

# USE OF REJUVENATORS FOR PRODUCTION OF SUSTAINABLE HIGH CONTENT RAP HOT MIX ASPHALT

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**Abstract.** Aged binder is the main obstacle for increasing the use of Reclaimed Asphalt Pavement (RAP) in production of asphalt pavements. RAP binder is much stiffer and therefore high RAP content mixes are perceived to be susceptible to fatigue and thermal cracking failures. Appropriate choice of rejuvenator may allow reducing the binder viscosity to attain the desired mixture workability and recover the necessary performance properties of the aged binder. Six differently originated rejuvenators were added to extracted binder at 12% dose including aromatic extract, waste engine oil, tall oil, organic oil, waste vegetable oil and waste vegetable grease. The rejuvenation potential of these products was evaluated by performing multiple empirical as well as performance tests and the results compared with a virgin binder. The rejuvenators proved to soften the extracted binder to the level of virgin and the kinematic viscosity results show that the optimum compaction temperature can be reduced by 15°C to 25°C compared to extracted binder. Performance Grade (PG) test results showed that none of the rejuvenators reduced the high PG of extracted binder to the level of virgin binder indicating increased rutting resistance. At the same time most are able to reduce low PG from -12°C of the extracted binder to at least the required -22°C. The low temperature performance correlated well with penetration index, proving that thermal cracking should not be an issue at appropriate rejuvenator dose. The binder fatigue performance was evaluated through Linear Amplitude Sweep (LAS) test. Only waste vegetable products were able to increase the fatigue resistance to the level of virgin binder at a 12% dose using LAS test while the Superpave  $G^* \cdot \sin \delta$  indicated that all rejuvenators pass the minimum fatigue requirement.

**Keywords.** Rejuvenators, softening agents, waste vegetable oil, linear amplitude sweep test.

## INTRODUCTION

Reclaimed Asphalt Pavement (RAP) binder properties depend on the composition of the original binder and aging during service. If the aggregates are of good quality, then the main obstacle for increasing the RAP dosage, is the aged RAP binder. The stiff binder has low workability and may cause fatigue and thermal cracking.

A successful use of rejuvenators should reverse the RAP binder aging process, restore the properties of asphalt binder for another service period and make the RAP asphalt effectively “available” to the mix, and hence allow a significant increase in the amount of RAP that could be used in Hot Mix Asphalt (HMA). However, it is necessary to carefully select the rejuvenator to provide the necessary short and long term properties, as follows.

1. 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.
2. Long term. Rejuvenators should reconstitute chemical and physical properties of the aged binder and maintain stability for another service period. The binder rheology has to be altered to reduce fatigue and low temperature cracking potential without over softening the binder to cause rutting problems. Sufficient adhesion and cohesion have to be provided in the mix to prevent moisture damage and raveling.

## 1 RESEARCH APPROACH AND MATERIALS

### 1.1 Objective

The objective of the research is to evaluate the use of rejuvenators for restoring the desirable properties of the aged RAP binder in order to produce very high content RAP hot mix asphalt.

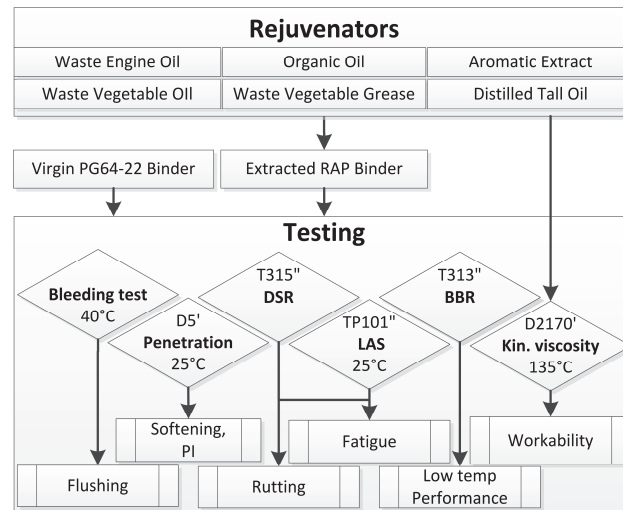


Fig 1. Research Test Plan

## 1.2 Test plan and Methods

The test methods used in this study for binder evaluation are summarized in Figure 1. Mixing of rejuvenators with the extracted binder demonstrates the effects of 100% blending; the penetration and viscosity tests were carried on unaged samples, but the blended samples for performance grading (PG) were aged according to standard procedure for virgin binders.

## 1.3 Binder

The binder was extracted from the RAP using toluene according to ASTM 2172, method A and recovered using rotary evaporator, according to ASTM D5404. Typically a PG 64-22 binder is used in this climatic area and therefore this grade was selected as a reference binder.

## 1.4 Rejuvenators

Six different rejuvenating agents are used in the study. The products have been labeled by generic descriptors that briefly define the origins of the products. Measured kinematic viscosity and the specific gravity along with the some basic characteristics obtained from manufacturers are included in Table 1. The rejuvenator dose selection (12% from binder mass) is based on the author's previous study (Zaumanis et al., 2013) as a compromise between all of the rejuvenators. The research results suggested that this is close to optimum dose for organic products, but lower than that required for petroleum products.

Table 1. Rejuvenator properties and description

Rejuvenator	Viscosity at 135°C	Specific gravity	Engineered or Generic	Petroleum or Organic	Refined or Waste	Molecular structure	Polarity	Traditional or Novel
Nustar PG 64-22	474	1.02	Generic	Petroleum	Refined	Ring and Strand	Mixed	Traditional
Aromatic Extract	9.20	0.995	Generic	Petroleum	Refined	Aromatic Ring	Very	Traditional
Waste Engine Oil	3.86	0.872	Generic	Petroleum	Waste	Aliphatic	Slight	Traditional
Distilled Tall Oil	5.60	0.950	Generic	Organic	Refined	Ring and Strand	Mild	Traditional
WV Grease	4.28	0.924	Generic	Organic	Waste	Ring and Strand	Mild	Novel
WV Oil	5.17	0.917	Generic	Organic	Waste	Ring and Strand	Non	Novel
Organic Oil	5.43	0.947	Engineered	Organic	Refined	Ring and Strand	Mild	Traditional

#### ***1.4.1 Aromatic extract***

An aromatic extract is a traditional rejuvenator with dominant polar aromatic rings. Recent findings show concern with unsaturated polar aromatic ring structure, which have been shown to be carcinogenic (The Petroleum HPV Testing Group, 2012). This research is not intended to promote the use of aromatic extract, but rather to allow comparing other products to a rejuvenator, that has been used historically and has demonstrated acceptable results. Aromatic extracts contain approximately 75% polar aromatic oils, resin compounds with balance less polar aromatics. Proponents claim polar aromatics associate with asphaltene molecules and in the process make binder less brittle, by balancing the chemistry of the oxidized aged binder.

#### ***1.4.2 Waste Engine Oil (WEO)***

WEO is produced from paraffinic oil with small dose of specialty compounds to improve viscosity characteristics, stability, cleaning and flammability for use in engines. WEO may contain short chain polar molecules that break apart during lubricating service. Recent interest in waste engine oil re-refineries around the world is making WEO increasingly difficult to obtain and more costly.

#### ***1.4.3 Refined tall oil***

Sylvatal™ D30E tall oil is produced by Arizona Chemical. It is a byproduct of paper manufacture being concentrated from kraft liquors. Tall oil is available either in crude form or as refined products. Crude tall oil contains fatty acids, resin acids and unsaponifiables in varying ratios depending on tree type used. Tall oils have a long history of use in hot mix manufacture with many emulsifiers, antistripping agents and warm mix additives.

#### ***1.4.4 Waste Vegetable Oil (WV Oil)***

WV Oil is increasingly used for bio-diesel production and has very strict compositional specifications including low free fatty acid content (<15%), less than 2% MIU (Moisture, Impurities, Unsaponifiables) (National Renderers Association, 2008). Derived from fast and convenience food frying oil, it is also referred 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.

#### ***1.4.5 Waste Vegetable Grease (WV Grease)***

WV Grease is also a food industry organic waste stream but semi solid at ambient temperatures due to predominance of saturated Lauric and Myristic triglycerides. The product used in study is also high in free fatty acids (>40%) but with its free glycerin and moisture removed industrially.

#### ***1.4.6 Organic oil***

Hydrogreen S™ is an engineered product by PVS Meridian Technologies, Inc and is designed to be binder rejuvenator and a low temperature additive. It is composed of fast pyrolysis of pine tree biomass (Harshavardhan et al., 2012) with other oils added to balance performance. The product is free flowing most of the year, but slight heating to around 10°C is necessary when used in cold weather.

## **2 PERFORMANCE GRADE**

The extracted RAP binder was tested for Performance Grade (PG) after addition of each of the rejuvenators and the results, along with the virgin binder PG 64-22, are illustrated in Figure 2. They show that RAP binder has aged significantly and grades as PG 94-12. The addition of all products allowed reducing both the high and low binder grade compared to the extracted binder suggesting not only decrease in viscosity but also increasing in elasticity. None of the products was quite able

to reduce the high grade to the level of virgin binder indicating lower susceptibility to rutting. This shows that, despite the general concern, if adequate dose of rejuvenator is used and good diffusion occurs in asphalt binder film, there is no danger of increased rutting susceptibility with the use of rejuvenators. Note that this assumes that there is 100% diffusion of the rejuvenators in the binder.

The required low performance grade (-22°C) was reached in all cases except when using WEO. Most of the products are actually able to reduce the PG much lower than required, and at the dose of 12%, organic oil and WV oil have provided the lowest cracking temperature.

Penetration Index (PI) was developed to describe the temperature susceptibility of binder. However, Burke et.al (Burke et al., 2011) in their research on field performance of binder that has been modified with re-refined waste engine oil residue suggested the use of PI of binder in respect to its expected thermal and fatigue cracking performance. Their research proposes PI as a good measure of steric hardening (asphaltene structure formulation over time) that promotes accelerated oxidative hardening and gel-type structure that retain higher stress levels at low temperatures. According to their research an increase in PI, despite sufficient low temperature performance grade, indicates low temperature cracking susceptibility.

PI was calculated from penetration results at temperatures of 10°C and 25°C according to Equations 1 and 2, developed by Pfeiffer and Van Doormaal (Whiteoak et al., 2003). Figure 3 shows good correlation of rejuvenated binder PG and PI, confirming that low temperature performance has been in fact improved. The virgin PG 64-22 likely has a different origin and therefore its PI is significantly different from rejuvenated binders.

$$PI = \frac{120 - 500 \cdot A}{1 + 50 \cdot A} \quad (1)$$

$$A = \frac{\text{Log}(\text{penetration at } T_1) - \text{Log}(\text{penetration at } T_2)}{T_1 - T_2} \quad (2)$$

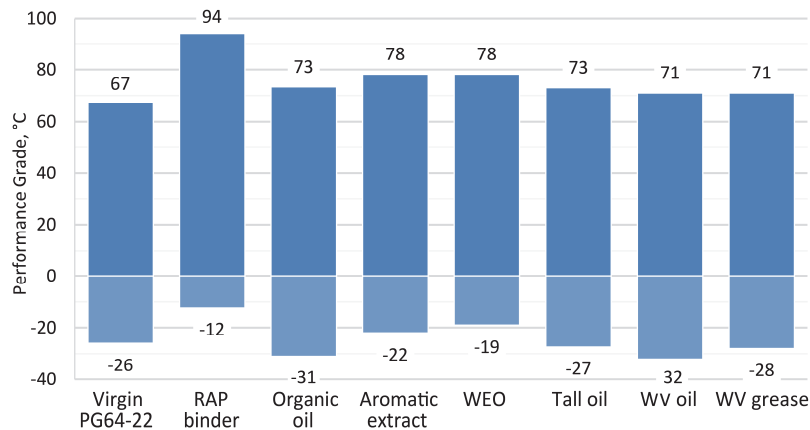


Fig. 2. True Performance Grade of Rejuvenated Binders

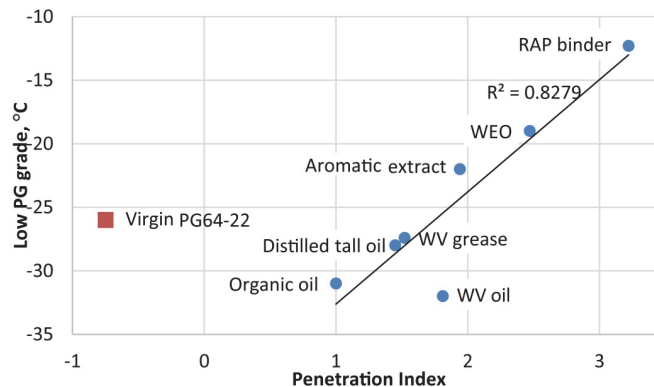


Fig. 3. Correlation of Low Temperature PG and Penetration Index

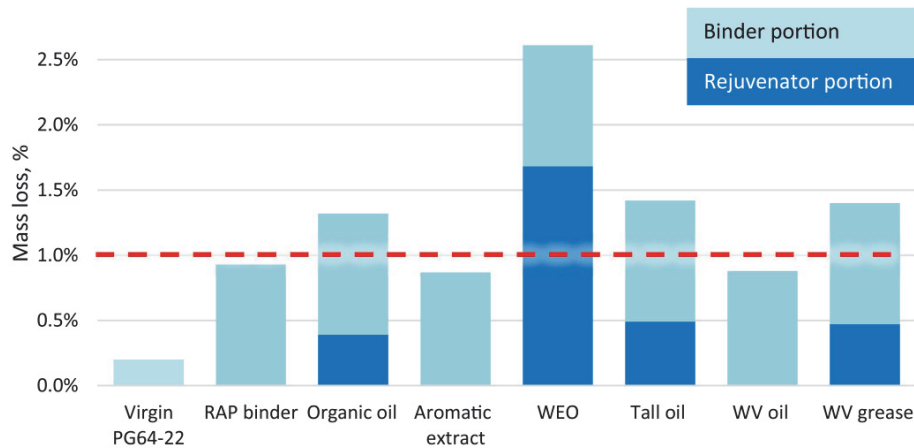


Fig. 4. Mass Loss After RTFO

## 2.1 Off-gassing

The mass loss results after aging the samples in RTFO, along with the Superpave requirement of less than 1%, are illustrated in Figure 4. The results show only small mass loss for virgin binder, but the extracted RAP binder exhibits significant reduction of volatile fractions. This may partly be a result of incomplete distillation of solvent during binder extraction. Most of rejuvenated binders exceed the allowable 1% mass loss, however, this is partly because of the high “initial” mass loss of RAP binder. Therefore, in order to demonstrate the “true” off gassing that is caused by the use of rejuvenators, the “initial” mass loss portion that is caused by RAP binder (0.93%) has been subtracted from total mass loss and indicated with lighter color. Consecutively the dark bar demonstrates the rejuvenator portion of mass loss. This shows that only WEO exceeds the allowable limits of off gassing, while other rejuvenators have less than 0.5% loss and WV Oil as well as aromatic extract doesn't cause any additional mass loss.

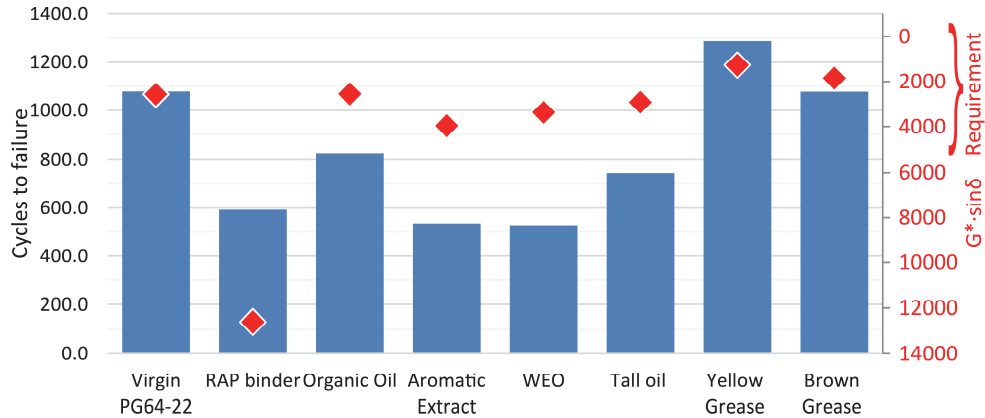
## 3 WORKABILITY

### 4 FATIGUE

The Linear Amplitude Sweep (LAS) (AASHTO TP-101-12) test has been proposed as a replacement to currently used PG grading intermediate temperature parameter  $G^* \cdot \sin \delta$ . The LAS procedure uses conventional DSR testing unit with 8mm plate and 2mm gap setting, but in contrast to the existing fatigue parameter ( $G^* \cdot \sin \delta$ ), the test is performed by means of cyclic loading employing, increasing load amplitude to characterize the non-linear strain response of binder.

The test is conducted in two steps. First, a frequency sweep is run from 0.1 to 30Hz at a strain level of 0.1% without damaging the sample. Second, a strain sweep test is performed at a constant frequency of 10Hz with linearly increasing strain from 0.1 to 30%. From the test results fatigue resistance is calculated using the viscoelastic continuum damage (VECD) approach. The results are expressed as cycles to failure, where the failure is defined as 35% reduction from undamaged  $G^* \cdot \sin \delta$ . The results can be calculated at any strain level, accounting for the differences in pavement structure or traffic loads (higher loads or thinner pavements will generate higher strain).

The test specimens were tested after short term (RTFOT) and long term (PAV) aging to simulate the pavement response at later stages of service. The test was performed in the Superpave intermediate temperature for PG64-22 (25°C). The results in Figure 5 are expressed at 5% strain which corresponds to 1000 microstrain in the pavement layer (Hintz et al., 2011) and was found to have good correlation with the mixture fatigue energy ratio test (Tran et al., 2012).



**Fig. 5.** Linear Amplitude Sweep Test Cycles to Failure at 5% Strain and  $G^* \cdot \sin \delta$

The results clearly demonstrate that the aged RAP binder has a reduced fatigue life compared to the virgin mixture. Addition of WV grease and WV oil has significantly increased the fatigue resistance and the number of cycles to failure is similar to the virgin mix. Tall oil and organic oil has slightly increased the fatigue life from RAP binder. However, surprisingly, WEO and aromatic extract were not shown to have any effect on the RAP binder. This result is unexpected considering that these products have been extensively used for rejuvenation.

The current Superpave intermediate temperature fatigue parameter  $G^* \cdot \sin \delta$  (complex shear modulus viscous portion) results are indicated with diamonds in Figure 5. For easy visual comparison with LAS test results they are plotted on a reverse scale and fitted in relation to virgin binder result. Results demonstrate that all rejuvenators have decreased the  $G^* \cdot \sin \delta$  from 12,600 kPa for extracted RAP binder to a level that passes the maximum Superpave requirement of 5,000 kPa and the results are similar to virgin binder.

#### 4.1 Compaction Temperature

Kinematic viscosity of rejuvenated binders was determined at 135°C and, along with the penetration results at 25°C, are summarized in Bitumen Test Data Chart (BTDC) that was developed by Heukelom (1969). BTDC was designed to determine the optimum mixing and compaction temperature range that corresponds to the viscosities that are generally recommended for good workability. The results in Figure 6 demonstrate that, by addition of rejuvenators, the production and compaction temperature can be reduced by around 20°C from that of the RAP binder, with WV Grease being the most effective. This reduces further aging of RAP and significantly decreases the greenhouse gasses from production (Zaumanis et al, 2012). None of the rejuvenators, however, have quite reduced the viscosity to the level of virgin PG 64-22 binder.

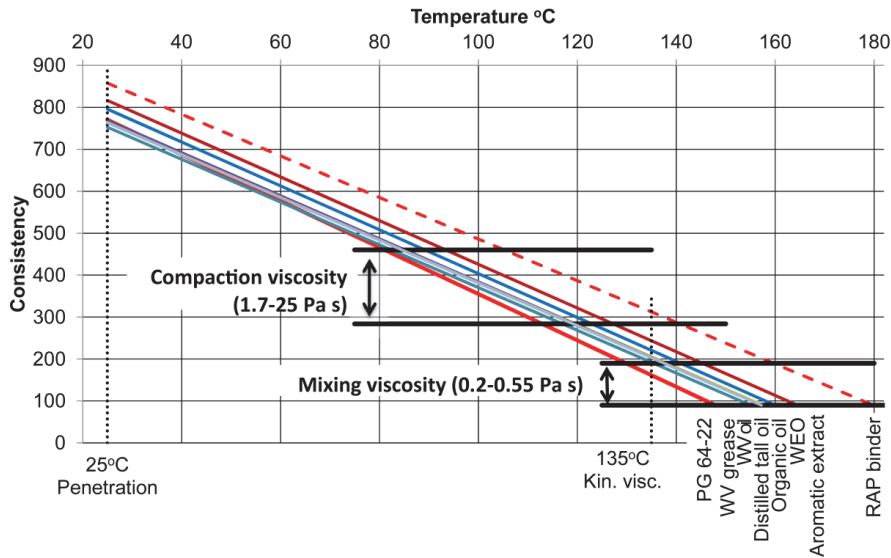


Fig. 6. Bitumen Test Data Chart

### 4.2 Calculation of viscosity

Refutas equation (3-5) (Maples, 2000) has been previously used to predict the viscosity of blends of petroleum products. It was used in this study to verify its suitability to calculate the viscosity rejuvenators and binder.

The results of measured versus calculated viscosity are presented in Figure 7 and show good correlation, meaning that the viscosity of the blend and hence compaction and production temperature can be predicted without the need to perform extraction and testing.

$$v_{blend} = \exp\left[\exp\left(\frac{VBI_{blend} - 10.975}{14.535}\right)\right] - 0.8 \quad (3)$$

$$VBI_{blend} = \sum_{i=1}^N f_i VBI_i \quad (4)$$

$$VBI_i = 14.354 \cdot \ln[\ln(v_i + 0.8)] + 10.975 \quad (5)$$

VBI – viscosity blending index of the components i,

v – kinematic viscosity, cSt (Table 1),

$f_i$  – mass fraction of component i (density in Table 1).

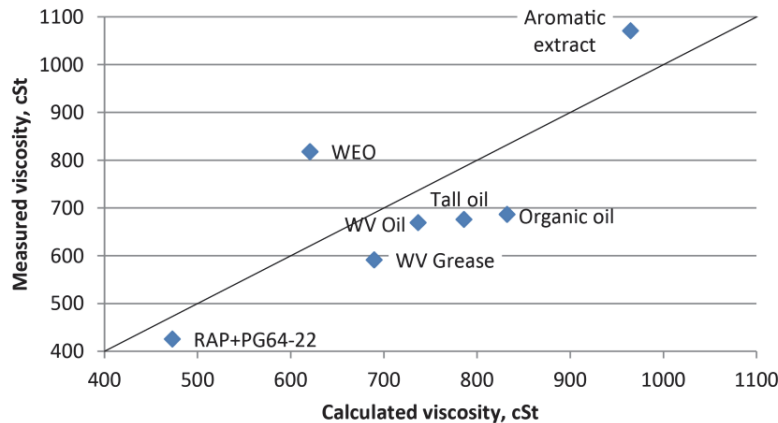


Fig. 7. Measured VS Calculated Viscosity of Rejuvenated Binder Blends

## 5 SOFTENING POTENTIAL

The penetration was determined at 25°C after addition of each rejuvenator at two different dosages. Figure 8 illustrates the changes in penetration based on the dose and shows that all of the rejuvenators can be used to soften the stiff extracted binder (penetration of 19 dmm) to the target level of virgin PG 64-33. WV Grease is the most effective, requiring only 8.2% of rejuvenator to reach 78 dmm, while 20.9% of aromatic extract must be added to have the same effect.

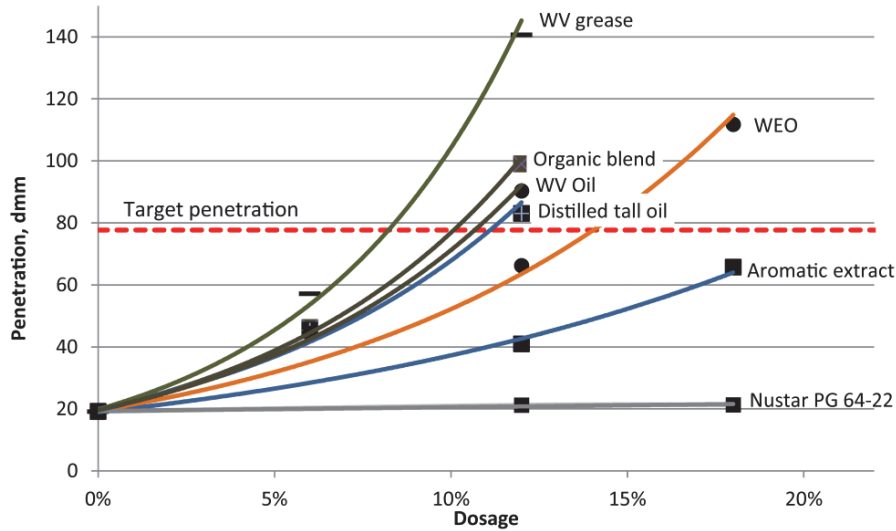


Fig. 8. Penetration at 25°C for Three Rejuvenator Dosages Fitted With Exponential Curve

An exponential relationship can be fitted extremely well on the data points with R values of more than 0.99. This means that the dosage to reach target penetration can be calculated on the basis of simple calculation (Equation 6) and requires only the penetration test result of extracted binder and rejuvenated sample at one rejuvenator dose.

$$\text{DOSE} = \frac{\log_e\left(\frac{PEN}{A}\right)}{B} \quad (6)$$

where

A – y-intercept of exponential function

B – base of exponential function

## 6 BLEEDING TEST

The compatibility of rejuvenators and binder needs to be verified in order to ensure a stable, safe and long-lasting mixture. In field studies with the use of incompatible products or inadequate dose or rejuvenators, a migration of oils toward the surface of the asphalt layer has been noticed, resulting in reduction of the friction of wearing course and compromised pavement performance. This has been described as instable rejuvenation resulting in bleeding or flushing (Karlsson et al., 2006; Kandhal, 1997). A hypothesis was framed that the lighter molecules of rejuvenators would tend to migrate towards the surface of the asphalt binder, and a simple test setup was developed for measuring the amount of oils that would result on the surface of the binder sample because of the influence of heat (Figure 9(a)). The test was executed as follows:

1. Extracted binder samples having PG 82-22 were mixed with two dosages of each rejuvenator and poured into aluminum foil containers at a thickness of about 35mm and allowed to cool.
2. A radiant heat lamp was placed above the samples at a distance to ensure 40±1°C in the binder sample. This was measured with a thermocouple inserted in a dummy sample.

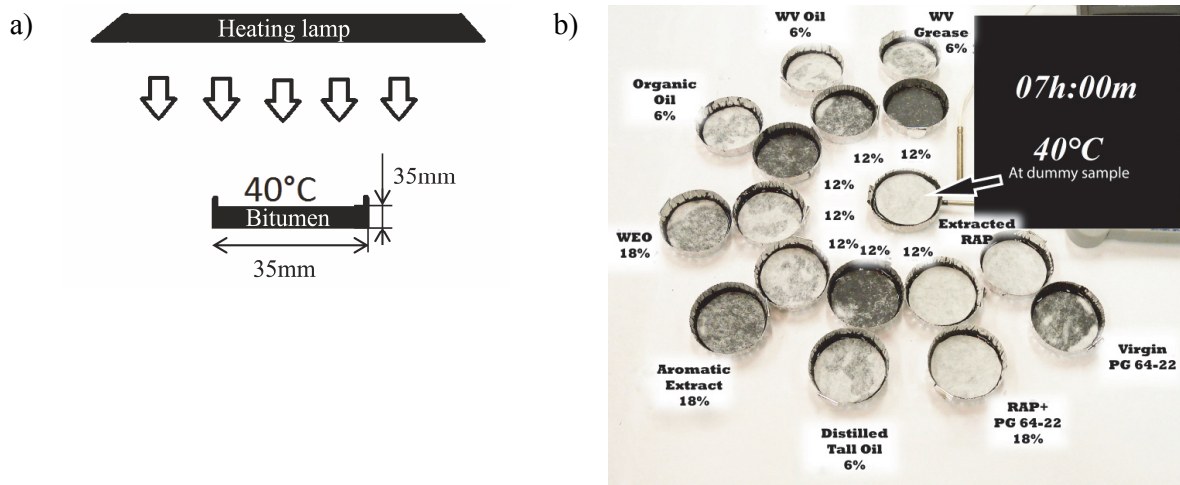


Fig. 9. Test setup (a) and an Image of Test Results After 7h (b)

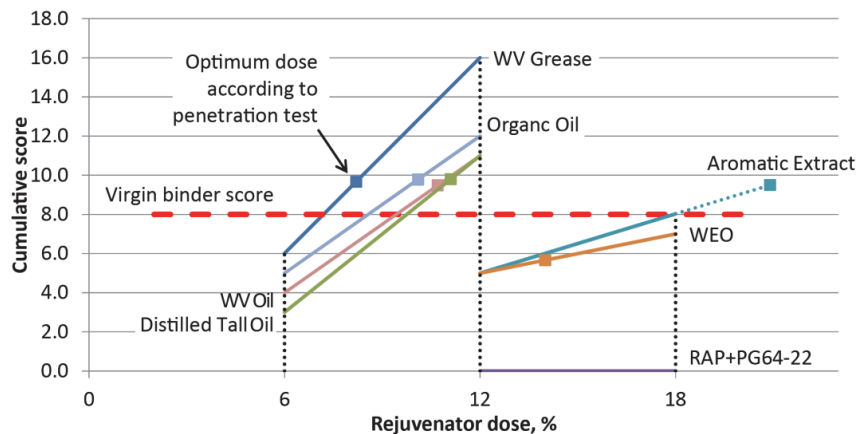


Fig. 10. Bleeding Test Results for Two Doses of Each Rejuvenator

- The samples were pre-heated for 5 minutes and covered with a filter paper that was applied to samples with constant pressure of  $0.6 \text{ g/cm}^2$  for 5 minutes at ambient temperature.
- The samples were placed back under the lamp and heated for 7 hours. A picture was taken each hour.
- The results were evaluated by visually assessing the darkness (wetness) of the filter paper at each hour on a scale from 0 to 3, where 0 – not wet and 3 – totally wet.

It is clear from Figure 9 (b) that RAP binder and the binders that are rejuvenated with virgin mixture do not exhibit any bleeding. This is likely because of the small amount of volatile fractions in the sample compared to unaged binder. The cumulative test results of two doses of each rejuvenator are illustrated in Figure 10 and the score of virgin binder is highlighted with a thick dotted line. The samples that are located above virgin binder score exhibit more bleeding, while the samples below the line did not wet the filter paper as much. As expected, a higher dose of a particular rejuvenator increases bleeding. If the results are evaluated according to the optimum dose from penetration results (Figure 8), it is clear that WEO will reduce the bleeding compared to virgin binder, while the other rejuvenators seem to have a little higher bleeding compared to virgin binder and therefore require more attention towards the diffusion and stability in long term.

## CONCLUSIONS

1. Rejuvenators are able to reduce the low temperature PG grade to level of virgin binder and PI can be used to predict the low temperature performance.
2. None of the rejuvenators are able to reduce the high temperature PG to the level of virgin binder indicating increased rutting resistance.
3. All of rejuvenators have provided fatigue parameter  $G^* \cdot \sin \delta$  results in the required Superpave range, but Linear Amplitude Sweep fatigue performance of the aged binder has been improved only by organic rejuvenators.
4. The penetration change after the use of rejuvenators can be predicted by simple exponential equation and the change in viscosity and mixing temperature can be accurately calculated by Refutas equation.
5. The bleeding test results suggest that it is necessary to evaluate the friction of the mix if pavement is to be used in pavement surface course.
6. At 12% dose the WV Grease and WV Oil showed the most rejuvenation potential, but increase in dosage for other products might allow to improve the performance.
7. Due to the fact that most rejuvenators are able to reduce the PG, when choosing rejuvenator, the focus should be on evaluation of fatigue.

## ACKNOWLEDGEMENTS

This work has been supported by Fulbright and Baltic-American Freedom Foundation sponsorship as well as European Social Fund within the project “Support for the implementation of doctoral studies”.

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