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Influence of six rejuvenators on the performance properties of Reclaimed Asphalt Pavement (RAP) binder and 100% recycled asphalt mixtures

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Highlights

- Rejuvenators can reduce performance grade to the level of virgin asphalt binder.
- Use of appropriate rejuvenator dose allows to pass rutting requirement.
- Rejuvenators improve mixture cracking resistance.
- Workability or rejuvenated mixtures remained lower than for virgin mixture.

Abstract

100% recycled hot mix asphalt lab samples were modified with five generic and one proprietary rejuvenators at 12% dose and tested for binder and mixture properties. Waste Vegetable Oil, Waste Vegetable Grease, Organic Oil, Distilled Tall Oil, and Aromatic Extract reduced the Superpave performance grade (PG) from 94–12 of extracted binder to PG 64-22 while waste engine oil required higher dose. All products ensured excellent rutting resistance while providing longer fatigue life when compared to virgin mixtures and most lowered critical cracking temperature. Rejuvenated samples required more compaction energy compared to virgin and some oils reduced moisture resistance slightly.

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1. Introduction

Although Hot Mix Asphalt (HMA) is a 100% recyclable material, the current prevalent practice in the US is to use only about 20% of Reclaimed Asphalt Pavement (RAP) in a given mix design [1]. The reluctance for higher recycling stems from the possibility that the stiff binder that is present in RAP would cause premature fatigue and low temperature cracking failures [2-6]. The increase in RAP proportion in pavements escalates the potential of such cracking [7] which is one of the main reasons for government agencies to set a limit on the maximum allowed RAP content [8,7,9]. Other reasons are the unknown amount of actual blending that occurs between virgin and RAP asphalt binders and the effective contribution of the RAP binder towards the total binder content of the mix [10,11].

The use of rejuvenators has the potential to address all of the above noted issues, by making the RAP asphalt binder effectively “available” for blending with virgin materials, reducing the RAP mixture stiffness and providing the required binder performance for another service period. These products have the potential to do so by restoring the rheology and chemical components of aged RAP binder [12,13]. Rejuvenators are sometimes also referred to as softening additives or recycling agents but due to lacking industry consensus on the means of categorizing these oils all products are called “rejuvenators” in this article.

Rejuvenators would also allow a significant increase in the amount of RAP used in HMA mix design, and, perhaps even provide a chance for a total (100%) hot-mix recycling [14]. This would require ensuring homogeneous RAP material with low fines content, adequate mix design, and modified production plants as demonstrated in video: http://youtu.be/coj-e5mhHEQ. Despite the economic and environmental benefits that the increase in RAP content promise, some state agencies are reluctant to allow the use of rejuvenators, mostly due to potential rutting damage [15], which may result from ineffective blending of the rejuvenator with the RAP asphalt and resultant low stiffness micro-layer on the surface of the RAP [16]. Hence, careful selection of the rejuvenator is required to provide the pavement the necessary short and long term properties, as follows:

- Short term. Rejuvenators should allow the production of high RAP content mixture by rapidly diffusing into the RAP binder and mobilizing the aged asphalt in order to produce uniformly coated mixtures. Rejuvenator should soften the binder in order to produce a workable mixture that can be easily paved and compacted to the required density without the hazard of producing harmful emissions. Major part of diffusion process should be completed before the traffic is allowed to avoid reduction of friction and increased susceptibility to rutting.
- Long term. Rejuvenators should reconstitute chemical and physical properties of the aged binder and maintain stability for another service period. The binder rheology should be altered to reduce fatigue and low temperature cracking potential without over softening the binder to cause rutting failure. Sufficient adhesion and cohesion have to be provided in the mix to prevent moisture damage and raveling.

2. Objective

The objectives of the research are as follows:

- Evaluate and compare the use of various rejuvenators for restoring the properties of aged RAP binder to satisfy Superpave requirements.
- Evaluate the use of rejuvenators to produce 100% recycled hot mix asphalt with performance properties similar to those of virgin mixture.

3. Materials and methods

3.1. Materials

3.1.1. RAP and mixture design

The mixture was produced from re-graded 100% RAP that had been milled from pavements of various layers and locations in the state of New Jersey (NJ). The original RAP was crushed and screened in asphalt production plant to nominal maximum aggregate size of 9.5 mm. This is probably the reason for high dust (filler) content (10.5%) in the resultant mix which did not meet the Superpave gradation requirements (Fig. 1) for 9.5 mm Nominal Maximum Aggregate Size (NMAS) mixture. The RAP also had a relatively high binder content of 6.2%. In order to reduce the binder and dust content and to meet the requirements of a 9.5 mm NMAS design the RAP was screened. A proportion of 85% remaining on 2.36 mm sieve and 15% passing it were used for the re-graded design. The final RAP aggregate composition satisfied Superpave gradation requirements (Fig. 1) having asphalt binder content of 5.3% and a dust content of 7.9%. The total binder content increased to 5.94% after the addition of 12% rejuvenator by mass of the binder and this rejuvenator content was kept constant for all mixtures evaluated in the study.

3.1.2. Asphalt binder

Typically a virgin PG 64-22 binder is used in the climatic area where the RAP was obtained (New Jersey, US) and therefore this grade was selected as a reference binder. This binder was also used for design of virgin reference mixture.

3.1.3. Rejuvenators

Six different rejuvenators were used in the study and their origins are briefly described below. These products were chosen by screening eleven products at an earlier stage of the research which is described in Zaumanis et al. [17]. The measured kinematic viscosity and specific gravity of the rejuvenators are included in Table 1. This table also contains some basic characteristics that were obtained from manufacturers and the approximate cost of each product. For comparison, the table includes characteristics of the virgin binder that was used in the study.

3.1.3.1. Waste Vegetable Oil (WV Oil). WV Oil is increasingly used for bio-diesel production with compositional specifications including low free fatty acid content (<15%), less than 2% MIU (Moisture, Impurities, Unsaponifiables) [18]. Derived from fast and
convenience food frying oil, it is also referred to as “yellow grease”. The product used in this study consists predominately of peanut, sunflower, and canola oils, with large concentrations of Oleic and Linolic acids.

3.1.3.2. Waste Vegetable Grease (WV Grease). WV grease is also a food industry organic waste stream but semi solid at ambient temperatures due to the predominance of saturated Lauric and Myristic triglycerides and needs to be heated to reach liquid phase most of the time. The product used in this study is high in free fatty acids (>40%) but with its free glycerin and moisture removed industrially.

3.1.3.3. Organic Oil. Hydrogreen S™ (formerly known as BituTech RAP™) is an engineered product from PVS Meridian Technologies, Inc. It is designed to be a binder rejuvenator and a low temperature additive and has been tested in multiple road and laboratory studies demonstrating good performance [19]. It is composed of products of fast pyrolysis of pine tree biomass with other oils added to balance performance. The product is free flowing at room temperature, but a slight heating may be necessary when used in cold weather.

3.1.3.4. Distilled Tall Oil. Tall oil is a byproduct of paper manufacturing and is concentrated from kraft liquors. Tall oil is available either in crude form or as refined product which was used in this study. Crude tall oil contains fatty acids, resin acids and unsaponifiables in varying ratios depending on the tree type used. Tall oils have a long history of use in hot mix manufacturing with many emulsifiers, anti-strip agents and warm mix additives.

3.1.3.5. Aromatic Extract. An Aromatic Extract is a traditional rejuvenator with dominant polar aromatic rings. Recent findings express concern with unsaturated polar aromatic ring structure that has been shown to be carcinogenic [20]. Therefore, most industries are moving away from polar aromatic oils and towards less polar substitutes. This research is not intended to promote the use of Aromatic Extract, but rather to allow for the comparison of other products to a rejuvenator that has been used historically and has demonstrated acceptable long term performance.

Aromatic Extracts contain approximately 75% aromatic oils and resin compounds with balance saturated oils. Polar aromatics are known to associate with asphaltene molecules and in the process make the binder less brittle, by balancing the chemistry of the oxidized aged binder.

3.1.3.6. Waste Engine Oil (WEO). Engine lubricating oil is produced from paraffinic base oils with small dose of specialty compounds added to improve viscosity characteristics, stability, cleaning (ability to remove deposits from cylinder walls and oil pathways), and flammability. WEO may also contain short chain polar molecules that break apart during lubricating service. Recent interest in waste engine oil re-refinery around the world is making WEO increasingly difficult to obtain and more costly [21]. Waste engine oil should not be confused with waste engine oil bottoms which is the residue from re-refining.

3.2. Methods

3.2.1. Experimental plan

The research plan and test methods with respective ASTM standard numbers are summarized in Fig. 2. Each rejuvenator was dosed at 12% of binder mass for both the extracted RAP binder and 100% RAP mixture. This dose was selected to provide equal total binder content for all mixtures and is based on the author's

<table>
<thead>
<tr>
<th>Table 1</th>
</tr>
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<tbody>
<tr>
<td><strong>Rejuvenator properties and description.</strong></td>
</tr>
<tr>
<td><strong>Rejuvenator</strong></td>
</tr>
<tr>
<td>Waste Vegetable Oil (WV Oil)</td>
</tr>
<tr>
<td>Waste Vegetable Grease (WV Grease)</td>
</tr>
<tr>
<td>Organic Oil</td>
</tr>
<tr>
<td>Distilled Tall Oil</td>
</tr>
<tr>
<td>Aromatic Extract</td>
</tr>
<tr>
<td>Virgin PG 64-22</td>
</tr>
</tbody>
</table>

Fig. 2. Research test plan and test methods with the respective ASTM standard number.
previous research of these products with this particular RAP [22]. In the study the extracted RAP binder was tested after adding various doses of each product to determine the range at which the reclaimed binder meets Superpave PG 64-22 requirements. Maximum dose was defined to ensure rut resistance (satisfy high PG temperature) while minimum dose was set by either low PG temperature or intermediate temperature parameter (the higher of the two was defined as the minimum so that the other parameter is also satisfied). The results in Table 2 demonstrate that the dose selected for this study (12%) is in the optimum range for all products but WEO.

Addition of rejuvenators to extracted RAP binder demonstrates the softening efficiency of the products and allows determining the required dose to satisfy the binder specification requirements. However, it artifically simulates full blending of rejuvenator and RAP binder, which might not be true in asphalt mixtures. Many research results have shown that in reality the blending is somewhere between full and no blending at all [10,23,24,11]. The part of RAP binder that does not significantly change its properties with addition of recycling agent is often attributed to as “black rock” and only performance-related mixture tests can demonstrate the effect of this phenomena. Use of incompatible rejuvenators or overdose are two other concerns that can be addressed by performing mixture testing since these effects would be highlighted by loss of cohesion and adhesion thus leading to raveling and moisture damage. For these reasons, 100% RAP rejuvenated mixtures were prepared for performance-related testing and compared with two reference mixtures:

- To quantify performance of this specific 9.5 mm gradation as a virgin mixture, aggregates were obtained by removing binder from the re-graded RAP in an ignition oven. They were blended with 5.94% of virgin PG 64-22 bitumen which is equal dose to that of the rejuvenated samples (binder + rejuvenator). This is labeled as “Virgin Mix” in the results.
- To evaluate the benefit of simply increasing binder dose rather than adding rejuvenators, virgin binder was added to the RAP at 12% of binder mass (equal to dose of rejuvenators). This mixture is named “RAP Mix” and used as representation of un-rejuvenated mixture to correlate with RAP binder in the figures.

The Superpave binder test results are reported first, followed by mixture test results and correlation, if any, between the two.

### 3.2.2. Binder sample preparation

Binder was extracted from the RAP using toluene according to ASTM D2172, method A and recovered using rotary evaporator, according to ASTM D5404. To ensure equal properties for all extracted batches, all extracted samples were blended together before batching in separate cans for belonging with 12% of each rejuvenator after 40 min of heating at 140 °C temperature.

### 3.2.3. Performance grade (PG)

The rejuvenated binder was graded according to the Superpave Performance Grading (PG) requirements defined in AASHTO M 320. Dynamic Shear Rheometer (DSR) at 10 rad/s frequency according to AASHTO M T 315 was used for determining high temperature properties using 25 mm plates with 1 mm gap. The low temperature stiffness and m-value (the ability to rapidly disperse the accumulating thermal stress) were recorded at 60 s using the Bending Beam Rheometer (BBR) test setup according to AASHTO T 313. The PG system specifies constant physical property requirements as indicated in Table 3 and the temperature at which binder can meet these requirements (with 6 °C increments) defines its high and low temperature grade. The required grade is based on the specific climatic conditions and traffic load. Because of the origin of the RAP that is used in this study the target PG was defined as 64-22. Intermediate temperature fatigue parameter $G’ \sin \delta$ for this grade is determined at 25 °C using DSR 8 mm plates at 2 mm gap at 10 rad/s frequency. Rotational viscosity was determined according to AASHTO T 316 and demonstrates workability of the binder.

Short term aging was performed using Rolling Thin Film Oven at 163 °C for 85 min (ASTM D2872) and long term aging was executed using Pressure Aging Vessel (PAV) for 20 h at 210 MPa pressure at 100 °C (ASTM D652). Agering before grading of all test specimens (including RAP and rejuvenated RAP) was performed in the same way as done for grading of virgin binder as indicated in Table 3. Original binder was tested for rotational viscosity. Original and short term aged binder were used for determining the high PG temperature. Short plus long term aging was performed before determining low PG temperature (BBR test) and intermediate temperature fatigue parameter $G’ \sin \delta$. Mass loss after short term aging with RTFO at 163 °C was calculated according to AASHTO T 240.

### 3.2.4. Mixture sample preparation

Mixing and compaction temperature of the mix samples was selected based on the viscosity results and was 145 °C for all the mixes. RAP and rejuvenator were heated at this temperature in an oven for 2 h before mixing them together (with 12% rejuvenator of RAP binder content) using a planetary mixer. All samples were short term conditioned for 4 h at 145 °C before compaction using gyratory compactor according to ASTM D6925. In order to perform volumetric calculations, the bulk specific gravity of RAP aggregates

### Table 2
Rejuvenator optimum dose based on Superpave PG tests [18].

<table>
<thead>
<tr>
<th>Rejuvenator</th>
<th>Max dose (%)</th>
<th>Min dose (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WV Oil</td>
<td>16.4</td>
<td>7.4</td>
</tr>
<tr>
<td>WV Grease</td>
<td>16.4</td>
<td>8.1</td>
</tr>
<tr>
<td>Organic blend</td>
<td>18.4</td>
<td>9.1</td>
</tr>
<tr>
<td>Distilled Tall Oil</td>
<td>18.8</td>
<td>9.4</td>
</tr>
<tr>
<td>Aromatic Extract</td>
<td>27.8</td>
<td>11.5</td>
</tr>
<tr>
<td>WEO</td>
<td>25.0</td>
<td>16.0</td>
</tr>
</tbody>
</table>

- Based on intermediate PG requirement.
- Based on low PG.

### Table 3
Superpave binder specification requirements (AASHTO M 320).

<table>
<thead>
<tr>
<th>Test method</th>
<th>Temperature</th>
<th>Test parameter</th>
<th>Binder state</th>
<th>Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dynamic shear rheometer</td>
<td>High PG</td>
<td>$G’ \sin \delta$</td>
<td>Original</td>
<td>$\geq 1.0$ kPa @ 10 rad/s</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>RTFO Residue</td>
<td>$\geq 2.2$ kPa @ 10 rad/s</td>
</tr>
<tr>
<td>Bending beam rheometer</td>
<td>Intermediate PG (test $\Theta 25$ °C for PG 64–22)</td>
<td>$G’ \sin \delta$</td>
<td>RTFO + PAV Residue</td>
<td>$\leq 5000$ kPa @ 10 rad/s</td>
</tr>
<tr>
<td></td>
<td>Low PG</td>
<td>Creep Stiffness</td>
<td>RTFO + PAV Residue</td>
<td>$\leq 300$ MPa</td>
</tr>
<tr>
<td>Rotational viscometer</td>
<td>135 °C</td>
<td>m-Value</td>
<td>Viscosity</td>
<td>$\leq 3$ Pa s</td>
</tr>
<tr>
<td>RTFO</td>
<td>163 °C</td>
<td>Mass loss</td>
<td>Before and after RTFO</td>
<td>$\leq 1$%</td>
</tr>
</tbody>
</table>
was determined according to ASTM C127 (coarse aggregates) and ASTM C128 (fine aggregates) after burning the binder in an ignition oven according to ASTM D6307. The same procedure was used to determine the RAP binder content and to obtain RAP aggregates for the reference virgin mixture. Automatic vacuum sealing method (Corelock™) was used for determining the bulk specific gravity of compacted samples according to ASTM D6752/D6752M and the maximum specific gravity of loose RAP according to ASTM D6857. The air voids for all mix samples were kept to 7 ± 0.5%. The VMA (Voids in Mineral Aggregate) ranged from 16.9% to 18.1% and VFA (Voids Filled with Asphalt) ranged 56.3–62.9% for the samples.

The mean value of two test results for fatigue test and Hamburg rutting test, three for tensile strength and creep compliance, and four for workability along with one standard deviation are reported for each test for every sample set. The creep compliance was determined according to the standard procedure which stipulates using trimmed mean from all three sample (dropping the maximum and minimum) displacement values before calculating the result; thus the standard deviation cannot be expressed in this case.

3.2.5. Hamburg wheel tracking test (WTT)

Hamburg wheel tracking test samples were prepared by gyratory compactor using 150 mm molds to approximately 60.5 mm height and then they were placed in WTT testing molds as illustrated in Fig. 3a. Two pairs of samples of each mixture type were tested for rutting according to AASHTO T324 submerged in 50 °C water. This test temperature was chosen based on the Texas DOT procedure for PG 64-22 binder where the test is used for accepted in the range of moisture damage and not rutting alone as illustrated. This is illustrated by calculating the inflection point in the rut depth versus loading cycles curve (Fig. 3b) using a third order polynomial function.

3.2.6. Workability

The changes in workability due to the addition of the rejuvenators were evaluated using binder and mixture data. The gyratory compacted samples prepared for the Hamburg WTT were also used for evaluation of the mixture workability by calculating the number of gyrations to 8% air voids according ASTM D6925. These results were compared to the rotational viscosity of the binder.

3.2.7. Critical cracking temperature

Three gyratory compacted samples (46.5 mm high and 150 mm in diameter) of each mix were tested for creep compliance at three temperatures (0, −10, −20 °C), followed by tensile strength test at −10 °C according to AASHTO T 322 standard on specimens in indirect tension configuration. Creep compliance was measured by applying static load to initiate asphalt deformation in the visco-elastic range (0.00125–0.0190 mm horizontal deformation at 1000 s). The deformation was measured with horizontal and vertical displacement transducers glued on both sides of the saw-cut sample (cutting improves the repeatability of results) to determine the time dependency of strain resulting from stress. Indirect tensile (IDT) strength was determined on creep compliance samples by applying 12.5 mm/min vertical loading rate. Only the vertical ram movement was measured and the uncorrected IDT strength was derived from the maximum load.

Creep compliance up to 100 s and tensile strength were used to determine the master relaxation function curve and fracture parameters in order to calculate the critical cracking temperature of the pavement. L1STRESS MS Excel™ spreadsheet (version from April 2012), developed by Christensen [25], was used for this calculation. The spreadsheet is based on mechanistic prediction model developed under the Strategic Highway Research Program (SHRP) [26]. The thermal stress is expressed as a hereditary integral, which includes the relaxation function, the shift factor function, the coefficient of thermal expansion of the mixture, the initial temperature, and the rate of temperature drop (5.6 °C/h in this case). The hereditary integral is integrated numerically to give thermal stress as a function of pavement temperature. To determine the fracture resistance of the mixture, the uncorrected IDT strength is corrected to account for the non-linear behavior as explained in NCHRP Report 530 [27] and correlated with field-core testing results by multiplying by 0.63 according to the data from Advanced Asphalt Technology, LLC [28]. The critical pavement cracking temperature (T_CR) is estimated as the temperature at which the surface thermal stress reaches the fracture resistance of the mixture. A simplified illustration of the calculation principle is demonstrated in Fig. 4.

3.2.8. Coaxial shear test (CAST)

The coaxial shear test (CAST) was developed at EMPA (Swiss Federal Laboratories for Materials Science and Technology) in Switzerland. It is a cyclic, axial loading system to determine the complex modulus (E*) and phase angle of asphalt mixtures [29]. The shear load is applied perpendicular to the specimen's circular

![Fig. 3](image-url) Typical sample after Hamburg WTT (a) and definition of stripping inflection point (b).

![Fig. 4](image-url) Critical cracking temperature calculation example.
surface through the central core, with lateral confinement provided by a metal ring surrounding the specimen as illustrated in Fig. 5. Such setup allows loading along the same axis as that of traffic while the lateral confinement simulates a semi-infinite situation experienced on the road. The complex modulus is calculated by the acquisition software integrated with a finite element model by taking into account the glue properties, specimen dimensions and geometry of the set-up as explained by Sokolov et al. using Eq. (1) [30,31].

\[
G' = \frac{F}{\delta_a} - A(G')
\]

where

- \( G' \) – complex modulus in shear.
- \( F \) – force amplitude along the steel core.
- \( \delta_a \) – displacement amplitude along the steel core.
- \( A(G') \) – coefficient function derived from finite element analysis by recursive iteration.

Tests were performed on long term aged (five days at 85 °C) donut shaped samples cut to 47 mm height and having a 55 mm hole in the center. A servo-hydraulic tension-compression machine was used for loading and the sample surface temperature during the test was maintained at 30 °C. Sinusoidal vertical deformation in a strain controlled mode with amplitude of 0.01 mm at the central core was applied at 10 Hz frequency.

4. Results and discussion

4.1. Performance grade

The extracted RAP binder was tested for PG after the addition of each of the rejuvenators and the results, along with continuous grade of virgin PG 64-22 binder, are illustrated in Fig. 6. The RAP binder had severely aged and grades as PG 94-12, but the addition of all products allowed reducing both the high and low binder PG temperature. The required low performance grade temperature of −22 °C was reached in all cases except when using WEO. The organic products (WV Oil, WV Grease, Organic Oil, Distilled Tall Oil) proved to be more efficient at the 12% dose compared to petroleum products (Aromatic Extract, WEO) and actually reduced the PG temperature more than required and below the temperature of the virgin binder (−26 °C). As described earlier (Table 3), the low PG temperature is determined by specifying the minimum m-value (0.3) and maximum stiffness (300 MPa) for BBR test sample. The lowest temperature at which both requirements are passed defines the low PG temperature. For the rejuvenated samples in this study in all cases the PG temperature was defined by the m-value requirement and for several rejuvenators the temperature at which the binder passes the stiffness requirement was significantly lower than the temperature defined by the m-value. For example, the temperature at which WEO and WV Oil rejuvenated samples passed the stiffness requirement was below −53 °C. This indicates that both for evaluating binder and mixture properties at low temperatures the ability to rapidly disperse the accumulating thermal stress (m-value) should be of primary interest when using high RAP content.

At the same time none of the rejuvenators reduced the high grade to the level of virgin binder thus showing increased resistance to rutting and providing grade sum that is greater than that of the virgin binder. This demonstrates that, despite the general concern, if adequate dose of rejuvenator is used and proper diffusion and blending occurs in the asphalt binder film, there is no danger of increased rutting susceptibility with the use of rejuvenators.

The Superpave intermediate temperature fatigue parameter \( G' \sin \delta \) (complex shear modulus viscous portion) results at 25 °C are indicated with diamonds in Fig. 6. All rejuvenators have decreased the \( G' \sin \delta \) from 12,600 kPa for extracted RAP binder to a level that passes the Superpave requirement of maximum 5000 kPa.

4.1.1. Susceptibility to aging

The volatilization results from aging the samples in RTFO, along with the Superpave requirement of less than 1% mass loss, are shown in Fig. 7. The virgin binder demonstrates only 0.2% mass loss while the extracted RAP binder exhibits significant reduction (0.93%) of volatile fractions and barely passes the requirement. This is likely a result of incomplete distillation of solvent from the RAP binder during extraction. During the RTFO aging any residual solvent would evaporate thus contributing to the mass loss. Most of the rejuvenated binders exceed the allowable 1% mass loss; however, this is mostly because of the high mass loss of RAP binder. Therefore, in order to demonstrate the “rejuvenator portion” of mass loss, the “binder portion” has been subtracted from the total mass loss and colored with a lighter shade in the plot. Consecutively, the dark bar demonstrates the “rejuvenator portion” of the mass loss. This shows that only the WEO exceeds the allowable limits of volatilization, while other rejuvenators have less than 0.5% loss and WV Oil as well as Aromatic Extract does not cause any additional mass loss compared to that of the RAP binder.

Superpave specifications require the determination of high PG temperature on both original and short term aged (RTFO) binder. Different minimum \( G' / \sin \delta \) requirement is defined at each of these states as indicated in Table 3 and temperature at which binder can pass these requirements is determined. Lowest of the two temperatures at which the requirement is met is defined as the high PG. While these requirements are empirical, they allow comparing the effect of rejuvenators on the aging of the samples. As illustrated in Fig. 8 both virgin and RAP binder PG temperature remained constant after aging whereas the use of WEO and WV Grease increased the temperature by around 4 °C. This shows that the resistance to rutting will increase over time, but may be an indicator of accelerated aging that can lead to cracking failures. Closer analysis of the data did not reveal dominance of either increased stiffness (\( G' \)) nor higher elasticity (reduced phase angle) as the sole cause of the different aging properties of the rejuvenators compared to the neat binder.

4.2. Rutting and moisture susceptibility

The Hamburg WTT results are illustrated in Fig. 9 along with the Texas DOT requirement for maximum rut depth (12.5 mm at 10,000 wheel passes for PG 64-22 binder). As expected due to
the aged binder, the RAP mixture has the highest rutting resistance. Rejuvenators have slightly increased the rut depth but all of them pass the rutting requirement. The only sample that fails this requirement is the virgin mixture. As noted, this sample was prepared by burning off binder from the 100% RAP mixtures and replacing it with virgin PG 64-22 binder at a dose that is equal to that of the rejuvenated samples (5.94%). The poor performance of this mix in comparison to the other mixes might be caused by (1) lower binder viscosity, (2) loss of fines during the burning process and therefore excessive binder content, (3) moisture damage.

Most of the rejuvenated mixtures did not reach stripping inflection point within the test period of 20,000 wheel passes as can be seen in Fig. 9. According to Colorado DOT [32] inflection point before 10,000 wheel passes indicates moisture susceptibility. WV Oil and virgin mix failed this requirement, but the Organic Oil, although reduced moisture resistance of the source RAP, passed this criterion.

Correlation of rut depth at 20,000 passes with binder high PG temperature is illustrated in Fig. 10. As expected, the results demonstrate increase in rut depth with decreasing PG temperature. The initial selection of rejuvenator maximum dose can therefore be based on the desired binder high PG temperature. However, this might hold true only if rejuvenator diffusion is completed and good blending with binder occurs (assuming no “black rock” effect). Insufficient blending may lead to increased rutting early in the pavement’s life due to soft outer layer of binder film dominating the pavement performance [8,5,33].
Fig. 10. Correlation of Hamburg WTT results and high PG temperature.

4.3. Workability

Rotational viscosity of the binders at 135 °C demonstrate that all recycling agents have reduced the viscosity of RAP binder from 4.3 Pa s to the Superpave requirement of less than 3 Pa s. These results are compared with mixture gyrations to 8% air voids in Fig. 11. It is hypothesized that, since the mixture compaction temperature (around 145 °C) is close to the test temperature of viscosity (135 °C), the two results should be in good agreement unless other factors affect mix workability. As expected, all of the recycled mixtures/binders align between the virgin and RAP data points both based on binder viscosity and on mix workability. They, however, show poor correlation, which might be a result of large statistical error of the gyratory test but might also be affected by diffusion rate of the recycling agents. The binder results by default provide 100% rejuvenator blending/diffusion, while in the mixture the extent of diffusion and binder activation depends on the time, temperature, rejuvenator and binder type [34]. It is possible that at the time of compaction part of RAP binder might be behaving as “black rock” [24], thus increasing the required compaction energy. The “black rock” binder content extremes in this case are the Virgin binder/mix and the RAP binder/mix (illustrated by squares). The former demonstrates the mixture workability of virgin mix with all of binder contributing to the mixture workability and the latter, along with highest viscosity, can be reasonably assumed to have the least activated binder content. Further research is necessary to evaluate this phenomenon.

At the least, these results demonstrate that caution should be used when evaluating the workability of rejuvenated binder since the results might not be reflected in compactivity of the mixture. Unlike for conventional binders, the increase in temperature will not only reduce the viscosity but also significantly facilitate the diffusion, affecting the effective binder content and lubricity of binder surface.

4.4. Low temperature properties

Neither stiffness nor strength alone determines when a mixture will crack. A stiff mixture will not crack if its strength is high enough; and a weaker mixture will not crack if it is sufficiently flexible [35]. Strength of rejuvenated mixes was tested by indirect tensile strength test, while creep compliance at low temperature shows the pavement’s potential to creep under thermal load stress at low temperatures.

Table 4 demonstrates the test results of creep compliance at 100 s at 0 °C, −10 °C and −20 °C. As expected RAP mix has the highest stiffness (lowest compliance) in most temperatures due to the presence of aged RAP binder. Virgin mix has the lowest stiffness at 0 °C while at other temperatures WEO and WV Oil are less stiff. Other rejuvenators have reduced the stiffness (except Distilled Tall Oil and Organic Oil at −20 °C) compared to RAP mix but the samples are more stiff than the virgin mix.

Fig. 12 shows the critical mixture low cracking temperature as calculated using LTSTRESS spreadsheet. The tensile strength at −10 °C is plotted using rhombs on a reverse scale relative to source RAP mix to provide visual comparison with cracking temperature.

Most of the rejuvenators have improved the cracking resistance compared to the source RAP mixture. The Aromatic Extract and WV Oil even provide cracking temperature similar to the virgin mixture. Note that, as shown by the rutting test, virgin mix would require reduction of binder content or change of binder grade thus likely the cracking temperature would increase (become warmer).

Most of the rejuvenated mixes have very similar tensile strength (rhombs in Fig. 12) compared to the source RAP Mix, hence the lowering in cracking temperature is generally caused by reduced stiffness as illustrated in Table 4. Only the Aromatic Extract has provided statistically higher tensile strength compared to RAP Mix, which is the cause of reduction in cracking temperature (the mixture stiffness by application of this additive was reduced similarly to others). The good performance of WV Oil rejuvenated mixture, conversely, is caused primarily by the reduction in mixture stiffness; WEO also provides relatively large reduction in stiffness, but due to lowered strength demonstrates only average $T_{crit}$. On the other hand, the relatively poor performance of Distilled Tall Oil sample is caused by insignifican change in mixture stiffness.

Critical cracking temperature of mixture is compared with the respective binder low PG temperature in Fig. 13. The samples that are rejuvenated using petroleum additives (illustrated as rhombs) provide similar mixture cracking temperature to the samples with

| Table 4 |
| Creep compliance at 0 °C, −10 °C, and −20 °C temperature. |

<table>
<thead>
<tr>
<th>Test temperature</th>
<th>RAP Mix</th>
<th>WV Oil</th>
<th>WV Grease</th>
<th>Organic Oil</th>
<th>Distilled Tall Oil</th>
<th>Aromatic Extract</th>
<th>WEO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Creep compliance at 0 °C, 1/GPa</td>
<td>0.113</td>
<td>0.325</td>
<td>0.158</td>
<td>0.138</td>
<td>0.130</td>
<td>0.149</td>
<td>0.201</td>
</tr>
<tr>
<td>Creep compliance at −10 °C, 1/GPa</td>
<td>0.059</td>
<td>0.154</td>
<td>0.116</td>
<td>0.087</td>
<td>0.063</td>
<td>0.091</td>
<td>0.120</td>
</tr>
<tr>
<td>Creep compliance at −20 °C, 1/GPa</td>
<td>0.050</td>
<td>0.076</td>
<td>0.058</td>
<td>0.046</td>
<td>0.044</td>
<td>0.054</td>
<td>0.079</td>
</tr>
</tbody>
</table>
organic products (squares). This is despite the fact that petroleum products have higher (warmer) PG low temperature at the selected 12% dose. There seems to be two almost parallel trends with a PG temperature shift of 10°C for correlation between the bitumen and mixture critical low temperature of rejuvenated mixtures using the petroleum and organic additives. The reasons for the superior performance of mixtures recycled with petroleum rejuvenators, however, are not identical in all instances as discussed previously.

4.5. Fatigue

Fig. 14 demonstrates the complex modulus of mixtures as determined using the coaxial shear test (CAST). In most cases, the mean of two results is reported for each mixture with error bars at the end indicating the average statistical deviation between the samples throughout the test. For the RAP mixture and VW Grease samples only one test was finalized since one of the specimens was damaged during testing. In addition to the mixtures used in the study a mixture with 9.5 mm NMAS design having 20% RAP content and produced using PG 64–28 binder was also tested. This mix was added to the test matrix in order to compare the rejuvenated mixtures with a conventionally designed mixture that has been approved for application on public roads. This mix was designed according to Superpave specifications by applying 50 gyrations for roads having traffic intensity of 0.3–3 ESAL's (Equivalent Single Axle Loads). All materials and mix design for this sample were obtained from a contractor in the state of Maine, US.

As expected due to the presence of aged binder, the RAP mixture has higher stiffness compared to the virgin and 20% RAP mixtures. Comparing to the RAP mixture most rejuvenators have reduced the stiffness while Distilled Tall Oil and, surprisingly considering the previous results, Organic Oil have increased it. The stiffness of all of these samples in general can be considered low. Other studies that have used CAST for up to 1,400,000 cycles at 30°C and 10 Hz loading frequency report modulus values above 800 MPa [29,36,30,37,38].

Traditionally, the number of repetitions of loading required to reach a 50% reduction in initial stiffness is considered to be an appropriate parameter for evaluating fatigue performance [39,40]. However, at the applied strain level (212 micro strain) during the 1.4 million cycles none of the samples exhibited such loss in modulus. Therefore, the procedure described in AASHTO D7460-10 (developed by Tsai et al. [41,42]) was used for extrapolating the result to failure point based on one-stage Weibull
Survivor Function according to Eq. (2). The equation is solved for cycles to failure (N) where the stiffness ratio (SR) equals 0.5.

\[ \ln(-\ln(SR)) = \gamma \cdot \ln(N) + \ln(\hat{\gamma}) \] (2)

where

- \( SR \) – stiffness ratio, beam stiffness at cycle \( i \)/initial beam stiffness (at 1000 cycles in this study).
- \( N \) – number of cycles.
- \( \gamma \) – the slope of the linear regression of the \( \ln(-\ln(SR)) \) versus \( \ln(N) \) as demonstrated in Fig. 15.
- \( \ln(\hat{\gamma}) \) – the \( y \) intercept of the linear regression of the \( \ln(-\ln(SR)) \) versus \( \ln(N) \) as demonstrated in Fig. 15.

The cycles to failure (50% reduction in modulus from initial stiffness at 1000 cycles) are illustrated in Fig. 16 on a logarithmic scale with bars indicating the maximum and minimum results for the two test samples. As demonstrated earlier in Fig. 14 Distilled Tall Oil sample had almost no change in the complex modulus therefore it is not illustrated in the figure.

Virgin and 20% RAP mixtures performed similarly. The WEO rejuvenated sample had the lowest cycles to failure compared to all other mixes and thus it had the highest susceptibility to fatigue cracking. All other samples demonstrated longer fatigue life compared to virgin mixture. This may seem counter-intuitive but in fact increased fatigue resistance of high RAP mixtures has been previously demonstrated by multiple laboratory studies [43,11,44–46,2]. Fatigue failure generally occurs when the applied loads are too high for the pavement structure or more repetitions of a given load are applied than accounted for in design [47]. If the structural design of pavement is sound and thus strain levels in HMA are ensured low, high stiffness materials of low viscosity (such as high RAP mixtures) can be expected to have high resistance to fatigue cracking [48]. A more thorough study of the samples by performing tests at other strain levels and temperatures and an analysis of the results with respect to traffic load and anticipated stress levels would be required to provide more conclusive evidences of the expected fatigue behavior of these samples. For example, if the traffic load at a certain temperature causes high strain in the pavement a mixture with high stiffness and high binder viscosity (as in the case of rejuvenated mixtures) would exhibit increased stress and the inability to relieve the stress by flow could lead to brittle pavement and potential fatigue cracking [49].

5. Summary

The performance of six different rejuvenators to improve performance-related properties of extracted RAP binder and 100% RAP mixtures was described. This was a phase of a broader study that also includes description of 100% RAP production methods, optimum rejuvenator dose determination, and testing of chemical and micromechanical properties of rejuvenators, and is summarized in video: http://youtu.be/y-rydGiebY.

The performance of each rejuvenator at 12% dose is discussed below and the results are summarized in Table 5. The following arbitrary scale is introduced in the table:

- Positive (+1): sample passes the specification pass/fail requirement or if none, performs equal or better than virgin binder/mix.
- Negative (-1): sample fails the specification pass/fail requirement or if none, performs equal or worse than RAP binder/mix.
- Neutral (±0): sample performs better than RAP binder/mix but not as good as virgin binder/mix. Applied only if no pass/fail criteria are available by specifications.

The summary row in Table 5 presents sum of the five measured performance parameters. Half of each property score is represented by binder test result and the other half is mixture test result (if both results are available for the specific property). The final score shows that overall Aromatic Extract showed the best performance passing all specification requirements and only having one parameter, workability, lower than the virgin mixture. However, the authors do not recommend the use of this product due to possible carcinogenic effects. Instead, one of the organic products can be used since with appropriate mix design procedure their performance likely can be optimized to comply with the requirements.

5.1. Virgin mixture

Virgin mixture was prepared by burning off the RAP binder and mixing the aggregates with a virgin binder at a dose that is equal to that of the rejuvenated mixtures (5.94%). This mixture was the only one to fail the Hamburg WTT test requirement, partly as a result of
higher than optimum binder content. Likely as a result of this, the virgin mixture has the best workability and high low temperature cracking resistance.

5.2. RAP mixture

RAP mixture has 12% of virgin bitumen added to equalize the total binder content with all other mixtures. As expected due to severe ageing of binder (PG 94–12) this mixture has the worst workability, high rutting resistance, and low thermal cracking resistance. The fatigue life of this mix at the test parameters was longer than that of the virgin mix while the RAP binder (extracted from non-modified RAP) demonstrated the lowest fatigue resistance.

5.3. WV Oil

Waste Vegetable Oil at the selected dose of 12% showed the most rejuvenation potential on the aged binder. Compared to all other rejuvenators it provided the most reduction in binder fatigue parameter $G^* \sin \delta$ and most reduction in performance grade. Along with mixture cracking temperature that is similar to virgin mixture and improved fatigue performance compared to virgin mix this promises good cracking resistance. However, the Hamburg WTT results suggest that this additive has increased the susceptibility to moisture damage and therefore use of adhesion promoting additives may be necessary.

5.4. WV Grease

Waste Vegetable Grease significantly reduced the low PG and demonstrated the best mixture workability from recycled mixtures. The mix cracking temperature, however, was only slightly lower than that of RAP mix. The selected dose of 12% is likely the maximum that should be used for this rejuvenator for the tested RAP in order not to cause plastic deformations. Possible effect of accelerated aging was observed based on the increase of complex shear modulus elastic portion.

5.5. Organic Oil

Organic Oil (Hydrogreen S™) significantly improved the mix workability and along with WV Oil provided the most reduction of low temperature PG and the highest grade sum. The mixture cracking temperature was also significantly improved compared to RAP mixture which concurs with other studies [19]. The Hamburg WTT results, although indicated slightly increased susceptibility to stripping, demonstrate high resistance to rutting. Along with Distilled Tall Oil, this rejuvenator also provided the best mixture fatigue performance.

5.6. Distilled Tall Oil

Distilled Tall Oil had an average performance in most of the tests. Critical mixture cracking temperature, due to almost no reduction of RAP mix stiffness, was the highest (most positive) from all samples. At the same time, low temperature PG was reduced even lower than that of virgin mixture. The mixture did not exhibit any loss of stiffness up to 1.4 million loading cycles thus potentially demonstrating the highest fatigue life at the test parameters.

5.7. Aromatic Extract

The 12% dose that was used in this study is likely too small for the rejuvenator, since it did not allow reaching the low PG of virgin binder, provided the least reduction in viscosity and, although it passed the Superpave fatigue requirement, the performance remained lower than that of virgin binder. The mixture low temperature cracking resistance, however, was significantly improved and became equal to that of the virgin mix. Overall, although the Aromatic Extract rejuvenated samples did not have the best performance in each specific test, the performance-related specification requirements (that were used in this study) were passed thus resulting in the highest score from all rejuvenators. Due to the fact that this is a carcinogen its use is not recommended.

5.8. Waste Engine Oil

WEO at 12% dose did not allow to reduce the PG to the required level of $-22^\circ C$, but despite slightly reduced low temperature strength of the mixture, the low temperature cracking susceptibility of the mixture was considerably improved. The Superpave fatigue parameter $G^* \sin \delta$ was reduced to the required level, but the mixture fatigue life was the lowest from all samples. WEO exhibited increased loss of volatile fractions in the mass loss test and indicated increased susceptibility to aging based on the DSR test before and after aging. The lowest overall score among the rejuvenators might be a result of the straight-chain aliphatic molecular structure of this product resulting in the inability to provide balanced chemical composition of the RAP (incompatibility). This has to be verified in chemical analysis.

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Table 5
Summary of test results.

<table>
<thead>
<tr>
<th>Property</th>
<th>Test method</th>
<th>Virgin mix/ binder</th>
<th>RAP mix/ binder</th>
<th>WV Oil</th>
<th>WV Grease</th>
<th>Organic Oil</th>
<th>Distilled Tall Oil</th>
<th>Aromatic Extract</th>
<th>WEO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fatigue</td>
<td>$G^* \sin \delta$ CAST, 50% stiffness loss</td>
<td>$&gt;5000$ kPa</td>
<td>$&lt;3000$ cycles</td>
<td>$&gt;5000$ kPa</td>
<td>$&lt;3000$ cycles</td>
<td>$&gt;5000$ kPa</td>
<td>$&lt;3000$ cycles</td>
<td>$&gt;5000$ kPa</td>
<td>$&lt;3000$ cycles</td>
</tr>
<tr>
<td>Summary</td>
<td></td>
<td>10</td>
<td>10</td>
<td>30</td>
<td>30</td>
<td>45</td>
<td>35</td>
<td>55</td>
<td>0</td>
</tr>
</tbody>
</table>

*pass/fail criteria unavailable, requirement based on virgin mix performance
*only the rejuvenator portion is considered (see section 4.1.1)

Legend: positive (+1), neutral (0), negative (-1)
6. Conclusions

The following conclusions can be drawn from the study:

- Five of the six tested rejuvenators at 12% dose ensured correspondence to the required PG 64–22 temperature and provided grade sum greater than that of the virgin binder. Organic additives proved to be more efficient at reducing PG temperature compared to petroleum products. All rejuvenators also ensured compliance to the Superpave binder fatigue and workability requirements.

- The high PG for all rejuvenated samples remained above that of virgin binder indicating increased rutting resistance. High PG temperature correlated well with the Hamburg WTT proving that all recycled mixtures have high rutting resistance.

- Low temperature mixture cracking test results showed that five of the six rejuvenators have decreased cracking susceptibility compared to RAP mixture. WV Oil and Aromatic Extract performed similarly to virgin mixture while others had slightly warmer cracking temperature.

- Despite having higher (warmer) binder low PG temperature the petroleum additives provided similar mixture low temperature cracking resistance to the organic additives.

- Fatigue resistance of recycled mixtures at the used test parameters was higher than that of virgin mixture for all except WEO-rejuvenated mixture.

- Workability of RAP mixture was increased by the use of all rejuvenators, but none was able to improve it to the level of the virgin binder or mixture.

- Overall the authors believe that most products that were used in this study can be applied as rejuvenators by optimizing the dose. Addition of anti-stripping additives might be necessary in some cases.

- The reported results reflect the rejuvenator performance only at the tested 12% dose. Use of same rejuvenators with a different asphalt binder may change the performance due to compatibility.

References


