EVALUATION OF REJUVENATOR'S EFFECTIVENESS WITH CONVENTIONAL MIX TESTING FOR 100% RAP MIXTURES

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ABSTRACT

This paper presents research evaluating effectiveness of rejuvenators for production of very high (40-100%) Reclaimed Asphalt pavement (RAP) content mixtures. Nine differently originated softening agents were tested, including plant oils, waste derived oils, engineered products, as well as traditional and non-traditional refinery base oils. Two different dosages of the agents were added to binder extracted from RAP to evaluate their softening potential through testing of kinematic viscosity and penetration at two different temperatures. At 25°C the softening efficiency varied by a factor of twelve between the most and least effective rejuvenators. Consistency results at different temperatures were used to express temperature susceptibility by means of Penetration Index (PI), Penetration-Viscosity Number (PVN) and Bitumen Test Data Chart (BTDC) of the softened binders. The PI results varied measurably depending on the rejuvenator and supported the low temperature mixture test results, showing that PI may be a good and simple measure of rejuvenation effectiveness. Low temperature mixture embrittlement was evaluated at -10°C through determination of the indirect tensile strength and creep compliance for rejuvenated 100% RAP mixture samples. It can be concluded that four of the nine tested rejuvenators reduced extracted binder consistency to the necessary level and reduced susceptibility of RAP mixtures to low temperature embrittlement. Of the four, two engineered products tested had notably different performance but neither was superior to similar generic oils.

INTRODUCTION AND SCOPE OF WORK

The substantial economic and environmental benefits of using Reclaimed Asphalt Pavement (RAP) material warrants implementable research on effectiveness and productivity of utilizing rejuvenators for producing high RAP content mixes. Currently high content RAP mixtures are defined as those having more that 25% RAP, but are usually not exceeding the maximum allowed RAP content by state agencies, which typically is not more than 40%. This research, however, focuses on possibility of restoring the properties of aged binders in order to produce up to 100% RAP mixtures, with the help of existing plants. Such mixtures have been produced previously *(1, 2)* with promising results, proving that the concept is feasible and the key to success may lie in details.

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. However, to successfully soften the oxidized binder and reach uniform dispersion within the asphalt to ensure required performance properties and longevity of the mixture, an appropriate dosage of a compatible rejuvenator has to be introduced. The application has to be carefully studied to improve the cracking performance without increasing the susceptibility to rutting and ensuring structural stability.

Objective

The goal of the study is to develop a simple method for evaluation and ranking of rejuvenators for high content RAP mixtures. The first phase of the research is reported in this paper and was aimed to investigate the effect of using different rejuvenators for restoring the properties of an aged RAP asphalt binder and determining the low temperature performance properties of 100% RAP mixtures.

Research Outline

Nine different rejuvenating agents are used in the study. Some of them are designed specifically for this purpose, others are versatile materials that are used in different industries and the rest are waste materials of other products that have been used for rejuvenating aged asphalt. The products have been labeled by generic descriptor that briefly describes the origin of the product. The Sasol Storbit Plus has Fischer-Tropsch wax as a compound for lowering the production temperature. The product was included in the research as recommended by asphalt producers; however since the components of this additive are significantly different from others included in this study, it is labeled with the trade name.

Several researchers *(3, 4, 5)* have indicated the weaknesses of the Superpave asphalt specification, the main ones of which are insufficient aging that does not correspond to the actual field oxidation and the insufficient conditioning in BBR. These shortcomings in some cases may lead to inadequate evaluation of the effective asphalt service temperature range and hence cause distresses, most often in the form of fatigue and thermal cracking. These types of distresses are the largest concern for high RAP content pavements, and therefore, the application of current asphalt test methods and/or conditions must be questioned to ensure long-lasting high RAP content pavement. The use of Penetration Index (PI) and Penetration-Viscosity number (PVN) have been reported as good indicators of oxidative hardening and cracking and therefore are evaluated in this study *(3, 6)*.

In conventional HMA production process the RAP is mixed together with virgin aggregates and fresh asphalt/rejuvenator. It is expected that during the short mixing time the aged asphalt attains sufficient viscosity to mix together with virgin asphalt and all the aggregates receive a homogeneous film thickness. At the same time sufficient diffusion of the rejuvenator is required in the aged asphalt to restore its properties to the required level. The diffusion is a function of temperature, viscosity, mixing, transportation and storage time, and it may continue in the pavement during the service period. Based on these considerations and according to the research goal of evaluating the rejuvenator effect on the binder and mixture properties, in order to avoid introduction of new variables, 100% RAP mixtures were tested. 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 nowadays. Therefore a Nustar PG 64-22 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 *(7, 8)*.

ASPHALT BINDER TESTING

Sample Preparation

Binder that is used in the study was extracted from RAP using toluene as a solvent. Based on the expected performance and industry experience on the effectiveness of the rejuvenators, two different dosage rates were applied (18.26% or 9% from asphalt 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. The sample was tested for penetration at temperatures of 4°C and 25°C according to ASTM D5-06 and later used for determination of kinematic viscosity at 135°C according to ASTM D2872.

Test Results

The test results are summarized in Table 1 and show that all products soften the reference unmodified RAP extracted binder which has a very low penetration value (4.0 1/10mm at 4°C, 16.3 1/10mm at 25°C) and very high kinematic viscosity (2054 mm^2/s). 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 viscosity at 25°C to a level that has been observed for virgin binders in this climatic region (around 80-90 1/10mm), while others have significantly smaller effect on the change in penetration. The column "softening effectiveness" represents the rate at which the penetration at 25°C changes per one percent of rejuvenator at the given dosage rate (equation 1). It shows that refined tallow is the most effective at reducing viscosity of the aged asphalt while the Storbit Plus is the least effective. This finding is important both in respect to the performance and the expenses of the production. The column also clearly shows that by using napthenic flux oil, Storbit Plus or Waste Engin Oil (WEO) bottoms it is not possible to reach the penetration level of virgin binder within a reasonable dosage rate. Overdosage of rejuvenator will lead to adhesion and stripping problems.

$$\text{Softening efficiency} = \frac{\text{Pen. rejuvenated binder, } \frac{1}{10\text{mm}} - \text{ Pen. RAP binder, } \frac{1}{10\text{mm}}}{\text{rejuvenator dosage, \%}} \quad (1)$$

Most of the rejuvenators have significantly decreased kinematic viscosity of the extracted binder and it has reached a level that is comparable with that of the virgin binder, which has a viscosity of 474 mm^2/s. The kinematic viscosity is closely linked with the required mixing and compaction temperature. Approximately 0.2 to 0.5 Pa·s is the recommended viscosity range for mixing to ensure sufficient coating of the aggregates and 1.7 to 25 Pa·s is the suggested range to guarantee adequate workability for paving *(9)*. WEO bottoms show significantly higher viscosity compared to other products meaning that mixing and compaction temperature would have to be increased by approximately 22°C compared to the virgin binder. Low viscosity at mixing temperature is even more important in a conventional recipe that consists of both virgin and RAP materials. It is necessary to ensure enough binder flow to guarantee similar film thickness of virgin and RAP aggregates and uniform blending of fresh and RAP binder. Temperature/viscosity dependence is critically important factor for ensuring this and since using very high temperature will damage binder, it is recommended to choose a rejuvenator that can ensure sufficient reduction of the high temperature viscosity.

TABLE 1 Bitumen Test Results

Rejuvenator	**Rejuvenator dosage, %**	**Penetration @ 4°C, 1/10mm**	**Penetration @ 25°C, 1/10mm**	**Softening efficiency @25°C, Δpen/dose%**	**Kincematic viscosity @135°C, mm^2/s**
Extracted RAP binder	0	4.0	16.3	na	2054
Virgin Nustar PG 64-22	0	8.7	85.0	na	474
Organic blend	9	9.3	54.0	6.3*	831
Refined Tallow	9	17.0	83.7	7.5	612
Paraffinic base oil	18	20.3	91.3	4.2	379
Aromatic Extract	18	14.3	95.0	4.4	406
Napthenic flux oil	18	11.3	51.3	1.9	699
Sasol Storbit Plus	18	8.3	28.0	0.6	1006
Waste engine oil bottoms	18	10.0	32.3	0.9	2054
Waste engine oil	18	20.3	87.7	4.0	457
Distilled Tall Oil	9	10.0	46.3	5.8*	893

*interpolated penetration value at 90 1/10mm from two penetration measurements was used for calculation
na = not appliciable

Penetration Index (PI)

Penetration Index (PI) describes the temperature susceptibility of an 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 *(9)*. S.Hesp et.al in their research on WEO modified asphalt *(3, 6)* suggest use of PI for ranking asphalt in respect to its expected performance of thermal and fatigue cracking. Their research proposes the PI and PVN as good measures 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 *(3)*. 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.

The most common method for calculating PI is Pfeiffer and Van Doormaal formula which requires determination of penetration at 25°C and softening point (equation 2). It is based on the hypothesis that at the temperature of softening point, the penetration is 800mm *(9)*. However, while this is true for most "normal" temperature susceptibility bitumen, modified binders, oxidized binders and binders from waxy crude oils may deliver erroneous PI due to a spread of actual penetration at softening point temperature and therefore require a different approach for the calculation of PI.

$$\mathrm{PI} = \frac{1952 - 500 \cdot \mathrm{Log}\, penetration - 20 \cdot softening\ point}{50 \cdot \mathrm{Log}\, penetration - softening\ point - 120} \tag{2}$$

Carrying out penetration test at two temperatures gives more consistent results for PI calculation. Therefore, in this research PI was calculated from penetration results at temperatures of 4°C and 25°C according to the formula in equations 3 and 4, developed by Pfeiffer and Van Doormaal *(9)*.

$$\mathrm{PI} = \frac{120 - 500 \cdot A}{1 + 50 \cdot A} \tag{3}$$

$$\mathrm{A} = \frac{\mathrm{Log(penetration\ at\ T_1) - Log(penetration\ at\ T_2)}}{\mathrm{T_1 - T_2}} \tag{4}$$

The results of PI for the rejuvenated asphalts are summarized in Figure 1. Most of the rejuvenators have decreased the PI, but Storbit Plus 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, but they still have significantly higher value compared to the reference virgin PG 64-22 binder with a PI of -1.06.

A drawback of this method is that the determination of penetration at low temperature (in this case 4°C) may have relatively large statistical deviation since the actual measured values are small; therefore small inaccuracies in measurement can significantly influence the calculated PI. For this reason the use of Penetration-Viscosity Number (PVN) as an alternative was also evaluated.

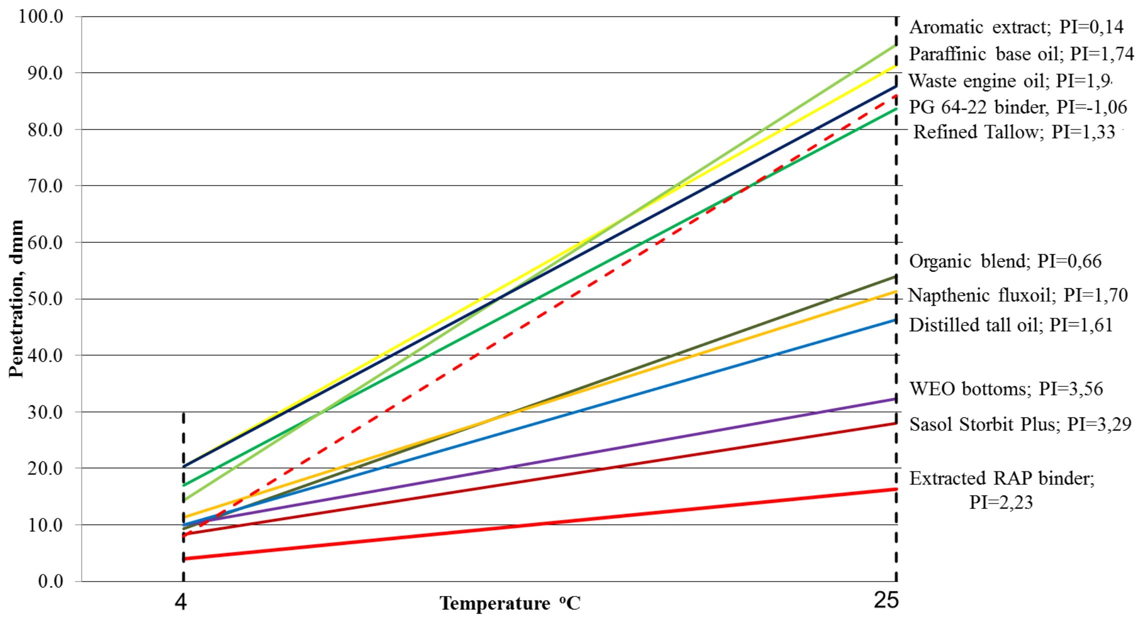

FIGURE 1 Penetration Values at 4°C and 25°C and Penetration Index (PI) of Rejuvenated Asphalt.

Penetration Viscosity Number

Penetration-Viscosity number (PVN) was developed by N.W.McLeod as an alternative to the PI *(10)*. PVN is calculated from the kinematic viscosity at 135°C and penetration at 25°C according to equations 5 to 7 *(10)*. High PVN indicates low thermal susceptibility while low PVN indicates high thermal susceptibility.

$$PVN = \frac{\log L - \log X}{\log L - \log M} \cdot (-1.5), \quad (5)$$

$$\log L = 4.2580 - 0.79674 \cdot \text{LogP} \quad (6)$$

$$\log M = 3.46289 - 0.61094 \cdot \text{LogP} \quad (7)$$

where
X – kinematic viscosity at 135°C, cSt;
L – viscosity at 135°C for a PVN of 0.0, cSt;
M – viscosity at 135°C for a PVN of -1.5, cSt;
P – penetration at 25°C, 1/10 mm;

All of the rejuvenators have decreased the PVN of the RAP extracted binder with exception of WEO bottoms (Figure 2). Five of the rejuvenated asphalts have PVN's that are in the same range or lower than that for Nustar virgin binder.

The calculated PVN results show significantly different numerical values compared to the PI and the results do not correlate with each other. In general the results are of smaller range and much lower than for PI. There are two reasons for this. Firstly, the PVN was intended to characterize unmodified binders and calibrated to have the same numerical values as the PI within the PI range of -1.5 to 0.0 *(10)*. Secondly, the actual measured consistency at the three measured temperatures does not form a linear relationship as, according to Heukelom's Bitumen Test Data Chart (BTDC) *(11)*, it would be for normal paving grade (S class) bitumen with limited wax content *(9)*. An example of the results, plotted in BTDC is shown in Figure 3. The available data allows assuming that the tested binders correspond to the B class (from Blown) *(9)*, with exception of Storbit Plus which has wax in it. The association with blown binders is reasonable, since the RAP binder has been oxidized during the service period. The non-linear temperature susceptibility in BTDC shows that the PVN for RAP binders may be beneficial for characterization of medium to high temperature properties but, since there is no linear correlation with low temperature viscosity, it cannot be used for characterization of low temperature properties and cracking.

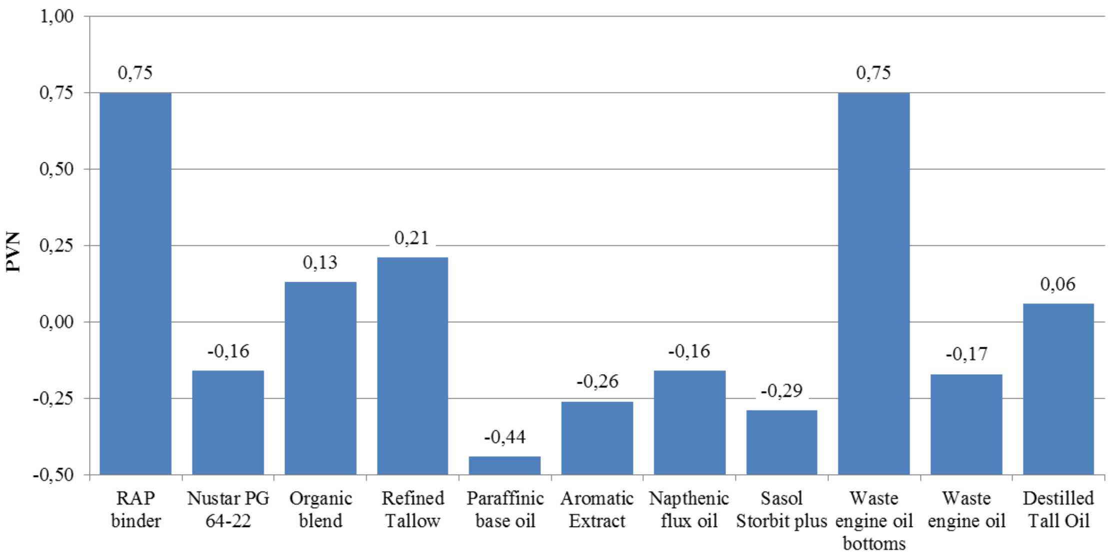

FIGURE 2 Penetration Viscosity Number (PVN).

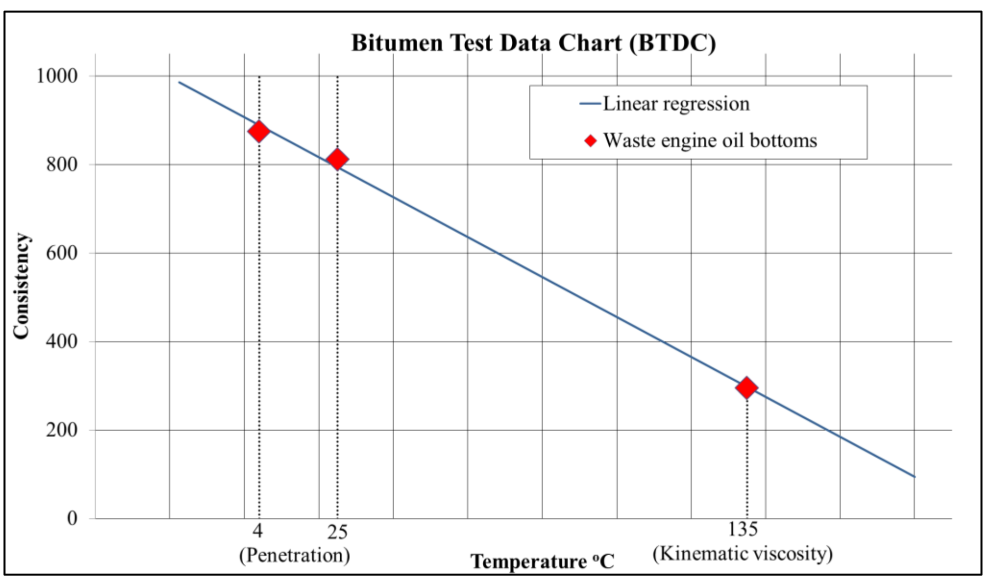

FIGURE 3 Bitumen Test Data Chart (BTDC) Example for WEO Bottoms.

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 Superpave 9.5mm aggregate gradation with high dust content (10%). No modifications were made to the gradation. The samples were mixed with the respective dosage of rejuvenator after 2 hour heating at 150°C and aged for 2 hours before compaction at the same temperature. 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 procedure A. Indirect tensile strength was calculated from the maximum load of IDT strength test without horizontal deformation measurements. The samples were compacted to 7%±0.5% air voids and the measurement for creep compliance samples was performed after cutting the core to the necessary height.

Rejuvenator Dosage.

The asphalt mixture samples were prepared based on the following considerations:

1) The penetration at 25°C for the rejuvenated samples must be similar, if possible without exceeding reasonable rejuvenator content;

2) All samples must have equal binder content and hence, film thickness.

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 was calculated after some additional penetration measurements and defined according to supposition as illustrated in Figure 4.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% (from binder mass) in order to reach equal film thickness with the rest of samples. For the mixtures for which lower dosage rate of rejuvenator was required to reach the target penetration, the difference between the binder contents was compensated by the 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) may not be the optimum procedure according to the mixture design principles. However, for the purposes of directly comparing the different rejuvenator performance, this method was found to be most suitable since it allows comparing 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 2.

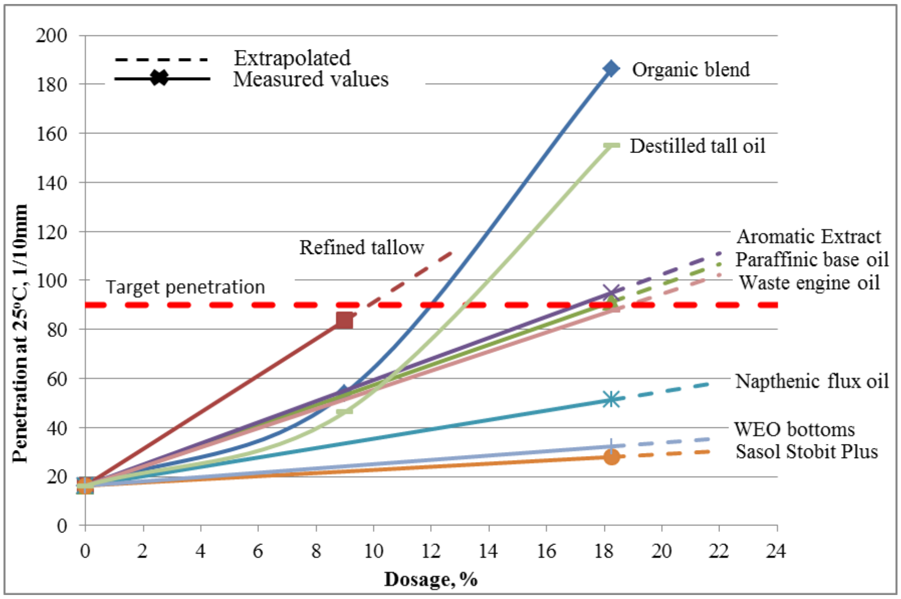

FIGURE 4 Required Dosage to Reach Target Penetration at 25°C.

TABLE 2 Rejuvenator and Virgin Binder Dosage in Mixture Samples

Sample ID	Rejuvenator dosage, % from asphalt mass	Virgin asphalt dosage, % from asphalt mass	Penetration at 25°C, 1/10 mm
Reference	na	18.26	NA
Organic blend	11.52	6.74	90.0*
Refined tallow	9.68	8.58	90.0*
Paraffinic base oil	18.26	na	91.3
Aromatic extract	18.26	na	95.0
Napthenic flux oil	18.26	na	51.3
Storbit Plus	18.26	na	28.0
Waste engine oil bottoms	18.26	na	32.3
Waste engine oil	18.26	na	87.7
Distilled tall oil	12.71	5.55	90.0*

*calculated based on test results
na = not appliciable
NA = not available

Test results

The mixture test results are presented in figures 5 through 7. 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 somewhat improved low temperature performance compared to the milled RAP material.

Creep compliance is a way of characterizing the stiffness of material. The less stiff the pavement is at low temperature, the lower the possibility of cracks. The results in Figure 5 show that in most cases creep compliance has been increased after addition of rejuvenating agent, therefore reducing cracking potential. The most effective is the paraffinic base oil; however, it is also reflected in the IDT strength (Figure 6) where this sample shows much lower result than the other mixtures. Reduction of the rejuvenator content may resolve this problem but doing so would not allow reaching the required penetration of 90 1/10mm.

Fracture energy is defined as the energy required to initiate fracture of the mixture and it is not dependent of loading rate *(12)*. Therefore, it can be used as an indicator of crack resistance. The results in Figure 7 show that all products except Storbit Plus have the same or higher fracture energy compared to the reference mixture, therefore reducing cracking potential.

The products that (compared to the reference mixture) have maintained or increased the creep compliance, without reducing the tensile strength and fracture energy, can be considered to have reduced the embrittlement of the mixture. Five rejuvenators comply with this provision: organic blend, refined tallow, aromatic extract, napthenic flux oil and distilled tall oil. This finding complies with the PI study where these five exact rejuvenators have showed five most significant reductions in the PI (see figure 1).

Sasol Storbit Plus 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.

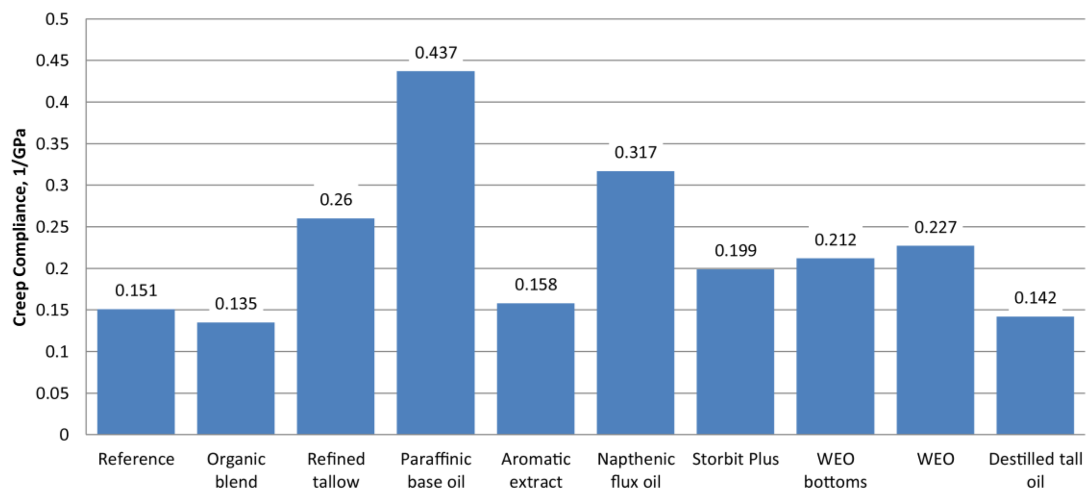

FIGURE 5 IDT Creep Compliance.

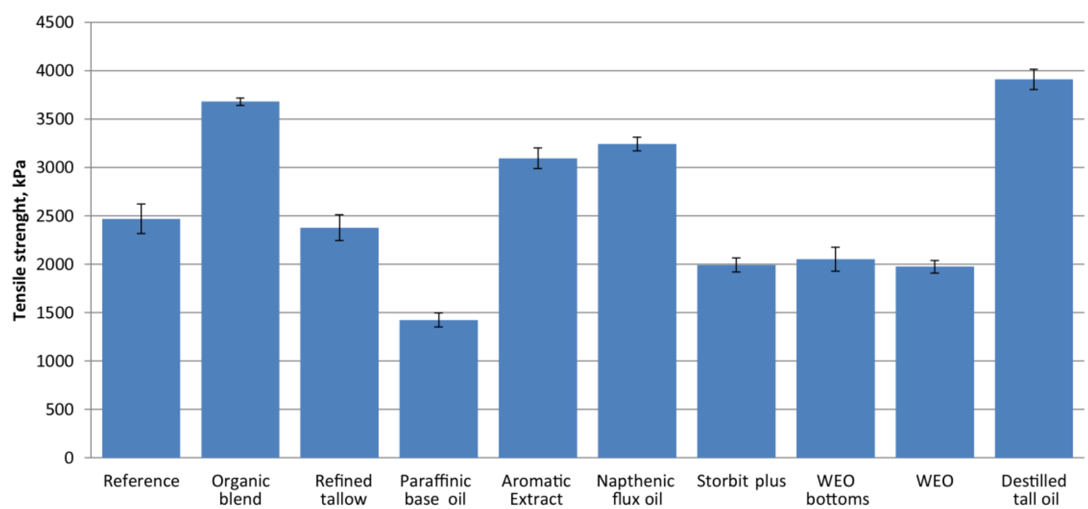

FIGURE 6 Tensile Strength.

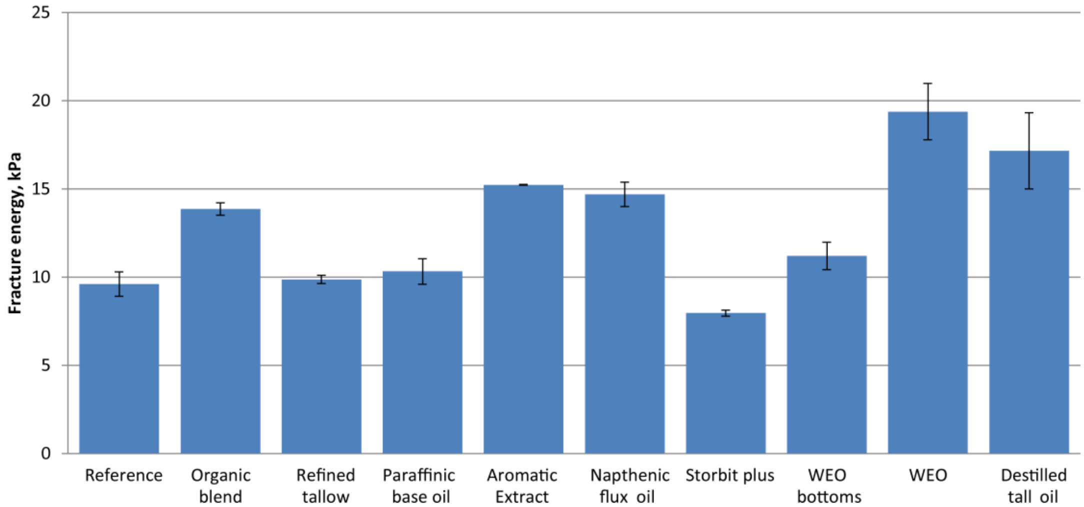

FIGURE 7 Fracture Energy.

SUMMARY

The testing data has shown that there are definite differences between the performances of the various rejuvenators that are included in the study. The results, that from the discussions above are considered relevant for evaluation of the rejuvenators in this part of the research, are summarized in Table 3. They are evaluated based on arbitrary requirement of achieving the required binder consistency and being more effective than simply increasing the AC content as done with the reference mixture:

● The softening effectiveness of 4.0 is required to reach the target binder penetration at 25°C of 90 1/10mm which corresponds to virgin binder. The selection criteria should also depend on mix properties, but this criterion is offered as a starting point in the process of selection of an appropriate rejuvenator.

● 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, tensile strength of more than 2469 kPa, 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 minimum values have been set within a reasonable error range and in some cases little lower values have been accepted;

The other parameters that were determined in the testing and were discussed before will be used in later stages of the research.

The summary of the results in Table 3 shows that four rejuvenators - organic blend, refined tallow, aromatic extract and distilled tall oil, have fulfilled the arbitrary requirements at this stage of the research. This, however, does not mean that the other products will be excluded from further study since rejuvenator content optimization in the next stages of the research may show a better way of characterizing the products and evaluating the rejuvenated RAP.

TABLE 3 Summary of the Relevant Results for Rejuvenator Acceptance

Sample ID	Soft. effic. @25°C, Δpen/%		PI		Creep compliance 1/GPa		Tensile strength, kPa		Fracture Energy, kPa		**Result**
Requirement:	**> 4.0**		**< 2.23**		**> 0.151**		**> 2469**		**> 9.6**		
Reference	na	na	2.23	na	0.151	na	2469	na	9.6	na	na
Organic blend	6.3	pass	0.64	pass	0.135*	pass	3679	pass	13.9	pass	**pass**
Refined Tallow	7.5	pass	1.33	pass	0.260	pass	2377*	pass	9.9	pass	**pass**
Paraffinic base oil	4.1	pass	1.74	pass	0.437	pass	1423	fail	10.3	pass	**fail**
Aromatic Extract	4.3	pass	0.14	pass	0.158	pass	3095	pass	15.3	pass	**pass**
Napthenic flux oil	1.9	fail	1.70	pass	0.317	pass	3243	pass	14.7	pass	**fail**
Storbit plus	0.6	fail	3.29	fail	0.199	pass	1991	fail	8.0	fail	**fail**
WEO bottoms	0.9	fail	3.56	fail	0.212	pass	2052	fail	12.2	pass	**fail**
Waste engine oil	4.0	pass	1.94	pass	0.227	pass	1974	fail	19.4	pass	**fail**
Distilled Tall Oil	5.8	pass	1.61	pass	0.142*	pass	3910	pass	17.2	pass	**pass**

na = not applicable

*accepted based on reasonable error range of the requirement

Research plans

The study is aimed in developing a simple method for evaluation of rejuvenator effectiveness and developing a procedure/method for accepting the rejuvenated asphalt. The future research plan includes testing of different asphalt binders according to the PG specification requirements and using mixture performance tests to characterize the fatigue and rutting performance. The low temperature properties will be also investigated through the application of thermal cracking models from test results at different temperatures. Finally mix design optimization will be performed to design a mixture with the required properties.

CONCLUSIONS

The following conclusions can be drawn from the study reported in the paper:

1) The penetration test is an easy and fast method for the evaluation of softening properties of the rejuvenators. Measuring penetration at two temperatures allows determining PI. This may be a good measure for predicting the low temperature mixture performance since the test ranked the rejuvenators in the same order as the mixture test results at -10°C.

2) PVN, because of the original calculation formula that was developed for characterizing binders of much lower PI than used in this study and the non-linear viscosity relation in the BTDC, does not give dependable results for asphalt binder characterization in the low temperature range.

3) The addition of five rejuvenators was found to be effective in maintaining or increasing the low-temperature creep compliance and at the same time increasing the IDT tensile strength and fracture energy, therefore improving low-temperature performance of the mixtures. Four of these rejuvenators also reduced the penetration of the RAP binder to the required level of virgin mixture. They are organic blend, refined tallow, aromatic extract and distilled tall oil.

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