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Development of a laboratory medium-term oven aging (MTOA) protocol with field validation for asphalt mixes containing RAP

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ABSTRACT

In this study, laboratory loose mix oven aging was compared to field aging to begin calibration for a medium-term oven aging (MTOA) protocol. Five different oven aging temperatures were implemented at different durations to evaluate the change in chemical and rheological aging parameters for four plant-produced asphalt concrete (AC) mixes. The sulfoxide (SUL) index failed to show a consistent trend with aging for extracted binders obtained from both loose mixes and field slabs. The carbonyl area (CA) index was found to show a linear trend with the binders' rheological parameters. The results of the field calibration indicate that loose mix oven aging of 20 h at 100 °C can simulate field aging at 0.5–0.6 in. (12 mm to 15 mm) depth after six years of pavement life in the climate region and for the material tested. Based on these results for a hot climate, loose mix aging of 20 h at 100 °C was proposed as the MTOA protocol for AC mixes for future studies.

1. Introduction

The implementation of balanced mix design (BMD) for asphalt concrete (AC) depends on the development of laboratory aging techniques that simulate both short- and longer-term aging of asphalt pavement. Some researchers have considered the asphalt binder in characterizing aging and others considered the asphalt mixes [1-3]. Two commonly used laboratory techniques to simulate the short-and long-term aging of asphalt binders are the rolling thin film oven (RTFO) and pressure aging vessel (PAV) tests, respectively. The aging process gets more complicated when physicochemical interactions between asphalt binder and aggregate occur in the presence of air voids for asphalt mixes [1,3]. The AASHTO R 30 test method is the most common method for short-term aging of laboratory-produced asphalt mixes, where 2 h of oven aging at 135 °C are specified on loose mixes to simulate short-term field aging [4]. To achieve long-term oven aging (LTOA), an additional 5 days of oven aging at 85 °C was suggested on the short-term oven-aged

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(STOA) compacted specimen. According to the AASHTO R 30 test method [4], this LTOA method typically corresponds to 7–10 years of service life. However, it was reported by Kim, Castorena [1] that the laboratory aging of the compacted specimens does not simulate the actual field aging trend of asphalt pavement because the oxidation gradient varies along the radius of the compacted specimen, while in the field a height-wise aging gradient is typically observed [1]. Also, differences in performance for laboratory and field compacted specimens have been reported by Jin, Boateng [5]. Therefore, several researchers have tried to use loose asphalt mixes to come up with a protocol for LTOA [1,6].

Homogenous aging and maintaining material integrity are the benefits of using loose mixes instead of compacted specimens for the LTOA. However, choosing a temperature for loose mixes in LTOA is still a point of concern for the research community. Petersen [7] reported that disruption of polar molecular groups and sulfoxide decay were found to be critical for asphalt mixes at temperatures greater than 100 °C. Therefore, several researchers have considered an oven aging temperature of 95 °C for the LTOA [1,8,9]. A decrease in both dynamic modulus values and fatigue resistance was reported by Rad, Elwardany [9] after considering the LTOA temperature of 135 °C compared to 85 °C for two out of three mixes. The binder's chemical properties were found to change differently compared to rheological properties while considering the carbonyl and sulfoxide absorbance peak as the chemical parameter at 135 °C [9]. Therefore, a temperature of 95 °C was selected by Kim, Castorena [1] for LTOA of AC mixes in the laboratory. However, a much longer laboratory oven aging period (1–37 days) was proposed by Kim, Castorena [1] to go with the lower temperature. The proposed laboratory aging period may not be compatible with the field requirements for quality assurance and quality control (QA/QC) purposes and may be too long for job mix formula approval as well. Therefore, a shorter aging period with higher oven aging temperature for LTOA was proposed by other researchers [6].

A study conducted at the National Center for Asphalt Technology (NCAT) proposed an oven aging protocol of 8 h at 135 °C for evaluating the field performance of AC mixes [6]. They reported a good correlation between the carbonyl area (CA) index and the binder's rheological parameters even at 135 °C oven aging [6]. A strong correlation between chemical and rheological properties was also reported by Kim, Castorena [1] while considering only the CA index as the chemical parameter instead of carbonyl and sulfoxide absorbance peak. Based on that, the chemical aging parameter sulfoxide may not be a stable element above 100 °C as reported by Petersen [7]. Therefore, there is a need to develop a reasonable laboratory oven aging protocol that results in rheological changes that match those occurring in the field, which will occur where the aging protocol produces chemical changes that match those occurring in the field. The medium-term oven aging (MTOA) aging protocol can be a way to solve this issue. This MTOA protocol is expected to allow a reasonably lower oven aging temperature with a shorter aging period and is expected to simulate the aging to mid-life (2-15 years, depending on the depth and the climate region) for asphalt pavement. Also, the aging indices change linearly after an initial fast rate of aging [2,10,11]. Therefore, the MTOA can also be used in predicting the aging parameters for LTOA. Another consideration is that the aging process gets more complicated for AC mixes containing reclaimed asphalt pavement (RAP) and recycling agents [3,12]. To construct more cost-efficient and more sustainable transportation systems, the asphalt industry has been using RAP and recycling agents for more than forty years [13–19]. The presence of RAP is anticipated to reduce the aging rate, recycling agents are expected to have the opposite effect. Therefore, there is a need to develop a reasonable aging protocol for AC mixes containing recycled materials. Based on that the objectives of this study are:

- To evaluate the variation of field aging parameters with depth for AC layers containing recycled materials,
- To evaluate the effect of different laboratory aging temperatures and duration on the aging of mixes containing RAP and recycling agents,
- To evaluate the correlation between binder rheological and chemical aging parameters for mixes containing RAP and recycling agents in both the laboratory and the field,
- To propose a draft medium-term oven aging (MTOA) aging protocol for mixes containing recycled materials.

Mix No.	Project ID	Mix ID	Gradation type	RAP/RAS content by TWM (%)	Base binder PG	%AC by TWM	% RA	RA type	No. of lots	Loose mix oven aging	Binder source	Field aging
1	SD	SD 76	Dense	25	PG 64–10	5.0	0	NA	1	Yes	California A	Yes
2	NAPA	NAPA 29	Dense	15	PG 64–16	5.0	0	NA	1	Yes	California B	No
3	SJ	Virgin-SJ	Dense	0	PG 64–28PM	4.7	0.0	NA	2	Yes	California B	No
4	SJ	RAP40 % RA1.5 %-SJ	Dense	40	PG 58–34PM	4.0	1.5	Tall oil- based	2	Yes	California B	No

Description of asphalt mixes considered in this study

Table 1

TWM = total weight of mix, TWB = total weight of binder, PMLC = plant-mixed, lab-compacted; NA = not applicable; PG = performance grade; PM = polymer modified; RAP = reclaimed asphalt pavement; RA = recycling agent.

2. Materials and methods

2.1. Experimental design

2.1.1. AC mix types

In this study, four different plant-produced loose AC mixes (labeled SD 76, NAPA 29, Virgin-SJ, and RAP40 % RA1.5 %-SJ) were considered to evaluate the effect of oven aging temperature and duration on the aging pattern. Table 1 shows the details of all four plant-produced AC mixes. The laboratory aging to field aging was compared for the SD 76 mix only. In 2016, an asphalt pavement was constructed on state highway 76 in northern San Diego County in California. During the construction, plant-produced asphalt mixes (SD 76) were collected from the asphalt plant and stored in a temperature-controlled room in sealed steel buckets. The asphalt pavement is comprised of four AC layers. For all AC layers, the same base binder (PG 64–10) was used with a 25 % inclusion of reclaimed asphalt pavement (RAP). The base binder for all four layers was collected from California A refinery source as shown in Table 1. In 2022, field slabs were collected from this highway after six years of pavement life to evaluate the gradient of aging in the field. In this study, field slab samples from this pavement were considered within 50 ft (15.24 m) pavement length to minimize the field variability in the horizontal direction during construction.

The NAPA 29 (NAPA county) plant-produced AC mix was considered to provide a different base binder refinery source (California B) for the study. This mix was collected in the year 2016 and stored in a temperature-controlled room in a sealed bucket. The effect of a high percentage of RAP with recycling agent in mixes was evaluated by comparing the aging gradient of RAP40 % RA1.5 %-SJ mix to the Virgin-SJ mix. Both mixes were used in the surface layer of state highway SJ 26 in the year 2022 in San Joaquin County. The same refinery (California B) was used for polymer-modified (PM) base binders used in the RAP40 % RA1.5 %-SJ and Virgin-SJ mixes. However, the base binder used for Virgin-SJ and RAP40 % RA1.5 %-SJ mixes were PG 64–28PM and PG 58–34PM, respectively. Also, a tall-oil based recycling agent (1.5 % by total weight of binder) was used to produce RAP40 % RA1.5 %-SJ mix.

2.1.2. Laboratory loose mix oven aging

Five different oven temperatures (85 °C, 100 °C, 110 °C, 120 °C, and 135 °C) for different durations were implemented to evaluate the change in chemical and rheological aging parameters. Fig. 1 shows the workflow diagram of this study. Before starting the oven aging at different temperatures, the stored plant-produced mixes were heated at 135 °C for 2–4 hours to make them workable. Then mixes were transferred to smaller pans from buckets. Each pan contains about 1.8 kg to 2.0 kg of loose mixes. Then loose mixes were oven-aged at five different temperatures for different durations (maximum of 185 hours). The aging period was selected based on the oven aging temperatures to provide comparisons.

2.1.3. Field slabs breakdown

The bulk specific gravity of field slab specimens was determined in the lab using AASHTO T331 test methods. To break down the field slabs collected at year 6 from the SD 76 project, a temperature of 100 °C for 40 minutes was used as shown in Fig. 1.

2.1.4. Laboratory testing

To collect binder samples from loose AC mixes and broken slab specimens, an auto-extractor was used to extract the asphalt binder from asphalt mixes and heated slab samples following the ASTM D 8159 method. The extracted binder was then recovered using the rotary evaporation process following ASTM D 5404. The following binder tests were conducted in the laboratory to characterize the aging of asphalt binder:

2.1.4.1. Chemical aging test. The chemical properties of the extracted binders were evaluated using Fourier transform infrared spectroscopy (FTIR). The spectra measured by the FTIR were recorded in a reflective mode, from 4000 to 400 cm⁻¹, at a resolution of



Fig. 1. Workflow diagram.

 4 cm^{-1} . For each measurement, an average value of 24 scans was recorded. Nine replicate measurements were taken to ensure that representative measurements were collected for each binder sample. The CA index and sulfoxide (SUL) area index determined from FTIR were used to track chemical properties with aging. The tangential integration of the component area calculated for CA index was in between 1671 and 1720 cm⁻¹ wavenumber [2]. To account the SUL component area was calculated in between 982 and 1050 cm⁻¹ wavenumber [2]. The aliphatic band at 2923 cm⁻¹ was used to normalize the spectra and eliminate any variability introduced by the operator and any background impacts between repeat measurements. Previous literature suggested that this aliphatic band structure is not affected by aging over time [20,21]. Eq. (1) was used to integrate the chemical component area index.

$$I_{i} = \int_{w_{l,i}}^{w_{u,i}} a(w)dw - \frac{a(w_{u,l}) + a(w_{l,i})}{2} \times (w_{u,i} - w_{l,i})$$
(1)

where: $I_i = \text{index}$ of area i $w_{l,i} = \text{lower}$ wavelengthintegral limit of area i $w_{u,i} = \text{upper}$ wavelengthintegral limit of area i a(w) = absorbance as the function of wavelength

2.1.4.2. *Rheological aging test.* Rheological properties were determined with a dynamic shear rheometer (DSR). The complex shear modulus (G^{*}) and phase angle (δ) values at four different temperatures (5, 10, 25 and 40 °C) and at 16 different testing frequencies (0.02–15.92 Hz) for all extracted binders were evaluated. A symmetric sigmoidal fit function was used to convert the frequency sweep data into a master curve at the reference temperature using the fit function in Eq. (2). The reference temperature considered in this study was 15 °C. Eq. (2) can be used to generate a binder master curve for shear complex modulus (G^{*}).

$$\log|G^*| = \delta + \frac{\alpha}{1 + e^{\beta + \gamma \log \omega f_r}}$$
⁽²⁾

where: $|G^*| =$ magnitude of complex modulus (kPa)

 α = fitting parameter (the high asymptote of the master curve)

 δ = fitting parameter (the lower asymptote of the master curve)

 β, γ = fitting parameters (the slope of the transition region of the master curve)

 $\omega =$ frequency (Hz)

 f_r = reduced frequency, which is the shifted frequency at the reference temperature from the frequency at the test temperature (Hz)

The reduced frequency can be calculated using the Williams-Landed-Ferry shift function, which is based on the time-temperature superposition as shown in Eq. (3) [22].

$$\log(\alpha_T) = \frac{-C_1(T - T_r)}{C_2 + (T - T_r)}$$
(3)

where: α_T = shift factor as a function of temperature T

T = test temperature in Kelvin (°K) T_r = reference temperature in Kelvin(°K) $C_1 and C_2$ = fitting parameters

Then Eq. (4) obtained from the Kramers-Kronig relation was used to develop the phase angle master curve [23]. The time-temperature shift factor was calculated using Eq. (3).

$$\delta(fr) = \frac{\pi}{2} \frac{\alpha \gamma}{\left(1 + e^{\beta - \gamma \log(f_r)}\right)^2} e^{(\beta - \gamma \log f_r)}$$
(4)

 α = fitting parameter (the high asymptote of the master curve)

- δ = fitting parameter (the lower asymptote of the master curve)
- β , γ = fitting parameters (the slope of the transition region of the master curve)

 $\omega =$ frequency (Hz)

 f_r = reduced frequency, which is the shifted frequency at the reference temperature from the frequency at the test temperature (Hz)

Several other aging parameters were calculated using the G^* and δ values as shown below:

1. Glover-Rowe (GR) Parameter: The GR parameter was calculated using G^* and δ values at 15 °C and 0.005 rad/sec as shown in Eq. (5). This parameter increases with an increase in aging level [24].

(5)

$$GR = \frac{G^* \cos^2 \delta}{\sin \delta}$$

- 2. G* at 64 °C and 10 Hz: The literature suggests that the binder' G* value at 64 °C and 10 Hz is appropriate for evaluating the aging of asphalt. This rheological parameter also increases with an increase in aging level [1].
- 3. Crossover modulus (G_c^*): The crossover modulus is defined as the modulus when the phase angle (δ) is 45°. The G_c^* values decrease with an increase in binder aging [3,25].
- 4. Crossover frequency (ω_C): The crossover frequency is defined as the frequency when the phase angle (δ) is 45°. The degree of aging also decreases the ω_C values.

3. Results and discussion

3.1. Climate region of SD 76 project

Based on the climate, California can be divided into nine different regions [26]. The field project considered in this study (SD 76 state highway) is located in the south coastal region of California [26]. Fig. 2 shows the variation in maximum daily air temperature for a nearby weather station (Los Angeles) starting from year 2016 to year 2019. The maximum daily weather data were collected from the National Climate Data Center (NCDC). Fig. 2 illustrates that the maximum daily air temperature was always greater than the freezing temperature of zero degree Celsius. Therefore, the cumulative degree-days (CDD) will be a direct sum of the total days to be considered. The CDD is defined as the accumulation of daily high temperatures above freezing temperature (0 °C) for all the days being considered. This parameter can account for both the effect of climate and in-service days on field aging [27]. As shown in Fig. 2, the highest value of maximum daily air temperature is 39.4 °C for the asphalt pavement location at the SD 76 project. Therefore, the pavement considered in this study is in a hot climatic region.

3.2. Field aging

Fig. 3 shows the rheological properties of the extracted binders at different depths for the SD 76 project after 6 years of service life. As discussed earlier, this asphalt pavement consists of four AC layers. In this study, the top AC layer (Layer 4) was subdivided into four equal sub-lifts to evaluate the effect of ultraviolet (UV) rays in the top lift. For all other layers (Layers 1–3) no sub-lift was considered. The typical thicknesses observed for Layer 1, Layer 2, Layer 3, and Layer 4 are 2.6 in. (66 mm), 2.2 in. (55 mm), 2.0 in. (50 mm), and 2.3 in. (58 mm), respectively. Field slabs were collected from two different locations (Spot 1 and Spot 2) within 50 ft (15.24 m) pavement length to minimize the field variability.

As shown in Fig. 3, different binder rheological parameters were found to convey similar aging trends at different depths of AC layers for both locations (Spot 1 and Spot 2). With aging, the GR and G^{*} values are expected to increase and G_c^* and ω_c values are expected to decrease [3,25]. As expected, the top sub-lift of surface layer (Layer 4) was found to show much higher aging compared to lower sub-lifts. The GR values found at the top and bottom of Layer 4 for Spot 1 were 2375 kPa and 222 kPa, respectively. A similar trend was also observed for the G^{*} values at 64 °C and 10 Hz. The G^{*} values observed at the top and bottom of Layer 4 for Spot 1 were 409 kPa and 92 kPa, respectively. This is mainly attributed to the exposure to UV rays and higher temperature at the top sub-lift compared to the bottom sub-lift of Layer 4. Similar findings were also reported by other researchers [1,25,28].

At a depth of 1.5 in. (37 mm) much lower aging indices were observed compared to other sub-lifts of the surface layer (Layer 4) as



Fig. 2. Variation in maximum daily air temperature for a nearby weather station from SD 76 project at different years, °C.



Fig. 3. Variation of rheological parameters (a) log (GR, kPa), (b) log (G* @ 64 °C and 10 Hz), (c) log (G_c^* , kPa), (d) log (ω_c) with depth in the field for SD 76 project. Note: (- - - - - -) line corresponds to aging parameters of losse SD 76 mixes for 20 hours @ 100 °C in the laboratory.

shown in Fig. 3. The GR and G* values found at 1.5 in. (37 mm) depth for Spot 1 were 18 kPa and 41 kPa, respectively. Therefore, lower aging indices were observed at 1.5 in. (37 mm) depth compared to even the bottom sub-lift of Layer 4 at 2.0 in. (50 mm) depth. As shown in Fig. 4, better compaction was observed at 1.5 in. (37 mm) depth compared to other sub-lifts for both locations. This results in less oxygen accessibility at 1.5 in. (37 mm) depth compared to other sub-lifts. Therefore, the degree of compaction plays an important role in controlling the aging of AC mixes in the field.

At Layer 2 and Layer 3, similar aging indices were observed as shown in Fig. 3. The GR values observed for Layer 2 and Layer 3 at Spot 1 were 200 kPa and 239 kPa, respectively. However, Layer 2 is expected to show less aging compared to Layer 3 due to less effect of high temperatures for Layer 2. This is attributed to higher air void contents for Layer 2 compared to Layer 3 (Fig. 4). The air void contents observed for Layer 2 and Layer 3 at Spot 1 were 5.0 % and 8.3 %, respectively. Also, Layer 1 was found to show less aging compared to Layer 3 and 2.3 respectively. Therefore, better compaction (air void contents of 5 %) and absence of higher pavement temperatures are proposed to result in a lower degree of aging for Layer 1. Fig. 4 (b) presents the variation of percent binder content by total weight of mix (TWM) with depths. The percent binder content varied between 4.7 % and 5.2 % by TWM. Both Spot 1 and Spot 2 were found to exhibit a similar trend in percent binder content.

Fig. 5 illustrates the variation in chemical aging (CA) indices with depth for SD 76 project after six years of service. The CA index was found to show a similar trend as the rheological parameters for both locations. Higher CA index values were observed at the top sub-lift for Layer 4 compared to other sub-lifts due to exposure to UV rays. The CA indices observed at the top and bottom of Layer 4 for Spot 1 were 1.91 and 1.15, respectively. At a depth of 1.5 in. (37 mm) much lower CA values were observed compared to other sub-lifts of the surface layer (Layer 4) due to better compaction at 1.5 in. (37 mm) depth. The CA values observed at 1.5 in. (37 mm) depth for Spot 1 and Spot 2 were 0.96 and 1.25, respectively. Also, Layer 2 was found to show somewhat similar or slightly greater CA values compared to Layer 3 due to poor compaction.

The SUL indices were found to show a similar trend of aging at Spot 1. However, greater variation in SUL indices was observed for Spot 2. The SUL value observed for Layer 1 at Spot 2 was 12.3 with a standard deviation of 8.3. Greater variation in SUL values was also observed for Spot 2 at a depth of 2 in. (50 mm) as shown in Fig. 5 (b). The SUL value found at a depth of 2 in. (50 mm) was 19.2 with a standard deviation of 7.1. The extraction and recovery process involved in getting those binders might cause this large variation. Therefore, SUL indices may not be a good parameter in tracking chemical changes with aging. Other researchers also reported greater variability in the SUL index compared to the CA index in tracking aging due to the decomposition of sulfoxide at higher temperatures [1,7,12].



Fig. 4. Variation of (a) % air voids and (b) % binder content with depth in the field for SD 76 project.



Fig. 5. Variation of chemical indices (a) CA index and (b) sulfoxide index with depth in the field for SD 76 project.

3.3. Field to laboratory oven aging

Fig. 6 illustrates the change in rheological and chemical aging parameters with time for different oven aging temperatures for SD 76 loose mixes in the laboratory. The CA index was found to match well with rheological parameters (GR and G* values) at all five temperatures. However, the SUL indices showed large variability even at a laboratory oven aging temperature of 85 °C. The aging indices observed after the breakdown/reheating of SD 76 mixes from bucket to pan were higher compared to aging indices observed in the field at Layer 1. For example, GR values found at Spot 1 of Layer 1 in the field and after reheating the bucket in the laboratory were 56 kPa and 474 kPa, respectively. The CA indices observed at Spot 1 of Layer 1 in the field and after reheating the bucket in the laboratory were 0.84 and 1.46, respectively. This is attributed to no exposure of Layer 1 to UV rays and high pavement temperatures. Also, the use of 135 °C at 2–4 h for the breakdown of buckets is the likely cause of these slightly higher aging indices after the reheating process. However, the reheating temperature was selected based on current practices followed to prepare plant-mix lab-compacted (PMLC) specimens. The main objective of developing MTOA is to evaluate the performance of plant-produced AC mixes in the laboratory which can be implemented in BMD guidelines. Therefore, a temperature of 135 °C for 2–4 hours was used to break down the AC mixes from buckets to smaller pans. Also, it is difficult to break down AC mixes from buckets at temperatures below 135 °C. However, extra reheating of buckets was avoided by continuous monitoring of the buckets. As soon as the mixes were found to be workable, the reheating process was stopped. The dotted black lines shown in Fig. 6 present the top sub-lift aging indices for SD 76 field samples.

Fig. 7 shows the hours of laboratory oven aging required for SD 76 loose mixes to reach the top surface aging in the field at different temperatures for the rheological and chemical aging indices. The increase in laboratory oven aging temperature was found to lower the aging duration required to produce similarly aged mixes, as expected. For example, approximately 170 hours of oven aging at 85 °C can be obtained using 8 hours of aging at 135 °C based on both rheological and chemical parameters. The CA index was found to show slightly higher oven hours compared to other rheological parameters (GR and G*). The oven aging required to reach field top sub-lift aging for SD 76 mixes at 85 °C based on GR and CA indices were 170 hours and 176 hours, respectively. The SUL index was not considered for Fig. 7 due to large variability at all oven temperatures. Other researchers also reported greater variability in the SUL index compared to the CA index [1,7,12]. To achieve the top surface GR values for the SD 76 mixes, the oven aging times required in the oven at 100 °C, 110 °C, and 120 °C temperatures were 64 hours, 34 hours, and 17 hours, respectively.

Fig. 8 shows the relationship between the CA index and rheological aging parameters for SD 76 loose mix for all laboratory aging times and temperatures and field slabs from different depths. A very strong correlation was observed between the CA index and different rheological parameters. Also, both field and loose mixes were found to follow the same path in the relationship plots. Rahman, Harvey [3] suggested that the relation between binder chemical and rheological parameters depends on the base binder grades and sources. For mixes with RAP and potentially also including recycling agents, the blended binder properties also play an



Fig. 6. Variation of aging parameters with oven aging in the laboratory for SD 76 mix (a) log (GR, kPa), (b) log (G* @ 64 °C and 10 Hz), (c) carbonyl area (CA) index and (d) sulfoxide index. Note: dotted black lines are the top sub-lift aging indices for SD 76 field samples.



Fig. 7. Hours of laboratory oven aging required at different temperatures to reach top surface aging for SD 76 mix.



Fig. 8. Relationship between CA index and (a) log (GR, kPa), (b) log (G* at 10 Hz and 64 °C, kPa), (c) log G_c^* , kPa), (b) log (ω_c , Hz) for all loose mix laboratory aging times and temperatures and field slab depths.

important role in defining the relation between chemical and rheological parameters [3]. Fig. 8 shows that the relationships between CA index and rheological indices for the loose mix for different laboratory aging protocols and field slabs at different depths, all with the same base binder and similar RAP, are consistent for the SD 76 materials, as expected.

3.4. Laboratory aging of loose plant-produced mixes

Figs. 9, 10, and 11 illustrate the change in aging parameters with hours of laboratory oven aging at different temperatures for NAPA 29, Virgin-SJ, and RAP40 % RA1.5 %-SJ mixes, respectively. The SUL index failed to show a consistent aging trend for all three mixes at all five temperatures. For example, The SUL index values observed for the NAPA 29 mix after reheating and reheating plus 120 hours of oven aging at 85 °C temperature were 7.07 and 6.14, respectively. The standard deviation values observed for reheating and reheating plus 120 hours of oven aging were 0.92 and 0.16, respectively. Therefore, 120 hours of extra oven aging at 85 °C for loose NAPA 29 mix was found to show a lower SUL index value (Fig. 9). Also, the SUL value observed after 165 hours of oven aging at 85 °C temperature was 13.05 with a standard deviation of 8.75. Therefore, the SUL index was found to show much greater variability. This trend of higher variability for SUL index values was observed for all three mixes at all five temperatures. Other researchers also reported a similar finding [1,7,12].

The CA index showed a much lower coefficient of variance (COV) compared to SUL indices. The average COV values observed for the CA index for NAPA 29, Virgin-SJ, and RAP40 % RA1.5 %-SJ mixes were 5.9 %, 7.2 %, and 8.8 %, respectively. The maximum COV values observed for the CA index values for NAPA 29, Virgin-SJ, and RAP40 % RA1.5 %-SJ mixes were 12.9 %, 12.0 %, and 14.9 %, respectively. The maximum COV found for the SUL index for NAPA 29, Virgin-SJ, and RAP40 % RA1.5 %-SJ mixes were 62.0 %, 52.0 %, and 56.4 %, respectively. Therefore, about four to five times higher maximum percent COV values were observed for the SUL index compared to the CA index. This is mainly attributed to the decomposition of sulfoxide at higher aging temperatures and longer aging periods.

Higher oven temperatures were found to achieve similar aging indices at much lower oven durations for all three mixes. The logarithm of GR value observed after oven aging of 168 h at 85 °C for the Virgin-SJ mix was 2.0 as shown in Fig. 10 (a). To achieve this



Fig. 9. Variation of aging parameters with oven aging in the laboratory for NAPA 29 mix (a) log (GR, kPa), (b) log (G* @ 64 °C and 10 Hz), (c) CA index and (d) SUL index.



Fig. 10. Variation of aging parameters with oven aging in the laboratory for Virgin-SJ mix (a) log (GR, kPa), (b) log (G* @ 64 °C and 10 Hz), (c) carbonyl area (CA) index and (d) sulfoxide (SUL) index.



Fig. 11. Variation of aging parameters with oven aging in the laboratory for RAP40 % RA1.5 %-SJ mix (a) log (GR, kPa), (b) log (G* @ 64 °C and 10 Hz), (c) CA index and (d) SUL index.

GR value, the time required at 100 °C, 110 °C, 120 °C, and 135 °C of oven temperatures were 56 h, 48 h, 16 hours, and 8 h, respectively. The CA index observed after oven aging of 168 hours at 85 °C for the Virgin-SJ mix was 1.45 (Fig. 10 (c)). To achieve this CA index value, the times required at 100 °C, 110 °C, 120 °C, and 135 °C of oven temperatures were 70 h, 30 h, 18 h, and 11 h, respectively. The CA index was found to require slightly higher oven aging durations compared to GR values for all temperatures except 110 °C. A similar trend of higher aging hours requirement based on CA index calculation compared to GR values was also observed for SD 76 mix.

The aging parameters observed for Mix RAP40 % RA1.5 %-SJ after reheating/breakdown of buckets were higher compared to the Virgin-SJ mix as shown in Figs. 10 and 11. The GR values observed for Virgin-SJ and RAP40 % RA1.5 %-SJ mixes after reheating of buckets were 12 kPa and 21 kPa, respectively. The CA index observed for Virgin-SJ and RAP40 % RA1.5 %-SJ after reheating of buckets were 0.59 and 1.27, respectively. For both mixes base binders were collected from the same refinery and a similar aggregate gradation was maintained. However, an increase in RAP content of 40 % is expected to cause greater chemical and rheological aging indices for the RAP40 % RA1.5 %-SJ mix. Also, two distinct aging trends of GR values at 85 °C oven temperature were observed for the Virgin-SJ and RAP40 % RA1.5 %-SJ mix as shown in Fig. 10 (a) and 11 (a)]. Distinct fast-rate and constant slow-rate of aging trends were observed for the RAP40 % RA1.5 %-SJ mix as shown in Fig. 11 (a) at 85 °C temperature. However, Virgin-SJ mix was found to show only a constant rate of aging at different aging durations [Fig. 10 (a)]. A similar trend was also observed for both mixes at 135 °C oven aging temperature. This might be attributed to the addition of RAP to Mix RAP40 % RA1.5 %-SJ. The increase in RAP content is expected to reduce the availability of aging-prone saturates of the total asphalt binder.

The CA index was found to follow a similar trend of rheological properties for all three mixes at different aging temperatures. Fig. 12 presents the relationship between the CA index and different rheological properties. A strong correlation was observed between the CA index and different rheological properties. The Virgin-SJ mix was found to show a different trendline compared to NAPA 29 and RAP40 % RA1.5 %-SJ mixes for G_c^* and ω_C parameters. Rahman, Harvey [3] also reported that altering the base binder and RAP types leads to a shift in the trendline between the CA index and rheological properties.

3.5. Comparison of proposed MTOA with field aging

The red dotted lines shown in Figs. 3 and 5 show the 20 h of laboratory oven aging at 100 °C for the SD 76 mix. It can be seen from Figs. 3 and 5 that 20 h of oven aging at 100 °C is expected to simulate field aging at 0.5–0.6 in. (12 mm to 15 mm) of depth after six



Fig. 12. Relationship between CA index and (a) log (GR, kPa), (b) log (G^{*} at 10 Hz and 64 °C, kPa), (c) log (G_c^* , kPa), (b) log (ω_c , Hz) for plant produced oven-aged loose mixes for all laboratory aging times and temperatures.

years of pavement life for this material and climate region and level of compaction. This level of aging can be considered as the medium-term aging for AC pavement. Several researchers have reported significant changes in binder chemical parameters (mainly sulfoxide) at an oven temperature of greater than $100 \,^{\circ}$ C [7,9]. A decrease in both dynamic modulus values and fatigue resistance was reported by Rad, Elwardany [9] after considering the laboratory aging temperature of 135 $\,^{\circ}$ C. Therefore, the oven aging temperature of 100 $\,^{\circ}$ C was considered for the development of draft MTOA. It should be noted that the chemical parameter CA index was found to show good correlations with binder rheological parameters for all four mixes considered in this study. The chemical parameter SUL index failed to follow the trend of rheological properties at a temperature of 85 $\,^{\circ}$ C (current AASHTO R 30 proposed oven temperature).

Fig. 13 presents the relationship between aging parameters after 20 h at 100 °C and 6 h at 135 °C oven aging for all four mixes. Both the GR and G* values were found to show a linear relationship between two different oven aging conditions for the four mixes. The RAP40 % RA1.5 %-SJ mix showed the highest and Virgin-SJ showed the lowest rheological aging parameters for both oven aging conditions (20 h at 100 °C and 6 h at 135 °C). Regarding the CA index, the Virgin-SJ mix exhibited the lowest value for both oven aging conditions. This binder source from the California central valley is known to have very low aging potential [22]. However, the other



Fig. 13. Relationship between aging parameters of loose mixes after 20 h= at 100 °C and 6 h= at 135 °C for (a) log (GR, kPa), (b) log (G* at 10 Hz and 64 °C, kPa), and (c) CA index.

three mixes were found to show similar CA indices for both 20 h at 100 °C and 6 h at 135 °C oven aging (Fig. 13 (b)). Therefore, the rheological parameters might be more susceptible to change in mix types compared to the chemical indices.

4. Conclusions

In this study, a draft MTOA protocol was developed by comparing laboratory loose mix oven aging with field aging for a set of rheological and chemical aging parameters for one mix. Also, four types of AC mixes were considered to evaluate the effect of oven aging temperatures and duration on the chemical and rheological properties of extracted asphalt binders. Based on this study following conclusions can be drawn:

- The degree of compaction or connectivity between pores greatly affects the aging of asphalt mixes in the field. The top lift of AC layers is more susceptible to aging due to exposure to UV rays and greater pavement temperatures. At the intermediate and bottom AC layers, the degree of compaction plays the most critical role in defining the aging gradient.
- Higher oven temperatures were found to achieve similar aging indices at much lower oven durations. Eight hours of oven aging at 135 °C was found to show similar aging indices compared to 170–180 h of oven aging at 85 °C. The AC mixes with recycled materials were found to show much higher aging indices compared to virgin mix at all aging temperatures and durations. This is mainly attributed to higher aging indices present in the RAP binder.

M.A. Rahman et al.

- The CA index is found to be a more stable chemical parameter in tracking the changes due to aging than the SUL index. The SUL index failed to show a consistent trend due to aging even at a lower oven aging temperature of 85 °C. The extraction and recovery process involved in this study may contribute to this inconsistency. About four times greater COV values were observed for the SUL index compared to the CA index. Also, CA index was found to show a good correlation with the rheological parameters of extracted binders obtained from both loose AC mixes and field slabs.
- Loose mix oven aging of 20 h at 100 °C was found to simulate field aging at pavement depths of 0.5–0.6 in. (12 mm to 15 mm) after six years of pavement life for the mix, climate region, and compaction levels for one mix. The asphalt pavement considered in this study is located in the relatively mild south coastal region of California with a maximum daily air temperature always greater than 10 °C. Therefore, 20 h of loose mix aging at 100 °C is proposed as the MTOA for QA/QC purposes. This MTOA ensures quick turnaround times for mix verification purposes without using a much greater oven temperature (i.e., 135 °C).

In the future, the proposed MTOA will be implemented in screening mixes containing RAP and recycling agents. Also, the extracted binder's aging parameters after 6 h at 135 °C were found to show good correlations with the aging parameters observed after 20 h at 100 °C for four mixes. Further investigation is required to evaluate the effect of 135 °C oven temperature on the cracking performance of AC mixes. In this study, field aging data from one site (SD 76) was considered to develop the MTOA protocol. Field samples from another project (NAPA 29) will be collected and laboratory aging parameters will be compared.

CRediT authorship contribution statement

Mohammad A. Rahman: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. John Harvey: Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. Hanyu Deng: Writing – review & editing, Investigation, Data curation. David Jones: Writing – review & editing, Supervision, Investigation, Conceptualization. Angel Mateos: Writing – review & editing, Supervision, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Author contributions

The authors confirm contribution to the paper as follows: study conception and design: MR, JH, DJ, AM; data collection: MR, HD; analysis and interpretation of results: MR, JH, DJ, AM; draft manuscript preparation: MR, JH, DJ, AM. All authors reviewed the results and approved the final version of the manuscript.

References

- [1] Y.R. Kim, et al., Long-term Aging of Asphalt Mixtures for Performance Testing and Prediction, Transportation Research Board, 2018.
- [2] Y. Liang, et al., Investigation into the oxidative aging of asphalt binders, Transp. Res. Rec. 2673 (6) (2019) 368–378.
- [3] M.A. Rahman, et al., Characterizing the aging and performance of asphalt binder blends containing recycled materials, Adv. Civ. Eng. Mater. 12 (1) (2023) 1–17.
 [4] AASHTO, R., 30-02 Standard Practice for Mixture Conditioning of Hot-Mix Asphalt (HMA). Standard Specifications for Transportation Materials and Methods and Methods and Tartine Mixture DO 2000 (1997)
- and Sampling and Testing Part II: Tests. Washington DC, 2002.
 [5] D. Jin, et al., A case study of the comparison between rubberized and polymer modified asphalt on heavy traffic pavement in wet and freeze environment, Case Stud. Constr. Mater. 18 (2023) e01847.
- [6] C. Chen, et al., Selecting a laboratory loose mix aging protocol for the NCAT top-down cracking experiment, Transp. Res. Rec. 2672 (28) (2018) 359–371.
- [7] J.C. Petersen, A review of the fundamentals of asphalt oxidation: chemical, physicochemical, physical property, and durability relationships, Transp. Res. Circ. (E-C140) (2009).
- [8] Al-Qadi, I.L., et al., Development of Long-Term Aging Protocol for Implementation of the Illinois Flexibility Index Test (I-FIT). 2019, Illinois Center for Transportation/Illinois Department of Transportation.
- [9] F.Y. Rad, et al., Investigation of proper long-term laboratory aging temperature for performance testing of asphalt concrete, Constr. Build. Mater. 147 (2017) 616–629.

- [10] Glover, C.J., et al., Evaluation of binder aging and its influence in aging of hot mix asphalt concrete: technical report. 2014, Texas. Dept. of Transportation. Research and Technology Implementation Office.
- [11] Y. He, et al., Evaluating diffusion and aging mechanisms in blending of new and age-hardened binders during mixing and paving, Transp. Res. Rec. 2574 (1) (2016) 64–73.
- [12] Harvey, J., et al., RAP and RAS in HMA Pilot Project on ELD 49: Material Testing, Observations, and Findings. UCPRC-TM-2022-04, 2023: p. 1-104.
- [13] Newcomb, D.E. and J.A. Epps, Asphalt Recycling Technology: Literature Review and Research Plan. 1981.
- [14] M.A. Rahman, et al., Evaluation of mix design volumetrics and cracking potential of foamed Warm Mix Asphalt (WMA) containing Reclaimed Asphalt Pavement (RAP). Int. J. Pavement Eng. (2021) 1–13.
- [15] M. Rahman, et al., Laboratory performance and construction challenges for plant produced asphalt mixes containing RAP and RAS, Constr. Build. Mater. 403 (2023) 133082.
- [16] M.A. Rahman, et al., Rutting and moisture-induced damage potential of foamed warm mix asphalt (WMA) containing RAP, Innov. Infrastruct. Solut. 6 (3) (2021) 1–11.
- [17] M.A. Rahman, et al., Evaluation of rutting and cracking resistance of foamed warm mix asphalt containing RAP, in: Civil Infrastructures Confronting Severe Weathers and Climate Changes Conference, Springer, 2018.
- [18] D. Jin, et al., Cold in-place recycling asphalt mixtures: Laboratory performance and preliminary ME design analysis, Materials 14 (8) (2021) 2036.
- [19] D. Jin, et al., Laboratory evaluation and field demonstration of cold in-place recycling asphalt mixture in Michigan low-volume road, Case Stud. Constr. Mater. 20 (2024) e02923.
- [20] B. Hofko, et al., Repeatability and sensitivity of FTIR ATR spectral analysis methods for bituminous binders, Mater. Struct. 50 (3) (2017) 1–15.
- [21] J. Lamontagne, et al., Comparison by Fourier transform infrared (FTIR) spectroscopy of different ageing techniques: application to road bitumens, Fuel 80 (4) (2001) 483–488.
- [22] M.L. Williams, R.F. Landel, J.D. Ferry, The temperature dependence of relaxation mechanisms in amorphous polymers and other glass-forming liquids, J. Am. Chem. Soc. 77 (14) (1955) 3701–3707.
- [23] X. Yang, Z. You, New predictive equations for dynamic modulus and phase angle using a nonlinear least-squares regression model, J. Mater. Civ. Eng. 27 (3) (2015) 04014131.
- [24] G. Rowe, Prepared discussion for the AAPT paper by Anderson et al.: Evaluation of the relationship between asphalt binder properties and non-load related cracking, J. Assoc. Asph. Paving Technol. 80 (2011) 649–662.
- [25] P. Singhvi, et al., Impacts of field and laboratory long-term aging on asphalt binders, Transp. Res. Rec. 2676 (8) (2022) 336-353.
- [26] Rahman, M.A., J.T. Harvey, and M. Elkashef, Update of the PG Binder Map in California Using the Enhanced Integrated Climate Model (EICM) and LTTPBind Online, in Airfield and Highway Pavements 2023. p. 429-441.
- [27] Yin, F., et al., Short-term laboratory conditioning of asphalt mixtures. Relationships of Laboratory Mixture Aging to Asphalt Mixture Performance, 2018: p. 21.
- [28] Bell, C.A., A.J. Wieder, and M.J. Fellin, Laboratory aging of asphalt-aggregate mixtures: field validation. 1994.