**2021 Final Report** **Project duration: 1 year**

**Project Title:** Develop a commercially-ready natural asphalt modifier using olive pomace to improve asphalt pavement performance

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## Executive Summary

The olive oil milling process generates a vast amount of olive pomace that could potentially cause environmental pollutions for soils and groundwater. This study explores the feasibility of recycling olive pomace as a bio-renewable modifier to improve the performance of asphalt pavement. The specific objectives of this one-year project are to (1) explore the processing methods for olive pomace so that it can be used as an asphalt modifier; (2) determine the optimum dosage of olive pomace used for asphalt modification based on rutting, cracking, and antioxidant performances of pomace-modified asphalt binders; and (3) evaluate the performance of pomace-modified asphalt mixes (blends of asphalt binder and aggregates) in the laboratory.

A coffee grinder was used to fine-grind olive pomace after drying. The performance results of pomace-modified asphalt binders showed that the processed olive pomace could improve asphalt binders' resistance to oxidization-induced cracking and fatigue cracking. The pomace-modified asphalt binders have comparable rutting resistance and slightly better low-temperature thermal cracking resistance than the control binder. To further improve the antioxidant effectiveness of olive pomace, mechanical sieving is used to separate the stone-rich fraction (>0.3 mm) and the pulp-rich fraction (<0.3 mm). Then, the pulp-rich fraction was blended to modify asphalt binders. It was found the pulp-rich fraction has better softening and antioxidant effectiveness than the unscreened ground olive pomace. The addition of a 5% pulp-rich fraction could neutralize the oxidation caused by asphalt mixing and production. The use of a higher dosage of pulp-rich fraction, e.g. 15% and 25%, could significantly extend the service life of modified asphalt binders prior to experiencing initial and severe oxidation-induced cracking. The pomace-modified asphalt mix exhibited better cracking resistance after long-term aging than the control mix. The use of pomace-modified asphalt binder could promote the incorporation of waste asphaltic materials to benefit the sustainability of both agriculture and asphalt paving industries.

Future works are recommended to investigate active ingredients in olive pomace to maximize antioxidant capacity and develop zero-waste recycling methods to utilize wastewater in olive pomace. Additional works are also needed to optimize the performance of pomace-modified asphalt mixes with recycled materials and conduct life cycle cost analysis to determine economic benefits.

## Introduction

The olive oil industry is one of the most important agricultural sectors for Mediterranean counties and areas (Iofrida et al. 2018 and Berg et al. 2018). In the recent ten years from 2008 to 2017, the annual production of olives in California increased from 66,800 tons to 192,300 tons (CDFA 2018). The olive milling extracts approximately 20-30 percent of ingredients being olive oils and the rest of 70-80 percent is the byproduct of olive pomace (La Rubia-García et al. 2012 and Khdair and Abu-Rumman 2020). Thus, there is an urgent need to recycle a vast amount of olive pomace, which generally contains about 35% solid residue (olive stone and pulp) and 50-65% moisture (Moreno-Maroto et al. 2019 and Difonzo et al. 2021).

Inappropriate disposal of olive pomace could cause environmental pollutions for soil and groundwater due to its acidity and high organic content, which has been extensively discussed and reviewed by Azbar et al. (2004), Niaounakis and Halvadakis (2006), Stamatakis (2010), and Khdair et al. 2019. The direct recycling of olive pomace has been explored for non-food applications, such as the use of olive pomace as soil fertilizer (Lacolla et al. 2019), polymer filler (Valvez et al. 2021), biocharred fuel (Braadbaart et al. 2016), or additive for clay bricks (La Rubia-García et al. 2012, Eliche-Quesada et al. 2016, and Eliche-Quesada and Leite-Costa 2016). However, the direct use of olive pomace as soil fertilizer may change pH, porosity, minerals of soil and cause reduction of seed germination (Lammi et al. 2018). The addition of olive stone ash decreased the strength of bricks (La Rubia-García et al. 2012, and Eliche-Quesada et al. 2016). Chemical recycling was also developed to extract phenolic compounds from olive pomace and use extracts as antioxidants in food, chemistry, and cosmetics (Rodrigues et al. 2015, Vitali Čepo et al. 2018, and Ribeiro et al. 2020). The use of chemical solvents and sophisticated processes to extract antioxidant ingredients from olive pomace may not be environmental-friendly and economic-feasible (Difonzo et al. 2021). Therefore, it is needed to recycle olive pomace in diverse applications. This study explores the feasibility of using olive pomace as a natural modifier for asphalt binders to improve the performance of asphalt pavement.

The rationale of using olive pomace as a natural modifier with antioxidant improvements for asphalt binder is based on the fact that olive pomace is rich in lignin and contains abundant phenolic compounds. The lignin-based additive could retard oxidization of asphalt binder because lignin could work as an inhibitor or scavenger of free radicals (Dizhbite et al. 2004, Pan et al. 2006, Williams and McCready (2008), and Pan 2012). Lammi et al. (2018) determined the lignin contents in crude olive pomace, pulp-rich fraction, and stone-rich fraction as 44.3%, 48.9%, and 37.8%, respectively. Similarly, Ribeiro et al. (2020) tested the lignin contents of 42.48-43.95% and 43.38-45.72% in the crude olive pomace and pulp-rich fraction, respectively. In addition, the radical scavenging capacity could be measured using oxygen radical absorbance capacity (ORAC). Ribeiro et al. (2020) determined the crude olive pomace, fractionated liquid-rich fraction, and pulp-rich fraction had an ORAC of 641.05-734.8 μM Trolox-equivalents (TE)/g, 1546.93-1585.46 μM TE/g, and 454.74-502.80 μM TE/g, respectively. An analogy work conducted by Calabi-Floody and Thenous (2012) was to use grape pomace as an antioxidant for asphalt binder, which the grape pomace had ORAC of 650 μM TE/g.

A few studies have investigated the influence of olive pomace on the performance of asphalt binders. Al-Masaeid et al. (1994) investigated the effects of olive husk on the performance of asphalt binder and mixture. It was found that the olive husk may work as a softening and anti-stripping agent, and the optimum dosage of olive husk added in asphalt binder and mixture was 10 percent. Yener and Yadollahi (2013) also mixed dry olive pomace at dosages of 1%, 3%, and 5% by weight of asphalt binder. Results showed the olive pomace could soften asphalt binder and increase the adhesive bond between aggregate and asphalt binder to improve durability. A recent study conducted by Khedaywi et al. (2020) pretreated olive waste by burning it at 600°C to obtain olive waste ash (OWA) for asphalt modification. As the dosage of OWA increased from 5% to 20% by volume of asphalt binder, the modified asphalt binders had decreased ductility and increased stiffness than the control binder. However, none of these studies have investigated the antioxidant performance of asphalt binder modified by olive pomace.

The specific objectives of this one-year project are to (1) explore the processing methods for olive pomace so that it can be used as an asphalt modifier; (2) determine the optimum dosage of olive pomace used for asphalt modification based on rutting, cracking, and antioxidant performances of pomace-modified asphalt binders; and (3) evaluate the performance of pomace-modified asphalt mixes (blends of asphalt binder and aggregates) in the laboratory.

## Materials

### Olive Pomace

The olive pomace used in this project was collected from the milling facility of the California Olive Ranch Inc., located at Artois, California. The olive pomace was dried in an oven at 110°C for 22-24 hours. The dried olive pomace was smashed manually using mortar and pestle and then ground using a coffee grinder, as shown in Figure 1. The specific gravity of the ground olive pomace was determined as 1.409 following the AASHTO T84 method.

The particle size distribution of the olive pomace after smashing using mortar and pestle and after fine-grinding was determined by mechanical sieving analysis. Figure 1 shows that the coffee grinder could effectively reduce the particle size of olive pomace from 4.75-1.18mm to 1.18mm-0.075 mm. Because the pulp-rich fraction in olive pomace is rich in lignin (Lammi et al. 2018 and Ribeiro et al. 2020) and contains higher residue oils (ANATOLIKI 2008), the grounded olive pomace was fractioned to obtain stone-rich fraction (≥ 0.3 mm) and pulp-rich fraction (< 0.3 mm).

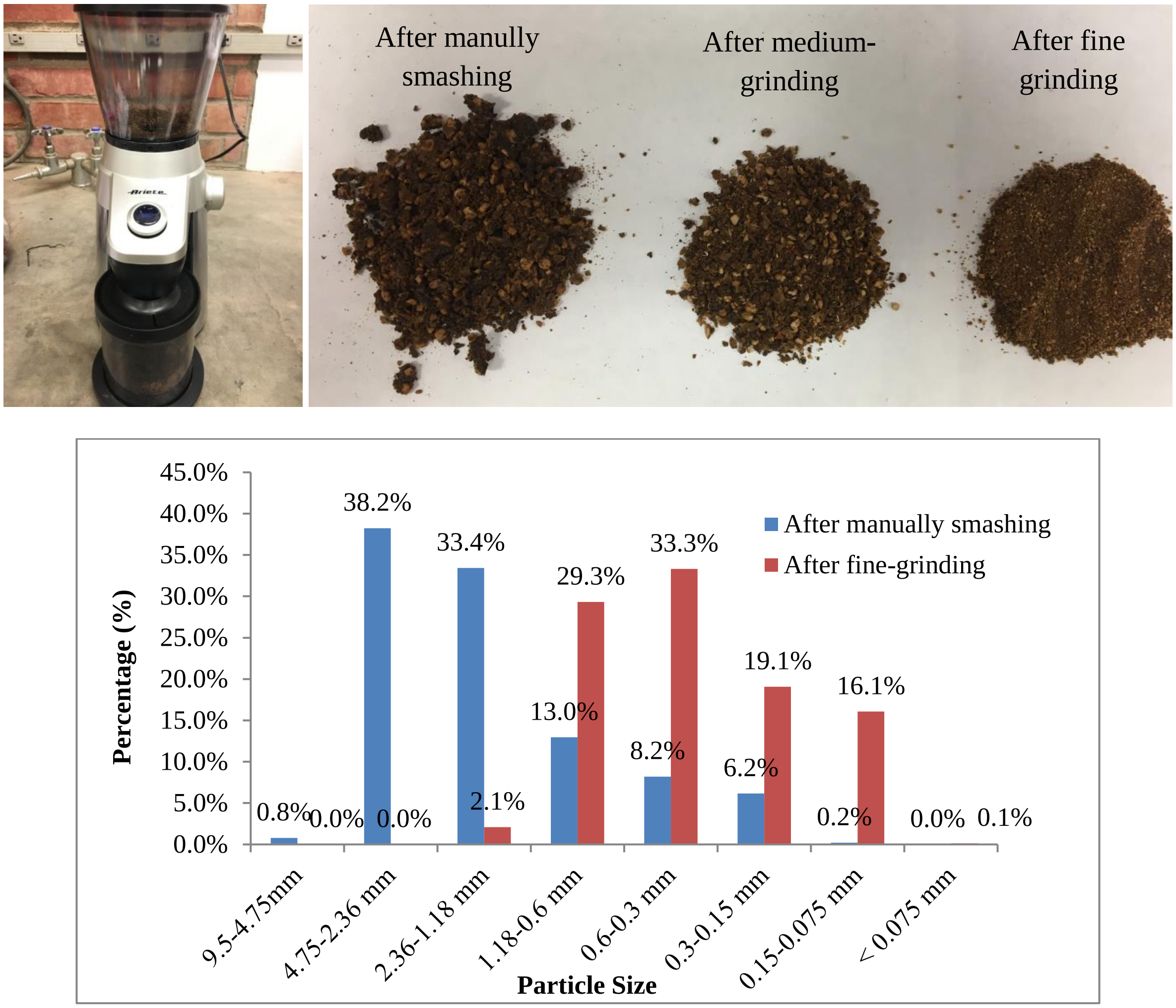


**Figure 1. Coffee grinder used to process olive pomace and particle size distribution of olive pomace after manually smashing and after fine-grinding using the coffee grinder.**

Asphalt Binders

The control asphalt binder is a commercial binder with the performance grade (PG) 64-22. The asphalt binder was heated in the oven at 160°C for 1 hour before mixing with pomace. The ground but unscreened olive pomace was added to the asphalt binder first at dosages of 1%, 3%, 5%, and 10% to modify the asphalt binder. The processed olive pomace (POP)-modified asphalt binders are referred to as POP1, POP3, POP5, and POP10 binders, respectively. The pulp-rich fraction was also added to modify the asphalt binder at the dosage of 5% (POP5p) to compare with the performance of the POP5 binder.

A high-speed shear mixer was used to blend asphalt binder with pomace at 2000 rpm for 30 minutes. A hot plate was used to maintain the mixing temperature of 160°C. The control asphalt binder without POP was also stirred using the shear mixer for 30 minutes to maintain the same aging history as pomace-modified asphalt binders.  The asphalt binders right after 30-minute stirring are considered to be unaged binders. The rolling thin film oven (RTFO) test was conducted at 163°C for 85 minutes to simulate short-term aging of asphalt binders, and the 20-hour pressure aging vessel (PAV) test was conducted at 100°C and 2.1 MPa to simulate long-term field aging of asphalt binders.

High dosages of pulp-rich fraction were also used to modify asphalt binders at 5%, 15%, and 25%, which are referred as to POP5p, POP15p, and POP25p, respectively. A longer stirring time of 85 minutes was used to blend pulp-rich fraction with asphalt binders at the temperature of 160°C. This is to evaluate the influence of oxidation due to asphalt mixing on the performance of the asphalt binder. The antioxidant performance of POP5p, POP15p, and POP25p was assessed after the long-term thermal aging using 20-hour PAV, 40-hour PAV, and 60-hour PAV tests. Figure 2 shows the flow chart of the tested asphalt binders and the testing plan.

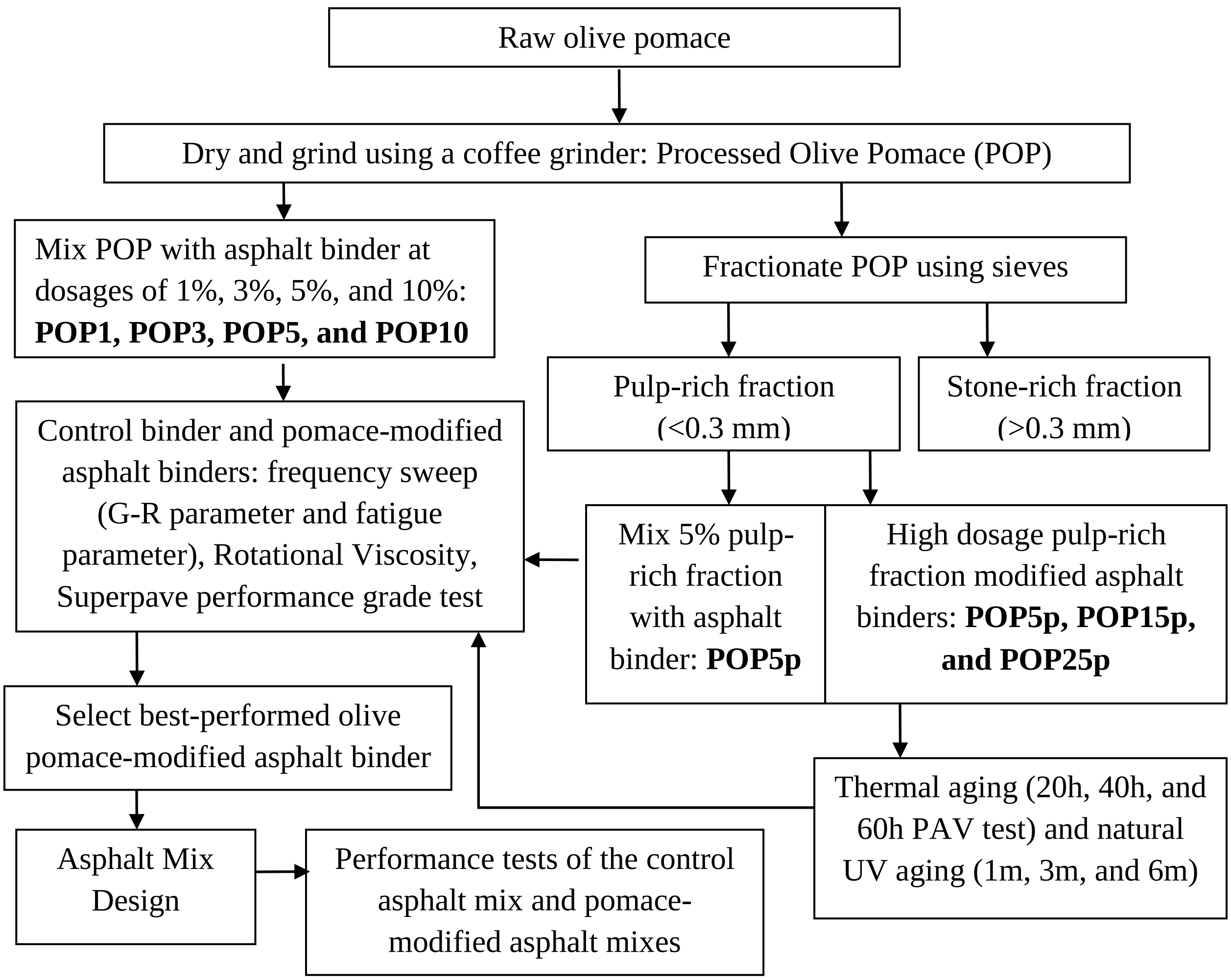


**Figure 2. Flow chart of the testing plan.**

In addition, the control binder, POP5p, and POP15p were conditioned outdoor to study the influence of ultraviolet (UV) aging on asphalt binders up to 6 months from November 2020 to June 2021 in this work. Asphalt binder specimens were prepared with a film thickness of 1 mm in a PAV pan as shown in Figure 3(a). A UV meter was used twice a week to record the UV irradiance (mW/cm$^2$) of UVA at 365nm wavelength every 5 minutes next to samples. A typical daily UVA irradiance from sunrise to sunset is shown in Figure 3(b) and the area below the curve is calculated as the radiant exposure (kJ/m$^2$).

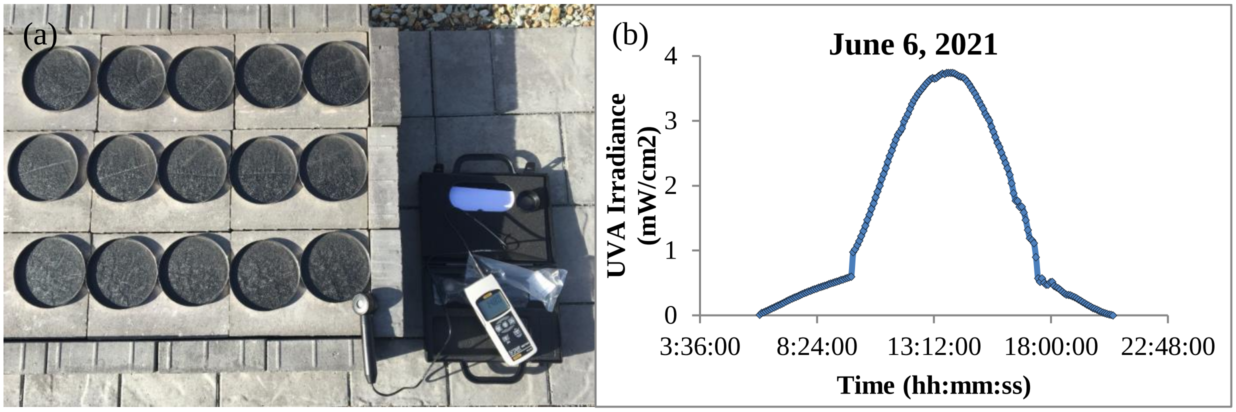


**Figure 3. (a) Asphalt samples of the control binder, POP5p, and POP15p conditioned outdoor and (b) a typical daily UVA irradiance at 365nm wavelength using a UV meter.**

The daily maximum UV index was extracted from theweathernetwork.com (https://www.theweathernetwork.com/us/forecasts/uv/california/chico) in Chico, CA from Nov. 23rd, 2020 to June 7th, 2021 (Figure 4). The relationship between the daily maximum UV index and measured radiant exposure is developed based on the data of 35 testing days (Figure 5). This regression is only valid for this study during the evaluated period at the specific location. Such regression will be changed when a full-year UV index and radiant exposure are recorded at different locations. Based on the regression, the daily radiant exposure from Nov. 23rd, 2020 to June 7th, 2021 was calculated and the cumulative radiant exposure was determined for asphalt samples aged outdoor in 1, 3, and 6 months.

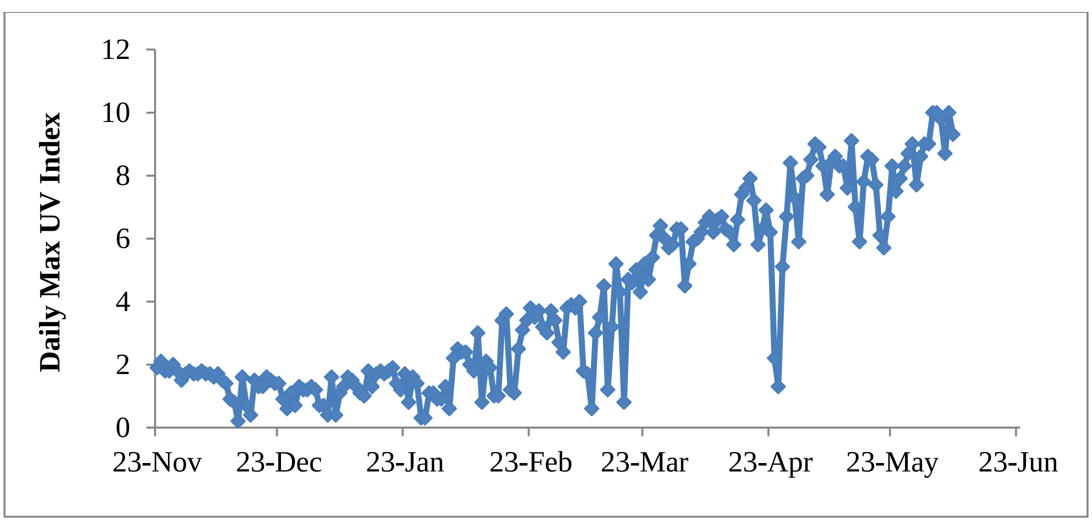


**Figure 4. The daily maximum UV index in Chico, CA from Nov. 23rd, 2020 to June 7th, 2021.**

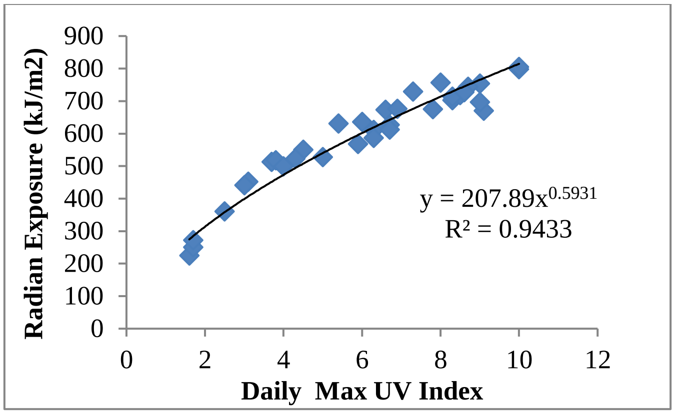


**Figure 5. Regression between daily maximum UV index and measured radiant exposure at UVA wavelength of 365nm.**

Asphalt Mixes

A 1/2” dense-graded asphalt mix was designed based on the Superpave mix design method. The aggregate gradation is shown in Table 1. The optimum binder content is 5.9 percent at air voids of 4.0%. The design mix had voids in mineral aggregate (VMA) of 14.0%, voids filled with asphalt (VFA) of 71%, and dust proportion of 1.1. These volumetric properties could meet with the volumetric requirement for 1/2” asphalt mix according to the AASHTO M323 “Standard Specification for Superpave Volumetric Mix Design”. Based on performance results of pomace-modified asphalt binder, the best-performed binder is POP15p so that this binder was used to fabricate pomace-modified asphalt mixes, which is referred to as POP15p mix with minor gradation adjustment to accommodate pomace in asphalt mix (Table 1).

In addition, to evaluate the benefit of using olive pomace to promote incorporations of waste asphaltic materials in asphalt mixes, the POP15p binder was also used to fabricate a 1/2” dense-graded asphalt mix with reclaimed asphalt pavement (RAP) at the aggregate replacement ratio of 25%. This mix is referred to as the POP15p-25%RAP mix, which had similar aggregate gradation as the control mix (Table 1) and the same total binder content of 5.9%. The RAP binder content was determined as 3.85% using the AASHTO T164 method. The binder replacement ratio is calculated as 16.0%. The volumetric properties of this mix were verified to have air voids of 3.6%, VMA of 13.8%, VFA of 74.1%, and dust proportion of 1.1.

**Table 1. Aggregate gradations in asphalt mixes.**

| Sieve Size (mm) / %Passing | 19 | 12.5 | 9.5 | 4.75 | 2.36 | 1.18 | 0.6 | 0.3 | 0.15 | 0.075 |
|---|---|---|---|---|---|---|---|---|---|---|
| Control Mix | 100 | 96.8 | 85.9 | 62.8 | 43.0 | 26.4 | 17.6 | 11.5 | 7.3 | 4.65 |
| POP15p mix | 100 | 96.7 | 85.7 | 62.5 | 42.4 | 26.0 | 17.4 | 11.3 | 7.2 | 4.53 |
| POP15p-25%RAP mix | 100 | 96.9 | 86.2 | 60.4 | 42.6 | 26.4 | 17.8 | 11.6 | 7.4 | 4.69 |

It shall be noted that the olive pomace used in the POP15p-25%RAP mix was collected in 2020, which was a different batch of olive pomace used in the rest of this study. A food processor instead of the coffee grinder was used to grind the olive pomace. The residue oil contents were determined for unscreened olive pomace, pulp-rich fraction, and stone-rich fraction collected in 2020. A pomace sample with about 10g was loaded in a WhatmanTM 33mm*130 mm extraction thimble, then the oil content was determined by a BÜCHI University Extractor E-800 (Flawil, Switzerland). A total of 155mL Hexane and 30 circles were applied for each of the sample extractions. Each of the pomace samples was tested by replicates. The residue oil content in the unscreened olive pomace was 9.34%±0.16%. The pulp-rich fraction had a higher residue oil content of 13.73%±0.13%, and the stone-rich fraction has a lower residue oil content of 8.64%±0.18%.

## Experiments

### Performance Tests of Asphalt Binders

#### *Frequency sweep test for oxidation-induced and fatigue cracking resistances*

A dynamic shear rheometer (DSR) was used to conduct the frequency sweep test of asphalt binders (8-mm in diameter and 2-mm in height) at temperatures of 5°C, 15°C, 25°C, 35°C, and 45°C and frequencies from 0.01592 Hz (0.1 rad/s) to 15.92 Hz (100 rad/s). The frequency sweep results were used to construct master curves of complex modulus (G*) and phase angle (δ) at the reference temperature of 15°C using the sigmoidal functions (Liu et al. 2020). The Glover-Rowe (G-R) parameter ($G^*(\cos\delta)^2/\sin\delta$) at 15°C and 0.005 rad/s was determined based on the master curves of G* and δ and used to evaluate the resistance of asphalt binder to the oxidation-induced cracking (Osmari et al. 2019 and Zhang et al. 2019). The fatigue parameter ($G^* \cdot \sin\delta$) was determined at the frequency of 1.592 Hz (10 rad/s) to characterize the fatigue cracking resistance of asphalt binders.

#### *Rotational viscosity test for workability evaluation*

To evaluate the influence of olive pomace on the workability of asphalt binders, the rotational viscosity (RV) of the unaged binders was determined at temperatures of 135°C, 145°C, 155°C, and 165°C following AASHTO T316. The viscosity results were used to determine the mixing and compaction temperatures based on the viscosity ranges of 0.15-0.19 Pa·s and 0.25-0.31 Pa·s, respectively.

#### *Superpave performance test for rutting and low-temperature cracking resistances*

The DSR test was also used to determine the rutting parameter ($G^*/\sin\delta$) of the unaged and RTFO-aged asphalt binders at temperatures of 64°C and 70°C following the AASHTO T315 method. The critical high failure temperature ($T_{c,\ high}$) is calculated based on $G^*/\sin\delta = 1.0$ kPa for the unaged binder and $G^*/\sin\delta = 2.2$ kPa for RTFO-aged binder according to AASHTO M323.

The low-temperature cracking performance of asphalt binders was determined using the bending beam rheometer (BBR) test following the AASHTO T313 method. The creep stiffness (S) and rate of relaxation (m-value) of PAV-aged binders were measured at -12°C and -18°C. The critical low failure temperatures ($T_c(S)$ and $T_c(m)$) were calculated based on the stiffness failure threshold (S=300 MPa) and m-value failure threshold (m-value = 0.300) following AASHTO M323. The delta $T_c$ ($\Delta T_c = T_c(S) - T_c(m)$) was reported as an indicator of binder quality and the acceptable threshold of $\Delta T_c$ is -5°C or above (Sharma et al. 2017). For DSR and BBR tests, two specimens of each asphalt binder were tested and the acceptable ranges of two test results (d2s%) shown in AASHTO T315 and T313 were used as the criterion to examine test variation. The average result of two specimens is reported and discussed.

Performance Tests of Asphalt Mixes

*Moisture Resistance*

The tensile strength ratio (TSR) test was used to assess the moisture resistance of asphalt mixes after freeze-thaw conditioning following the AASHTO T283 standard. The specimens were compacted at the target height of 95 mm with air voids of 7% ±0.5%. Three specimens were prepared and tested to determine the indirect tensile (IDT) strength without freeze-thaw conditioning (Dry IDT Strength in Equation 1) at 25°C. Another three specimens were compacted, saturated to 70-80 percent, and conditioned using one freeze-thaw cycle to determine the conditioned IDT strength. Figure 6(a) shows the test set-up of the IDT strength. The TSR value is calculated using Equation (1). The designed asphalt mixes shall have a minimum TSR of 0.8 to have sufficient moisture resistance based on the AASHTO M323 standard.

$$tensile\ strength\ ratio\ (TSR) = \frac{Conditioned\ IDT\ Strength}{Dry\ IDT\ Strength} \quad (1)$$


**Figure 6. (a) Test set-up of indirect tensile (IDT) strength for moisture and cracking resistances and (b) Hamburg wheel tracking test for rutting resistance.**

*Cracking Resistance and Antioxidant Resistance*

The IDT strength test performed at 25°C is also used to characterize the cracking resistance of asphalt mixes after short-term and long-term aging conditions. The loose mixes of three specimens were short-term aged at 60°C for 16 hours and 145°C for 2 hours before compaction to the height of 95mm with air voids of 7% ±0.5%. Another three specimens were mixed and the loose mixes were long-term aged at the compaction temperature of 145°C for 2 hours, following by 85°C for 5 days, and then conditioned at 145°C for 2 hours before compaction. The results of the IDT strength test were analyzed using the IDEAL cracking testing (IDEAL-CT) analysis method (Zhou 2019). The cracking tolerance index ($CT_{index}$) was determined as (Zhou 2019):

$$CT_{index} = \frac{t}{62} \times \frac{G_f}{|m_{75}|} \times \frac{l_{75}}{D} \tag{2}$$

where $CT_{index}$ is the cracking tolerance index, asphalt mix with a higher $CT_{index}$ has better cracking resistance; $t$ is the thickness of specimen; $G_f$ is the failure energy that is the total area under the load-displacement curve divided by the area of cracking face; $l_{75}$ is the loading head displacement corresponding to 75 percent of the peak load in the post-peak curve; $D$ is specimen diameter; and $|m_{75}|$ is the post-peak slope at load decrease to 75 percent of the peak load.

*Rutting Resistance*

The Hamburg wheel tracking test (HWTT) was used to characterize the resistance to permanent deformation following the AASHTO T324 standard. The specimens were mixed and conditioned at 135°C for 4 hours, and then compacted to the target height of 60 mm and air voids of 7% ±0.5%. The testing temperature of HWTT was controlled at 50°C and wheel loads were set at 158 lbs. The result of HWTT is also used to evaluate the moisture resistance of asphalt mixes. Figure 6(b) shows the test set-up of HWTT.

## Test Results and Discussion

### Performance Results of Asphalt Binders

*Resistance to oxidation-induced cracking*

Figure 7(a) shows the G-R parameter of the control binder, POP1, POP3, POP5, POP10, and POP5p after RTFO and 20-hour PAV aging in the laboratory. The asphalt binder with a lower G-R parameter has better resistance to oxidation-induced cracking (Osmari et al. 2019). The result shows that the POP-modified asphalt binders have better resistance to oxidation-induced cracking than the control binder. As the dosage of POP increases, the antioxidant performance of asphalt binders improves. The POP10 binder had 21.7% lower in the G-R parameter than the control binder as shown in Figure 7(b). By comparing the POP5 and POP5p binders, the pulp-rich fraction had a better antioxidant effect than the unscreened ground olive pomace.

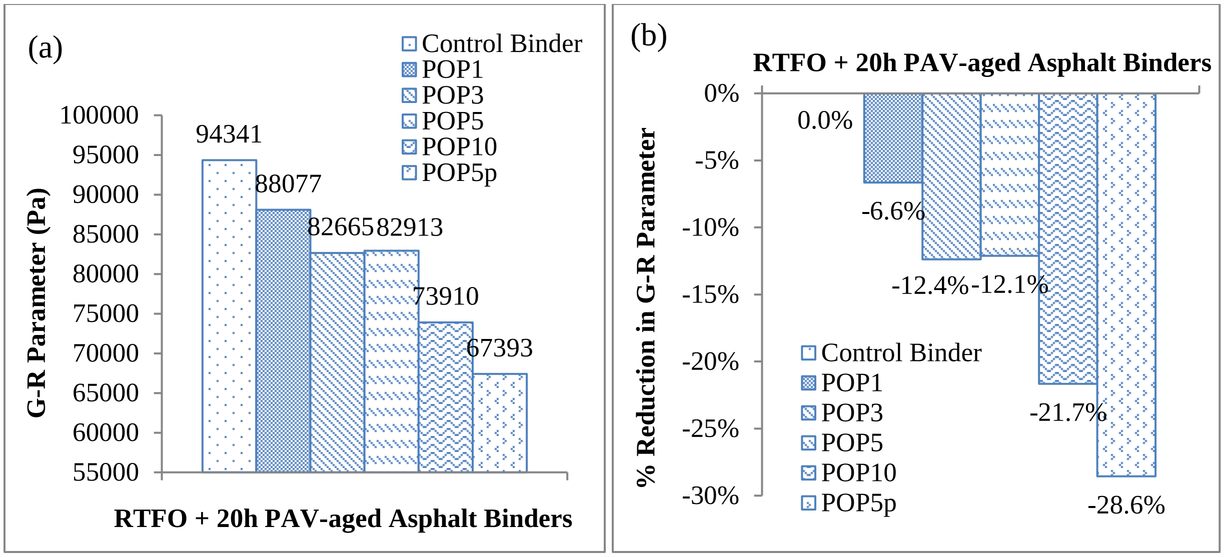


**Figure 7. (a) G-R parameter and (b) percent reduction in G-R parameter of the control and POP-modified asphalt binders.**

Figure 8(a) shows the G-R parameter of the control binder and POP5p, POP15p, and POP25p binders before and after 85-minute stirring. As expected, the 85-minute stirring will cause oxidation of the control binder as the G-R parameter increased. However, the G-R parameter decreased after 85-minute stirring for POP5p, POP15p, and POP25p binders. This indicates that the addition of pulp-rich fraction help prevent aging during the production or even rejuvenate asphalt binder when the dosages of the pulp-rich fraction are at 15% and 25%.

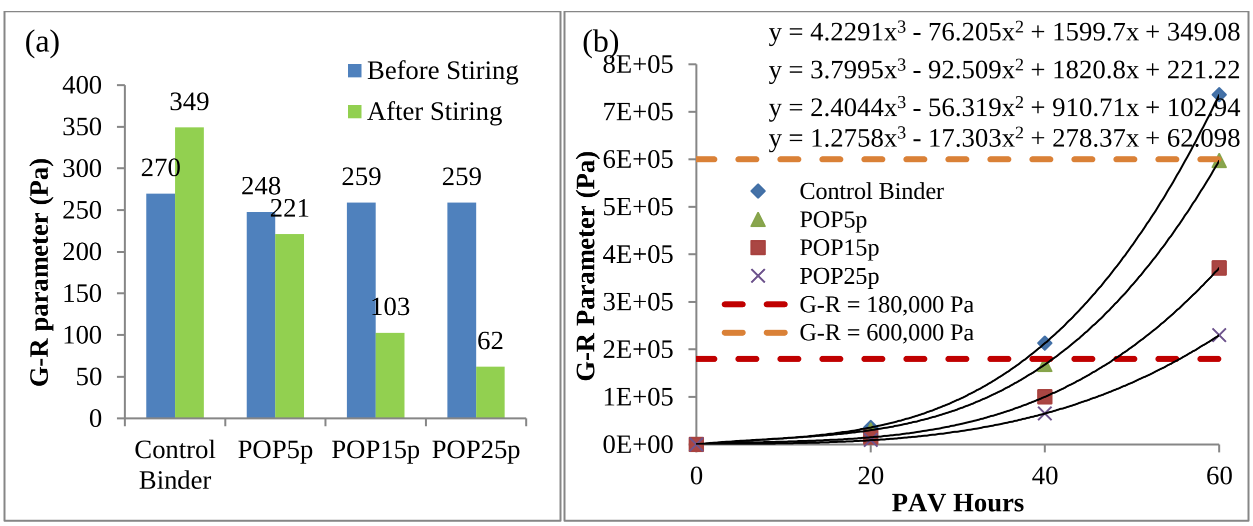


**Figure 8. (a) G-R parameters before and after 85-minute stirring at 160°C and (b) G-R parameters after the long-term PAV aging (20h, 40h, and 60h).**

The G-R parameter of the control binder, POP5p, POP15p, and POP25p binders after 20, 40, and 60-hour PAV aging are shown in Figure 8(b). The threshold values of the G-R parameter for onset and severe cracking damages are G-R = 180,000 Pa and 600,000 Pa, respectively (Osmari et al. 2019 and Haghshenas et al. 2021). As the dosage of pulp-rich fraction increased, the modified asphalt binder exhibited better antioxidant performance and had extended service life prior to experiencing onset and severe oxidation-induced cracking damages.

The 3$^{rd}$ order of the polynomial regression is used to quantify the change of G-R parameter of tested binders with PAV aging hours. Table 2 summarizes the PAV aging hours to cause each asphalt binder to have onset and severe oxidation-induced cracking damages according to the regression. The control binder would experience onset and severe cracking damages after 37.8 hours and 56.2 hours of PAV thermal aging, respectively. The POP15p and POP25p would experience severe cracking damages after 69.6 hours and 81.6 hours of PAV thermal aging, respectively. Table 2 also shows the percent time extensions for POP5p, POP15p, and POP25p binders to experience onset and severe oxidation-induced cracking damages. Generally, the POP15p and POP25p binders are expected to have approximately 25% and 45% time extension to experience onset and severe oxidation-induced cracking damages than the control binder. These results showed that the olive pomace could rejuvenate and retard thermal oxidation of asphalt binders to increase the service life of asphalt pavement.

**Table 2. PAV-aging hours for asphalt binders to reach the G-R parameter of 180,000 Pa and 600,000 Pa.**

| | **PAV hours to onset cracking damage (G-R=180,000 Pa)** | **PAV hours to severe cracking damage (G-R=600,000 Pa)** | **%Time increase to onset cracking damage** | **%Time increase to severe cracking damage** |
|---|---|---|---|---|
| **Control** | 37.8 | 56.2 | --- | --- |
| **POP5p** | 40.9 | 60.1 | 8.3% | 6.9% |
| **POP15p** | 48.0 | 69.6 | 27.1% | 23.8% |
| **POP25p** | 55.5 | 81.6 | 46.8% | 45.2% |

For outdoor aged samples, since asphalt binders were prepared on different dates, they received different cumulative radiant exposures after 1 month, 3 months, and 6 months conditionings. Table 3 shows the outdoor conditioning periods and corresponding cumulative radiant exposures for tested binders. It is seen that the control binder and POP5p binder received a similar amount of UV radiant exposures since they were prepared just two days apart. While the POP15p binder was prepared two weeks later, the POP15p binder received approximately 10% more UV radiation exposures after 6-month conditionings.

**Table 3. Outdoor conditioning periods and cumulative radiant exposure for control binder, POP5p, and POP15p.**

| Binder Type / Cumulative Radiant Exposure (kJ/m2) | 1 Month Conditioning | 3 Months Conditioning | 6 Months Conditioning |
|---|---|---|---|
| **Control Binder** | Nov.24-Dec.22 | Nov.24-Feb.22 | Nov.24-May 22 |
| | 7439.7 | 23189.1 | 73682.0 |
| **POP5p Binder** | Nov.26-Dec.24 | Nov.26-Feb.24 | Nov.26-May 24 |
| | 7271.4 | 23467.6 | 74480.8 |
| **POP15p Binder** | Dec.8-Jan.6 | Dec.8-Mar.6 | Dec.8-June 6 |
| | 6391.5 | 24266.7 | 80950.2 |

The changes of the G-R parameter of tested binders are plotted with the cumulative radiant exposure and shown in Figure 9. For the first one month and three months, the G-R parameter values of these binders are close to each other as they received similar cumulative radiant exposure. After 6-month outdoor aging, the control binder and POP5p binder had similar G-R parameters, while the POP15p binder had the highest G-R parameter after 6-month conditionings, which is mainly because the 6-month aged POP15p binder received approximately 10% higher cumulative radiant exposure than the control binder and POP5p binder.

As shown in Figure 9, the change of the G-R parameter has an exponential relationship with the cumulative UV radiant exposure. The outdoor UV aging could cause significant oxidation and damage to asphalt binders. In the studied short period, the pomace did not exhibit noticeable anti-UV aging effectiveness. However, as shown in Figure 8(b), the difference in the anti-thermal oxidation performance of asphalt binders becomes more pronounced after long-term thermal aging of 40-hour and 60-hour PAV tests. Therefore, long-term outdoor conditioning or accelerated UV aging in the laboratory needs to be conducted to evaluate the long-term anti-UV oxidation performance of pomace-modified asphalt binders.

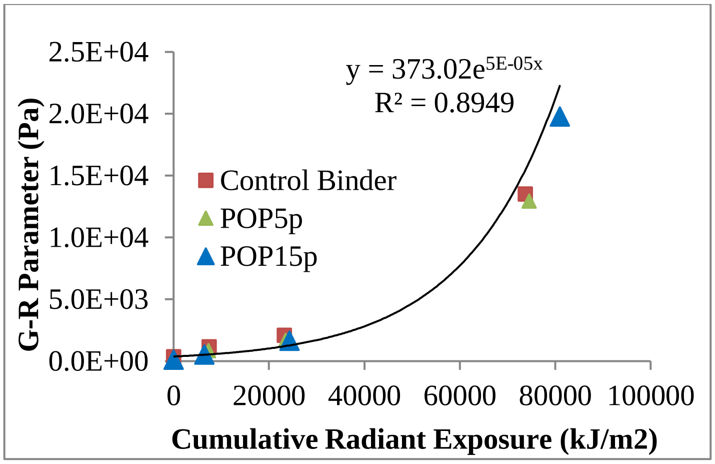


**Figure 9. Changes of G-R parameters with cumulative radiant exposure.**

*Resistance to fatigue cracking*

Figure 10 shows fatigue parameter (G*·sinδ) results tested at 25°C and the frequency of 10 rad/s. A lower fatigue parameter means a better fatigue resistance for asphalt binders. All tested pomace-modified asphalt binders had better fatigue resistance than the control binder. As the dosage of POP increases, the POP-modified asphalt binders had improved resistance to fatigue cracking. Comparing between POP5 and POP5p binders, the pulp-rich fraction has better effectiveness to improve fatigue resistance than the unscreened ground olive pomace. The POP5p binder has even better fatigue and antioxidant performance than the POP10 binder.

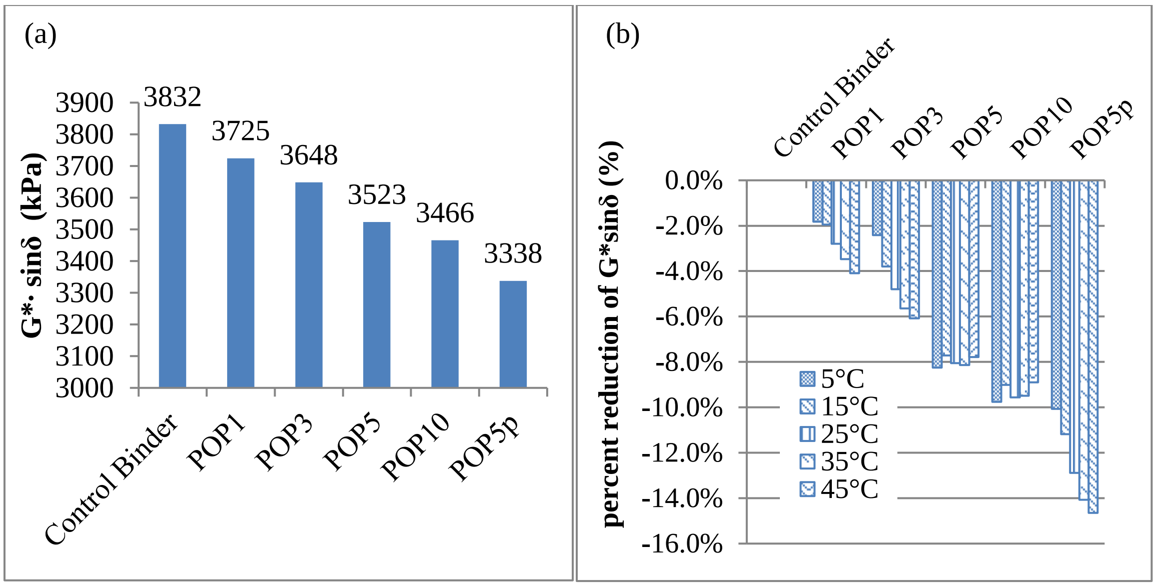


**Figure 10. (a) parameter (G* · sinδ) results at 25°C and (b) percent reduction of fatigue parameter of pomace-modified asphalt binders.**

Similar to the G-R parameter, the fatigue parameter of the control binder increased after 85-minute stirring at 160°C due to oxidation, as shown in Figure 11(a). The fatigue parameter of the pulp-rich fraction modified asphalt binders decreased after the 85-minute stirring. The addition of 5% or more pulp-rich fraction can retard oxidation of asphalt binders during the production or even soften asphalt binders. This allows longer storage time and longer hauling distance of asphalt mixes without causing severe oxidation during asphalt production.

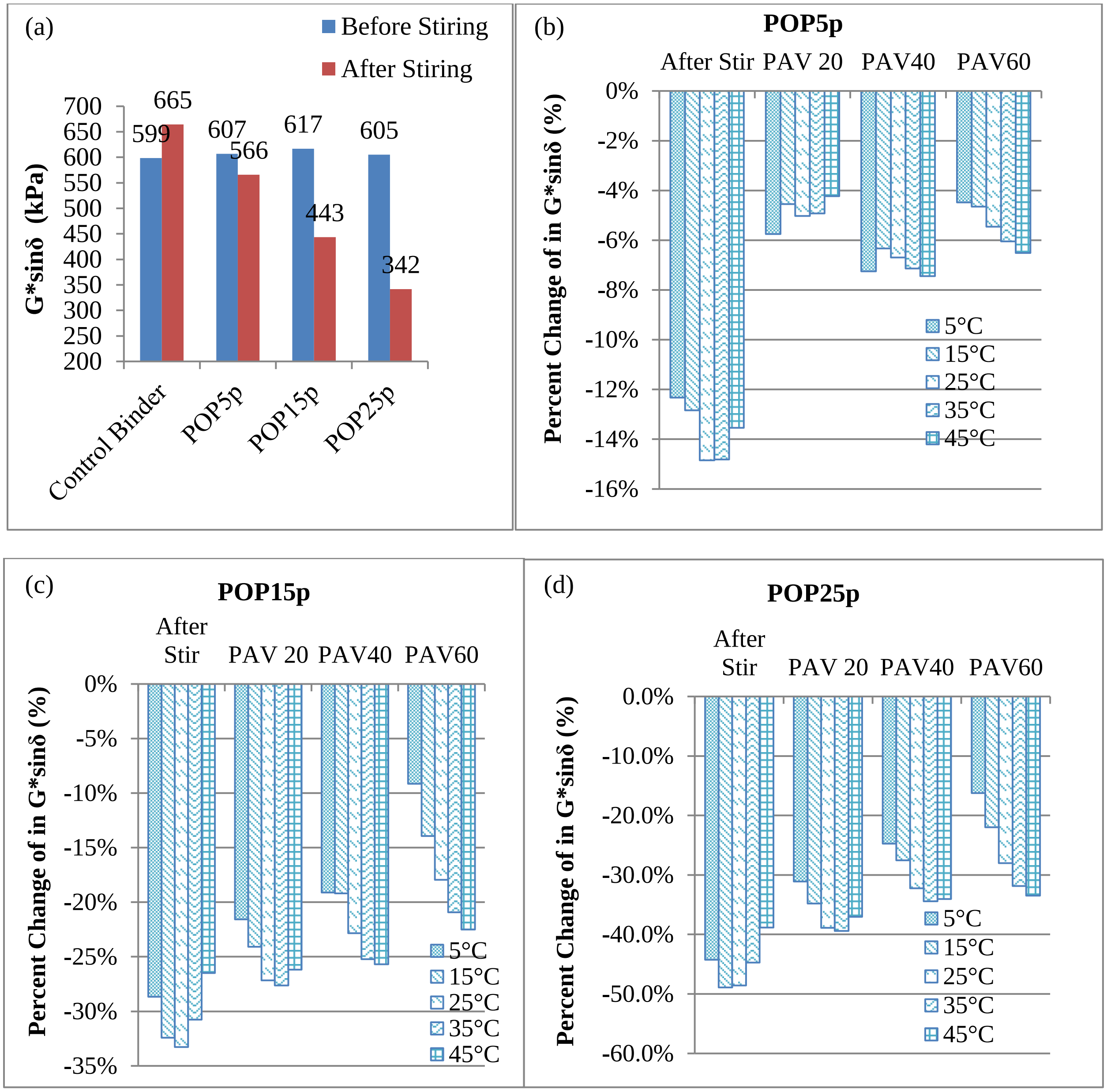


**Figure 11. (a) Fatigue parameters (G*·sinδ) at 25°C before and after 85-minute stirring and percent changes in G*·sinδ for (b) POP5p, (c) POP15p, and (d) POP25p binders compared with the control binder after 85-minute stirring and long-term PAV aging.**

*Rotational viscosity test for workability evaluation*

Figure 12 shows the rotational viscosity results of pomace-modified asphalt binders. The mixing and compaction temperatures of the control and pomace-modified asphalt binders were determined based on the viscosity results and shown in Table 4. Generally, the addition of pomace up to 15% does not influence the workability of the asphalt binder. However, the addition of 25% pulp-rich fraction in asphalt binder caused significantly increased viscosity. This consequently requires higher mixing temperature (183-194°C) and compaction temperature

(163-172°C). Therefore, the POP25p is not recommended to blend with aggregates to produce asphalt mixes, although it has the best antioxidant performance and resistance to fatigue cracking.

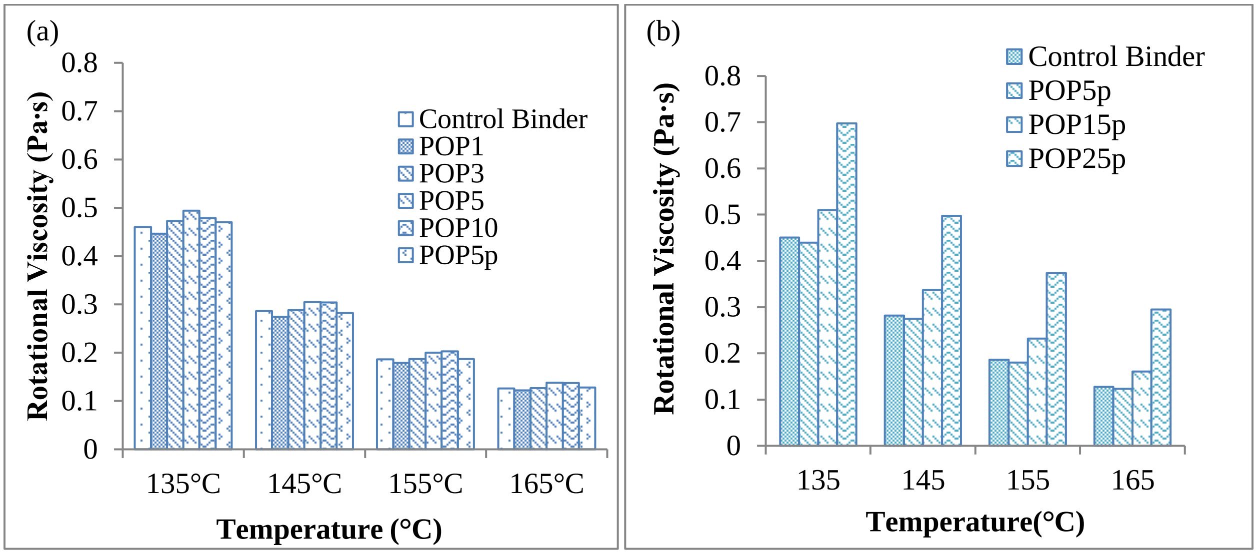


**Figure 12. Rotational viscosity results of (a) pomace modified asphalt binders and (b) high dosage pulp-rich fraction modified asphalt binders after 85-minutes stirring.**

**Table 4. Mixing and compaction temperatures of control and pomace-modified asphalt binders.**

| Asphalt Binders | Mixing Temperature (°C) | Compaction Temperature (°C) |
|---|---|---|
| | Viscosity range of 0.15-0.19 Pa·s | Viscosity range of 0.25-0.31 Pa·s |
| **Control Binder** | 154-160 | 143-148 |
| **POP1** | 154-160 | 143-147 |
| **POP3** | 155-161 | 144-148 |
| **POP5** | 157-163 | 145-150 |
| **POP10** | 156-163 | 144-149 |
| **POP5p** | 155-161 | 144-148 |
| **Pulp-rich fraction modified asphalt binders after 85-min. stirring** | | |
| **Control Binder** | 155-161 | 143-148 |
| **POP5p** | 154-160 | 142-147 |
| **POP15p** | 160-167 | 147-153 |
| **POP25p** | 183-194 | 163-172 |

*Superpave performance test for rutting and low-temperature cracking resistances*

Rutting resistance (high-temperature performance)

The critical high failure temperature ($T_{c,\ high}$) for unaged binders at $G^*/\sin\delta = 1.0$ kPa and RTFO-aged binders at $G^*/\sin\delta = 2.2$ kPa are presented in Figure 13. Generally, the addition of unscreened olive pomace up to 10 percent in the asphalt binder does not change the high-performance grade (PG). All tested binders could be classified with high PG of 64°C.

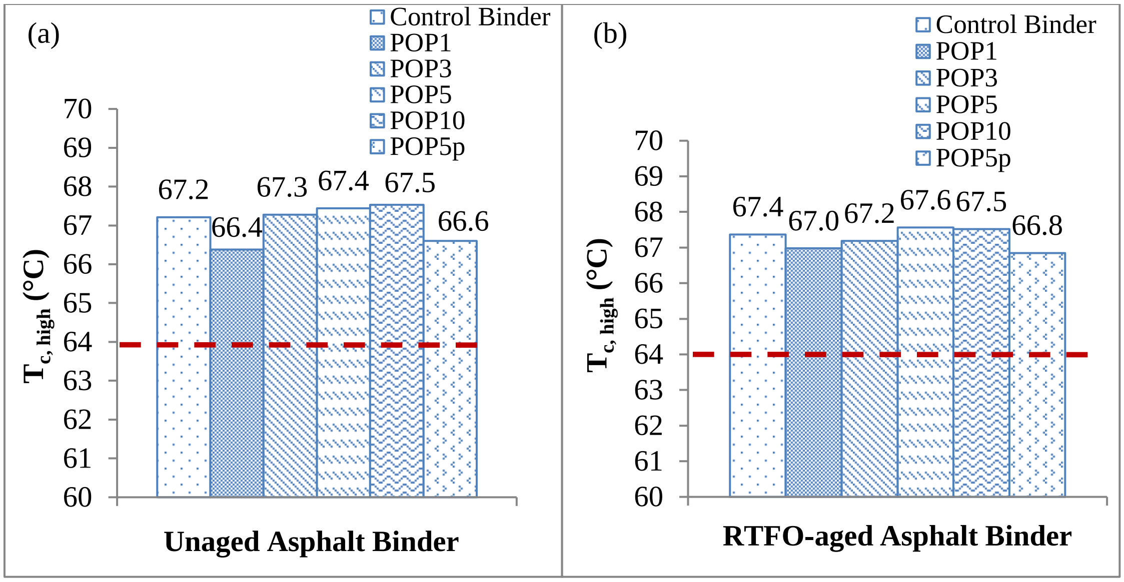


**Figure 13. Critical high failure temperature ($T_{c,\ high}$) of unaged and RTFO-aged asphalt binders.**

The critical high failure temperature ($T_{c,\ high}$) for high dosages of pulp-rich fraction modified asphalt binders is shown in Figure 14. Again, the 85-minute stirring using a high-speed shear mixer could cause oxidation of the control binder as the $T_{c,\ high}$ increased after stirring. The RTFO aging will further increase the $T_{c,\ high}$ of the control binder. The addition of 5% pulp-rich fraction in asphalt binder neutralized the oxidation caused by 85-minute stirring at the temperature of 160°C, as the $T_{c,\ high}$ is maintained at the almost same level before and after 85-minute stirring for POP5p binder. For the POP15p binder, the addition of 15% pulp-rich fraction will not only neutralize the oxidation caused by 85-minute stirring but also further soften the asphalt binder. After RTFO aging for POP15p binder, the $T_{c,\ high}$ was maintained at the almost same level before 85-minute stirring. The addition of 25% pulp-rich fraction in the asphalt binder leads to the decreased $T_{c,\ high}$ after 85-minute stirring, and the POP25p binder barely meets the high PG of 64°C. Therefore, the addition of excessive pulp-rich fraction in asphalt binders, e.g. 25%, may compromise the rutting resistance. Based on the viscosity and high PG results, the POP15p binder is considered the best-performed pomace-modified binder in this study and used to blend with aggregates to fabricate pomace-modified asphalt mixes.

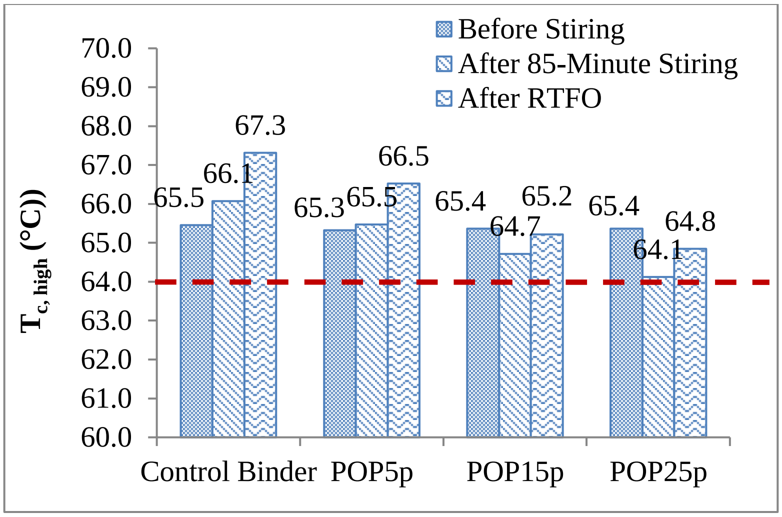


**Figure 14. Critical high failure temperature ($T_{c,\ high}$) of high dosages of pulp-rich fraction modified asphalt binders before and after 85-minute stirring and after RTFO aging.**

Low-temperature thermal cracking resistance

The critical low failure temperature ($T_{c,\ low}$) results are shown in Figure 15. The pomace-modified asphalt binders had similar $T_{c,\ low}$ and improved $\Delta T_c$ values with the control binder. All of the tested binders could be classified with the low PG of -22°C. Overall, the addition of olive pomace in the asphalt binder will not compromise the high- and low-temperature performance of asphalt binders.

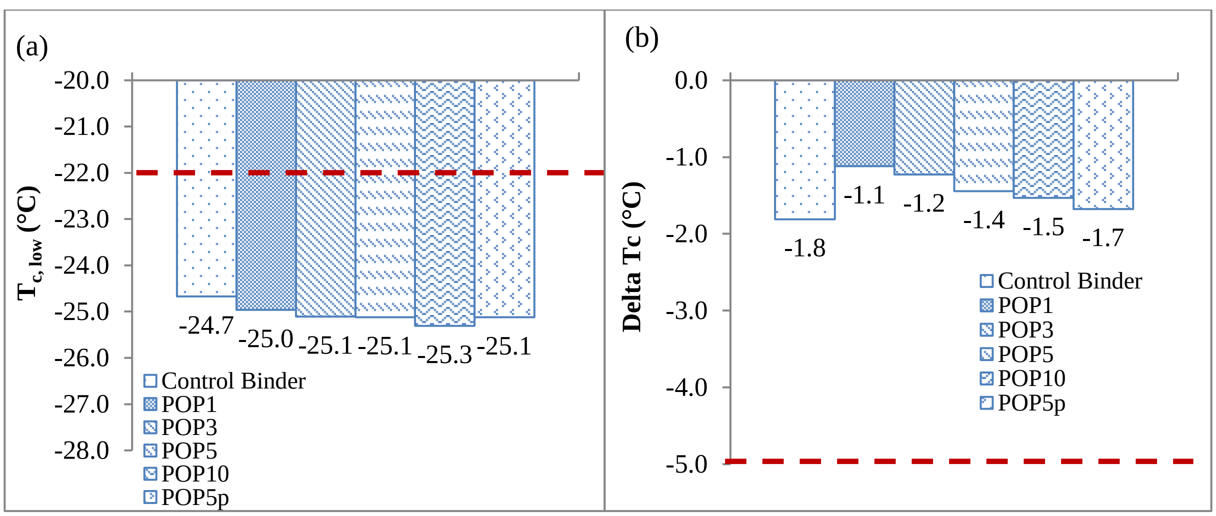


**Figure 15. (a) Critical low failure temperature ($T_{c,\ low}$) and (b) $\Delta T_c$ results of the control binder and pomace-modified asphalt binders.**

*Summary of Performance Results of Pomace-Modified Asphalt Binders*

Based on the performance results of pomace-modified asphalt binders, the addition of unscreened ground olive pomace or olive pulp-rich fraction improves asphalt binders' resistances to the oxidation-induced cracking and fatigue cracking. The pomace-modified asphalt binders have comparable rutting resistance and slightly better low-temperature thermal cracking resistance than the control binder. The olive pulp-rich fraction has better softening and antioxidant effectiveness than the unscreened ground olive pomace. The addition of a 5% pulp-rich fraction could generally neutralize the thermal oxidation that occurred during the production of asphalt mixes. The use of a 15% or 25% pulp-rich fraction could significantly improve the antioxidant performance and fatigue resistance of asphalt binders subjected to long-term thermal oxidation. The outdoor UV radiant exposure caused severe oxidization of asphalt binders. The olive pomace did not exhibit anti-UV oxidation in the short studied period.

Although the POP25p binder showed the best antioxidant performance and fatigue resistance among the tested binders, the addition of a high dosage of pomace in the asphalt binder causes the difficulty of stirring and requires high production temperatures. Therefore, in this study, the POP15p binder is considered as the best-performed pomace-modified asphalt binder and used to blend with aggregates to fabricate pomace-modified asphalt mixes.

## Performance Results of Asphalt Mixes

### *Moisture Resistance*

The IDT strength of the control mix, POP15p mix, and POP15p-25%RAP mix is shown in Figure 16(a). The addition of pulp-rich fraction softened the asphalt binder and resulted in a 30% lower in the IDT strength for the POP15p mix, compared with the control mix. The addition of 25% RAP increased the IDT strength of the POP15p-25%RAP mix, which is because the RAP material typically contains the oxidized asphalt binder.

After the freeze-thaw conditioning, all mixes showed reduced IDT strength as shown in Figure 16(a). The tensile strength ratios are 85.9%, 88.2%, and 88.2% for the control mix, POP15p mix, and POP15p-25%RAP mix, respectively, and shown in Figure16 (b). All mixes could meet the AASHTO M323 requirement of a minimum TSR of 80%. However, these mixes experienced moisture stripping in the HWTT. The addition of pulp-rich fraction seems not to improve or compromise the moisture resistance of an asphalt mix.

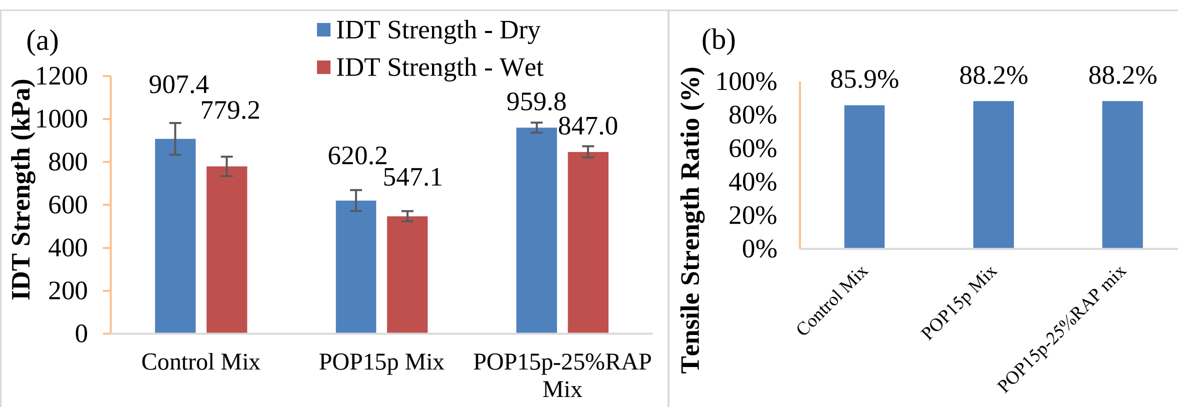


**Figure 16. Results of (a) dry and wet IDT strength and (b) tensile strength ratio of mixes.**

*Cracking Resistance and Antioxidant Resistance*

As shown in Figure 17, the POP15p mix had the highest CT_index after short-term and long-term aging. This means the POP15p mix is more ductile and has better cracking resistance than the control mix and POP15p-25%RAP mix. The addition of RAP typically causes a lower cracking resistance, while the POP15p-25%RAP mix had comparable cracking resistance with the control mix in this study. This means the pomace-modified asphalt binder could alleviate the stiffening effect caused by RAP materials.

The long-term aging caused significant increases in IDT strength and compromised the cracking resistance of these mixes shown in Figure 17. The CT_index ratio between long-term aged specimens and short-term aged specimens is 6.944, 3.517, and 3.524, for the control mix, POP15p mix, and POP15p-25%RAP mix, respectively. This shows the use of pomace-modified asphalt binder helps improve the cracking resistance and antioxidant resistance of asphalt mixes, especially after the long-term thermal aging.

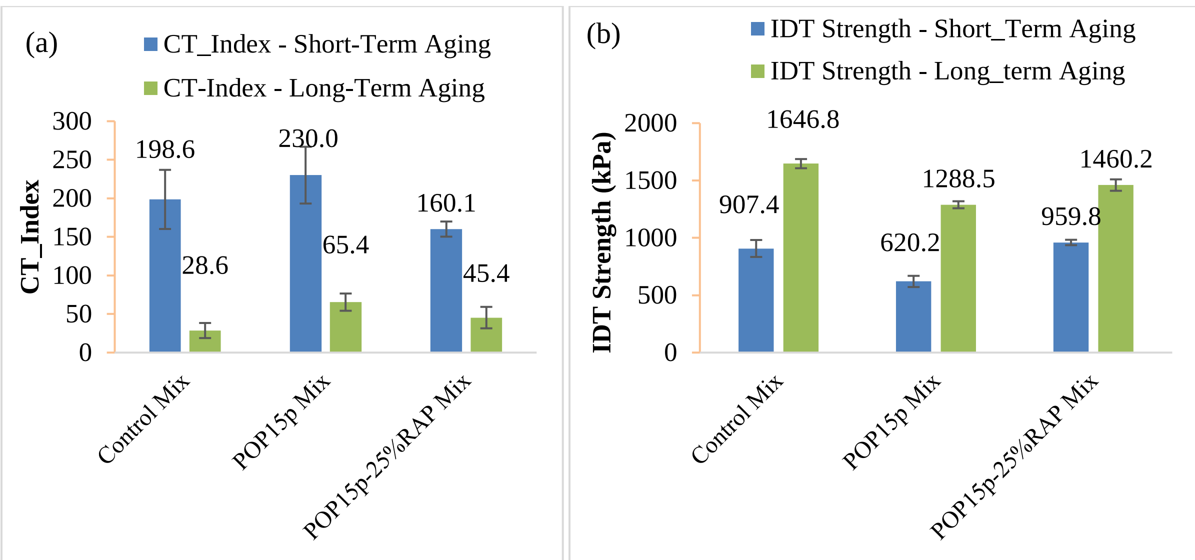


**Figure 17. (a) Cracking tolerance index (*$CT_{index}$*) and (b) IDT strength of mixes after short-term aging (60°C for 16 hours and 145°C for 2 hours on loose mixes) and long-term aging (145°C for 2 hours, 85°C for 120 hours, and 145°C for 2 hours on loose mixes).**

*Rutting Resistance*

The rut depth of asphalt mixes was tested using HWTT and the results are shown in Figure 18. Although the POP15p mix had a lower IDT strength than the control mix, it had comparable rutting resistance with the control mix. This result is consistent with the performance results of asphalt binders, as the control binder and POP15p binder are both classified as PG 64 binders based on the rutting parameter. The use of olive pomace could soften the asphalt binder but not compromise the rutting resistance. The POP15p-25%RAP mix had the best rutting resistance among the studied mixes, which is expected because the aged binder in RAP could increase the stiffness of an asphalt mix.

However, all of the asphalt mixes tested in this study failed to meet the Caltrans' specification that the minimum number of passes for the asphalt mix with PG 64 binder is 15,000 before rut depth reached 12.5 mm (Caltrans 2018). The reason is that moisture damages of asphalt mixes were observed, as fine aggregates were eroded from specimens during HWTT. Again, the pulp-rich fraction does not improve or compromise the moisture resistance of asphalt mixes. An anti-stripping additive, such as lime or liquid anti-stripping additive, is needed to improve the moisture and rutting resistances of the designed asphalt mixes.

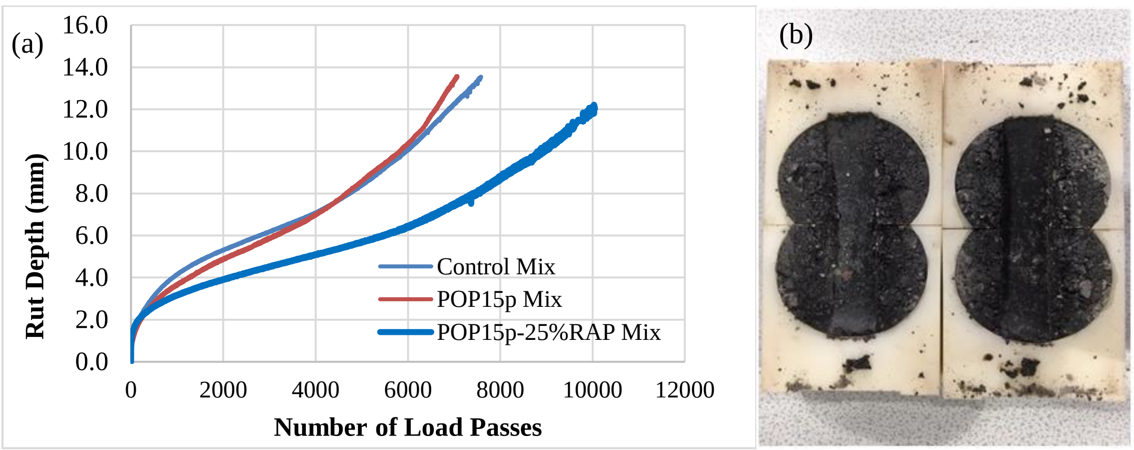


**Figure 18. (a) HWTT results and (b) evidence of moisture stripping for the control mixture.**

*Summary of Performance Results of Pomace-Modified Asphalt Mixtures*

The addition of olive pulp-rich fraction could improve the cracking resistance and antioxidant resistance of asphalt mixes, especially subjected to long-term thermal aging conditions. The pulp-rich fraction could soften the asphalt mix, but not compromise the rutting resistance of the asphalt mix. However, the pulp-rich fraction does not exhibit anti-stripping effectiveness. An anti-stripping additive is needed to improve the resistance to moisture damages of designed asphalt mixes.

The use of pulp-rich fraction allows incorporating waste asphaltic materials, such as RAP, in asphalt paving materials. The combined uses of bio-renewable olive pomace and RAP in asphalt mixes promote waste management and sustainability for both agriculture and road industries. Additional mixes shall be designed and tested to determine the optimal dosages of RAP and pomace when they are used together.

## Conclusions and Recommendations

This study explored the feasibility of using olive pomace as a natural asphalt modifier to improve the performance of asphalt pavements. The olive pomace was dried and processed using a coffee grinder. The mechanical and antioxidant performances of pomace-modified asphalt binders and mixes were evaluated. The processed olive pomace could improve resistances to cracking and oxidization for asphalt binders and mixes, especially subjected to long-term thermal aging conditions. The olive pulp-rich fraction has better softening and antioxidant effectiveness than the unscreened ground olive pomace, which is probably due to a higher residue oil content in the pulp-rich fraction. The addition of a 5% pulp-rich fraction could generally neutralize the oxidation of asphalt binder that occurred during production. The use of a higher dosage of the pulp-rich fraction to modify asphalt binder, e.g. 15% that is considered as the optimum dosage in this study, could extend the service life of asphalt pavements by retarding the oxidation-induced cracking. In addition, the use of pomace-modified asphalt binder allows incorporating recycled asphalt materials, which promotes sustainability for both agriculture and road industries.

The future works shall investigate the chemical composition in the processed olive pomace and identify active ingredients to increase antioxidant capacity, especially subjected to UV oxidation. Additional tests are needed to optimize the design of pomace-modified asphalt mixes with anti-stripping additives. There is also an urgent need to develop zero-waste recycling methods to utilize wastewater in olive pomace and conduct the life cycle cost analysis to determine the economic benefit of using olive pomace to improve the performance of asphalt pavements.

## Acknowledgments

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