BIOFLUXED BITUMENS - EXPERIENCES FROM FIELD TESTS AND FURTHER DEVELOPMENTS

Markus Simonen[1], Terhi Pellinen[2], Jarkko Valtonen[2], Timo Blomberg[3]

[1]A-Insinöörit Suunnittelu Oy, Espoo, Finland
[2]Department of Civil and Environmental Engineering, School of Engineering, Aalto University, Espoo, Finland
[3]Nynas Oy, Vantaa, Finland

ABSTRACT

Stockpiled patching mixtures for temporary and permanent pavement repairs are made of cut-back bitumens, which allow workable mixture at relatively cold temperatures. However, cut-back bitumens contain volatile organic compounds (VOCs) that contribute to climate change and produce other harmful environmental effects. In this follow-up study, mixtures manufactured and laid down two years ago using a new renewable fluxing agent, bioflux (branched and linear C10 – C20 alkanes), were investigated. Biofluxed bitumens emit less VOCs than the cut-back bitumens and pose no threat to the environment. However, the rate of curing in cut-back bitumens is regulated by solvents. Therefore, by replacing solvents with bioflux the rate of curing most likely is altered, which would in turn influence the stability of mixtures. An experiment was conducted where two low volume roads (less than 1000 vehicle per day) were paved with the mixtures made of biofluxed bitumen. The follow-up samples were obtained from the stockpiled mixtures and from test roads and compared to the original as-constructed samples. Additionally, the test roads were inspected visually. The simulated distillation studies suggested that the evaporation rate for bioflux was moderate, which was confirmed by the moderate increase in the indirect tensile strength of the mixture. Despite of the modest strength gain, after two years of traffic loading, the test roads were in excellent condition apart from minor rutting. These results are encouraging for replacing cut-back bitumens with biofluxed bitumens.

Keywords: Bioflux, Environment, Emissions, Workability, Patching

1. INTRODUCTION

1.1 Background

Oil gravel was developed in the 1950s to provide an affordable pavement solution for low volume roads. However, the slow-curing cut-back bitumen (BL2K) in the oil gravel contained a considerable amount of volatile organic compounds constituting to the climate change; therefore in the 1990s its use in Finland was restricted only to the stockpiled patching mixtures. In 2008, Nynas Ab introduced a new fluxing agent, bioflux, to replace the volatile solvents used in cut-back bitumen. Bioflux consists of linear and branched C10 – C20 alkanes and it is the product of a chemical process called NExBTL, which is patented by Neste Oil. The feed of NExBTL-process consists of vegetable oils, animal fats and hydrogen. The benefits of using bioflux instead of petroleum based fluxing agents may contribute to lower emissions of volatile organic compounds, in safety to aquatic organisms and in the use of renewable raw materials.

In the wintertime, when mixing plants in Finland were closed, it was a common practice to manufacture oil gravel and stockpile it at the back of the mixing plant. This mixture was then used as a temporary cold patching compound for low volume roads and streets. Although, soft asphalt mixtures (referred as PAB-V and made of neat V1500 or V3000 bitumen) could be used to replace the oil gravel as a paving material, it cannot be used as patching material because mixture loses its workability due to the excessive hardening during storage.

Table 1 gives some typical biofluxed bitumen properties compared to the Finnish Asphalt Specification [1] requirements for the road oils. The heavier flux in biofluxed bitumen brings along a better flash point. The flash point exceeds the critical temperature of 100 °C, being about 130 °C. This gives considerable safety factor when using hot mix plants in oil gravel production. It is expected that mixture will retain good workability over longer period of time because of the heavier flux. The biofluxed products should be used with anti-stripping agent similar to the conventional road oil.

Table 1: Properties for road oils.

Property	Unit	Biofluxed bitumen Typical values *)	Road oil Specification
Viscosity 60 °C	mm^2/s	750	350-650
Flash point, PM closed cup	°C	130	> 55
Density 15 °C	kg/m^3	970	965
Distillation			
- distillate at 225 °C	vol-%	0	0
- distillate at 260 °C	vol-%	0	< 1
- distillate at 315 °C	vol-%	0	< 8
- distillate at 360 °C	vol-%	2	< 12
Distillation residue **) viscosity 60 °C	mm^2/s	1500	2000-4000
Stabilized binder ***) viscosity 60 °C	mm^2/s	1600	

*) Typical values for the biofluxed bitumen introduced in 2008
**) Obtained at 360 °C
***) 24 h at room temperature + 24 h at 50 °C + 24 h at 85 °C (film thickness 1 mm)

1.2 Stockpiled patching mixtures

The primary requirements for stockpiled patching mixtures are: suitability for cold or warm mixing, workability, stability, water resistance and safety to environment and health. Cold and warm mixing should be possible, since stockpiled soft asphalt mixtures are typically produced in mobile asphalt plants with lower mixing temperatures than in the stationary plants. Stockpiled mixtures should be workable even after long storing times. Respectively, the stability right after spreading and compaction should be high enough, so that patches or temporary pavements would sufficiently resist environmental and traffic loading. Water resistance is emphasized in mixing at low temperatures especially with mobile asphalt plants using steam for aggregate heating. The requirements are partly opposite, e.g. good workability of a mixture is likely to relate to lower stability. Below is a list of properties that bituminous patching mixture should have [2]:

- *Stability*, to allow the patch to resist displacement by traffic.
- *Stickiness*, so the patch will adhere to the sides of the pothole.
- *Resistance to water action*, to keep the binder from stripping off the aggregate.
- *Durability*, so that the patch has satisfactory resistance to disintegration.
- *Skid resistance*, should be similar to the pavement in which the patch is placed.
- *Workability*, to enable the material to be easily shoveled and shaped.
- *Storageability*, so the mixture can be stockpiled without hardening excessively or having the binder drain off the aggregate.

1.3 Objectives and research approach

This paper presents certain results of a follow-up study on the development of mixtures made of biofluxed bitumens. An experiment was conducted where two low volume roads (less than 1000 vehicle per day) were paved (see Figure 1) with the mixtures made of biofluxed bitumen in 2008 [3]. Additionally, in 2009 small amount of biofluxed mixture was plant produced and stored at asphalt plant at Maantiekylä for further investigation of curing rates, see Figure 2. Although results were encouraging, more research was needed since only one composition of binder was studied in 2008. Therefore, the objective was to investigate the behavior of storage mixtures fabricated with different blends of bioflux and type of base binder.

The study is divided into two steps as follows:

Step 1 – Test road follow-up studies

- The mechanical and chemical properties of the mixtures obtained from the test roads and stockpiles
- Visual inspection of test roads

Step 2– Further developments of mixtures made of biofluxed bitumens

- Laboratory study of biofluxed bitumen with different composition: binder testing and mixture preparation and testing

Figure 1: Paving Test Roads in 2008.

Figure 2: Stockpiled patching mixtures at Maantiekylä in 2009.

2. TESTED MATERIALS

2.1 Preliminary research

In Punkalaidun and Iitti (mixing plant at Elimäki) two different soft asphalt mixtures were field produced having viscosity graded soft bitumen V1500 and biofluxed bitumen as binding agents, see Table 2. The maximum aggregate size of the Punkalaidun mixtures was 16 mm and it was a typical local aggregate used in the low volume roads [1] while the Iitti mixtures had 11 mm top size and gradation was specially proportioned for the job. Quality control tests showed that for both plants the produced mixture gradation matched the target gradation. The stockpiled aggregate had 2.6% moisture content in Punkalaidun mixing plant and 3.8% in Iitti. At Maanitekylä two mixtures were produced and stockpiled one having biofluxed binder and the other one being a reference mix with BL2K road oil.

Table 2: Materials used on Test Roads.

Mixing plant	Binder blend	Viscosity mm^2/s at 60°C	Binder content (%)	Aggregate moisture content (%)
Punka-PAB	Bitumen V1500	1260	3.5	2.6
Punka-Bio	Biofluxed bitumen	529		
Eli-PAB	Bitumen V1500	1490	3.6	3.8
Eli-Bio	Biofluxed bitumen	557		
Maantiekylä Stokpiled mixtures	Biofluxed bitumen BL2K	~600	3,8	0

For biofluxed bitumen, the soft base bitumen V1500 and bioflux was mixed in the tank, while delivering the blend from the refinery. There was no need for high-shear mixing because fluids were able to flow easily and mix together. Iitti and Punkalaidun mixtures were produced by turbo mixing plant that uses steam to warm up the aggregate. The mixing temperature ranged from 50 to 55 ºC. At Maantiekylä mixing took place in a hot mix patch plant using 105 ºC mixing temperature and aggregate was dried in a drum before mixing. Aggregate used was crushed gravel from Hiiskula quarry. Antistripping agent was added in the refinery. Punkalaidun mixtures had 0.7 %, Iitti mixtures 0.8 % and Maantiekyä mixtures 1.0% of antistripping agent by weight of the binder.

2.2 Follow-up Study

Four different blends of biofluxed bitumen were prepared in laboratory for the tests conducted in the Step 2. Base bitumens were viscosity-graded bitumens (V 1500, V3000 and V6000) and penetration graded 160/220 bitumen. The binder used in test roads was similar to V15BF blend. The target viscosity for the blends was 600 mm^2/s at 60 °C, which equals to the viscosity of BL2K used in the oil gravel. Additionally, BL2K was chosen as a reference binder for the laboratory tests. Table 3 presents the compositions and viscosities of the blends and the reference binder and Figure 3 shows the temperature dependency of blends.

Table 3: The compositions and viscosities (at 60 °C) for the biofluxed bitumens and the reference binder (BL2K)

ID	Base bitumen	Solvent	Composition (base bitumen % / solvent %)	Viscosity (60 °C) mm^2/s
V15BF *)	V1500	Bioflux	94.5 / 5.5	674
V30BF	V3000	”	90.8 / 9.2	587
V60BF	V6000	”	88.5 / 11.5	622
B20BF	160/220	”	82.0 / 18.0	646
BL2K	**)	Kerosene + gas oil	86.0 / 7.0 + 7.0	551

*) Similar to the biofluxed bitumen introduced and used in the two test roads in 2008
**) Similar to V6000

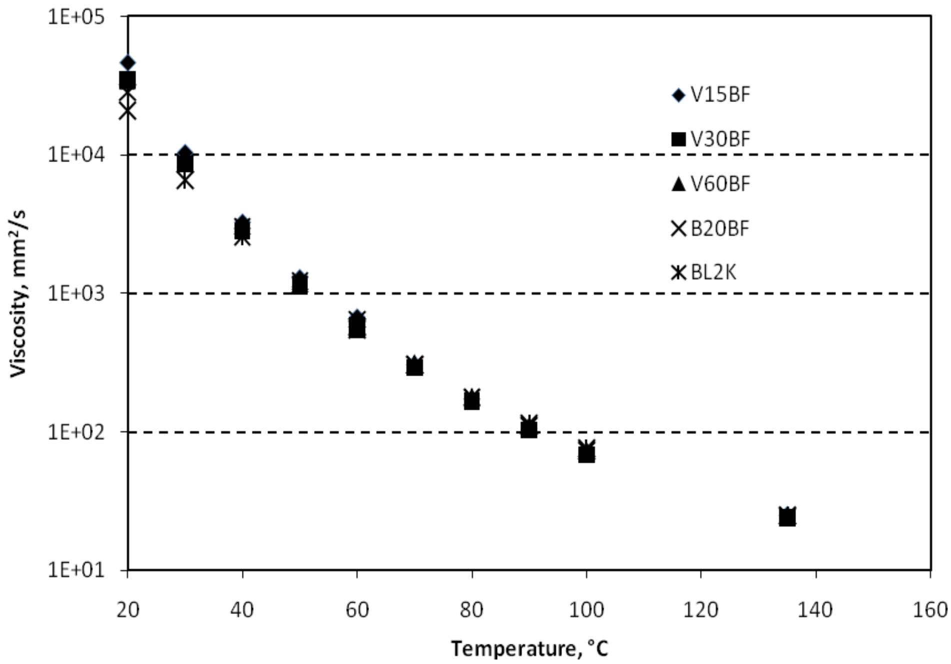

Figure 3: Viscosity of the biofluxed bitumens (20 - 100 °C – rotational, 60 and 135 °C – capillary)

The soft asphalt mixtures made of the five binder blends and crushed gravel from Hiiskula quarry was prepared in a laboratory with cold-mixing at 22 ºC temperature. The nominal maximum aggregate size of the gravel was 8 millimeters and the binder content was 3.7 %. Samples were compacted using gyratory compactor with 600 kPa pressure and 1.00° tilt angel. All samples were competed using 100 gyrations, which produced approximately 11 – 12 percent air void content.

3. EXPERIMENTS

3.1 Field experiments

The visual inspection of the test roads was conducted twice in 2010. In addition, rutting of the test roads was measured with a straight edge and tape ruler. Samples were taken from both test roads to determine the curing of the mixtures by indirect tensile strength test. The field tests included also samples from five-month-old stockpiled mixtures. Stockpiling took place during the paving work.

3.2 Laboratory testing

The cut-back bitumens (comprising the four biofluxed bitumens and the reference binders) were investigated to determine the effect of chemical composition on their rheological properties. The generic fractions (saturates, aromatics, resins and asphaltenes) of base bitumens were quantified by thin layer chromatographic method with flame ionization detection (IATROSCAN MK-6s) and the rheological properties of the base bitumens and the fresh cut-back bitumens were studied with a rheometer (Physica 301). In addition, the flashpoints of the cut-backs were determined for the safety evaluation.

The tests performed to the asphalt mixtures made of the cut-back bitumens included water sensitivity (EN 12697-12 method C), workability with blade resistance test (Figure 4; modified from a Canadian method LS-289) and stability as indirect tensile strength (Figure 5; EN 12697-23), thus the tests covered the primary performance requirements.

Workability and stability was tested from samples with different curing times using five replicates at each curing time. The water sensitivity test involves mixing 1-hour-old soft asphalt mixture with water in a glass beaker, and measuring the material that has come loose by filtering. In the blade resistance test a steel blade is penetrated into mixtures slightly compacted in wooden boxes, while the maximum resistive force is measured. Indirect tensile strength test is conducted by loading a cylindrical specimen diametrically along the direction of the cylinder axis and measuring the peak load applied at break. The tensile strength (in Pa) is then calculated from the peak load and the dimensions of the specimen.

Testing the workability of bituminous mixtures has proven to be challenging. Most of the current tests include a subjective evaluation performed by a skilled technician or are high in variation [4]. Both the subjectivity and variation decreases the general acceptability of the results. A blade resistance test simulating the penetration of a scoop or shovel in a stockpile mixture is a standard method for determining the workability of patching materials in Northern America. The standards published by Ontario Ministry of Transportation [5] (MTO; LS-289) and American Society of Testing and Materials [6] (ASTM; D6704-8) are practically equal. The both standards include a steel blade that is penetrated into mixtures lightly compacted in wooden boxes, while the maximum resistive force is measured. Additionally the loading speed and loading time (50 mm/min. for 30 seconds) as well as the dimensions of the steel blade are the same (3 x 50 x 130). However, the MTO's method suggests larger test specimen size and using the same specimen in three separate tests, which may lead to disturbed test specimen with changed test temperature. Maher et al. (2001) got high variation in blade resistance tests (the MTO's method), although the repeatability of D6704-8 is determined to be 10 % within a laboratory.

Figure 4: Blade resistance test

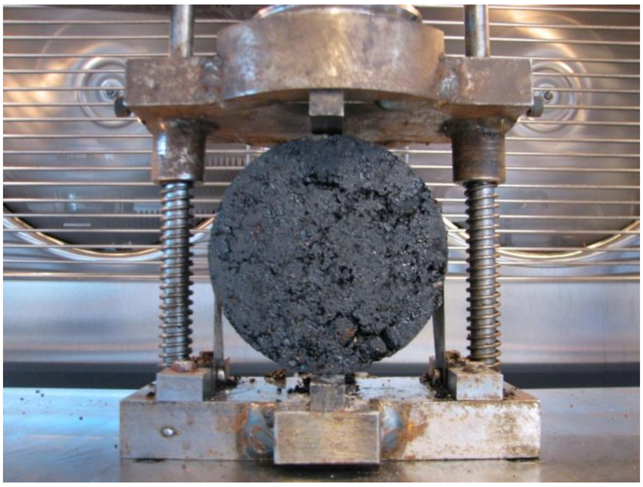

Figure 5: Indirect tensile strength test

4. RESULTS

4.1 Field observations

Both test roads were generally in good condition after two years of trafficking, see Figure 6. There were no signs of raveling, cracking or other major distresses. However, the rut measurements with the straight edge revealed, that both test lanes (nominal pavement thickness was 40 mm) containing biofluxed bitumens had rutted about 10 millimeters. Surprisingly, the maximum rut depth was observed in the middle of the lane (see Figure 7) and no rutting was observed on the reference lane made of neat V1500 bitumen. Since both lanes, the one made of V1500 and the other made of biofluxed bitumen, had equal base course, sub-base and sub-grade in both test sites, it may be concluded that the observed rutting resulted from low stability of the pavement made of biofluxed bitumen. Additionally, for biofluxed mixtures the largest stone particles had sunk into the pavement, which led to an extremely smooth surface in both test sites. All in all, the observations from the test roads suggested that the mixtures made of biofluxed bitumen might have stayed too soft.

Figure 6: Iitti test road: Biofluxed binder on the left and V1500 on the right.

Figure 7: Rutting measurements.

4.2 Evaporation of the bioflux

Based on SFS-EN 14895 testing, the evaporation of bioflux proved to be moderate compared to BL2K based on curing both in laboratory and in the samples obtained from the field. Table 4 shows that after the recovery the solvent content of BL2K had dropped 50 % meanwhile the bioflux content had decreased only 10 – 20 % due. The results suggested that the laboratory recovery of biofluxed bitumens and BL2K corresponded 1 – 2 years of evaporation in a road or in stockpile (see Table 5). The results comply with the field experiences of BL2K. Thus, the standard recovery procedure could be used in determining the evaporation rate of bioflux during the first couple of years. The stabilization procedure at excessively high temperatures effects the binder compositions radically. The relation between the stabilization and field aging may not be reliably established based on the data after only two years.

Table 4: Nominal and observed solvent contents after the laboratory aging procedures. (boiling point ≤ 360 °C)

ID	Nominal, %	Original, %	Recovered, % *)	Stabilized, % *)
V15BF	5,5	4,8	4,4	1,9
V30BF	9,2	8,2	6,6	1,8
V60BF	11,5	10,4	8,3	2,2
B20BF	18,0	17,0	13,9	5,2
BL2K	14,0	14,1	7,1	4,6

*) according to SFS-EN 14895

Table 5: Nominal and observed solvent contents of the field samples. (boiling point ≤ 360 °C)

Testsite	Binder	Nominal, %	Observed, %
Punkalaidun	Punka-Bio	5,5	5,9
Elimäki	Eli-Bio	6,4	5,0
Maantiekylä	Biofluxed V1500	5,3	4,8
Maantiekylä	BL2K	14,0	7,8

4.3 Stability

Figures 8 and 9 are presenting stability measurements from the test roads. Early results are from the preliminary research [3] and the later results are from the follow-up study of the test road mixtures. It can be seen from the figures, that there were no strength gain in the mixtures made of biofluxed bitumen in Punkalaidun. Also, results from Punkalaidun test road suggest that for cut-back bitumen curing took place in the first 4 months but after that there were no strength gain. The Iitti test road suggests a slight strength gain for the biofluxed mixture and very clear strength gain for the mixture made of cut-back bitumen. Figures also show the error bars (2·standard deviation) for the average test results. The coefficient of variation (CV) ranged between 2.6% and 12.2% being 6.9% in average in the preliminary testing. In the follow-up testing, CV ranged from 3.9 % to 14.4 % average being 8.1%, whish is slightly higher than in the filed mixtures.

Table 4 shows the stability test results from the stockpiled mixtures. During the five months of storage, mixture with road oil (BL2K) had cured faster than the biofluxed mixture gaining 55% higher IDT strength. Faster curing and the higher strength of the mixture most likely also contributed to the higher testing variation.

Table 6: IDT Strength of stockpiled Mixture at Maantiekylä.

	IDT Strength at 10°C		
Mix type	Average (kPa)	Standard Deviation (kPa)	CV%
Maantie-Bio	103	3.9	3.8
Maantie-BL2K	160	11.2	7.0

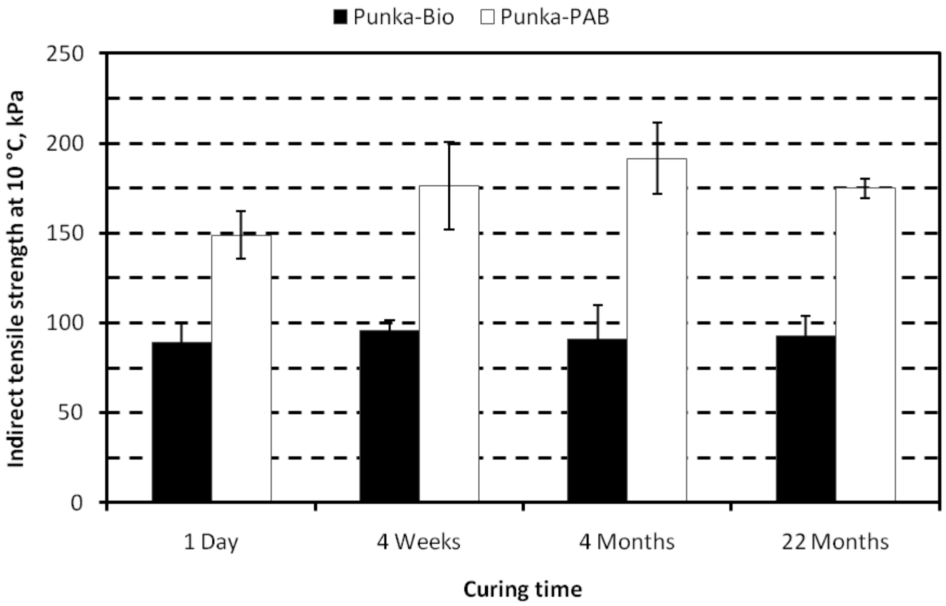

Figure 8: The average indirect tensile strength of Punkalaidun test road mixtures at 10 °C (1 Day, 4 Weeks and 4 Months from [3])

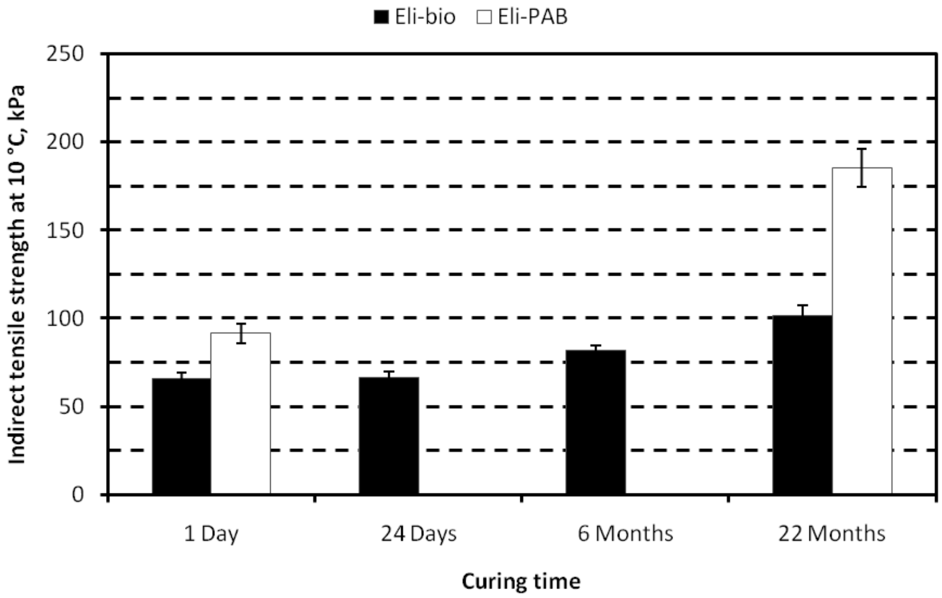

Figure 9: The average indirect tensile strength of Iitti test road mixtures at 10 °C (1 Day, 24 Days from [3])

Figure 10 shows the stability results for the mixtures made of the new binder blends shown in Table 3. The stability of the mixtures increased only moderately during the first month. The largest increases in stability were observed from the mixtures made of biofluxed bitumens with V6000 and 160/220 as the base bitumens and from the BL2K mixture. The stability of the V15BF and V30BF mixtures stayed practically unchanged implying that only minor evaporation occurred during the first month, while the other reason might be the soft base bitumens. The consistency of the base bitumen and the solvent amount affected the curing of the mixtures. The development of bitumen structure may have contributed to the strength increase in addition to the solvent evaporation.

The magnitude of the IDT strength for the laboratory mixtures is lower than that of the plant produced field mixtures. One reason may be the smaller aggregate size used. In addition, laboratory mixing may not be as effective as the field mixing, which takes place in large quantities. Another reason may be the use of antistripping agents. Laboratory mixtures were fabricated with no antistipping agent but all filed mixtures had antistripping agent 0.7 to 1.0% by weight of binder added at the refinery. When antistripping agent is added at the refinery, usually excess of 0.5% is used to compensate degradation during transport.

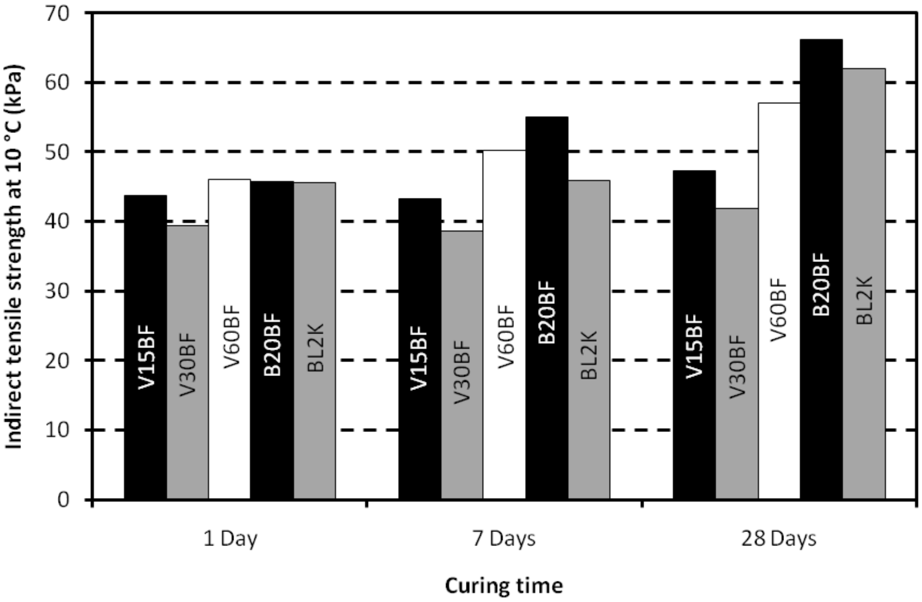

Figure 10: The average indirect tensile strength after three curing times at 10 °C

4.4 Workability

Figure 11 shows that the moderate evaporation of bioflux led to only minor changes in workability during the first five and a half months According to literature [7] 2 kN (at -10 °C) is considered to be the acceptance limit for stockpiled patching mixtures, thus the mixture made of cut-back bitumen (PL2K) remained workable for the whole observation time. A mixture made of neat V1500 bitumen (V15B in Figure 11) and the crushed gravel exceeded the limit. Based on field experience, mixtures containing V1500 are not generally considered workable at ambient temperatures. Therefore, the blade resistance test results and the 2 kN acceptance limit supported the subjective workability experiences. The stockpiled soft asphalt mixtures made of BL2K (oil gravels) are known to remain workable even after very long storage times, which encourages to assess the workability of the mixtures made of biofluxed bitumens to be at least as good. B20BF with the hardest base bitumen may form a rigid crust on the surface of the stockpile, if excessive amounts of bioflux would evaporate.

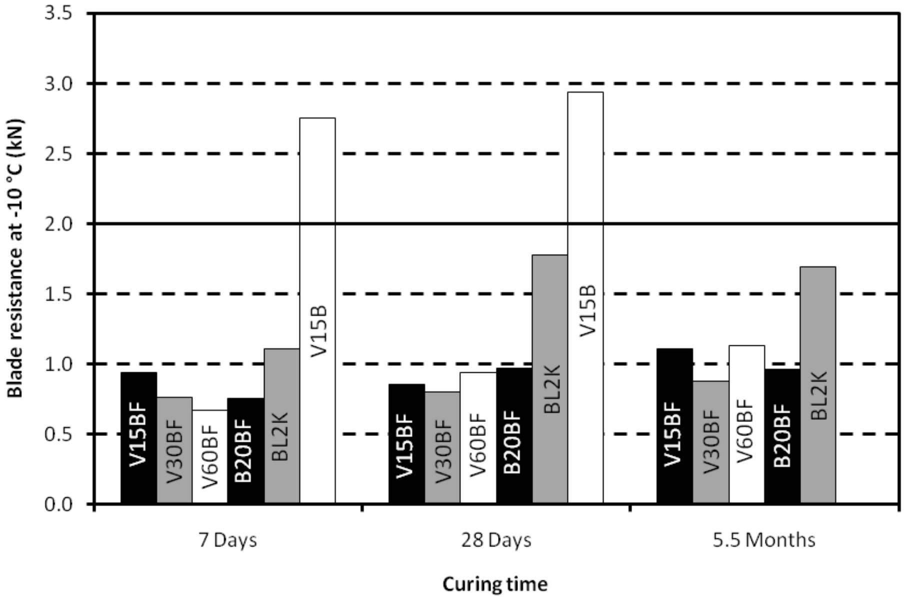

Figure 11: The average blade resistance of the mixtures after three curing times at -10 °C (V15B was made of neat V1500 bitumen)

5. DISCUSSION

Based on the literature, cold mixing is possible for bituminous binders having viscosity less than 600 mm^2/s at 60 °C [8]. Respectively, for warm mixing the binder viscosity at 60 °C should be less than 3000 mm^2/s. Thus, the fresh biofluxed bitumens may be used in cold and warm mixing. The flashpoints of biofluxed bitumens were between 110 – 141 °C and the flashpoint of BL2K 80 °C. The higher flashpoint of biofluxed bitumens increases the safety during mixing especially at excessively high temperatures. Additionally, BL2K is classified to be harmful for aquatic organisms ("may cause long-term adverse effects in the aquatic environment") according to the EU's regulations for chemicals (REACH/CLP) due to the used solvents (kerosene and gas oil), whereas biofluxed bitumen is not classified to posses any risk for the environment. Therefore the negative environmental impacts of stockpiled mixtures made of BL2K may be reduced with biofluxed products.

The results of this research provided useful information on the properties and utilization of biofluxed bitumens. However, the studied biofluxed bitumens cover only a minor part of all possible compositions. It could be recommended, based on the laboratory tests and field observations, to increase the viscosity of biofluxed bitumens to 800 – 1000 mm^2/s, so that better initial stability would be achieved without excessively degrading the workability. Respectively, the type of the base bitumen affected the curing of the mixtures.

For the future, the different applications for stockpile patching mixtures should be investigated, and perform field tests in the actual use of the patching mixtures. Additionally, a connection between laboratory test results and field performance should be established, so that the laboratory tests could be used in the evaluation of various patching materials.

6. CONCLUDING REMARKS

In conclusion, the studied biofluxed bitumens were found suitable for stockpiled soft asphalt mixtures. The recently published (2011) Finnish Asphalt Specifications now includes biofluxed bitumen called BL2Bio, which can be used in stockpiled soft asphalt mixtures according to the specifications. The major difference between the biofluxed bitumens and the BL2K is the curing rate, thus the binder design approach must be considered accordingly. t. The slow curing of the biofluxed bitumens is both advantageous and disadvantageous. The stability of the mixture made of biofluxed bitumens may remain moderate. However, the advantage is that the workability remains excellent for long times.

REFENRENCES

[1] Asfalttinormit 2008 (Finnish Asphalt Specifications). Pavement Advisory Council, PANK, ISBN 978-952-99985-0-0, Edita, Finland, 2007.

[2] Evaluation of Pothole Patching Materials. FHWA 2001-02, Maher, A., Gucunski, N., Yanko, W., Petsi, F., New Jersey, U.S., 2001.

[3] Development of Environmentally Friendly Cold Patching Compound, Valtonen, J., Pellinen, T., Blomberg, T., International Society of Asphalt Pavements, ISAP the 11th International Conference of Asphalt Pavements, August 1 to 6, 2010 Nagoya, Japan.

[4] Development of a Slump-Based Workability Test for Bituminous Maintenance Mixtures, Chatterjee, S., Smit, A.d.F., White, R.P & Prozzi, J.A., Transportation Research Board, 85th Annual Meeting., Transport Research Board, Washington D.C., 2005.

[5] LS-289: Test Methods for Cohesion of Cold Bituminous Patching Material by Blade Resistance Method, Ontario Ministry of Transportation, 1996.

[6] Standard Test Method for Determining the Workability of Asphalt Cold Mix Patching Material. Annual Book of ASTM Standards, (Vol.04.03) American Society of Testing and Materials. 2001.

[7] OPSS 307: Construction Specifications for Stockpiling of Patching Materials and Patching of Asphalt Pavement, Ontario Provincial Standard for Roads and Public Works, Ontario, Canada, 2007.

[8] http://onlinepubs.trb.org/onlinepubs/nchrp/nchrp_rpt_648.pdf