Low temperature properties of rejuvenated 100% reclaimed asphalt pavement mixtures

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ABSTRACT. *Nine differently originated softening agents were tested with 100% RAP mixtures to evaluate their effect on penetration at two temperatures and low temperature embrittlement. Penetration Index (PI) was evaluated as an indicator for rejuvenation effectiveness and to predict the cracking potential. Low temperature mixture performance was tested at -10°C through determination of indirect tensile strength and creep compliance. The mixture test results confirmed the PI results and showed that the use of four from the tested rejuvenators reduced the binder consistency to the necessary level and reduced the susceptibility of the recycled mixture to low temperature embrittlement.*

KEYWORDS: *high content RAP, softening agents, rejuvenator.*

1. Introduction and scope of work

Because of the presence of stiffer (aged) binders, high RAP content mixes are perceived to be more susceptible to fatigue and thermal cracking failures than mixes with virgin binders. In order to restore the necessary properties of the aged asphalt, rejuvenating additives can be used. The application has to be carefully studied to ensure uniform dispersion, improved cracking performance without increasing the susceptibility to rutting and ensuring structural stability.

In order to simplify the study and evaluate the effect of rejuvenators on the aged binder, this research focuses on possibility of restoring the properties of 100% RAP mixtures. Such mixtures have been produced previously (Mallick *et al.,* 2010) with promising results, proving that the concept is feasible and the key to success may lie in details.

1.1. *Objective*

The research was aimed to investigate the effect of using multiple rejuvenators for restoring the properties of an aged RAP asphalt binder and determining the low temperature performance of 100% RAP mixtures. This is also the first phase of a study to develop a simple method for ranking rejuvenators based on their performance.

1.2. *Research Outline*

Nine different rejuvenating agents are used in the study. The products have been labeled by generic descriptor that briefly describes the origin of the product and some or their basic characteristics are included in Table 1. The WEO plus Fischer-Tropsch (FT) wax is an outliner from these products since it can be used for the production of Warm Mix Asphalt (Zaumanis *et al.*, 2012); however evaluation of its properties as WMA additive is out of the scope of this research.

RAP that is used in the reported study has been reclaimed in the state of New Jersey, where typically a PG 64-22 binder is used. Therefore it was selected as a reference binder and used in mixture design, where necessary. The mixtures were tested for creep compliance and indirect tensile strength at -10°C for thermal cracking potential since these tests have been recognized as accurate ways of describing mixture properties at low temperatures (Christensen *et al.,*2004; Christensen, 1998).The use of Penetration Index (PI) has been reported as good indicator of oxidative hardening and cracking; therefore it is evaluated in this study (Burke *et al.,* 2011; Hesp *et al.,* 2010).

Table 1. *Rejuvenator characteristics*

Rejuvenator	Spec. gravity	Visc. 135°C, $mm^{2/s}$	Designed or Generic	Petroleum or Organic	Refined or Waste	Molec. structure	Polarity
Organic blend	0.947	5.4	Designed	Organic	Refined	Ring and Strand	Mild
Refined Tallow	0.891	3.0	Generic	Organic	Refined	Strand	Mild
Paraffinic base oil	0.867	2.7	Generic	Petroleum	Refined	Aliphatic	Non
Aromatic Extract	0.995	9.2	Generic	Petroleum	Refined	Aromatic Ring	Very
Napthenic flux oil	0.940	11.2	Designed	Petroleum	Refined	Ring and Strand	Mild
WEO+ FT wax	0.857	40.1	Designed	Petroleum	Refined	Aliphatic	Slight
WEO bottoms	0.917	300.1	Generic	Petroleum	Waste	Aliphatic	Slight
Waste engine oil	0.872	3.9	Generic	Petroleum	Waste	Aliphatic	Slight
Distilled Tall Oil	0.950	5.6	Generic	Organic	Refined	Ring and Strand	Mild

Recent findings show concern with unsaturated polar aromatic ring structure being carcinogenic (Petroleum HPV Testing Group, 20012). Therefore, most industries worldwide are moving away from polar aromatics oils and finding less polar substitutes. 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 historically used and has demonstrated acceptable results.

2. Asphalt binder testing

2.1 *Sample Preparation*

Binder that is used in the study was extracted from RAP using toluene as a solvent. Based on the literature review and industry experience on the effectiveness of the rejuvenators, one of the two different dosage rates was applied (18.26% or 9% from asphalt binder mass). A sample was prepared with each of the nine rejuvenators by thoroughly mixing the product with asphalt binder after 40 minutes of heating at 135°C. After that it was tested for penetration at temperatures of 4°C and 25°C according to ASTM D5-06.

2.2. ***Consistency***

The bitumen test results are summarized in the Table 2 and show that all products soften the reference unmodified RAP extracted binder which has very low penetration values (4,0 1/10mm at 4°C and 16,3 1/10mm at 25°C) and high viscosity. The virgin Nustar PG 64-22 has been included in the table for reference purposes to demonstrate the consistency results of a typical binder that is used in this climatic region. The consistency of the RAP rejuvenated binder varies significantly among the different products. Some of the rejuvenators have reduced the penetration at 25°C to a level that has been observer for virgin binders in this climatic region (around 80-90 1/10mm), while others have significantly smaller effect on the penetration.

2.3. ***Penetration Index (PI)***

Penetration Index (PI) describes the temperature susceptibility of asphalt and the PI ranges in this system vary from around -3 for highly temperature susceptible asphalt to +7 for highly blown or low-temperature susceptible (high PI) asphalt (Whiteoak *et al.,* 2003). Burke et.al in their research on WEO modified asphalt (Burke *et al.,* 2011) suggest use of PI for ranking asphalt in respect to its expected performance of thermal and fatigue cracking. Their research proposes the PI as a good measure of steric hardening (structure formulation) that promotes accelerated oxidative hardening and gel-type structure which retains higher stress levels in cool temperatures. When bitumen ages, its PI usually increases, indicating more structured and brittle material that is less able to flow and thus more prone to cracking (Burke *et al.,* 2011). A successful restoring of aged RAP binder properties should reverse this process and thus the decreasing of PI compared to the extracted asphalt may be a good indicator of the rejuvenation quality.

PI was calculated from penetration results at temperatures of 4°C and 25°C according to the formula in equation [1], developed by Pfeiffer and Van Doormaal *(9)*.

$$\mathrm{PI} = \frac{120 - 500 \cdot A}{1 + 50 \cdot A} \qquad [1]$$

$$\mathrm{A} = \frac{\mathrm{Log(penetration\ at\ T_1) - Log(penetration\ at\ T_2)}}{\mathrm{T_1 - T_2}}$$

The results of PI for the rejuvenated asphalts are summarized in Table 2. Most of the rejuvenators have decreased the PI, but WEO+FT wax and WEO bottoms have increased it, meaning they are more temperature sensitive compared to the extracted binder and possibly have higher potential of cracking. Aromatic extract and organic blend have decreased the PI considerably more than the other rejuvenators.

Table 2. *Bitumen Test Results*

Rejuvenator	Rej. dose, %	Pen. 4°C, 1/10mm	Pen 25°C, 1/10mm	Binder PI	Kin. visc. 135°C, mm^2/s
Extracted RAP binder	0	4,0	16,3	2.23	2054
Virgin PG 64-22	0	8.7	85.0	-1.06	474
Organic blend	9	9,3	54,0	0.66	831
Refined Tallow	9	17,0	83,7	1.33	612
Paraffinic base oil	18	20,3	91,3	1.74	379
Aromatic Extract	18	14,3	95,0	0.14	406
Napthenic flux oil	18	11,3	51,3	1.70	699
WEO+FT wax	18	8,3	28,0	3.29	1006
WEO bottoms	18	10,0	32,3	3.56	2054
Waste engine oil	18	20,3	87,7	1.94	457
Distilled Tall Oil	9	10,0	46,3	1.61	893

2.4. Calculation of viscosity

Refutas equation [2-4] (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 predict the viscosity rejuvenators and extracted binder.

$$\nu_{blend} = \exp[\exp\left(\frac{VBI_{blend} - 10.975}{14.535}\right)] - 0.8 \quad [2]$$

$$VBI_{blend} = \sum_{i=1}^{N} f_i VBI_i \quad [3]$$

$$VBI_i = 14.354 \cdot \ln[\ln(\nu_i + 0.8)] + 10.975 \quad [4]$$

VBI – viscosity blending index of the components i,
ν – kinematic viscosity, cSt (Tables 1 and 2),
f_i– mass fraction of component i (density in Table 1).

The results of measured versus calculated viscosity are presented in Figure 1 and show high correlation, meaning that the viscosity of the blend can be predicted with high precision, without the need to perform extensive amount of extraction and testing. This can allow screening the rejuvenators that can not soften the RAP binder enough to produce workable mixture. The results also demonstrate an interesting trend: saturated aliphatic petroleum products have aligned to the left of 45° line while larger MW and more polar products are located to the right of this line. This may demonstrate compatibility of the two oils but has to be verified in further studies.

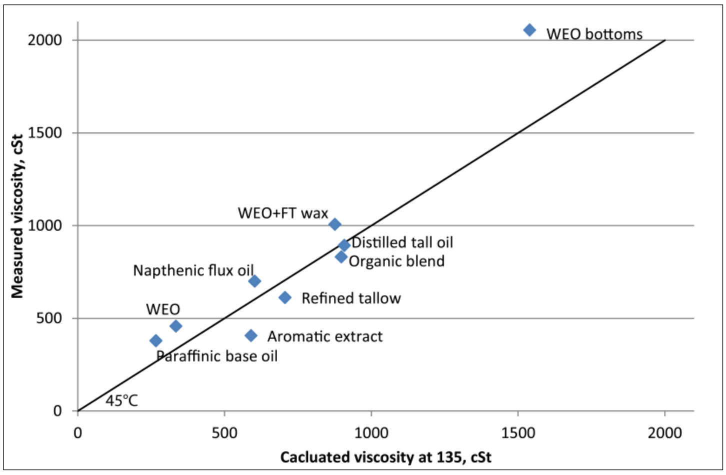

Figure 1. *Measured VS Calculated viscosity of rejuvenated binder blends*

3. Mixture testing

Ten different sets of 100% RAP mixture samples were prepared, including reference and nine rejuvenated mixes. The RAP material had a binder content of 5,1% and aggregate gradation that corresponded to Superpave 9,5mm design. No modifications were made to the gradation. The mixtures were tested for low temperature properties at -10°C using IDT creep compliance test for 1000 seconds according to AASHTO T322 and indirect tensile strength test according to ASTM D6931 on 100 mm diameter samples. They were compacted using gyratory compactor to 7%±0,5% air voids after two hour aging in forced draft oven and the density for creep compliance samples was measured after saw-cutting the core.

3.1. *Rejuvenator Dosage*

The target binder penetration at 25°C of the rejuvenated samples was defined as 90 1/10mm which is close to the Nustar PG 64-22 penetration. The dosage of all the rejuvenators was calculated to reach this penetration. Some additional penetration measurements for organic blend and refined tallow were performed to determine precise dose and the relationship is defined according to supposition of exponential increase (Zaumanis *et al.*, 2013) as illustrated in Figure 2. The required rejuvenator amount to attain the required penetration for the WEO, aromatic extract and paraffinic base oil was close to the dosage used in the asphalt binder study; therefore the verified amount (18,26% of asphalt binder mass) was chosen for the preparation of the respective asphalt mixture samples.

The products that cannot ensure the required penetration, without exceeding reasonable dosage range, were added at a rate of 18,26% in order to reach equal film thickness with the rest of samples. For the mixtures were lower dose was required to reach the target penetration, the difference between the binder contents was compensated by addition of virgin Nustar PG 64-22 asphalt. It should not affect the resulting penetration since the penetration values of rejuvenated and fresh binders are very close. Such normalization of binder content to 6,03% (5,1% in the RAP + 0,93% virgin materials) allows to directly compare the performance in testing of different agents without introducing new variables (e.g. virgin aggregates to reduce binder content). In fact the relatively high final binder content allows highlighting the rejuvenator performance. The rejuvenator and virgin binder dosages that were used for the mixture study are summarized in Table 3.

Figure 2 clearly shows that refined tallow is the most effective at reducing consistency of the aged asphalt, but by using napthenic flux oil, WEO+FT wax or Waste Engine Oil (WEO) bottoms it is not possible to reach the penetration level of virgin binder within a reasonable dosage rate. Over dosage of rejuvenator will lead to adhesion and stripping problems.

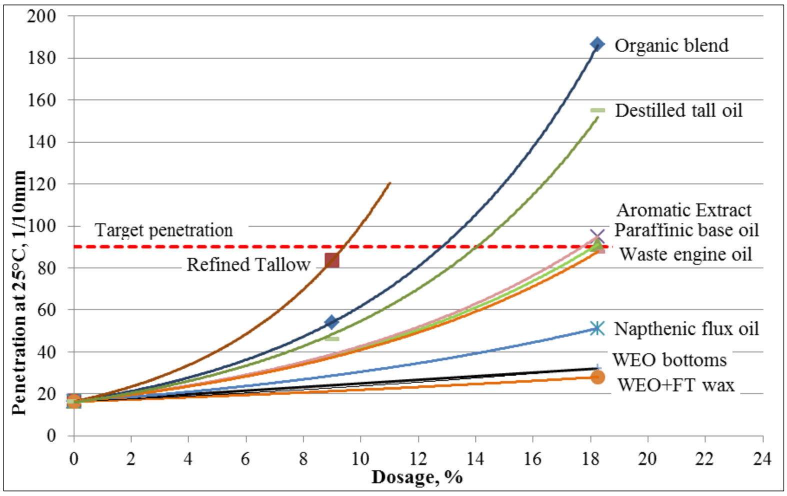

Figure 2. *Required Dosage to Reach Target Penetration at 25°C.*

The price per liter of each of the rejuvenator and the virgin asphalt (used as rejuvenator for reference sample) in the US are included in Table 3. The rejuvenator expanse per ton of asphalt, accounting for the calculated dosage and rejuvenator density, is also presented in Table 3. It is clear that the price of engineered products is higher, while the waste products are the cheapest option per ton of mix even accounting for the fact that they have to be applied at a higher dose.

Table 3. *Rejuvenator and Virgin Binder Dose in Mixture Samples and prices in US*

Sample ID	Rejuvenator dosage, % from asphalt mass	Virgin asphalt dosage, % from asphalt mass	Penetration at 25°C, 1/10 mm	Rejuvenator price/liter, USD	Rejuvenator price per ton of asphalt, USD
Reference	-	18,26	-	0,72	6,60
Organic blend	11,52	6,74	90,0*	2,38	14,78
Refined tallow	9,68	8,58	90,0*	1,34	7,45
Paraffinic base oil	18,26	-	91,3	0,97	10,40
Aromatic extract	18,26	-	95,0	1,26	13,50
Napthenic flux oil	18,26	-	51,3	1,00	9,94
WEO+FT wax	18,26	-	28,0	-	-
WEO bottoms	18,26	-	32,3	0,58	5,87
Waste engine oil	18,26	-	87,7	0,66	7,06
Distilled tall oil	12,71	5,55	90,0*	1,59	10,83

NOTE. – *calculated based on test results

3.2. *Test results*

The creep compliance results are presented in Figure 3 and tensile strength with fracture energy in Figure 4. The reader should keep in mind that the reference mixture has 18,26% of PG 64-22 virgin binder added to it, in order to reach equal asphalt content compared with the rejuvenated mixtures. Therefore, it can also be attributed as having improved low temperature performance compared to the milled RAP material.

The creep compliance, which is a way of characterizing the stiffness of material, in most cases has been increased after addition of rejuvenating agent. The most effective in reducing the low temperature stiffness is the paraffinic base oil.

Tensile strength and fracture energy were obtained from IDT strength test. Four rejuvenators have increased the strength while others have decreased it. However, while tensile strength is often used as parameter for evaluation of cracking resistance, it is not a fundamental parameter since it depends on loading conditions and loading mode. It is believed that Fracture energy is more important property for characterizing the cracking potential. It is defined as the energy required to initiate fracture of the mixture and it is not dependent of loading rate *(*Roque *et al., 2009)*. It was derived from the stress tests by calculating the area below the stress-strain curve up to the point of maximum stress. The results in Figure 4 show that all products except WEO+FT wax have the same or higher fracture energy compared to the reference mixture, therefore probably reducing cracking potential.

The products that compared to the reference mixture have maintained or increased the creep compliance, without reducing the fracture energy, can be considered to have reduced the embrittlement of the mixture.

WEO+FT wax didn't demonstrate the expected performance. It is produced as asphalt rejuvenator and WMA additive in one. Lowering of production temperature, as intended by the developers of this additive, may highlight the benefits of using this additive; however such evaluation is out of the scope of this paper.

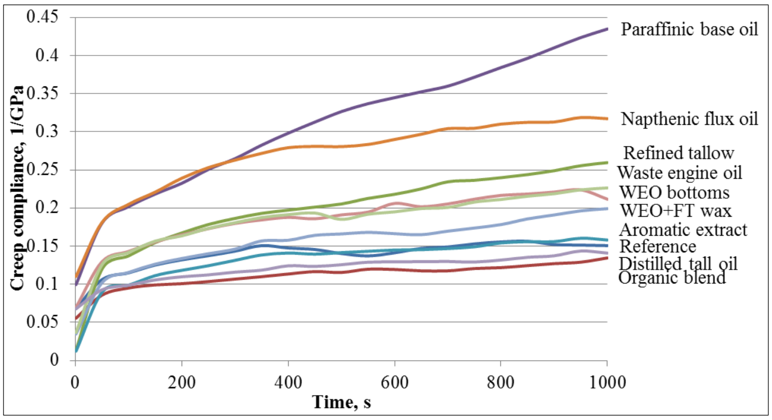

Figure 3. *IDT Creep Compliance.*

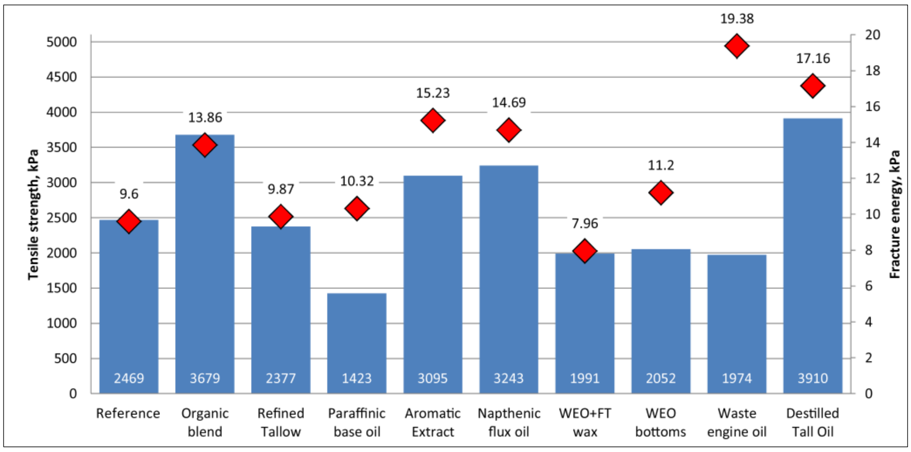

Figure 4. *Tensile Strength.*

The creep compliance results correlate reasonably well with calculated consistency at -10°C (Figure 5). Binder consistency in this graph is extrapolated based on linear regression from the two measured penetration values and expressed according to the Heukelom consistency calculation from BTDC (Heukelom, 1969). The graph shows reasonably good correlation, especially by keeping in mind that the mixture samples unlike binder samples in some cases have virgin binder added to it (see Table 3). This complies with the theory that at negative temperature mixture can be attributed as linear viscoelastic material (Christensen et al., 2004).

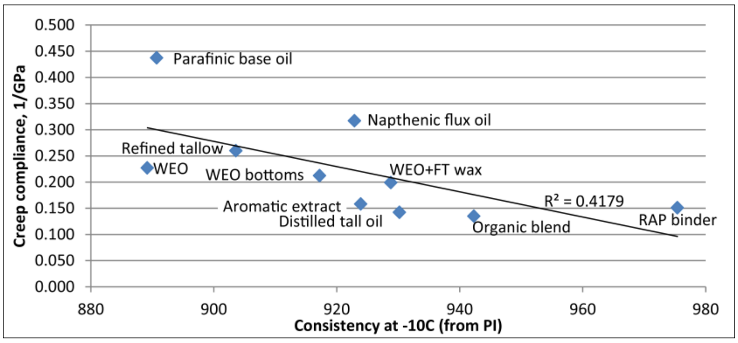

Figure 5. *Correlation between consistency at -10°C and creep compliance*

4. Summary

The test results are summarized in Table 4. They are evaluated based on previous discussions to ensure that addition of rejuvenator has improved low temperature performance:

– Rejuvenator softening effectiveness to reach penetration of a virgin binder (90 1/10mm) at a reasonable dosage rate (<20% from binder mass) is required,

– PI of max 2,23 is required to reduce the cracking potential of the RAP extracted binder,

– Creep compliance of more than 0,0151 1/GPa and fracture energy of more than 9.6 kPa are arbitrary requirements to make sure that the use of rejuvenating agent is more effective in improving low temperature performance than simply increasing the AC content.

The summary of the results in Table 4 shows that seven rejuvenators - organic blend, refined tallow, paraffinic base oil, aromatic extract, WEO and distilled tall oil, have fulfilled the arbitrary requirements at this stage of the research.

Table 4. *Summary of the Relevant Results for Rejuvenator Acceptance*

Sample ID	Dose to reach virgin binder penetration		PI		Creep compliance 1/GPa		Tensile strength, kPa	Fracture energy		Result
Required:	<20%		< 2,23		> 0,151			>9.6		
Reference	-	-	2,23	-	0,151	-	2469	9.6	-	-
Organic blend	11,5	pass	0,64	pass	0,135*	pass	3679	13.9	pass	**pass**
Refined Tallow	9,5	pass	1,33	pass	0,260	pass	2377	9.9	pass	**pass**
Paraffinic base oil	18,3	pass	1,74	pass	0,437	pass	1423	10.3	pass	**pass**
Aromatic Extract	18,3	pass	0,14	pass	0,158	pass	3095	15.3	pass	**pass**
Napthenic flux oil	>20	fail	1,70	pass	0,317	pass	3243	14.7	pass	**fail**
WEO+FT wax	>20	fail	3,29	fail	0,199	pass	1991	8.0	fail	**fail**
WEO bottoms	>20	fail	3,56	fail	0,212	pass	2052	12.2	pass	**fail**
Waste engine oil	18,3	pass	1,94	pass	0,227	pass	1974	19.4	pass	**pass**
Distilled Tall Oil	12,7	pass	1,61	pass	0,142*	pass	3910	17.2	pass	**pass**

NOTE. – *accepted based on reasonable error range of the requirement

5. Conclusions

The following conclusions can be drawn from the accomplished testing:

1) Penetration test is an easy and fast method for evaluation of softening potential of rejuvenators and PI results may be used to predict cracking performance.

2) Refutas equation can be used to predict the kinematic viscosity of rejuvenator and binder blends.

3) The addition of seven rejuvenators was found to be effective in improving low-temperature performance of the mixtures. In the order of cost-effectiveness these rejuvenators are waste engine oil, refined tallow, paraffinic base oil, distilled tall oil, aromatic extract, and organic blend.

5.1. *Future work*

The study is aimed in developing a simple method for evaluation of rejuvenator effectiveness and developing a balance mix design method for accepting rejuvenated asphalt. Future research plan includes testing of rejuvenated asphalt binders according to the PG specification requirements and using mixture performance tests to characterize the fatigue and rutting performance

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