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A systematic study of bituminous binders extended with a renewable material

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Abstract

Road construction and maintenance are progressively moving towards more sustainable solutions, for example, by an increased use of various materials from renewable resources. In this paper, a plant-based oil (PBO) from the forest and paper industry was systematically studied as potential renewable bitumen extender. First, laboratory analysis was conducted on a selected oil. Then, different bituminous binders were prepared containing PBO and studied extensively in terms of quality and performance. It was found that the PBO studied was fully miscible with the bitumen, and by properly selecting a base bitumen and a dosage of the oil, desired standard binder grades were obtained. The extended binders showed an improved resistance to long-term aging as assessed by the pressure aging vessel (PAV), and an improved adhesion with stone aggregates as evaluated by the rolling bottle test and the indirect tensile strength ratio (ITSR). Regarding other performance properties, such as asphalt stiffness, fatigue and permanent deformation, no significant differences were observed between extended and reference bitumens. Two full-scale field trials were also carried out, and HSE observations and pavement performance follow up is presented. In addition, further experimental aspects on recyclability of this type of materials will be addressed.

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1. INTRODUCTION

Nowadays, reduction of the carbon footprint and the use of sustainable solutions are becoming more important in road construction and maintenance. Over the years, much work has been done to enhance pavement durability, to reduce energy consumption using different innovative techniques, to increase the use of recycled materials, and to explore available renewable materials and alternatives in bitumen and asphalts, with aims of minimizing the environmental impact of asphalt paving and contributing to sustainable development. A biogenic material derived from plants, not subjected to depletion, could be a renewable component for bitumen [1-10]. However, it remains rather difficult to quantify the benefit of using such a material in terms of carbon footprint because of potential uncertainties on material source management and lack of a well-developed or commonly accepted methodology.

There are many publications on studying different applications of plant-based oils [3-10], e.g. as bitumen modifier, extender and emulsifier, or as a warm mix additive and rejuvenator. In spite of great research efforts, renewable materials generally have not been widely used in bituminous binders and asphalt pavements, especially when it comes to bitumen extender or as partial replacement of bitumen. There could be several reasons, for example, the long-term performance of plant-based materials has not been demonstrated or, knowledge on their HSE aspects may be insufficient. Also, recyclability of such materials needs to be investigated, as asphalt today is 100% recyclable, and adding any other materials to asphalts should not negatively affect this aspect. Moreover, availability of the materials, as well as their cost-effectiveness, could be a concern.

In this paper, a plant-based oil (PBO) from the forest and paper industry has been systematically studied as a potential renewable bitumen extender. First, laboratory analysis was conducted on plant-based oil samples from different supply sources. Then, various bituminous binders were prepared containing the oil and studied extensively in terms of quality and performance. Full scale trials were also carried out and pavement performance has been followed up.

2. ANALYSIS OF PLANT-BASED OILS

Plant-based oil (PBO) samples from three different manufactures (coded S1, S2 and S3) were analysed by various physical property tests, including viscosity (EN 12595), flash point (ASTM D 6450), and density (ASTM D 1475). Chemical analyses were also carried out; those included acid number, elemental composition, simulated distillation (SIMDIS), Fourier Transform Infrared Spectroscopy (FTIR), and generic fractions or SARA (saturates, aromatics, resins, asphaltenes) analysis by thin-layer chromatography with flame ionization detection (TLC-FID, known as Iatroscan).

Table 1 shows typical properties and elemental composition of PBO. Large differences can be seen in viscosity between the PBO samples. It is known that the oil from the forest and paper industry is a complex mixture composed of numerous chemicals, the most common of which are rosin acids, fatty acids and neutral compounds. This is reflected by a high acid number found for the material.

Table 1. Properties and elemental composition of the PBO investigated

Parameter	S1	S2	S3
Kinematic viscosity 60°C, mm^2/s	674	2235	712
Flash point, °C	247	247	> 220
Density, g/cm^3	0.984 (at 50°C)	1.005 (at 15°C)	0.995 (at 50°C)
Acid number, mg KOH/g	37.7	89.1	--
Carbon, %	80.7	80.1	80.5
Hydrogen, %	11.2	11.3	11.3
Nitrogen, %	< 0.5	< 0.5	< 0.5
Oxygen, %	7.6	7.8	7.6
Sulfur, %	0.38	0.34	0.31
H/C ratio	1.66	1.69	1.67

Regarding the elemental composition, there are no significant differences between these oil samples. Compared to typical bitumen which consists of about 80 to 85% carbon, 10% hydrogen, 0.5% nitrogen, 0.5% oxygen, and 3 - 5% sulfur [11]. The PBOs contain a similar level of carbon and slightly higher hydrogen, resulting in higher H/C ratios than that for bitumen (about 1.2). More importantly, the PBO samples contain much higher oxygen content, mostly in the form of acids and esters, as already shown by high acid numbers, as well as FTIR analysis on the functional groups.

The FTIR spectra of the PBOs used are quite similar, and typical examples are shown in Figure 1. Identified functional groups are: methylene chains with four or more carbons at about 716 cm^{-1}, C-O at about 1160 cm^{-1}, C-H in methylene and methyl groups at 1450 and 1370 cm^{-1}, C=C (aromaticity) at 1600 cm^{-1}, C=O in carboxylic acids at 1700 cm^{-1}, C=O in esters at 1730 cm^{-1}, and CH_2 and $-CH_3$ at 2920 and 2850 cm^{-1}. As expected, esters and carboxylic acids are rich in the tested PBOs.

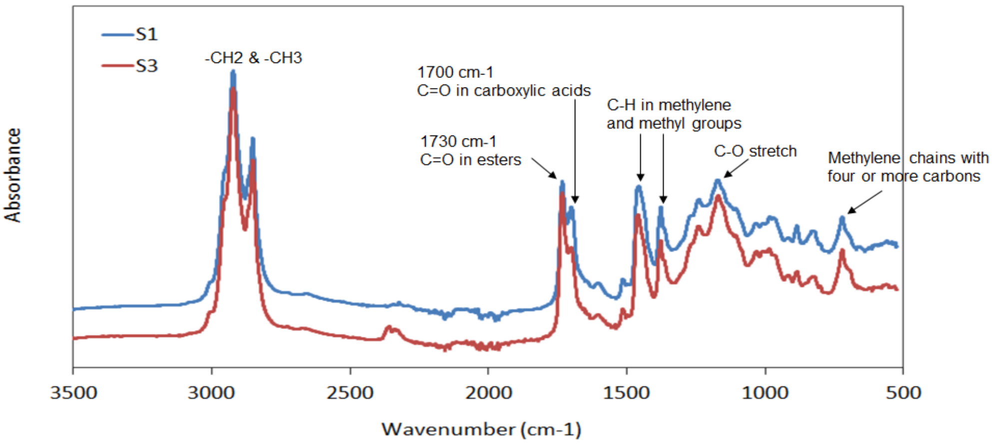

Figure 1: Typical FTIR spectrum of PBOs (S1 and S3)

SIMDIS (ASTM D 6352) shows that, S1, S2 and S3 have IBP (initial boiling point) of 384, 373 and 361°C, respectively, and at the boiling point of around 402°C (corresponds to normal paraffin C25), the recovered mass is 5 to 7 %. Considering their amount (< 30%) in bitumen, this should suggest no negative impact from an emission point of view. From SIMDIS, a high temperature gas chromatography (HTGC) chromatogram is exemplified in Figure 2. In this figure, possible normal paraffins corresponding to different peaks are indicated. However, it seems unlikely that those normal paraffins are present as a significant type of molecules in the PBO. According to a previous study [12], by using n-heptane as a development solvent in the Iatroscan SARA analysis, n-alkanes ranging from C_{20} to C_{40} are measured in a fraction of saturate, while no saturates are determined for the PBO, as illustrated in Figure 3. Note that SARA here are determined according to IP 469, where the fraction polars (II) is called asphaltenes; differences may occur if other standards (e.g. ASTM D 4124, IP 143) are used.

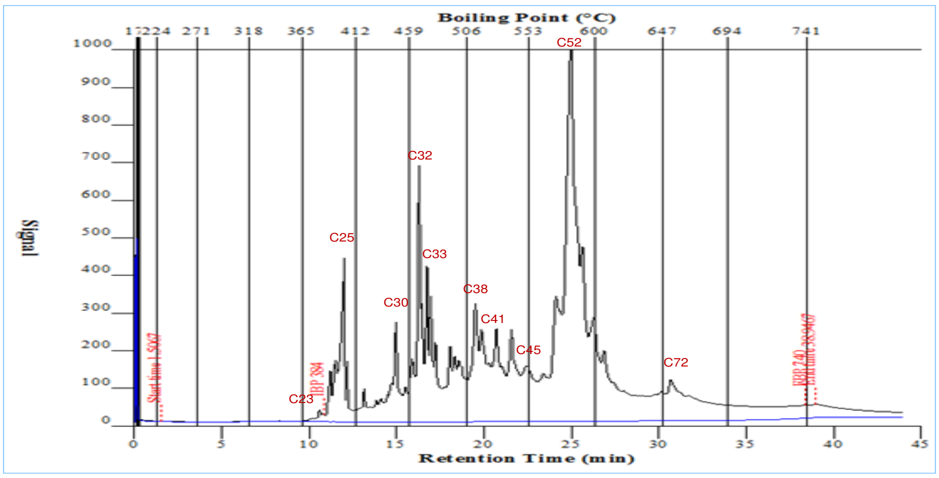

Figure 2: HTGC chromatogram of PBO (S1)

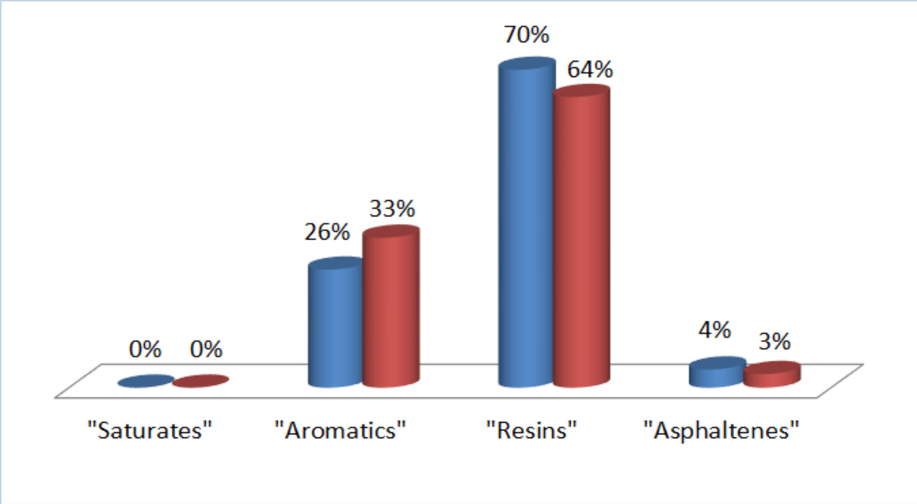

Figure 3: Iatroscan SARA analysis of PBOs (blue bars for S1, and red bars for S3)

3. ANALYSIS OF BITUMENS EXTENDED WITH PLANT-BASED OILS

In sample preparation, different base bitumens and different dosages (5 - 30%) of PBO were selected to target penetration grades of 70/100 and 160/220. To determine blending ratios, small amounts (< 100 g) of samples were prepared by manually homogenizing a hot bitumen (150°C) and PBO (100°C) for about 1 minute. For full binder analysis and asphalt tests, bitumen and PBO blends were prepared at 150°C using a laboratory low shear mixer with a speed of 500 rpm and a mixing time of about 10 min. The base bitumens used were normal paving grade bitumens complying to EN 12591. Blending proportions were made such that appropriate 70/100 and 160/220 grades according to EN 12591 were obtained (Cf. Table 2).

Binder analysis was performed according to EN 12591. Table 2 shows test results for several PBO and bitumen blends, one targeted to 70/100 (coded EB 80), and other four targeted to 160/220 (coded EB 200, EB 190, EB 180, and EB 170). As can be seen, all these blends fulfil the CEN specification.

The PBO used is also believed to be compatible with bitumen. To confirm this, two bitumen blends containing more than 10% PBO (by weight) were investigated by a storage stability test or phase separation test (EN 13399). The tests were performed at 155°C with the same procedure as for polymer modified bitumen. Test results in Table 3 indicate that no phase separation occurs during the hot storage.

The PBO extended bitumens are further investigated with regards to long-term aging by PAV (pressure aging vessel). PAV test was conducted at the standardized conditions (i.e. 100°C and 20 h) and on samples after RTFOT. The PAV aged samples were evaluated by a dynamic shear rheometer (DSR). Complex moduli at 20°C and 10 rad/s were used to calculate long-term aging index, i.e. modulus ratio between PAV aged and RTFOT aged samples. As can be seen from Figure 4, PBO has no detrimental effect on bitumen long-term aging. On contrary, it somehow improves the aging resistance of the bitumen.

Long-term durability of the PBO extended bitumen is also shown by ΔTc, a binder durability parameter defined as LST (temperature at 300 MPa stiffness) minus LmT (temperature at 0.300 m-value) [13], which in this study are determined from a bending beam rheometer (BBR). A more positive value of ΔTc means a higher durability [14]. As an example, for EB 180 in Table 1, after RTFOT and PAV, ΔTc was found to be +1.9°C, which is much more positive than many normal 160/220 penetration bitumen that generally have negative ΔTc.

Table 2. Analysis of PBO extended bitumen according to EN 12591

Parameter	Extended 70/100	EN 12591 70/100		Extended 160/220				EN 12591 160/220	
	EB 80	*Min*	*Max*	EB 200	EB 190	EB 180	EB 170	*Min*	*Max*
Penetration 25°C, 1/10 mm	75	70	100	198	190	182	167	160	220
Softening point	46.4	43	51	35.8	36.4	37.8	38.4	35	43
Dyn.viscosity 60°C, Pas	213.2	90		61.1	62.5	74.7	51.3	30	
Kin.Viscosity 135°C, mm^2/s	377.9	230		226	242	236	199	135	
Fraass breaking point, °C	-13		-10	-22	-23	-18	-23		-15
Flash point COC, °C	292	230			294	266	310	220	
Solubility in toluene, %	99.95	99.0		99.5	99.96	99.95	99.95	99.0	
After RTFOT 163°C									
Change of mass, %	-0.02		0.8	-0.08	-0.24	-0.15	-0.12		1.0
Penetration 25°C, 1/10 mm	53			125	113	118	79		
Softening point, °C	50.4			40.6	41.8	42.4	45.8		
Incr. in softening point, °C	4		9	4.8	5.4	4.6	7.4		11
Retained penetration, %	71	46		63	60	65	47	37	

Table 3. Results of storage stability tests

Tube test at 155°C, 72 h	EB 180	EB 150
Pen-top, 1/10mm	181	150
Pen-bottom, 1/10 mm	176	149
Diff pen, 1/10 mm	5	1
R&B-top, °C	38.2	39.6
R&B-bottom, °C	37.6	40.6
Diff R&B, °C	0.6	-1.0

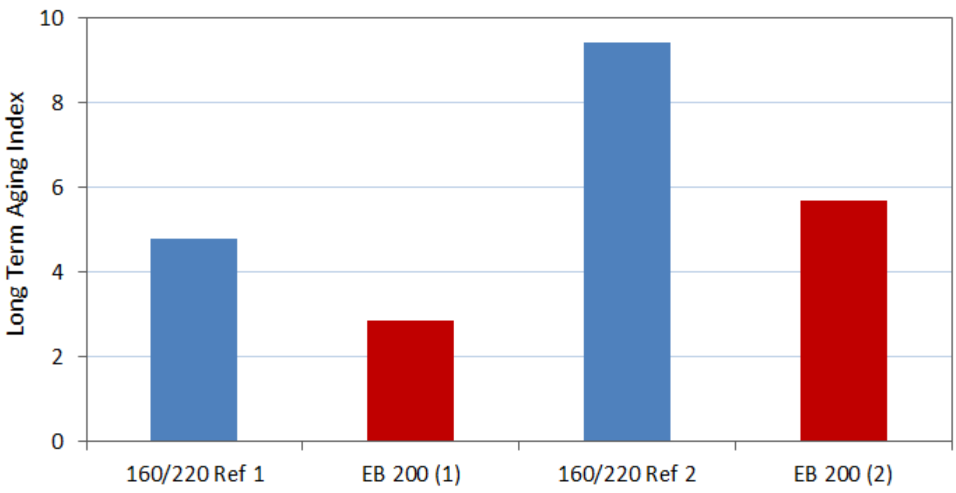

Figure 4: Effect of a plant-based oil on bitumen long-term aging. EB (1) has a base bitumen of the same source as 160/220 Ref 1, while EB (2) has a base bitumen of the same source as 160/220 Ref 2.

4. ASPHALT EVALUATION

In asphalt tests, five EB and three reference bitumens were selected (see Table 4), covering 160/220 and 70/100 grades, also considering variations in bitumen origin, PBO source and dosage. The base bitumens used in EB 180, EB 190 and EB 200 have the same origin as the 160/220 Ref 1, whereas EB 170 has a base bitumen of a same origin as 160/220 Ref 2. Asphalts were prepared in accordance with a Swedish standard ABT11. It is a dense-graded asphalt with maximum aggregate of 11 mm, and of about 6.0% (by weight) binder content and 3.5% air voids. In a first phase evaluation, the aggregates used were crushed granite from Södertälje in Sweden. Performance evaluation of the asphalts includes adhesion, stiffness, resistance to fatigue cracking, and resistance to rutting. More asphalt evaluations were then performed using a reference aggregate from the Swedish Transport Research Institute (VTI). In both cases, no adhesion promoter was used in the asphalts.

Table 4. List of the binders used in asphalt tests

Binder	Penetration, 1/10 mm	Softening point, °C	Kin viscosity 135°C, mm²/s	Dyn viscosity 60°C, Pa.s	Fraass breaking point, °C
160/220 Ref 1	192	37.8	199	64.0	-21
160/220 Ref 2	167	40.6	181	52.0	-17
70/100 Ref	83	45.4	345	187	-17
EB 200	198	35.8	226	61.1	-22
EB 190	190	36.4	242	62.5	-23
EB 180	182	37.8	236	74.7	-18
EB 170	167	38.4	199	51.3	-23
EB 80	75	46.4	377.9	213.2	-13

4.1. Adhesion

Rolling bottle method (EN 12697-11) was used to assess the affinity between aggregate and bitumen. In the test, granite aggregates of 8-11 mm size (510g) and bitumen (15.6g) were heated to 150°C prior to mixing. The mixed material was split into three parts, each weighting 150 g, and transferred into three bottles with distilled water. The bottles, with glass rod inside, were placed on the rolling machine and the rolling procedure was started. The rolling of the bottles at room temperature was stopped after 6 and 24 hours. Two operators carried out independently a visual determination of the aggregate area covered by bitumen. Results for the extended bitumen and corresponding reference bitumen are compared in Figure 5. It is evident that the PBO studied significantly increases the coverage, indicating improved adhesion between the aggregates and the EB binders. The improvement was found to increase with concentration of the PBO.

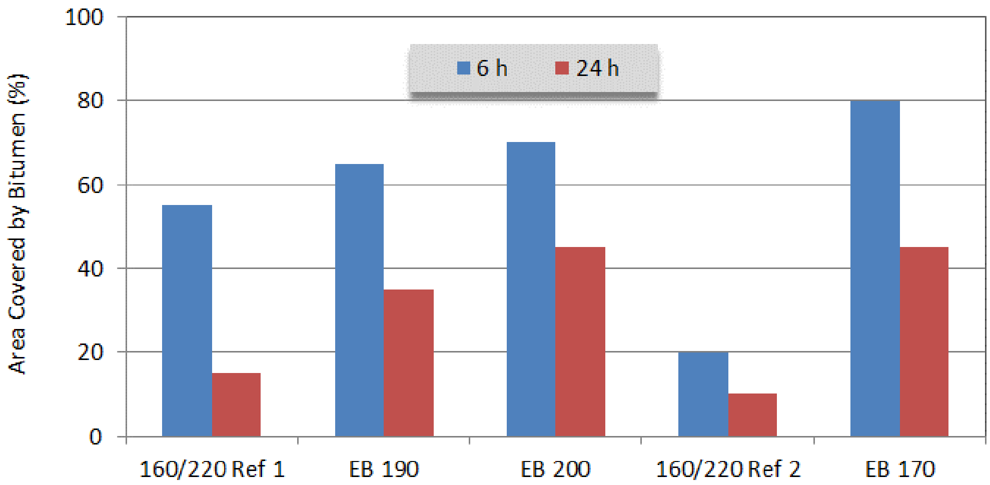

Figure 5: Results of rolling bottle test

Water sensitivity of asphalts was also assessed by indirect tensile test (EN 12697-23) in accordance with Swedish standard TRVMB 704. For each asphalt mixture, 10 specimens (diameter 100 mm, height 60 mm) were drilled from a laboratory compacted slab. A subset of five specimens was kept at room temperature (or dry conditions), and the other subset of five specimens was saturated (3 h) and then conditioned in a water bath at 40°C water for seven days (wet conditions). The indirect tensile tests were performed at 10°C, and indirect tensile strength ratios (ITSR) were calculated. As shown in Table 5, all the asphalts display low values of ISTR, probably due to poor quality aggregates. At the same time, it was also observed that, in water condition, the swelling (volume increase) of the specimens was relatively high. A high swelling may worsen ISTR results, but it is not necessarily related to water damage. Nevertheless, the asphalts made with the extended bitumens show lower water sensitivity as compared to those with the corresponding reference bitumen. More ITSR tests were performed using a different aggregate from VTI. As shown in Figure 6, the EB binders behave quite similarly to the reference bitumens. At the same time, the overall levels of ITSR, as well as ITS values, are much better than those presented in Table 5, mainly reflecting aggregate effect.

Table 5. Water sensitivity of asphalts assessed by indirect tensile strength ratio (ITSR)

Binder	ITS (dry), kPa	ITS (wet), kPa	ITSR, %
160/220 Ref 1	1444	522	36
EB 190	1445	695	48
EB 200	1066	434	41
160/220 Ref 2	1480	996	67
EB 170	1434	1080	75

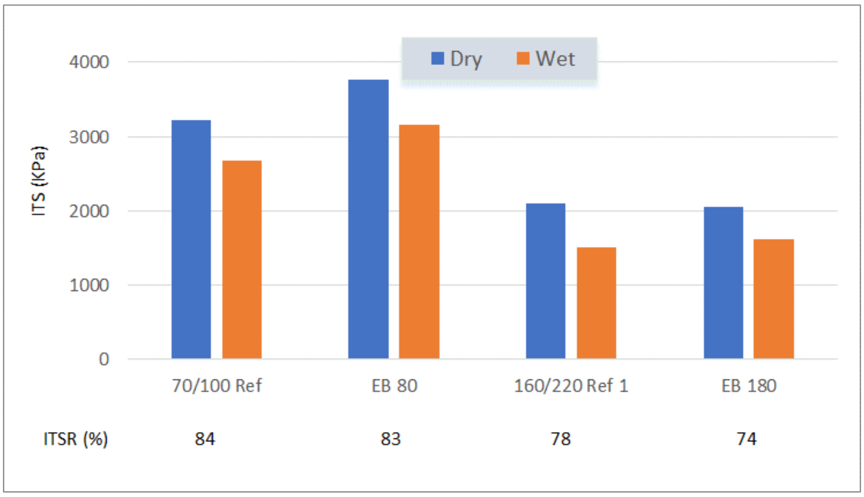

Figure 6: Water sensitivity assessed by ITSR

4.2. Stiffness

Stiffness measurements were performed at different temperatures using indirect tensile test (IDT) according to EN 12697-26 annex C. Specimens of 100 mm in diameter and 50 mm in height were prepared using a gyratory compactor. As illustrated in Figure 7, there are no significant differences in stiffness between the asphalts made with the extended bitumen and the corresponding reference bitumen. Moreover, these asphalts display similar temperature susceptibility, indicating the tested PBO will most probably not affect asphalt low temperature cracking.

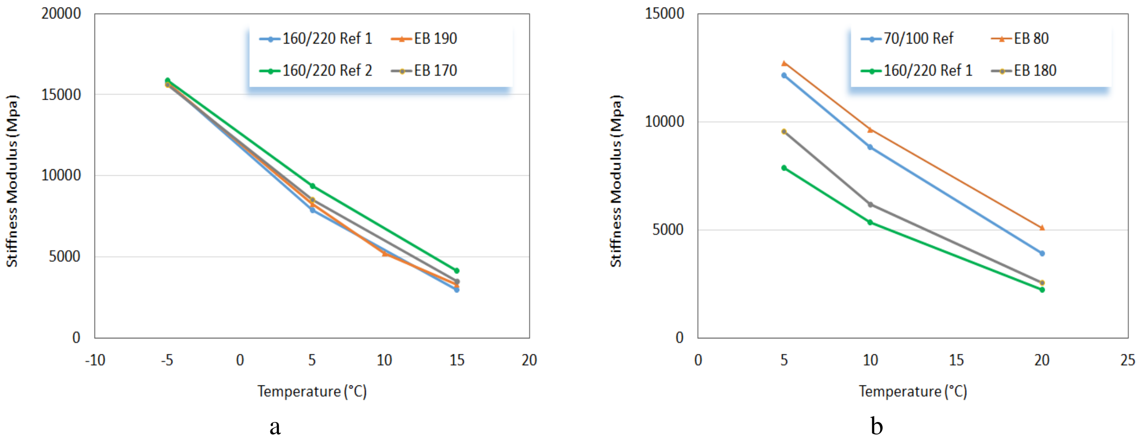

Figure 7: Asphalt stiffness as a function of temperature (a – aggregate from Södertälje; b – aggregate from VTI)

4.3 Fatigue

Fatigue tests were carried out at 10°C on gyratory-compacted specimens (diameter 100 mm, height 50 mm) also using indirect tensile test (IDT) as described in EN 12697-24 annex E. In the IDT fatigue test, a cylindrical specimen is exposed to a repeated haversine loading with a loading duration of 0.1 s followed by a 0.4 s rest period (a frequency of 2 Hz) through the vertical diametral plane. The resulting horizontal deformation of the specimen is measured and used to calculate tensile strain at the centre of the specimen. The initial strain is calculated after stabilization of the horizontal deformation, which normally occurs after 60 load repetitions. Fatigue life is defined as the total number of load repetitions when fracture of the specimen occurs. To establish the fatigue relationship at a test temperature (10°C in this study), 12 specimens of each asphalt mix were tested under a range of loading levels.

The obtained fatigue constants are summarized in Table 6 and fatigue lines compared in Figure 8. It is evident that the asphalts made with two reference bitumens behave differently. However, blending the PBO into the bitumen does not seem to have a significant effect on the fatigue behaviour.

Table 6. Summary of fatigue tests

Binder	$N_f = K\,(1/\varepsilon)^n$		
	K	n	Regression R^2
160/200 Ref 1	9.6E+11	2.94	0.99
EB 190	9.7E+11	2.98	0.96
EB 200	5.9E+11	2.90	0.98
160/220 Ref 2	1.9E+13	3.59	0.97
EB 170	2.8E+13	3.70	0.99

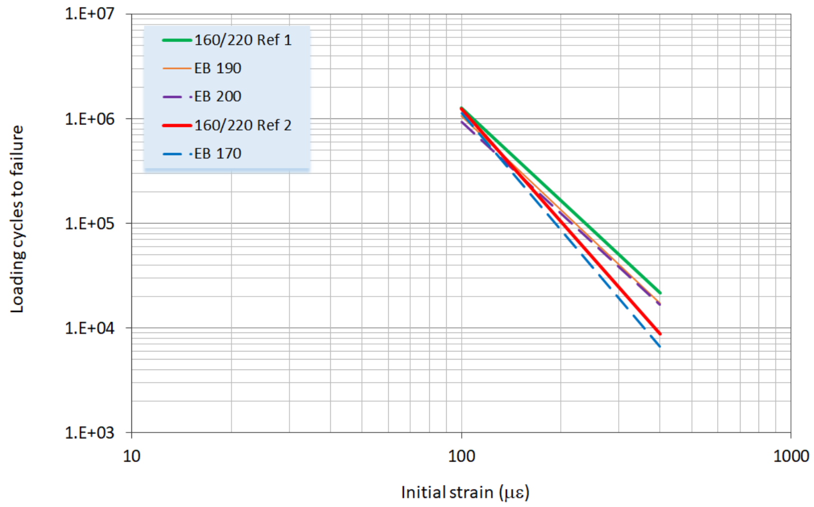

Figure 8: Fatigue lines at 10°C for the asphalts made with the extended bitumens as compared to reference bitumens

4.4. Rutting

The rutting performance of asphalts was evaluated using wheel tracking test. It was carried out at 45°C in air (dry) as well as in water with a small device according to procedure B as described in EN 12697-22. Asphalt specimens were prepared by a roller sector compactor according to "TP Asphalt 33/2007", a compaction program pre-installed by the manufacturer of the compactor. Mixing temperature was 150 ± 5°C. The specimens have been "let to rest" at room temperature for seven days prior to testing. For each asphalt mix, two specimens were tested. The average data obtained at dry condition are illustrated in Figure 9, as well as in Table 7. As indicated, the PBO extended bitumens show similar or slightly improved rutting resistance compared to the reference bitumen. It can also be seen that the two reference bitumens behave quite differently in the wheel tracking test.

Regarding the tests in water condition, unfortunately, relevant results were not achieved, either because specimens failed earlier (< 10000 cycles) or repeatability between two specimens was too poor to make a reasonable average. It was felt that this type of test is probably not suitable for ABT 11 containing a soft bitumen like 160/220 at the same time without adding an adhesion promoter.

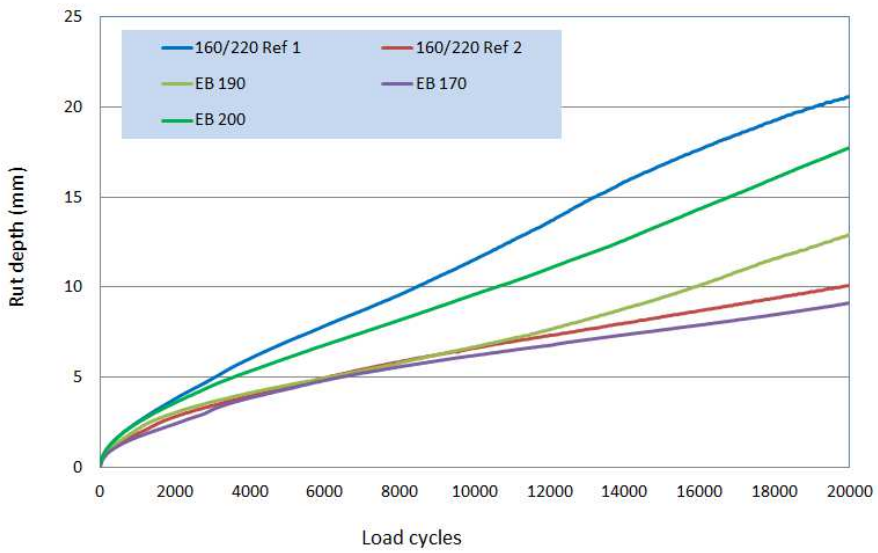

Figure 9: Wheel tracking tests at 45°C in air

Table 7. Wheel tracking test results obtained at 45°C in air

Rutting parameters	160/220 Ref 1	EB 190	EB 200	160/220 Ref 2	EB 170
Proportional rut depth (%)	54.2	33.1	44.4	25.8	23.4
Ruth depth at 20000 cycles (mm)	20.6	12.9	17.8	10.1	9.1
Wheel track slope (mm/1000 cycles)	1.81	1.25	1.63	0.70	0.58

5. FIELD TRIALS

Two field trials were carried out on the extended bitumen, EB 170 and EB 80, respectively. The trial on EB 170 was made in October 2016 in a residential area in Västerås. Asphalt layer was made with a dense mix (ABT11) with a nominal binder content of 6.0% and with Wetfix as adhesive agent; no RAP was used in the mix. During production and paving operation of the asphalt, a different smell was noticed, but it was not annoying or irritating. It should be pointed out that, prior any field trial, fumes from PBO and PBO extended bitumen were generated in the laboratory at elevated temperatures, and the fume composition was analysed. No compounds were detected at any level that triggered concerns of increased health risk for asphalt workers. Regarding performance, the pavement surface has been inspected yearly since 2016; no distress has been observed (Figure 10).

2016-10-19 2017-05-30 2018-04-18 2019-06-20

Figure 10: Field trial on EB 170 and performance follow up

A second field trial was conducted on the 10th of September 2018 on a road of higher traffic in Arboga. Four sections were constructed with 40 mm asphalt layer (Figure 11): (1) SMA 16 with 70/100 Ref, without RAP; (2) SMA 16 with EB 80, without RAP; (3) SMA 16 with EB 80, with 20% RAP, and (4) SMA 16 with 70/100 Ref, with 20% RAP. The paving was started in early morning with section 1, and after about 3 h, switched to section 2, both without RAP. It was completed later in the day by sections 3 and 4 with RAP. The whole process of mixing and paving went smoothly, no operative differences were reported. Similar to the first field trial, the asphalt workers involved all commented that the smell was different but still acceptable. Drawing a general conclusion on this aspect was not possible due to lack of quantitative data and only qualitative observations were available. The sections were also inspected in June 2019 after a first winter. As expected, all the sections remain in a good condition (photos not shown).

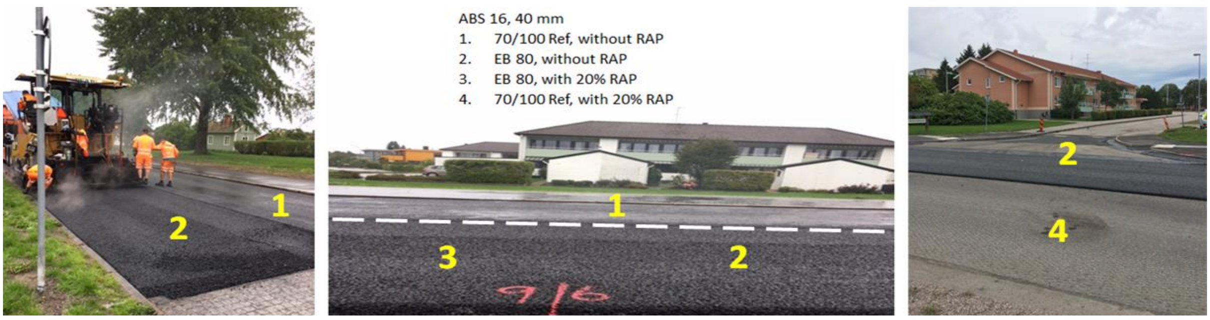

Figure 11: Field trial on EB 80, including two reference sections

In connection to the second field trial, fresh binders and those recovered/extracted from loose mixes (without RAP) were analysed. Test results obtained by BBR are shown in Figure 12. As illustrated, the limiting temperatures at 300 MPa (LST) are quite similar for the extended bitumen and reference bitumen, and no significant differences are observed between fresh and recovered samples. However, relatively large differences are seen on LmT (temperatures at 0.300 m-value), resulting in a more positive ΔTc for the extended bitumen than the reference bitumen, particularly for the samples recovered from loose mixes. This implies that the PBO may enhance bitumen durability, consequently, could improve asphalt performance in terms of resistance to surface cracking. To make further verification, follow-up on the field performance of the test sections will be carried out.

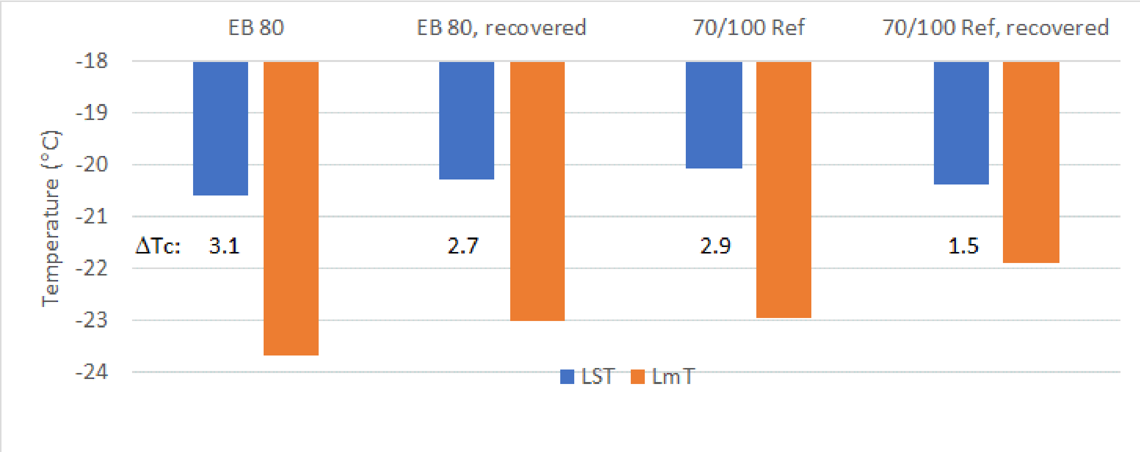

Figure 12: BBR measurements of the binders used in the field trial in 2018

6. CONCLUSIONS

The plant-based oil (PBO) studied is a complex mixture composed of numerous chemicals, including fatty acids and esters. The PBO used is fully compatible with bitumen and no phase separation occurs during handling or hot storage.

The PBO extended bitumens tested fulfil specification EN 12591. They also show improved long-term durability when measured in terms of ΔTc.

The PBO extended bitumens show improvement in asphalt adhesion especially when poor aggregates are used, as assessed by rolling bottle test and ITSR.

There are no significant differences in stiffness between the asphalts with the PBO extended bitumen and those with the corresponding reference bitumen. The asphalts also display similar temperature susceptibility, implying the PBO will most probably not affect asphalt low temperature cracking.

Asphalts made with different bitumens behave differently in fatigue. However, blending the PBO into the bitumen does not seem to have a significant effect on the fatigue behaviour of the asphalts.

In the wheel tracking test, the PBO extended bitumens show similar or slightly improved rutting resistance as compared to reference bitumens.

Two field trials were carried out on the PBO extended bitumens, and all sites have been found to be in a good condition over time. Performance follow up will be continued.

Prior to the field trials, fumes from PBO and PBO extended bitumen were generated in the laboratory at elevated temperatures, and the fume composition was analysed. No compounds were detected at any level that triggered concerns of increased health risk for asphalt workers. Future field trials are expected to include occupational workplace monitoring.

For future work, investigation on recyclability of the materials is also recommended, including performance evaluation of the extended bitumen with RAP, and assessment on if asphalts made with the extended bitumen is recyclable.

7. ACKNOWLEDGEMENTS

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