27. C=O Index Results for Change in Additive (64-22)

Accelerated Aging Methods: Upscaled Pressure Device

Methodology

An alternative method explored to accelerate aging was increasing temperature and thereby increasing the rate of oxidation. However, as demonstrated in literature search using a temperature greater than 100°C would trigger reaction pathways that normally do not occur in field conditions and lead to a fundamentally differently aged binder than what occurs in field. Another influencing factor explored was to pressurize the specimen that can similarly increase the rate of oxidation. Hence, the proposed alternative aging method was mainly used for providing benchmarks for the long-term accelerated aging of mixtures within a short period. Moreover, work was conducted to transform these methods from their pilot stage to an advanced stage regarding technological and implementation readiness. Changes adopted for the pressure device included using a dehydrating rack to increase airflow, the quantity of loose mix able to age, and a quick-connect system that is more practical. Figure 7.28 illustrates the pressure device along with the current setup.

Figure 7.28. Pressure Device and Aging Racks

Results and Discussion

This protocol adopted the long-term aging process from NCHRP 09-54 as the ultimate benchmark and the short-term aging from TxDOT. Three replicate samples were exposed to 2 hours of short-term aging at its respective compaction temperature and long-term oven aging at a constant temperature of 95°C for 22 hours. A study was performed to determine the uniformity of aging between top and bottom tiers using three replicate samples to assure the mixtures are evenly aged. Figure 7.29 illustrates the IDT tensile strength and Figure 7.30 shows the CT-Index comparison. The top and bottom tier specimens are similar in strength and aging process, since the top specimens' IDT values range between 137 psi to 139 psi, and 136 psi to 140 psi for the bottom specimens.

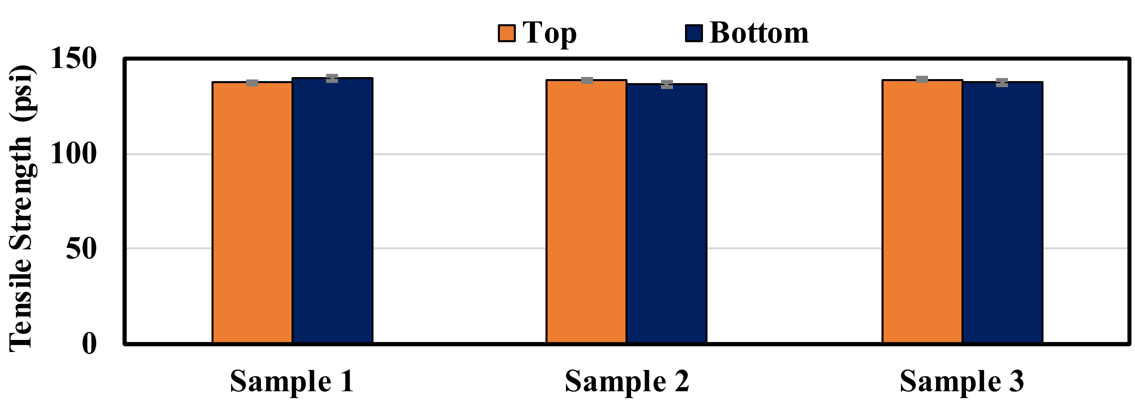


Figure 7.29. Tensile Strength Comparison Using Protocol 2

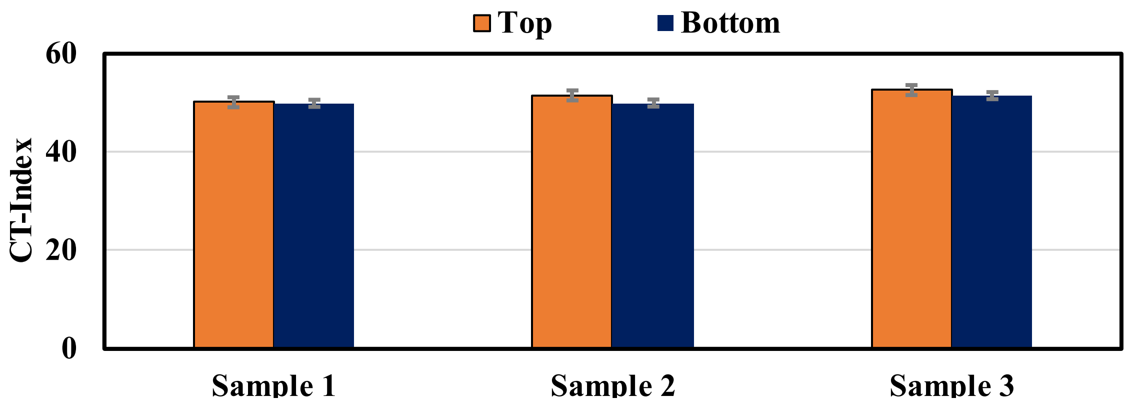


Figure 7.30. CT-Index Comparison Using Protocol 2

Extended Evaluation: Materials

An additional 15 mixtures were evaluated with different binder PG, aggregate types, and admixtures to assure the feasibility of this method. All of the control mixtures and variations listed in Table 7.1 were evaluated in the pressure device. Table 7.3 presents the details related to the constituents of these 15 mixtures. Each mix was subjected to aging regimes of NCHRP 09-54 (5 days of aging at 95ºC) as a benchmark, and 24 hours and 48 hours of aging at 80 psi. The IDEAL-Cracking Test was performed on all mixtures, and the results are discussed below.

The IDEAL-CT indices and IDT tensile strengths at different aging conditions are compared in Figures 7.31 and 7.32, respectively. All the mixes met or exceeded the benchmark values in 48 hours or less.

Table 7.3. Mixtures for Validation of Protocol 3

Mix	Aggregate Type	Binder Grade	Mix Design Characteristics
1	Limestone	PG 70-22	SP-C: 10% RAP, HydroFoam IEQ, 5.4% AC
2	Limestone	PG 70-22	SP-C: 20% RAP, HydroFoam IEQ, 5.1% AC
3	Igneous	PG 64-22	SP-C: 10% RAP, N/A, 5.4% AC
4	Igneous	PG 70-22	SP-C: 15% RAP, N/A, 5.3% AC
5	Igneous	PG 76-22	SP-C: 10% RAP, N/A, 5.4% AC
6	Limestone/Igneous	PG 70-22	SP-C: 15% RAP, Evotherm, 5.5% AC
7	Limestone Dolomite	PG 70-22*	SP-D: 10% RAP, Evotherm, 6.1% AC
8	Limestone Dolomite	PG 70-22*	SP-D: 20% RAP, Evotherm, 6.1% AC
9	Limestone Dolomite	PG 70-22*	SP-D: 30% RAP, Evotherm, 6.1% AC
10	Limestone Dolomite	PG 64-22*	SP-D: 30% RAP, Evotherm, 6.0% AC
11	Limestone Dolomite	PG 64-22	SP-D: 30% RAP, Evotherm, 6.0% AC
12	Gravel *(SAC A)*	PG 70-22*	SP-C: 10% RAP, ZycoTherm, 5.3% AC
13	Gravel *(SAC B)*		SP-C: 10% RAP, ZycoTherm, 5.3% AC
14	Gravel *(SAC A)*	PG 70-22*	SP-C: 10% RAP, ZycoTherm, 5.3% AC
15	Gravel *(SAC B)*		SP-C: 10% RAP, ZycoTherm, 5.3% AC

**NOTE: Mixes 7 to 13 have three different binder sources.*

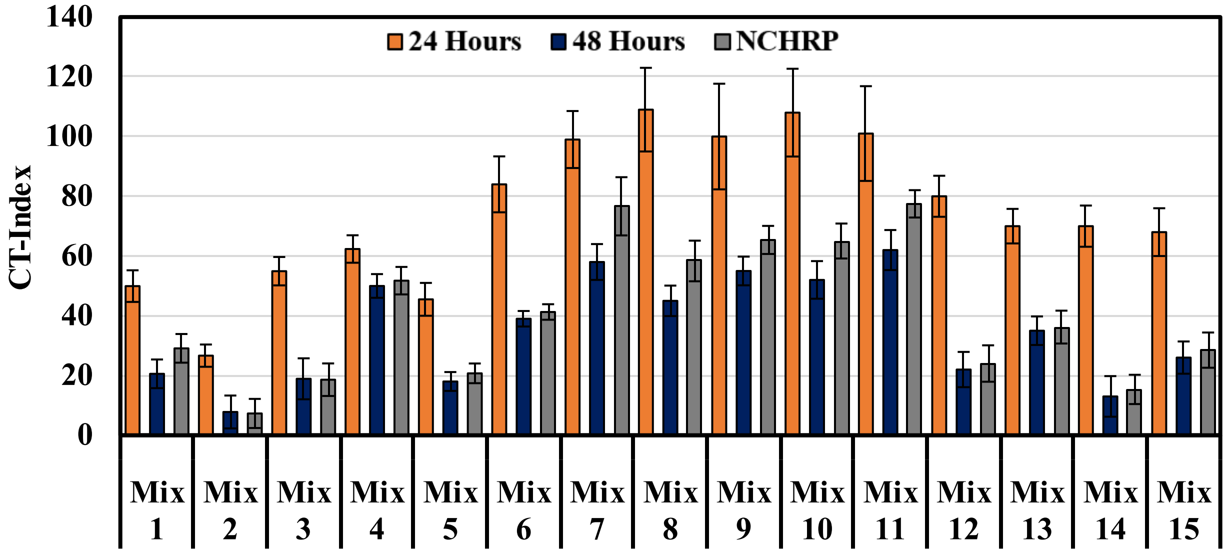


Figure 7.31. IDEAL-CT Index Value Comparison Using Protocol 3

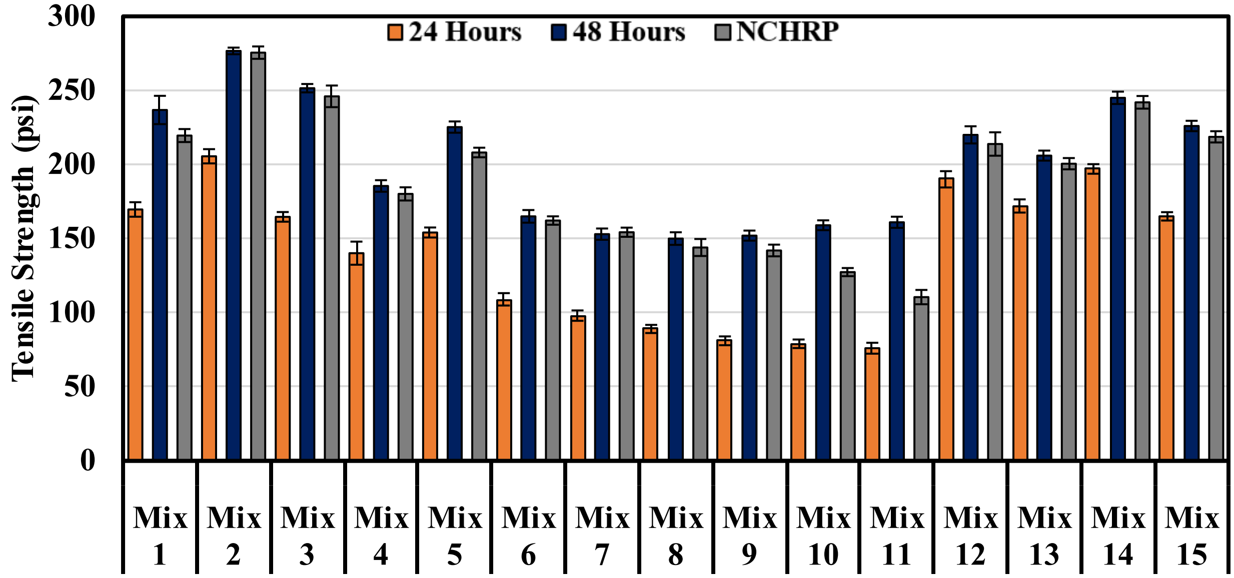


Figure 7.32. IDT Strength Comparison Using Protocol 3

Accelerated Aging Method: Ozone Enriched Method

For this protocol, an experimental method was developed for the accelerated simulation of long-term aging of loose asphalt mixtures using highly oxidative gas within one day (22 hours). The feasibility of this method was verified through mixtures prepared using five different binders during the initial evaluation regarding this method. Further development of the method has been conducted since and preliminary results are provided in the section below.

Materials used for Further Evaluation

The first part of the extended study focused on the evaluation of asphalt binders using Vocus PTR-TOF mass spectrometer. For this, three different unmodified RTFO aged binders were chosen from different geographical regions in Texas. The conventional properties of the materials are shown in Table 7.4.

Table 7.4. Performance Grade (PG) of binders used

Name	High Temperature PG (RFTO, Unaged) °C	Low Temperature PG (m-Value, Stiffness) °C
Binder-1	68.1, 70.7	-22.0, -22.3
Binder-2	71.1, 70.3	-24.2, -26.5
Binder-3	65.9, 65.5	-22.8, -24.9

In the second part, loose asphalt mixtures were aged using the developed ozone-based aging method. For this, three plant mixed mixtures were used, and the mix design followed the Superpave criteria (SP-C) and represented surface (wearing) courses. Table 7.5 shows the nomenclature, mix type and binder content of the mixes. Figure 7.33 illustrates the aggregate gradation of the mixes.

Table 7.5. Mix type and binder content of the mixes

Name	Mix Type & Binder Grade	Binder Content (%)
Sample-A	344-SP-C: 70-22	5.7
Sample-B	347-TOM-C: 76-22	6.5
Sample-C	344-SP-D: 76-22	5.6

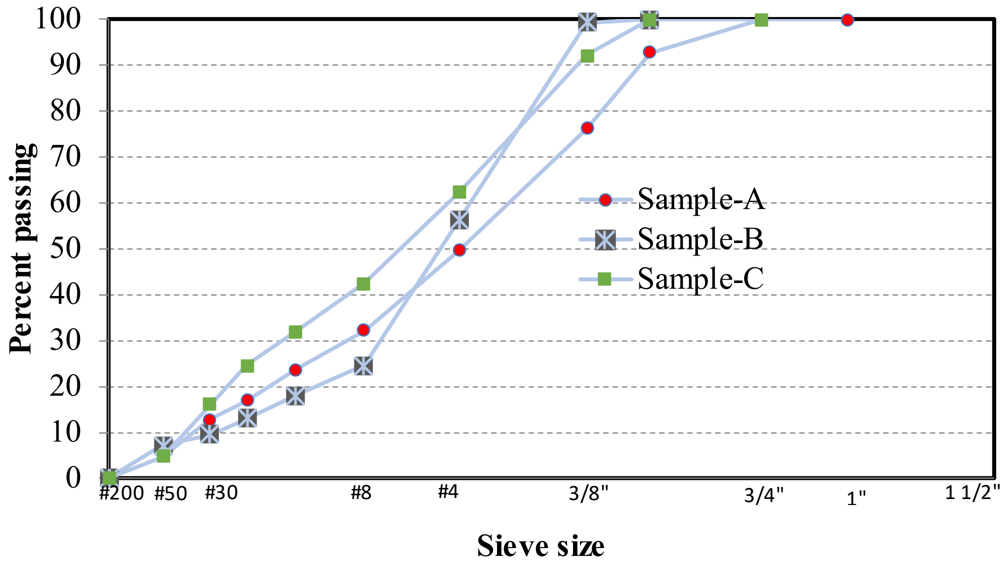


Figure 7.33. Grading Curve of Mixtures used in the Study

Experimental Methods: PTR-TOF

A Vocus 2R PTR-TOF-MS (Aerodyne, Inc., Billerica, MA) was used in this study to characterize the real time oxidation products of binders formed in an ozone and an air-based environment. This instrument has sub parts per trillion (ppt) levels of resolution and allows for the detection of a vast number of semi-volatile and low volatility volatile organic compounds (VOCs) and their oxidation products in real time.

The experimental setup is shown in Figure 7.34. 0.5 g of each binder (post RTFO aging) was poured in clear glass vial and placed in a glass tight bottle with gas tight fitting connections to create a flow through system. The sample chamber was placed inside a gas chromatography (GC) oven to control the temperature during the course of the experiments. The fittings were connected to two lines which supplied gas into the chamber and the other line served as an outlet to the PTR-TOF instrument for characterization. Two different gas blankets were used in this study namely, VOC free air (VOCFA) and VOCFA enriched with ozone. The ozone-based gas was produced by passing VOCFA through an ozone generator and the resultant gas had an ozone concentration of around 70 ppb. The flow rate of the flow-through chamber was set at 0.5 L/min. The binder samples were heated from 25°C to around 95°C in the GC oven which was held throughout the duration of the experiment. The VOFCA blanket and the ozone blanket were applied in succession for a period of about 1.5 hours where a steady state of volatiles could be observed in the real time preview displayer of the instrument. Following this, the initial data processing was conducted using PTRwid to obtain a list of feasible chemical formulas and their corresponding mass deviations from the specific m/z ratio. Further analysis and quality control of the data was done using MATLAB.

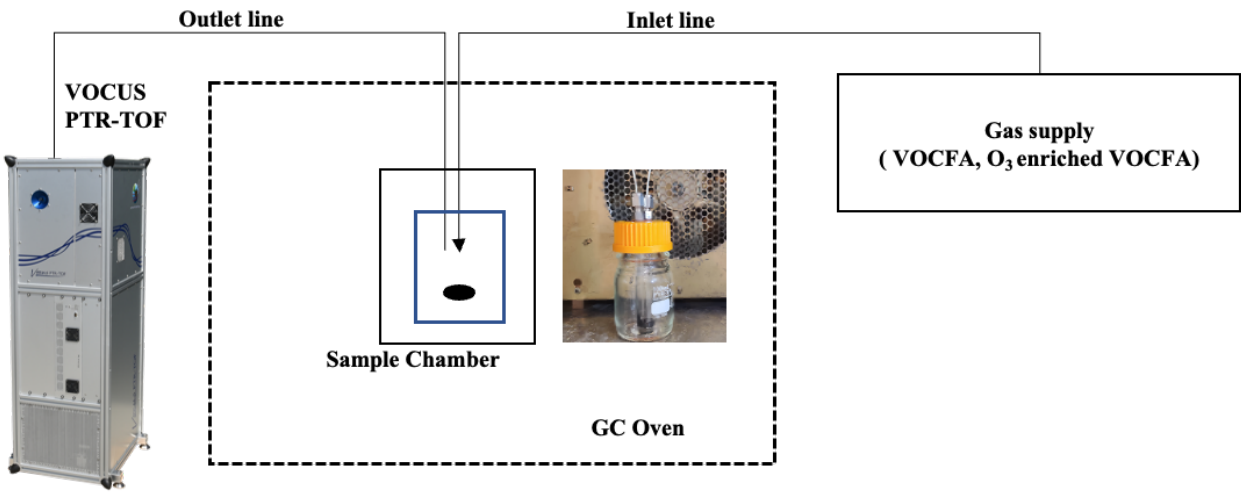


Figure 7.34. Experimental setup for characterization of binders using Vocus PTR-TOF

Upgradation of the Ozone-based Aging Setup

As described previously, the aging setup developed consisted of three main stages: (i) the production of ozone and reactive oxygen species (ROS) (ii) the aging of loose mixtures in an enclosed and heated tank, and (iii) the degradation of oxidative gases for release into an exhaust hood. The setup is illustrated in Figure 4. Purified compressed air at a pressure 138 kPa and flow rate around 500 ml/min was passed through an ozone generator manufactured by Oxidation Technologies, LLC. In the generator, a fraction of oxygen in the compressed air would be oxidized to ozone.

Following this, the produced ozone along with the ROS was led into aging tanks which were daisy chained together in series as illustrated. The tanks were enclosed in a laboratory oven which was used to control the temperature of loose mixtures in the tank. Inside each aging tank, around 3 kg of loose mixtures was placed around the perimeter of the wire mesh basket. This was done in order to keep the thickness of the sample consistent around 1 inch (2.54 cm). Following this, the gas was passed into a degradation unit filled with diesel engine fluid and exhausted into a fume hood. All fittings and tubing used in the set up were chosen to be compatible with ozone and ROS.

Firstly, large quantities of loose mixtures listed in Table 6 were short term aged as per Tex-206-F. This involves placing samples in a laboratory oven at 160°C for 2 hours. Following this, the samples were long term aged using two different protocols. In the first method, samples of loose mixtures were aged as per the NCHRP 09-54 recommended protocol. This involves a simple technique of placing measured quantities of loose mixtures in a laboratory oven for five days at 95°C. The climatic region of Texas determined the time duration of the test and was intended to replicate aging of about eight years (20mm below pavement).

The aging of around 9 kg of each loose mix sample was also conducted using the ozone aging method for a period of 22 hours at 95°C. This period was chosen based on practical considerations wherein the whole process of long-term aging could be achieved in around 24 hours i.e., 2 hours of short-term aging and 22 hours of ozone and ROS aging. Subsequently, the binders from the various mixtures i.e., short-term aged, ozone aged, and 5-day aged samples were extracted as per AASHTO T164.

Results and Discussion

Exceptionally complex emission profiles were observed for the different binders that displayed a large number of different compounds with specific mass to charge ratio (m/z). The increase in the complexity of the emission profiles with temperature is expected due to the enhanced volatility at higher temperatures, which boosts emission rates and the concentration of compounds that can be detected in the gas phase above the detection limit of the instrument. The emission rates observed were seen to span several orders of magnitudes as commonly observed with such complex organic compounds. The emission profiles of the three binders under the VOCFA and the ozone-enriched blanket are shown in Figure 7.35. The y-axis in the figure is a direct measurement of the emission concentration of individual molecules represented by their respective m/z values in the x-axis. For the three binders tested in this study, it could be seen that the emission rates of molecules were significantly higher under the ozone-enriched blanket as compared to the VOCFA blanket. Many of the different compounds detected were also multi-oxygen containing compounds and the magnitude of their intensity under the ozone-enriched blanket was considerably higher when compared to VOCFA, indicating higher extents of oxidation. As the flushing rate used in this study was considerably high (0.5 L/min), it is highly likely that the increased products and molecules observed under the ozone-based blanket are direct emissions of products formed during ozone-bitumen interaction, both at the surface and bulk level. This molecular level characterization of oxidized species provides evidence to support the previous hypothesis of using ozone to accelerate the oxidation process of asphalt mixtures.

The oxidation series of benzene, toluene and xylene & ethylbenzene detected during the experiments are shown in Figure 7.36. This figure illustrates the molecular abundance detected with the increase in oxygen molecules (averaged for the three different binders) for each series. The observation of such multi-oxygenated compounds firstly illustrate that the instrument is sensitive to observe even trace level low-volatility species. Secondly, the multi-oxygen compounds detected are naturally the products of real-time oxidation during the course of the experiments. It can be noted that there is an increased production of oxygenated species under the ozone-enriched blanket as compared to the VOCFA for each series, which indicates that this environment facilitates the accelerated oxidation of the constituent molecules of the binder. Moreover, the similarity of oxidation products detected under the two blankets suggests that the oxidation under ozone would likely lead to molecular transformations and structures akin to those naturally occurring during in field conditions.

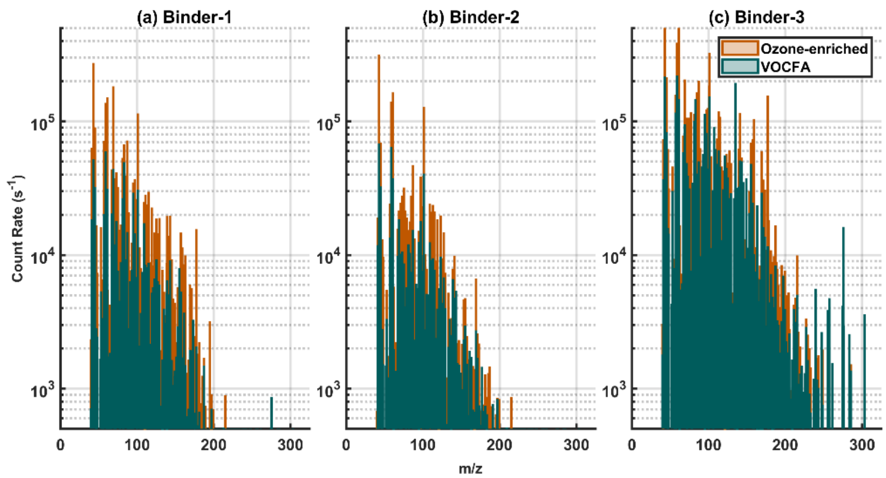


Figure 7.35. Molecular abundance under ozone enriched VOCFA and VOCFA

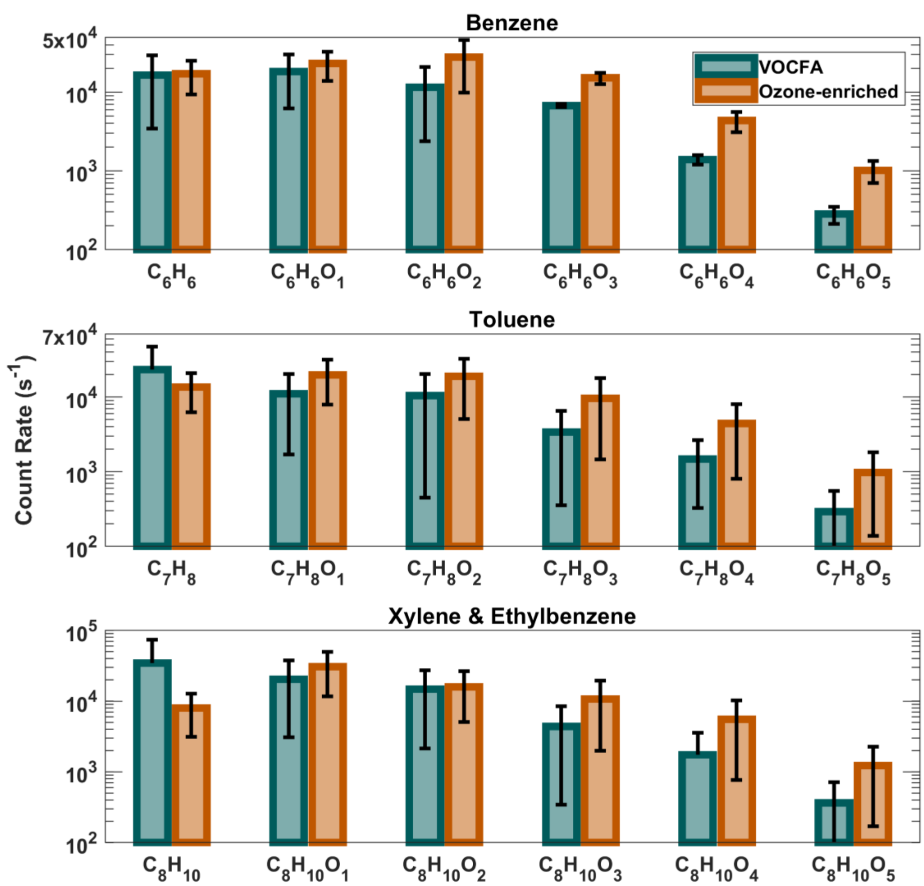


Figure 7.36. Oxidation series of benzene, toluene, and xylene & ethylbenzene

IDEAL-CT Results

The samples listed in Table 7.2 were tested post aging protocols using the procedure outlined for the IDEAL-CT test. The CT_{Index} values for the different mixtures are presented in Figure 7.37. It is well known that the aging induces crack propensity of mixtures due to loss of ductility of binders. Higher values of CT_{Index} are associated with lower levels of aging and less cracking propensity. Both long-term aging procedures showed significantly lower values for CT_{Index} as compared to the short-term aged specimens. For Sample-A, Sample-B and Sample-C, the CT_{Index} values for conventional long-term aged mixes were 82.7%, 50.9% and 79.6% lower than the values for short-term aged mixes respectively. Also, for the mixes aged under ozone, the CT_{Index} values for Sample-A, Sample-B and Sample-C were lower than the values for short-term aged mixes by 88.5%, 36.7% and 87.0% respectively. When comparing the two long-term aging protocols, similar results were obtained indicating very high crack propensity and consequently likely high oxidation levels of binders constituting those mixtures.

In terms of the tensile strength values, similar to the CT_{Index}, the 5-day aged samples and ROS aged samples were found to be similar, whereas the short-term aged samples showed lower tensile strength. The variation between ITS values for all the three mix types under these two aging conditions was less than 10%. This indicates that the ROS aging method which was only conducted for 22 hrs causes an equivalent increase in tensile strength as conventional five-day aging. Overall, the findings from the IDEAL-CT index test show analogous results for the ROS aging and 5-day aging methods, which highlights the comparability of aging levels of mixtures under these two methods.

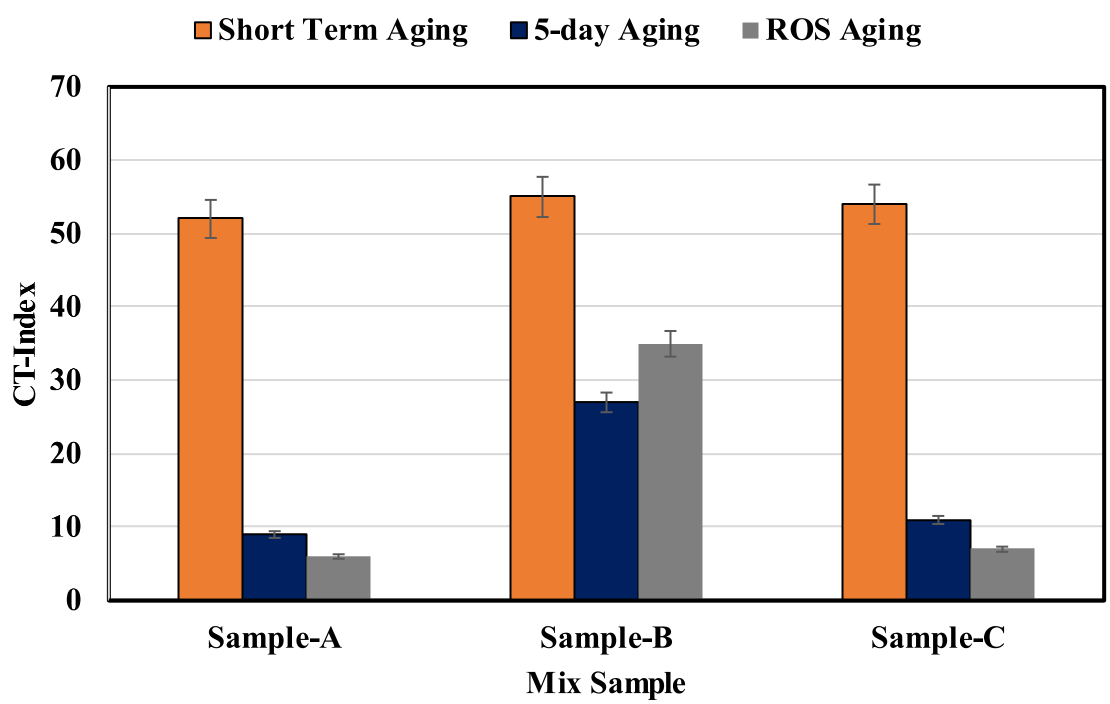


Figure 7.37. CT-index comparison of mixes aged under the three aging protocols

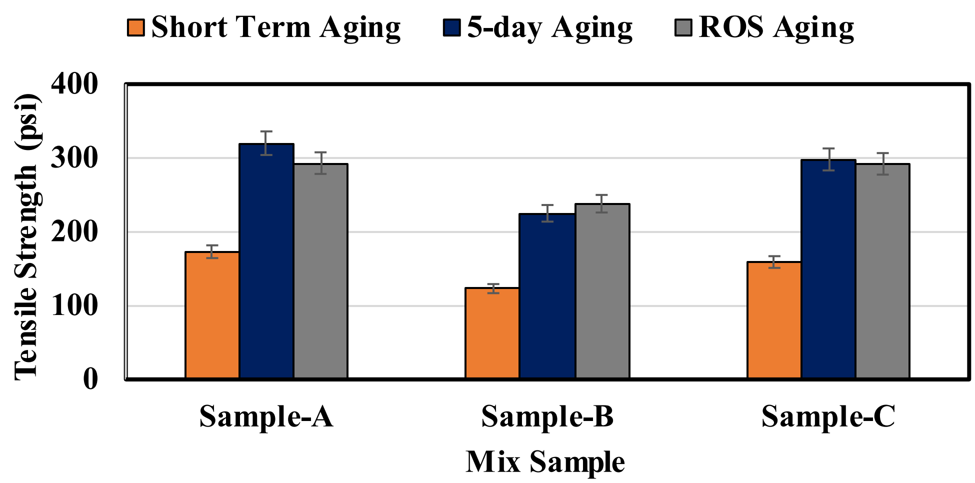


Figure 7.38. Tensile strength of mixes aged under the three aging protocols

Rheological Evaluation of Binders

Post mixture evaluation, the binders from the mixtures were extracted and the rheological and chemical analyses were conducted to further comprehend the aging levels of different mixtures.

The high temperature PG of the extracted binders were tested the results are summarized in Figure 7.39. The results were calculated based on the value of G*/sinδ parameter using the RTFO aged binder criterion in AASHTO T316. When comparing the extracted binders from the aging protocols, it was seen that in all three series, the binders aged using the ROS method for just 22 hours showed a greater high temperature PG compared to the binders from the five-day aging method. The difference between failure temperatures for Sample A, Sample B and Sample C under ROS aging and conventional five-day aging were 8.8°C, 8.8°C and 18.7°C respectively. This would constitute at least a one-grade increase as per PG specifications.

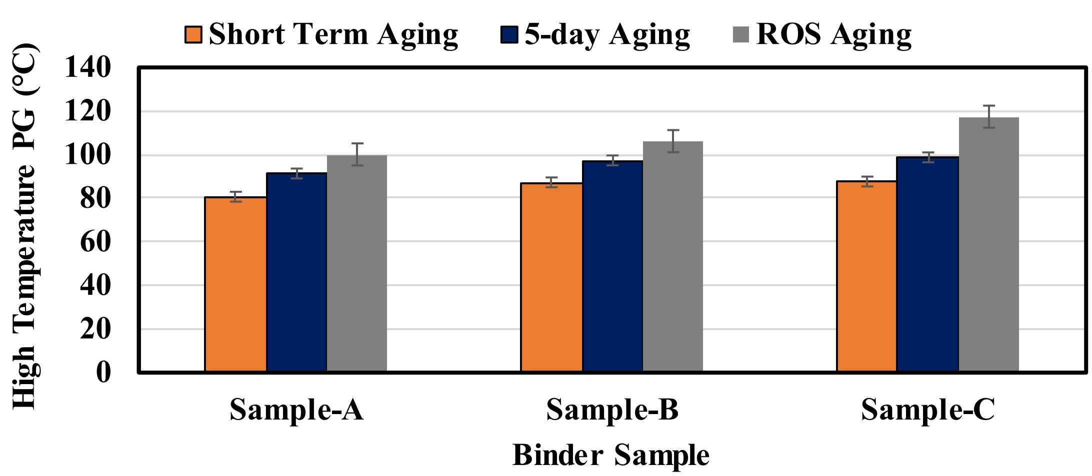


Figure 7.39. High temperature PG of binders under the three aging protocols

The low temperature properties of the extracted binders were also evaluated. The stiffness and m-value true grades of the binder are presented in Figure 7.40 (a) and (b) respectively. When comparing the same binders exposed to different aging methods, a higher value of the stiffness and m-value true grade indicates the presence of a stiffer binder that has undergone comparatively more oxidation and aging. The results obtained were consistent with the high PG results i.e., the binders aged using the ROS method showed higher values for stiffness and m-value as compared to the 5-day aging method. Overall, the evaluation of various extracted binders as per Superpave criteria clearly indicates that the ROS aging method imparts higher or similar levels of stiffness to the concomitant binders as compared to the 5-day aging method.

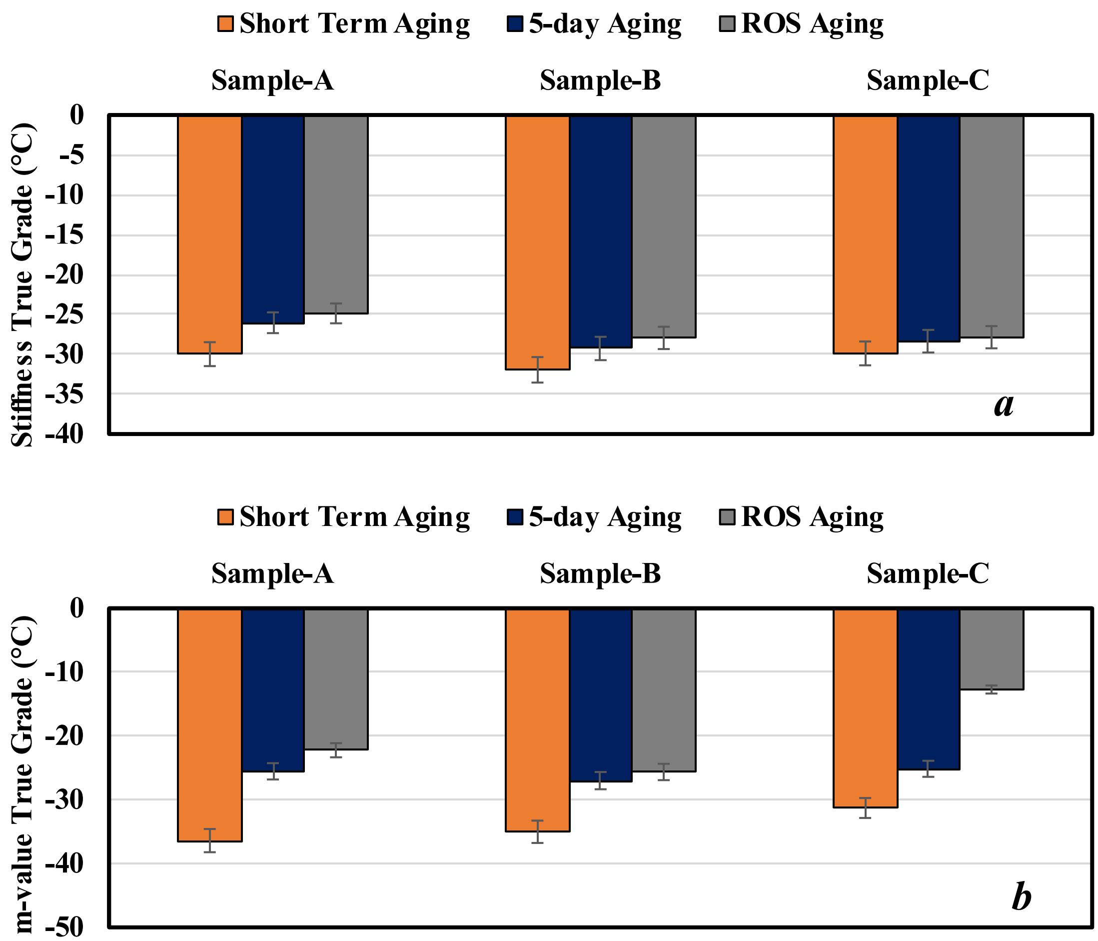


Figure 7.40. Low temperature PG of binders under the three aging protocols (a) Stiffness true grade, (b) m-value true grade

Evaluation by FTIR

The extracted binders aged using the 5-day aging and ROS aging were tested using the FTIR. The oxidation indices viz. carbonyl and sulfoxide groups were determined and are shown in Figure 7.41. For the three different samples, it was observed that carbonyl index values for binders aged using the ROS method were higher as compared to the 5-day long-term aged binders. Similarly, the sulfoxide index values were also comparable for all the binders under the two long-term aging conditions. Remarkably, the aging indices reveal that ROS aging employed only for 22 hours has a significant impact on the formation of carbonyls and sulfoxides and thus on binders aging. Overall, the results confirm the equivalency or enhanced aging potential of the ROS aging protocol over conventional long-term aging procedures.

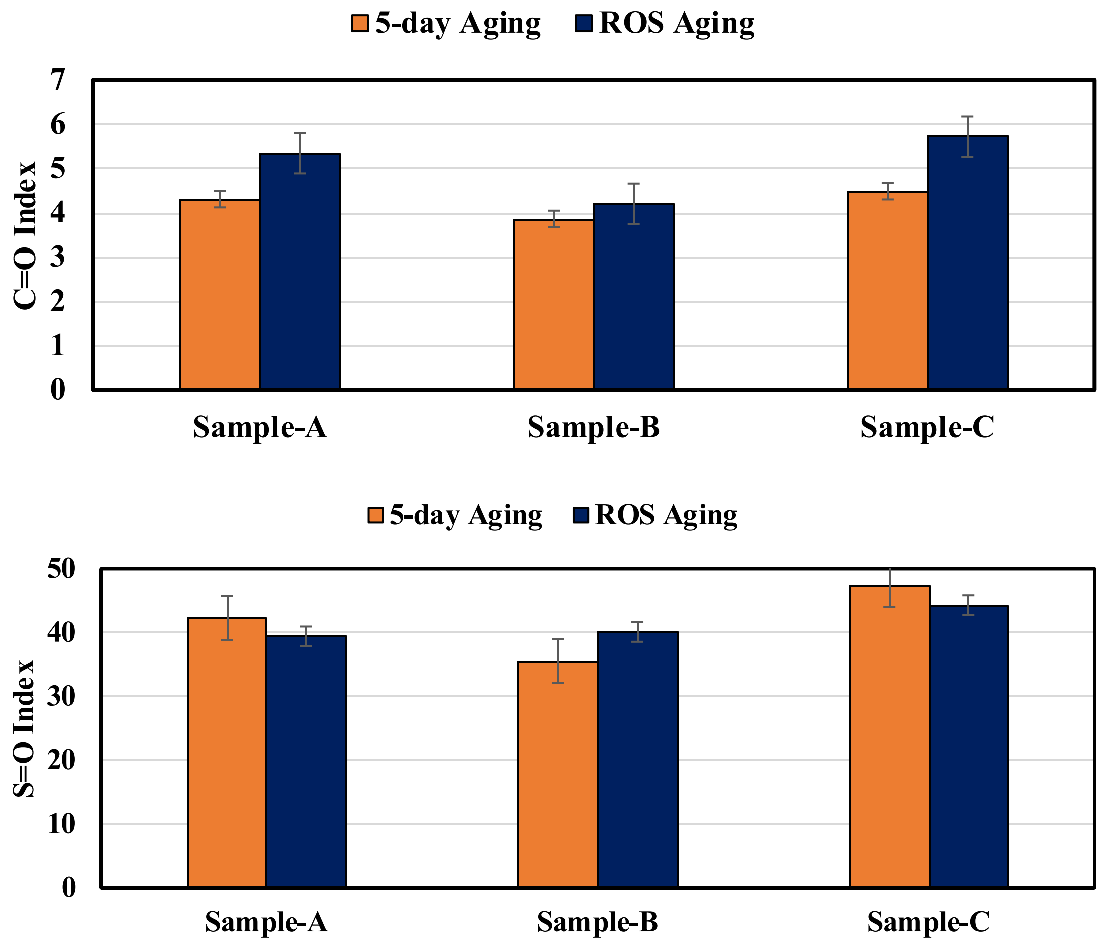


Figure 7.41. Aging indices of long-term aged binders (a) Carbonyl Index, (b) Sulfoxide Index

Summary

This chapter documented the findings from the Extended Evaluation of Aging Potential of Asphalt Concrete with Refined Laboratory Aging Protocols and documented its findings. The optimal aging protocols identified earlier in the project were further evaluated, including studying the influence of various mix design variables on aging characteristics. Temperature, duration, pressure, and aging environment varied among these protocols. The following points summarizes the main findings of this study based on the range of materials tested:

- Protocol 1 is a promising protocol for routine testing, but it is highly impractical to wait prolonged periods to determine a mixture susceptibility to cracking.
- For Protocol 1, mixture and corresponding binder tests showed a consistent increase in aging related metrics with increased testing time.
- Detailed analysis indicated that a minimum time to achieve levels of long-term aging would to relatively rank mixtures be close to 24 hours. Hence, the steady state of aging for different mixes may be adequately reflected after a period of 24 hours under Protocol 1 (including 2 hours of short-term aging).
- The aging trajectory remained generally the same when considering the use of different mix designs and variables.

- Mixture results especially in terms of tensile strength, CPR, and CFE best illustrated the steady state concept. Other mixture and binder properties was correlated to a lesser extent.
- The efficiency of Protocols 2 and 3, pressure aging and ozone gas, has been clearly demonstrated and its use may be considered as accelerated techniques for determining the mechanical properties using more accurate long-term aging.
- The rate of change of rutting characteristics are minimal beyond 2 hours of short-term oven aging.

Chapter 8 Verification of Preliminary Laboratory Aging Protocols

Verification of Preliminary Laboratory Aging Protocols

This chapter documents the preliminary work and research approach that was conducted in the parametric study relating to the verification of laboratory aging protocols. The parametric study was related to observing the influence of different components of the mix design and their effect on aging behavior and engineering properties. This chapter will provide data pertaining to field aging and mechanical performance of pavement sections.

Quantification of Aging Conditions

For an initial verification laboratory of aging protocols, the research team evaluated the field performance of asphalt concrete layers from relevant construction projects. To develop a database with field and lab performance data, a representative number of pavement sections relevant to the concept of this research project was used. A field evaluation and verification were conducted during *pre-construction*, *construction,* and *post-construction* stages to document every phase of the project. After the completion of the field evaluation activities, the pavement sections were revisited in six-month intervals as allowed by the length of this project. The collection of field cores is for further forensic study under the appropriate test methods. Relevant data from current and past TxDOT projects were monitored through condition surveys and performance testing.

Figure 8.1 illustrates examples of the performance of three pavement sections. The hollow symbols, representing the *pre-construction* evaluation of the mixes, have CPR values of about 0.30 and fall within the balanced region. The black symbols represent the evaluation during *construction* with similar CPR values. The gray symbols represent the *post-construction* evaluation with higher CPR values ranging from 0.35 to 0.45. Shortly after a mixture is placed, it is most prone to rutting. As the pavement ages, it becomes brittle and becomes more susceptible to cracking. If the performance diagram criterion were incorporated in the aged mixture design, Mix 3 would not have been accepted. As the material ages, the rutting characteristics of a mixture improves and, therefore, the early-age rutting of mixtures is considered for further evaluations.

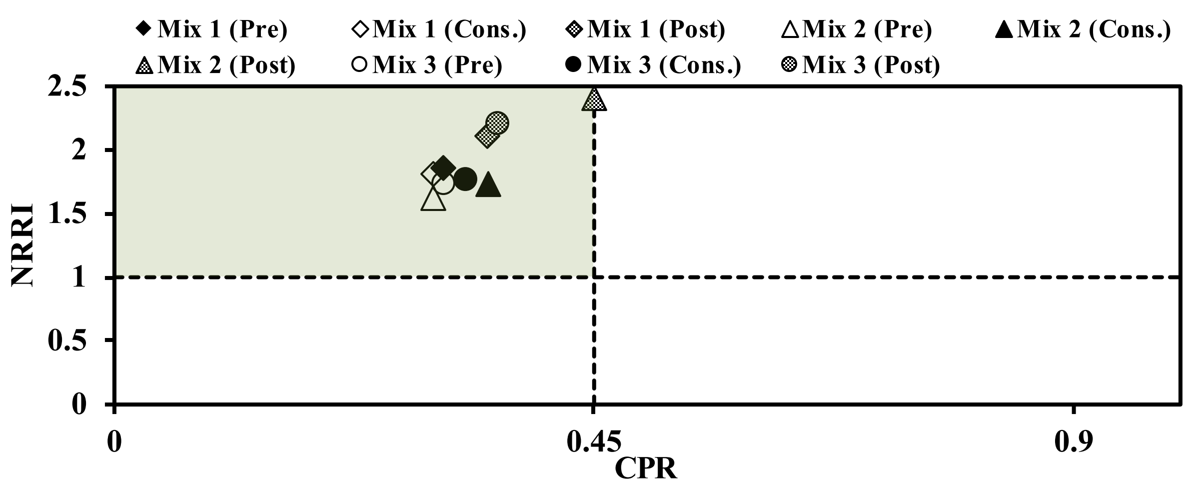


Figure 8.1. Interaction Performance Plot with a BMD Mix

Several AC mixes from across Texas that were evaluated to implement the concept of the Balanced Mix Design (BMD) into current practices were used. Table 8.1 summarizes the five projects selected for evaluation to determine the steady-state of the mix obtained from the implementation of the BMD. For the extended evaluation, five different mix variations were investigated including the RAP content, binder grade, aggregates, asphalt content, and additives. The binder grade and aggregate changes were also evaluated. To that end, different binder sources and grades, and different classification and gradation of the mix were considered.

Table 8.1. Overview of AC Mixtures from BMD Project

Mix No.	Mix ID	Mix Type	Binder Grade	Binder Percent	Additives	RAP
1	Yoakum SH-71	SP-D	70-22	6.1	Evotherm	20%
2	San Antonio FM-3009	SP-D	64-22	6.0	Evoflex	30%
3	San Angelo US-67	SP-C	70-22	5.3	N/A	10%
4	Childress US-70	DG-D	70-28	5.6	Blackledge	8%
5	San Antonio SH-85	SP-C	70-22	5.8	N/A	20%

Relevant data were extracted from the Implementation of the BMD project, and pre-construction information were considered as the trial batch of mix verification process. During this process, the optimum characteristics for each project were identified. *Construction* data were considered during the placement of the project and is often a plant-mix laboratory-compacted (PMLC) mix. Post-construction were cores taken during six-month intervals for further forensic study under the appropriate test methods and validation of the proposed aging protocols. The pre-construction and construction data available for the projects selected are presented in Table 8.2 and Table 8.3, respectively. However, many of the sections were only placed recently, so long-term evaluation was not possible at this stage of the project.

The cross plot of CPR and CFE shown in Figure 8.2 demonstrates examples of three *balanced* pavement sections with "good" crack performing mixtures and its progression as it ages. Previous sections from the DSS sections were not balanced using the rutting and cracking acceptance criteria. The hollow symbols represent the initial evaluation of the mixes that exhibits a satisfactory performance for CPR and CFE. The shaded symbols represent CPR from a field core 12-months post-material placement. These data points showed slightly worse CPR values but still fell within the acceptance criteria in terms of cracking. The patterned symbols represent the CPR and CFE values obtained on a core extracted 25-months post-material placement. Overall, as the pavement ages the cracking performance deteriorates. However, these balanced mixtures do not undergo major CPR changes as the pavement ages.

Table 8.2. AC Mixture Pre-Construction Performance Data from IAC BMD

Mix ID	IDT		OT		HWTT	
	CT Index	Tensile Strength, psi	CPR	CFE, in.lbs/in^2	NRRI	No. Cycles
ATL US-59	256	95	0.29	1.83	1.69	20000
YKM SH-71	31	158	0.30	1.92	1.78	20000
SAT FM-3009	226	79	0.28	1.34	1.33	20000
SJT US-67	30	166	0.36	2.87	1.73	20000
CHS US-70	99	119	0.30	1.71	1.74	20000
SAT SH-85	52	117	0.39	1.80	2.05	20000

Table 8.3. AC Mixture Construction Performance Data from IAC BMD

Mix ID	IDT		OT		HWTT	
	CT Index	Tensile Strength, psi	CPR	CFE, in.lbs/in^2	NRRI	No. Cycles
ATL US-59	71	110	0.32	2.69	1.73	20000
YKM SH-71	27	175	0.35	2.04	1.91	20000
SAT FM-3009	144	88	0.34	1.80	1.90	20000
SJT US-67	-	-	-	-	-	-
CHS US-70	-	-	-	-	-	-
SAT SH-85	-	-	-	-	-	-

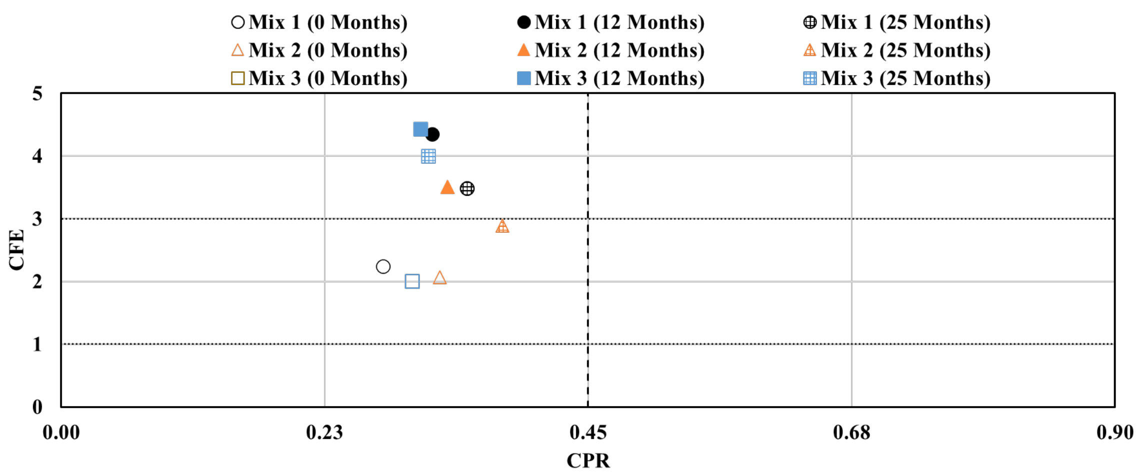


Figure 8.2. Interaction Diagram for Balanced Mixes (FM 3129)

Figure 8.3 illustrates examples of the proposed methodology for calibration of aging protocols. The hollow symbols represent a mixture used under the initial evaluation and its CPR progress. The cross symbols represent the field cores from during construction (first point) and 11-month post-material placement (second point). If the performance diagram criterion were used in the aged mixture design, this mixture would be equivalent to roughly 8 hrs to conventional oven aging.

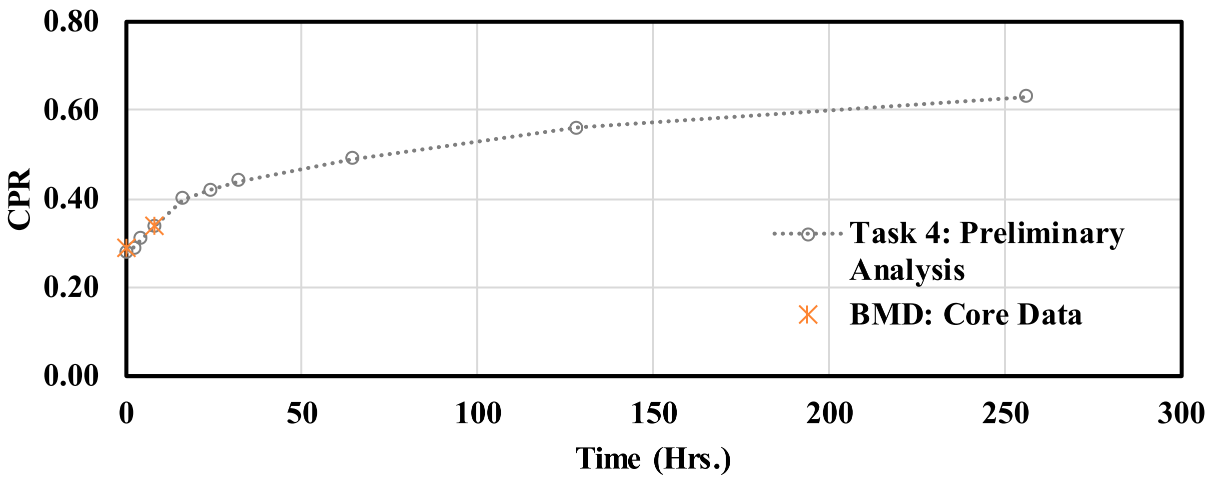


Figure 8.3. Laboratory and Field Cores Proposed for Calibration

Binder Performance Results

The figures below illustrate the results from extracted binders from three pavement sections from the balanced mix design project (FM 3129). The binders were extracted from cores at different times in its pavement life such as 0, 2, 15 and 25 months, respectively. The high temperature PG of the extracted cores is illustrated in Figure 8.4. The G*/sinδ parameter, as listed in AASHTO T316, was used to evaluate the data as per the RTFO aged binder criterion. It is understood that with an increase in aging of binders, there is an increase in complex modulus and a decrease of phase angle. Therefore, a larger value for high temperature PG can be associated with larger degrees of oxidative aging for the same binders aged under different conditions. The results show the clear increase in binder stiffness with increase in pavement age. The stiffness and m-value true grade are the main properties associated with low temperature properties of the extracted binders and the results are shown in Figure 8.5 and Figure 8.6, respectively. According to AASHTO T313 standards, 10°C below the minimum temperature for any binder where the stiffness does not surpass 300 MPa is defined as the stiffness true grade. Correspondingly, 10°C below the minimum temperature where the m-values surpasses a value of 0.3 is defined as the m-value true grade. For a given set of binders that were aged for various durations, higher values of stiffness and m-value true grades reflect higher levels of stiffness and hence more aged material. The results obtained were coherent with the high PG results, i.e., the extracted binders aged for longer durations showed higher values for these two parameters. This approach of analyzing extracted binders were used for the extended evaluation of extracted binders from cores in upcoming tasks, as detailed in previous sections.

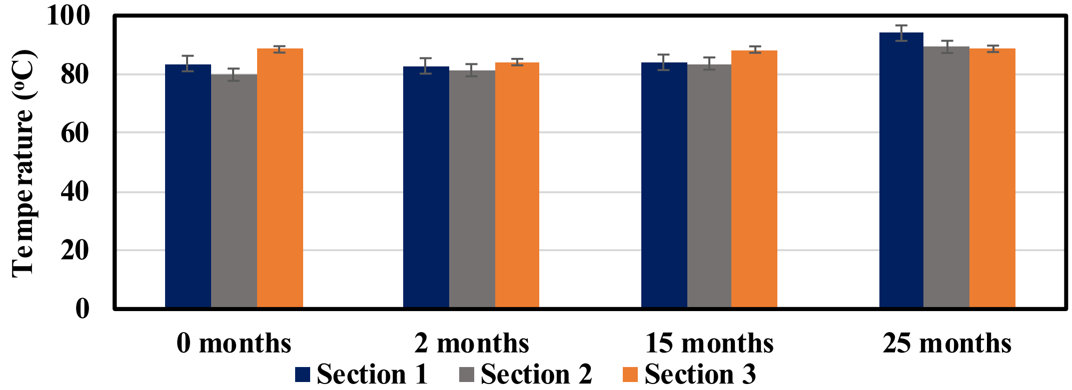


Figure 8.4. High temperature PG of extracted cores

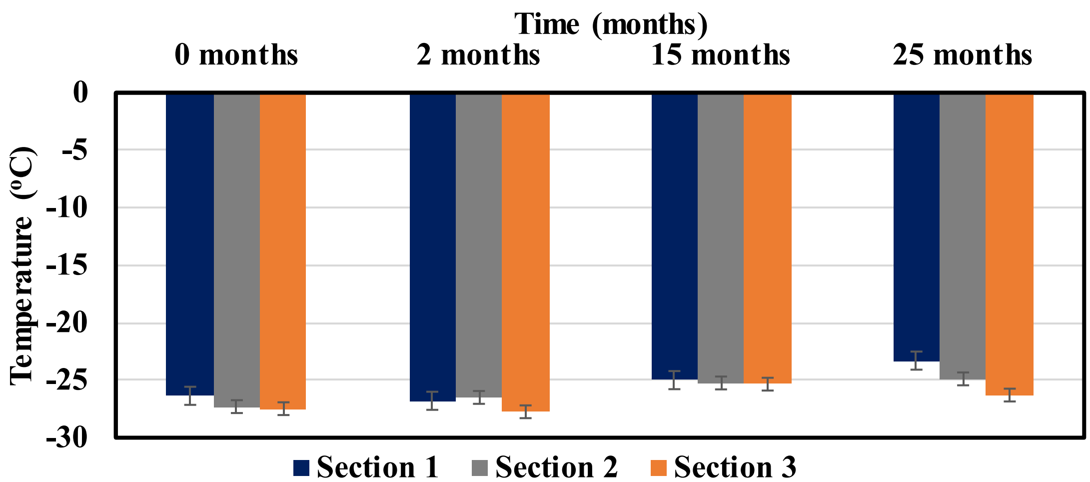


Figure 8.5. Low temperature PG- (Stiffness based) of extracted cores

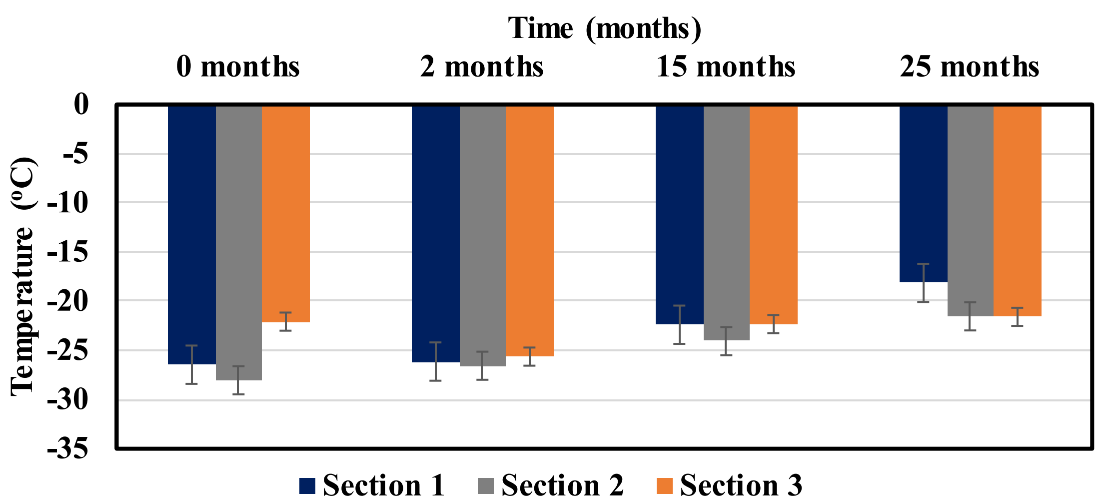


Figure 8.6. Low temperature PG (m-Value based) of extracted cores

Summary

This chapter corresponds to the preliminary verification of the proposed laboratory aging protocols, and documents the progress for this task. Interim data concerning field aging and mechanical performance of pavement sections is documented along with the primary performance indices that were tracked. Further performance indices from both mixture and binder aspects can be evaluated as field cores become available.

Chapter 9 Optimization of Laboratory Aging Protocols and Specifications

Optimization of Laboratory Aging Protocols and Specifications

This chapter presents preliminary, optimized lab protocols, specifications, and guidelines for the evaluation of aging performance based on the results of the study so far.

Three aging protocols were established by considering temperature, duration, and pressure as illustrated in Table 9.1. All the factors were considered in order to determine the selection of most suitable aging protocol. Optimized laboratory protocols and specifications are presented in this report for Protocol 1. Protocol 2 and Protocol 3 were mainly used for providing benchmarks for the long-term accelerated aging of mixtures within a short period. Work is currently being conducted to transform these methods to an advanced stage in terms of technological and implementation readiness and optimized laboratory specifications were presented at a later part of the project as part of the proposed extension.

Table 9.1. Optimized Laboratory Aging Protocol

Protocol No.	Temperature	Pressure	Environment	Curing Times*
1	Cured at 95°C	Atmospheric	Air	0, 10, 22, 46, 118 hours
2	Cured at 95°C	80 psi	Air	22 & 46 hours
3	Cured at 95°C	Atmospheric	Ozone and ROS	22 hours

** All protocols were exposed to 2 hours of short-term aging as per Tex-206-F. Curing times shown above are beyond this 2-hour short-term aging but include 2-hour high temperature conditioning used immediately prior to compaction.*

Summary

This chapter refers to the preliminary laboratory aging protocols and presents preliminary, optimized lab protocols, specifications, and guidelines for the evaluation of aging performance based on the results of the study. Draft protocols for both short- and long-term aging of loose mixtures are presented, and this is expected form the basis for inclusion in technical guidelines of agencies.

Test Procedure for *Proposed Standard for Short- and Long-term Aging*

LOOSE MIXTURE AGING OF ASPHALT MIXTURES

TxDOT Designation: XXXXX

Effective Date: DRAFT

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1. SCOPE

1.1 This test method simulates the field aging of loose asphalt mixtures in a laboratory setting. The protocol will simulate the two critical aging states needed to design an asphalt mixture to resist rutting and cracking and provide information on how curing effects the physical and engineering performance of binders and mixtures.

1.2 The rutting and cracking resistance of mixtures evaluated from these tests is expected to be a part of a mix design process (e.g., as in the case of a balanced mix design).

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2. APPARATUS

2.1 *Forced-draft oven,* capable of attaining the temperatures specified in the procedure.

2.2 *Balance,* Class G2 in accordance with Tex-901-K, with a minimum capacity of 10,000 g.

2.3 *Thermometers*, covering the range from 122°F to 500°F (50°C to 260) readable to the nearest 2°F (1°C).

2.4 *Miscellaneous equipment*, shallow metal pan for aging loose mix, metal spatula or spoon for stirring, oven gloves.

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3. MATERIALS

3.1 For selection and design of mixtures refer to Tex-204-F.

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4. MIXING

4.1 For mixing procedures of lab produced mixture, refer to Tex-204-F.

4.2 For sampling procedures of plant produced mixtures, refer to Tex-222-F.

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5. PROCEDURE

5.1 *Short-term Oven Aging*

5.1.1 The short-term oven aging is used to condition mixes prior to preparing specimens for mechanical testing and this procedure applies to laboratory-prepared loose mixtures only.

5.1.2 Place the required amount of loose mixture in a pan, and spread it to an even thickness of 2 in. Place the loose mixture and pan in the conditioning oven for 2 hrs ± 5 min at the compaction temperature of the mixture as per the guidelines listed in Tex 206-F.

5.1.3 After 2 h ± 5 min, remove the mixture from the forced-draft oven. The conditioned mixture is now ready for further conditioning or compaction and must be immediately transferred.

5.2 *Long-term Oven Aging*

5.2.1 The long-term oven aging procedure is used for the mixture mechanical property testing procedure and applies to laboratory-prepared mixtures that have been subjected to the short-term conditioning for the mixture mechanical property testing procedure described in Section 5.1, plant-produced mixtures, and other applicable mixtures.

5.2.2 Following a mixture's exposure to short-term oven aging or plant production, transfer the mixture to a conditioning oven for 120 ± 0.5 hrs at a temperature of 95 ± 3°C (203F). The mixtures must be spread in a pan and the layer thickness should be retained at 2 in

5.2.3 After 120 ± 0.5 hrs, immediately transfer the mixtures to an oven set at the compaction temperature of the mix in order to compact the samples. Do not touch or remove the specimen until it has cooled to room temperature.

Note 1—Cooling to room temperature will take approximately 12 hrs.

5.2.4 After cooling to room temperature, the long-term oven aged specimen is now ready further testing as required.

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6. PRECISION

6.1 The precision of this test method has not been established.

Chapter 10 Conclusions and Recommendations

Conclusions

The main focus of this study was on long term aging and its impact on cracking performance. To that end, the following conclusions were drawn based on the extensive work conducted in this study:

- Protocol 1 is a promising protocol for routine testing, but it is highly impractical to wait prolonged periods to determine a mixture susceptibility to cracking.
- For Protocol 1, mixture and corresponding binder tests showed a consistent increase in aging related metrics with increased testing time.
- The steady state of aging i.e., the aging time and duration after which the relative rank order of cracking performance does not change for different mixes may be adequately reflected after a period of 24 hours under Protocol 1 (including 2 hours of short-term aging).
- The efficiency of Protocols 2 and 3, pressure aging and ozone gas, has been clearly demonstrated and its use may be considered as a more efficient alternative for day-to-day operation of realistically simulating long-term aging for rigorous evaluation.
- The rate of change of rutting characteristics are minimal beyond 2 hours of short-term oven aging.

Recommendations

- This study recommends Protocol 1 as a s testing method that can be employed during routine mixture study, optimized laboratory protocols, and procedures have been established in this study. The steady state of aging for different mixes is achieved after a period of 24 hours under Protocol 1 (including the adopted 2 hours of short-term aging).
- Protocol 2 and Protocol 3 showed good promise as benchmarking tests for the realistic long-term accelerated aging of mixtures within a short period. However, work is still required to transform these methods to an advanced stage in terms of implementation readiness, and development of optimized laboratory specifications. The research team recommends the further consideration of these methods in future studies.

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