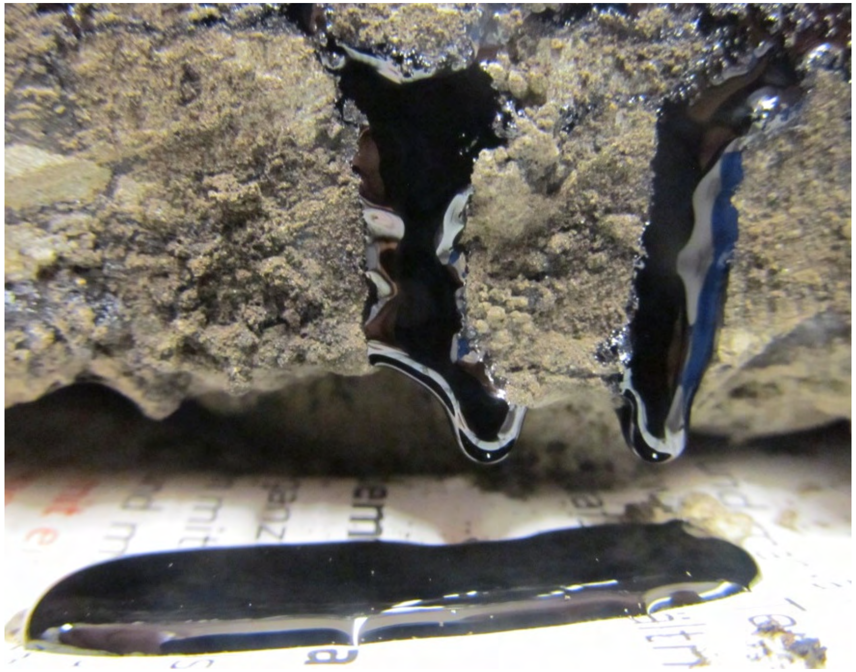

FORENSIC INVESTIGATION OF SURFACE DRESSINGS FAILURE IN ICELAND

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Alvaro Guarin, Ali Butt, Abdullah Khan

KTH Royal Institute of Technology
Nov-8-2013

Pavement Structures

Material	Thickness	Year	Bitumen, pen 160/220 + Binder + adhesive	Status
Road 75-02 Section 3870				
Surface dressing	3 cm	2012	Ethyl ester from fishoil 7,0 %	No-bleeding*
Bitumen stabilized Gravel	10 cm 50 cm	2000 1980		
Road 75-02 Section 4575				
Surface dressing	3 cm	2012	Ethyl ester from fishoil 7,0 %	No-bleeding*
Bitumen stabilized Gravel	7 cm 50 cm	2000 1980		
Road 1-K8 Section 5400				
Surface dressing	3 cm	2012	FAME from Rape seed oil 8 % + Wetfix	Bleeding
Crushed rock Crushed rock Gravel	20 cm 15 cm	2011 2011 1982		
Road 1-K8 Section 8600				
Surface dressing	3 cm	2011	FAME from Rape seed oil 8 % + Wetfix	Bleeding
Crushed rock Crushed rock Gravel	10 cm 15 cm	2011 2011 1982		
Road 1-K8 Section 11070				
Surface dressing	3 cm	2011	Ethyl ester from fishoil 7,0 %	Bleeding
Cement stabilized Gravel	15 cm 50 cm	2011 1982		
Road 1-K9 Section 2115				
Surface dressing	5 cm	2011	FAME from Rape seed oil 7,5 % + Wetfix	Bleeding
Crushed gravel Gravel	20 cm 50 cm	1992 1992		

Road Sampling

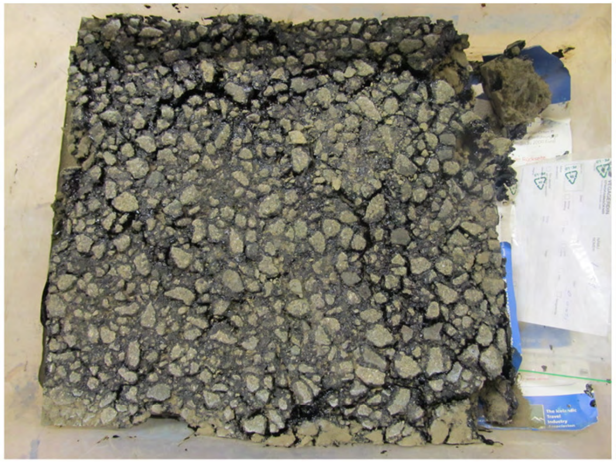
KTH VETENSKAP OCH KONST

Laboratory Testing Plan

	TEST
Binder Grading	Penetration (Original and recovered binder)
	Brookfield viscosity
	Dynamic Shear Rheometer DSR (Original, RTFOT residue, and PAV residue)
	Rolling Thin Film Oven Test RTFOT (Mass loss)
	Pressure Aging Vessel PAV
	Bending Beam Rheometer BBR (PAV residue)
Binder Chemical Characterization	Saturated, Aromatic, Resin, and Asphaltene fractions SARA
	Differential Scanning Calorimetry DSC
	Atomic Force Microscopy AFM
	Surface Free Energy (Sessile drop)
	Universal Sorption Device USD
Tomography	X-ray tomography

Visual Inspection

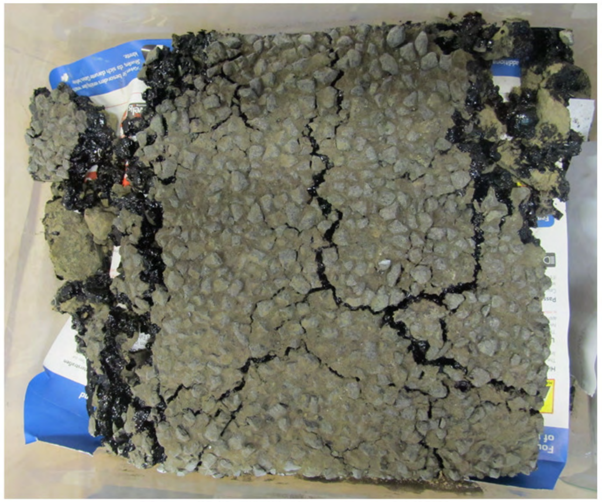
Road 75-02 Section 4575



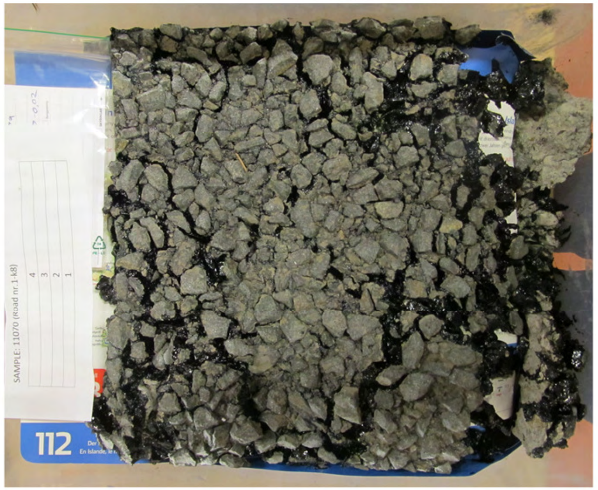
Road 1-K8 Section 8600

Road 1-K9 Section 2115

Road 75-02 Section 3870

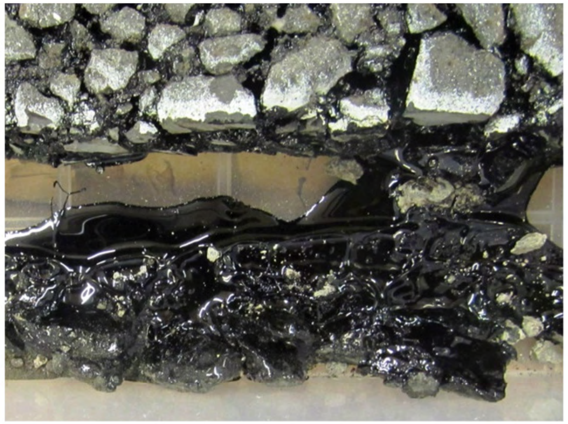


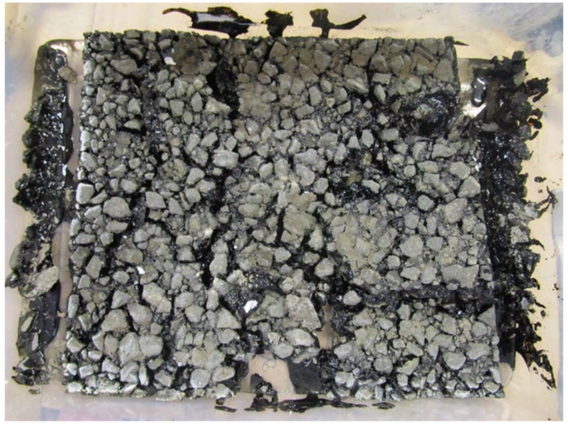
Road 1-K8 Section 5400

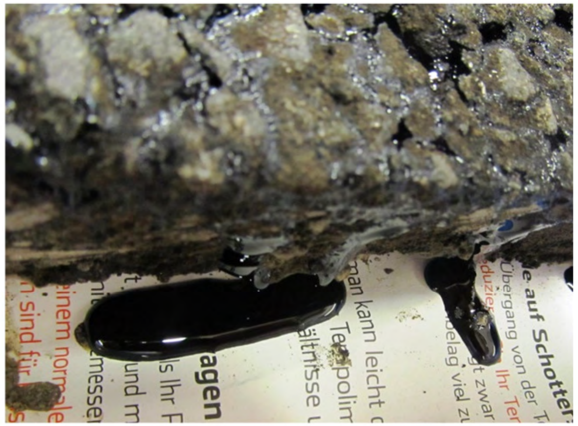


Road 1-K8 Section 11070

Visual Inspection





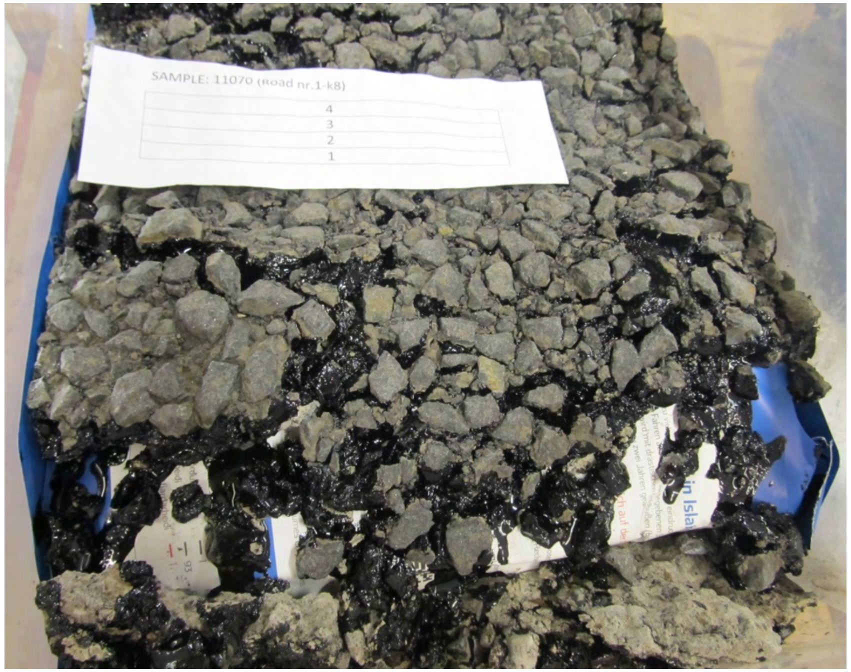


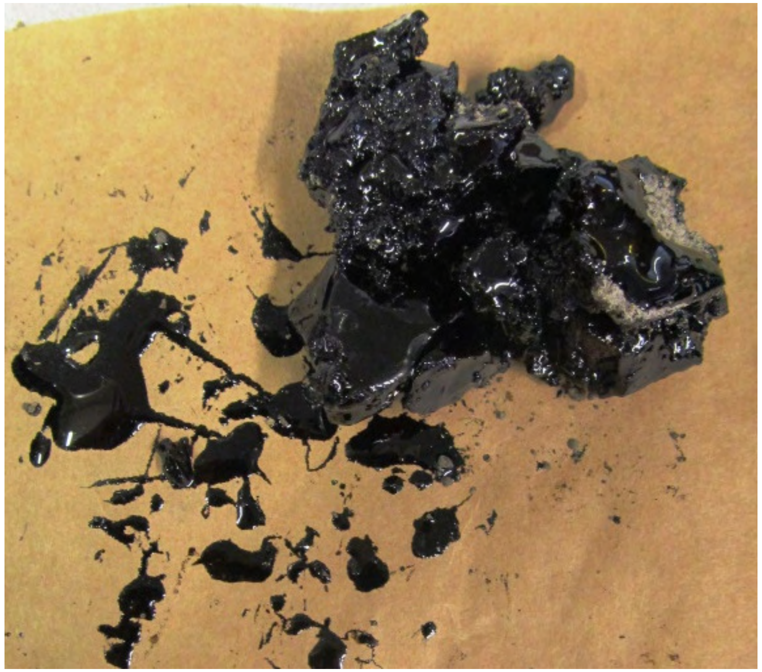
Road 75-02 Section 4575

Road 1-K8 Section 8600

Road 1-K9 Section 2115

Visual Inspection

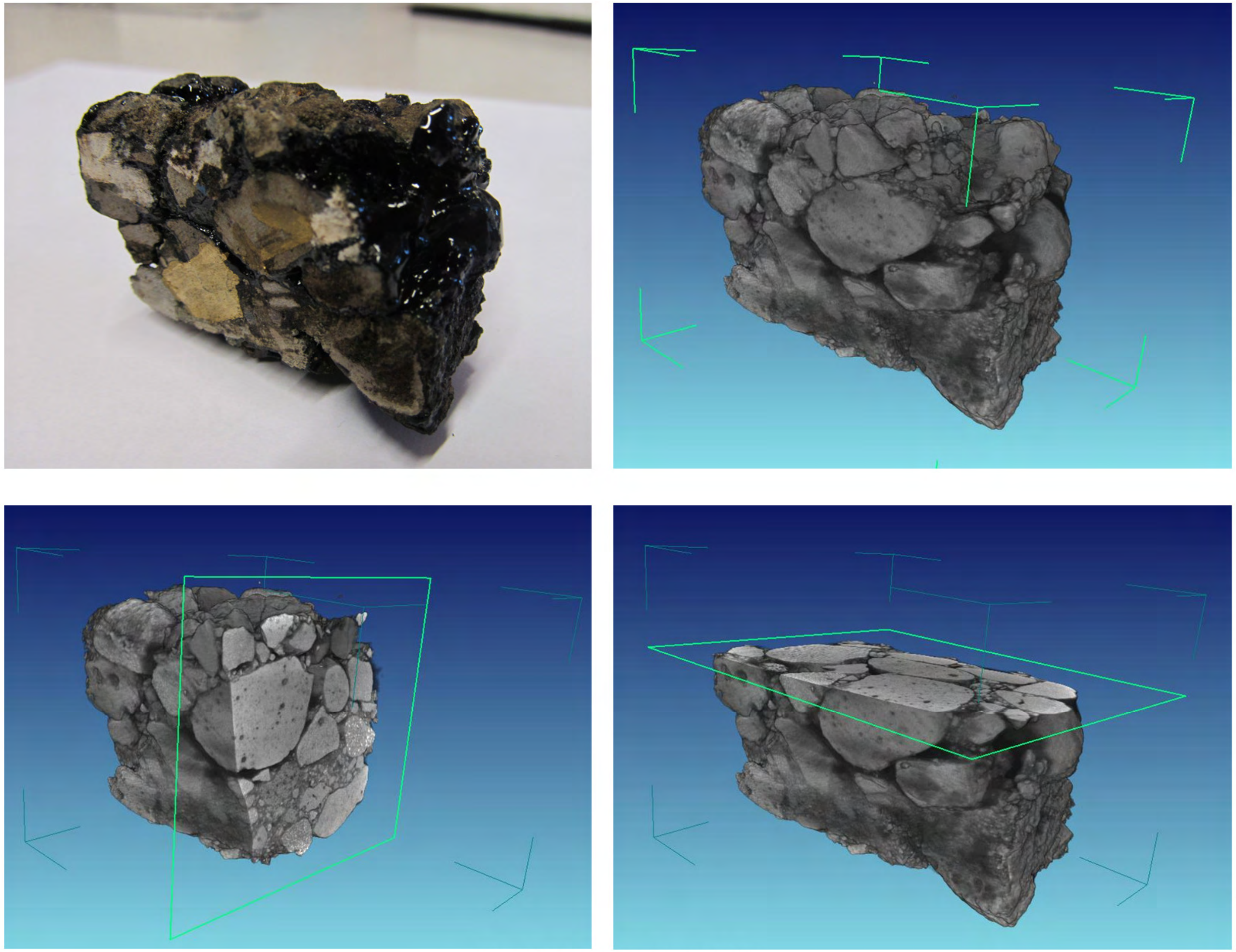




According to the visual inspection, the Road 1-K8 Section 11070 was the worst material; such surface dressing is extremely weak because of the very low viscosity of the binder causing significant binder bleeding. The surface dressing could not even take the own weight of the aggregate particles.

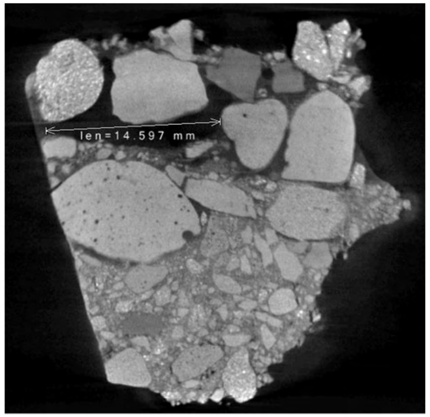
X-Ray Computed Tomography

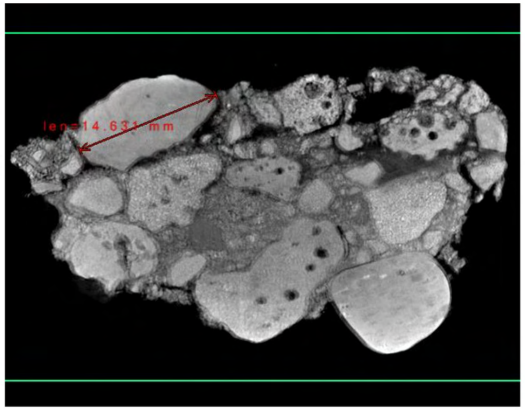
X-Ray Computed Tomography

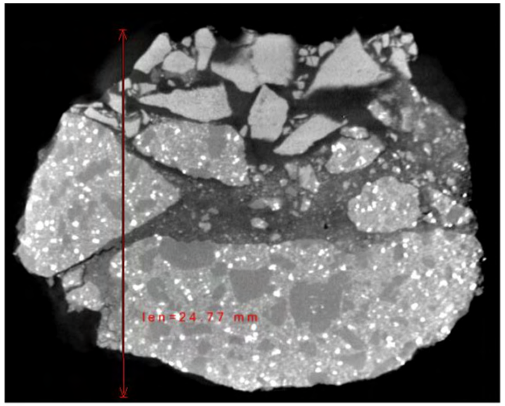


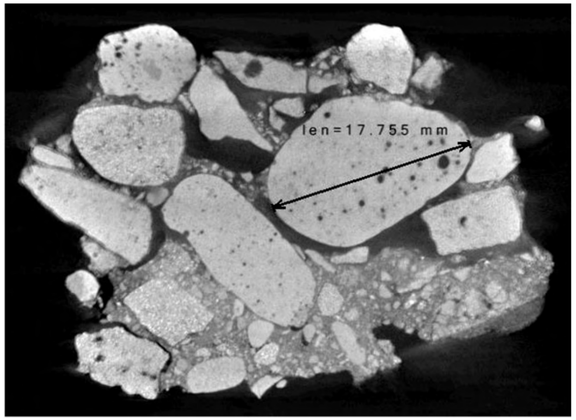
Road 75-02 Section 4575

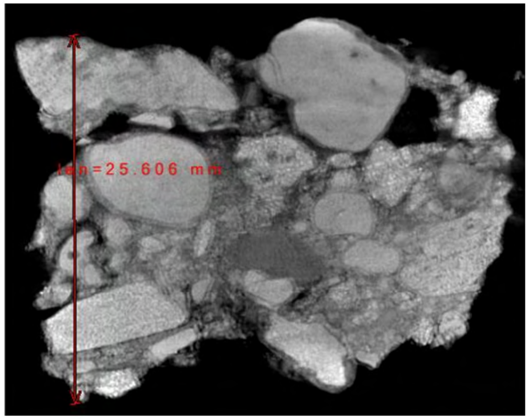
X-Ray Computed Tomography

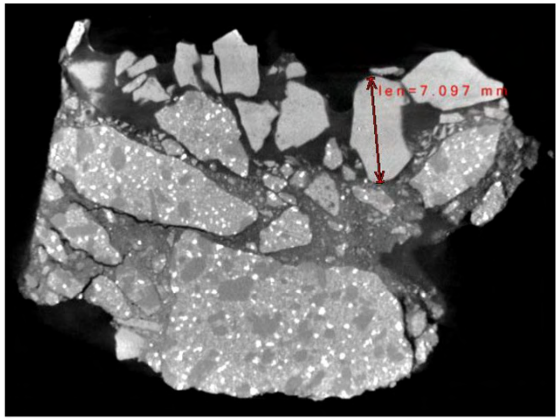






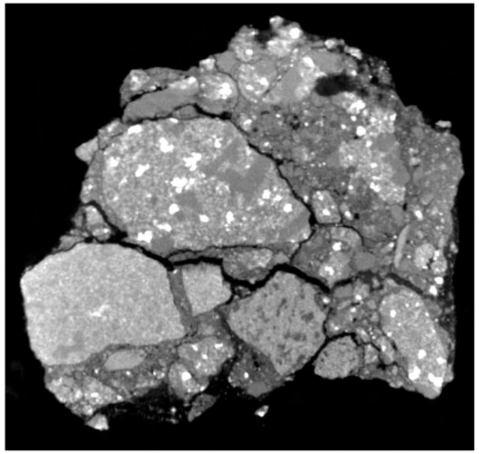


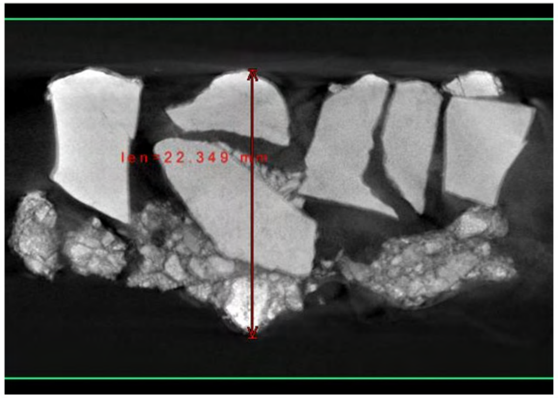


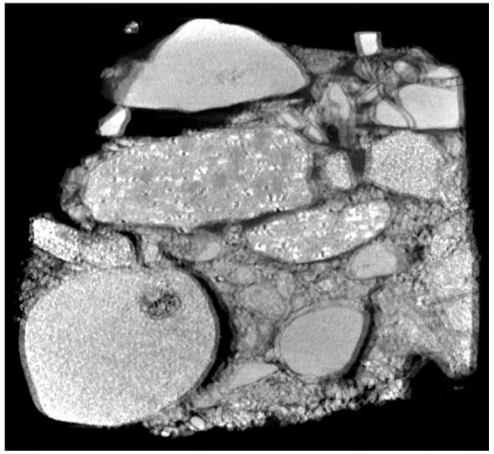


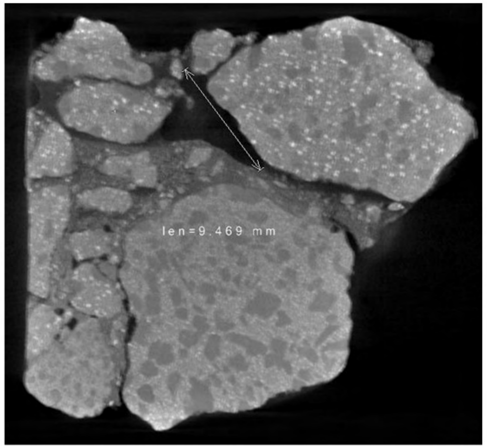
Road 75-02 Section 3870 **Road 75-02 Section 4575** **Road 1-K8 Section 5400**

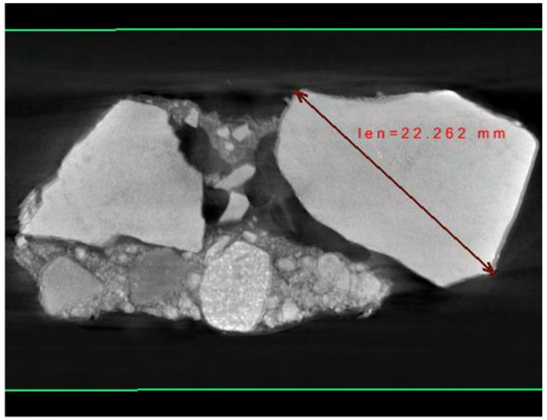
X-Ray Computed Tomography

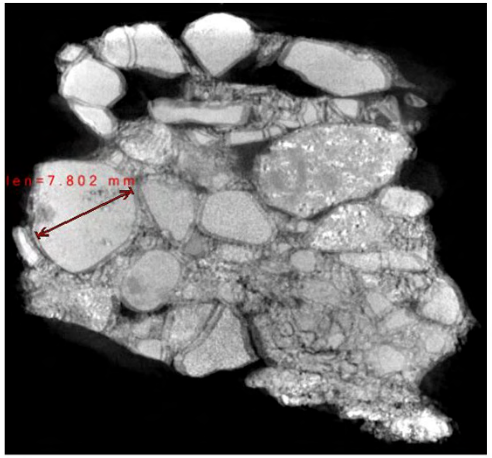






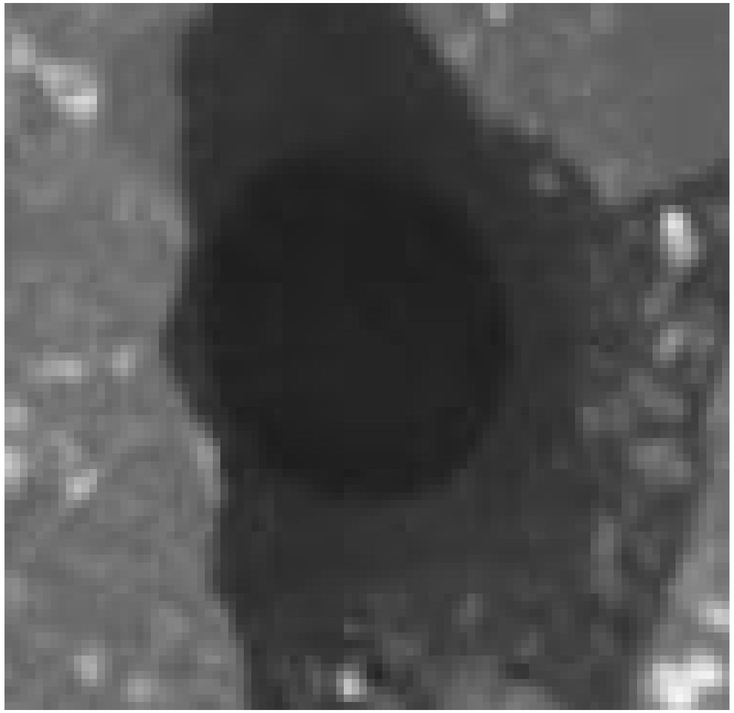


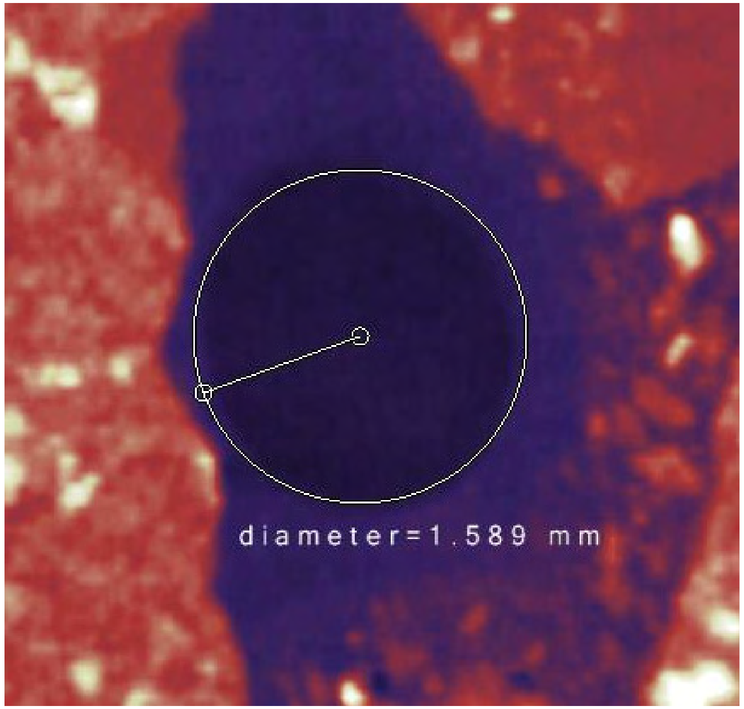




Road 1-K8 Section 8600 **Road 1-K8 Section 11070** **Road 1-K9 Section 2115**

X-Ray Computed Tomography





LABORATORY TEST RESULTS
Binder Rheology

Binder Penetration Grade

Three binder types tested in the laboratory:

Bitumen Penetration Grade 160/220 (Original) “N”

Bitumen Penetration Grade 160/220 + Ethyl Ester from fishoil 7,5 % “K8”

Bitumen Penetration Grade 160/220 + Fatty Acid Methyl Esters FAME from Rape seed oil 7,5 % “K9”

BINDERS	Penetration at 25°C (dmm)	Kinematic viscosity at 60C (mm^2/s.)
Unaged N	155	NA
Unaged K8	Too soft	10 500
Unaged K9	Too soft	15 600
Recovered Binder 1-K8 Section 11070	Too stiff	115 000
Recovered Binder 1-K9 Section 2115	Too stiff	93 500

Binder Penetration Grade

Strictly speaking, the binder N does not meet the penetration grades requirements to be classified as 160/220; however, it could also be a 160/220 originally, which after handling, heating history, and normal testing variability, it has become slightly stiffer.

The two unaged modified binders: K8 and K9 were too soft for the penetration test at 25°C; even at 15°C; consequently, they were subjected to kinematic viscosity tests. On the other hand, the recovered binders from 1-K8 Section 11070 and 1-K9 Section 2115, were too stiff to be tested for penetration at 25C and kinematic viscosity at 60°C, therefore dynamic viscosity test at 60°C was performed.

The general observation is that binders are a lot stiffer after recovery (mixing, laying, field aging, laboratory recovery); probably fairly close to the unaged original bitumen (Binder N).

Superpave Binder Grade

Binders	**N**			**K8**			**K9**		
Viscosity at 135 °C Max, 3 Pas	0.2			-			-		
PG, max pavement design temperature, °C	**46**	**52**	**58**	**46**	**52**	**58**	**46**	**52**	**58**
Dynamic shear (10 rad/s) G*/sin δ, Min 1.00 kPa	4.48	2.00	0.93	0.44	0.22	-	0.74	0.36	-
After RTFOT									
Mass loss, max 1%	o.6			2.85			1.71		
Dynamic shear (10 rad/s) G*/sin δ, Min 2.20 kPa	10.88	4.50	1.98	2.29	1.10	-	2.38	1.08	-
After PAV (100°C)									
Temperatures	**13**	**10**	**7**	**7**	**4**	**1**	**4**	**1**	**-2**
Dynamic shear (10 rad/s) G*sin δ, Max 5000 kPa	4170	6320	-	3490	3660	5530	2860	4470	6540
Min pavement design temperature, °C	**-12**	**-18**	**-24**	**-24**	**-30**	**-36**	**-24**	**-30**	**-36**
Creep stiffness (60 s)									
S, Max 300 MPa	61	178	Sample Broke	-	264	563	m-value < 0.1		
m-value, Min 0.300	0.434	0.358		-	0.363	-			
Estimated Performance Grade	**PG 52-28**			**Failed**			**Failed**		

Superpave Binder Grade

Binder N fulfilled Superpave mass loss requirement; however, the modified binders, K8 and K9, failed this criterion. This indicates that the addition of fish oil or rape seed oil dramatically increased the percent mass loss which may have negative impacts on road performance such as reduced binder-aggregate adhesive strength, bleeding and/or reduced lifetime of the surface layer.

Binder N met the DSR requirement for unaged binders, while the modified binders, K8 and K9, also failed this parameter. All the binders fulfilled the DSR specifications for both RTFOT and PAV residues.

The BBR results showed that the binders N and K8 complied the Superpave requirements; but binder K9 failed again.

Based on the Superpave grading system, the binder N was classified as PG 52-28. This suggests that the binder N is not very good to be used in hot climatic conditions; however, good low temperature performance can be expected.

The modified binders, K8 and K9, did not meet the Superpave binder requirements. It appears that the oil modification diminishes the high temperature performance of binder N; however, the low temperature performance of binder N seems to be improved with the addition of oils.

LABORATORY TEST RESULTS
Binder Chemistry

Saturated, Aromatic, Resin, and Asphaltene SARA

Sample	Saturates (wt%)	Aromatics (wt%)	Resins (wt%)	Asphaltenes (wt%)
N	9.0	49.6	22.4	19.0
K8	7.6	44.3	27.2	20.9
K9	7.1	43.6	30.4	18.9

Asphaltenes constituents seem constant in both modified and unmodified binders.

Aromatics in the original binder are approximately 5-6wt% higher as compared to modified ones.

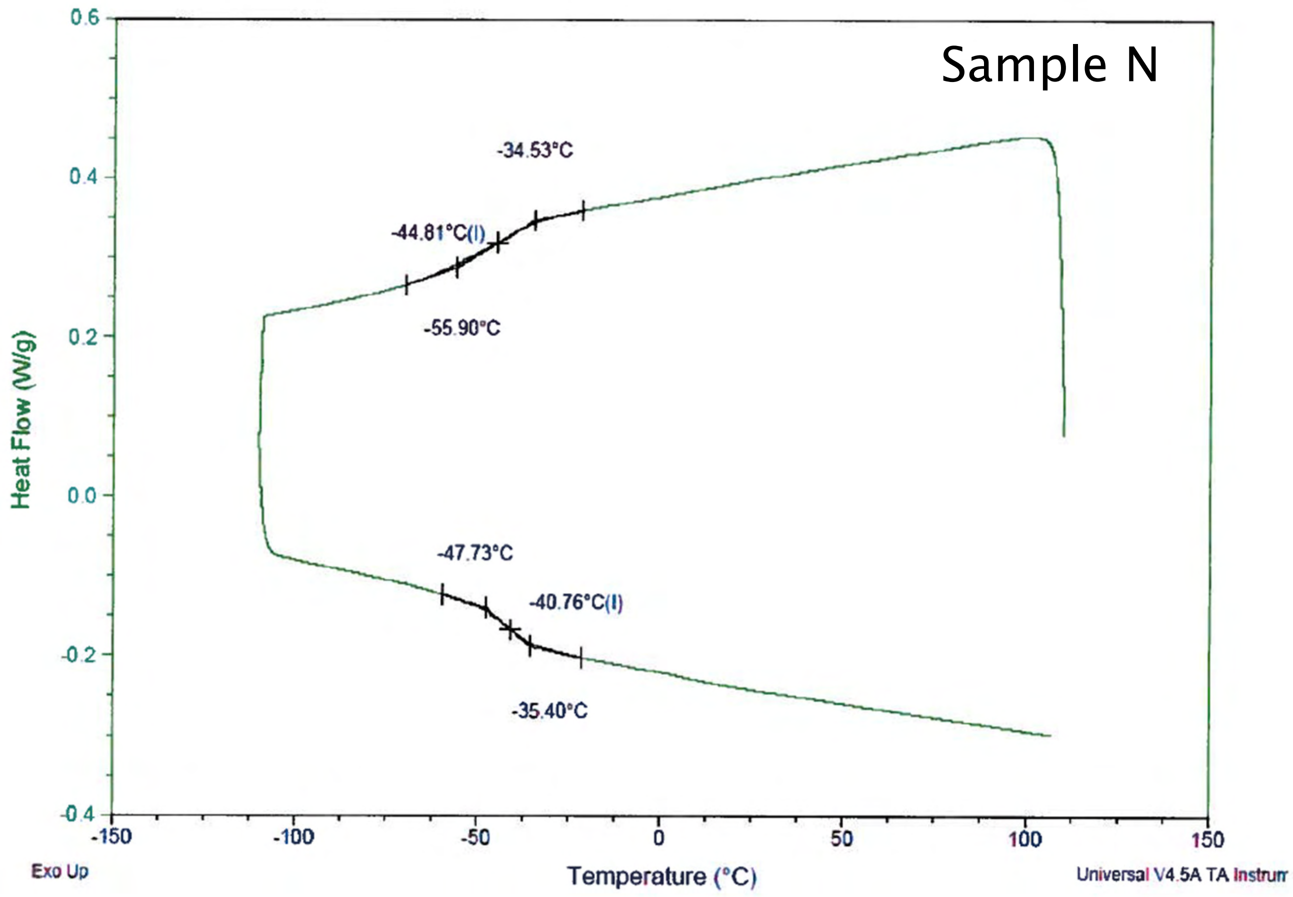
Differential Scanning Calorimetry DSC

Sample	T_g (^oC) down by DSC	T_g (^oC) up by DSC
N	-44.8	-40.8
K-8	-25.0	-25.9
K-9	-40.9	-37.4

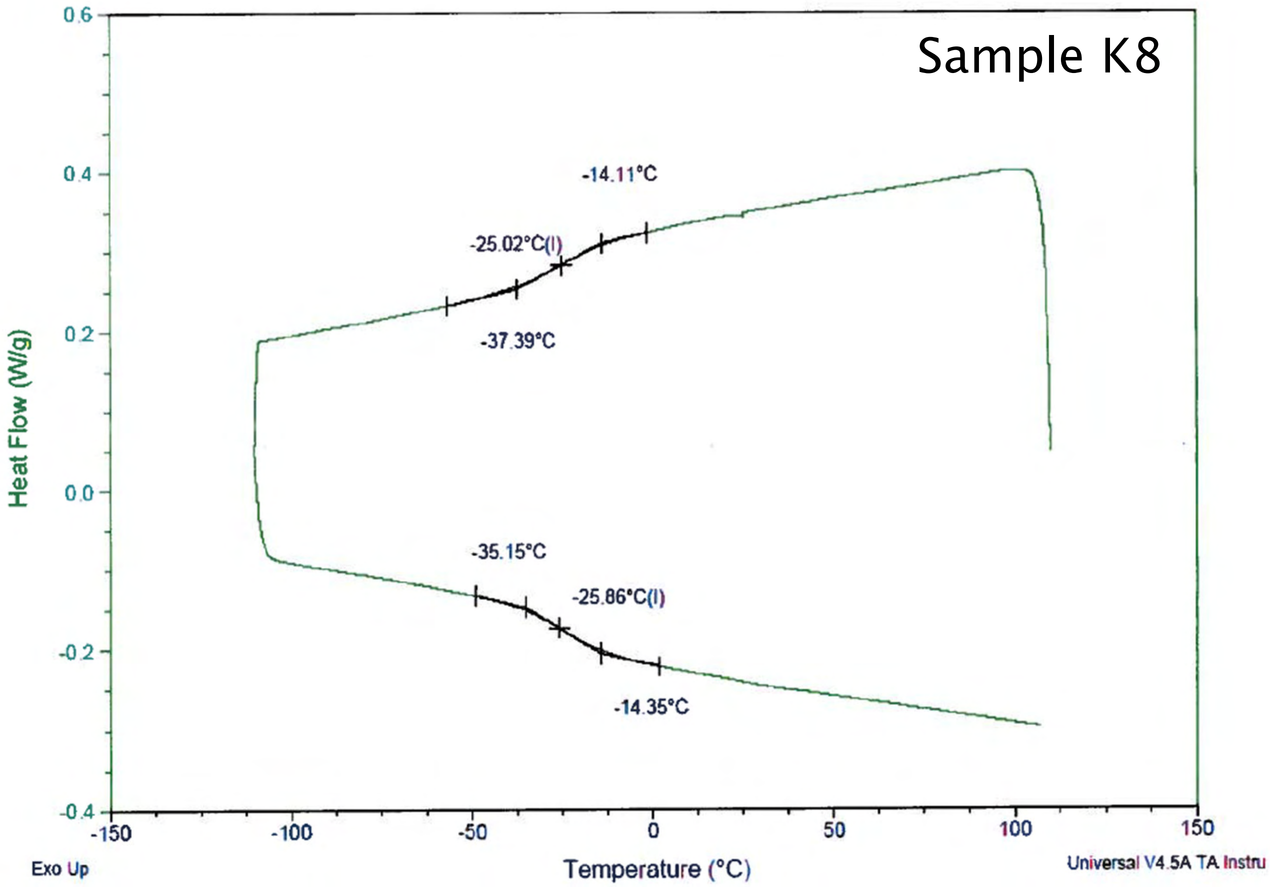
There was no indication of wax contents in all samples and glass transitions are calculated and graphs are shown below.

Figures 13 to 15 present the heat flow vs temperature plots. As these materials have short range order in their structure so they have glass transition temperatures in certain ranges and not a fixed value as in case of crystalline materials. It has been investigated in BBR test that K8 binder has T_g around −30ºC.

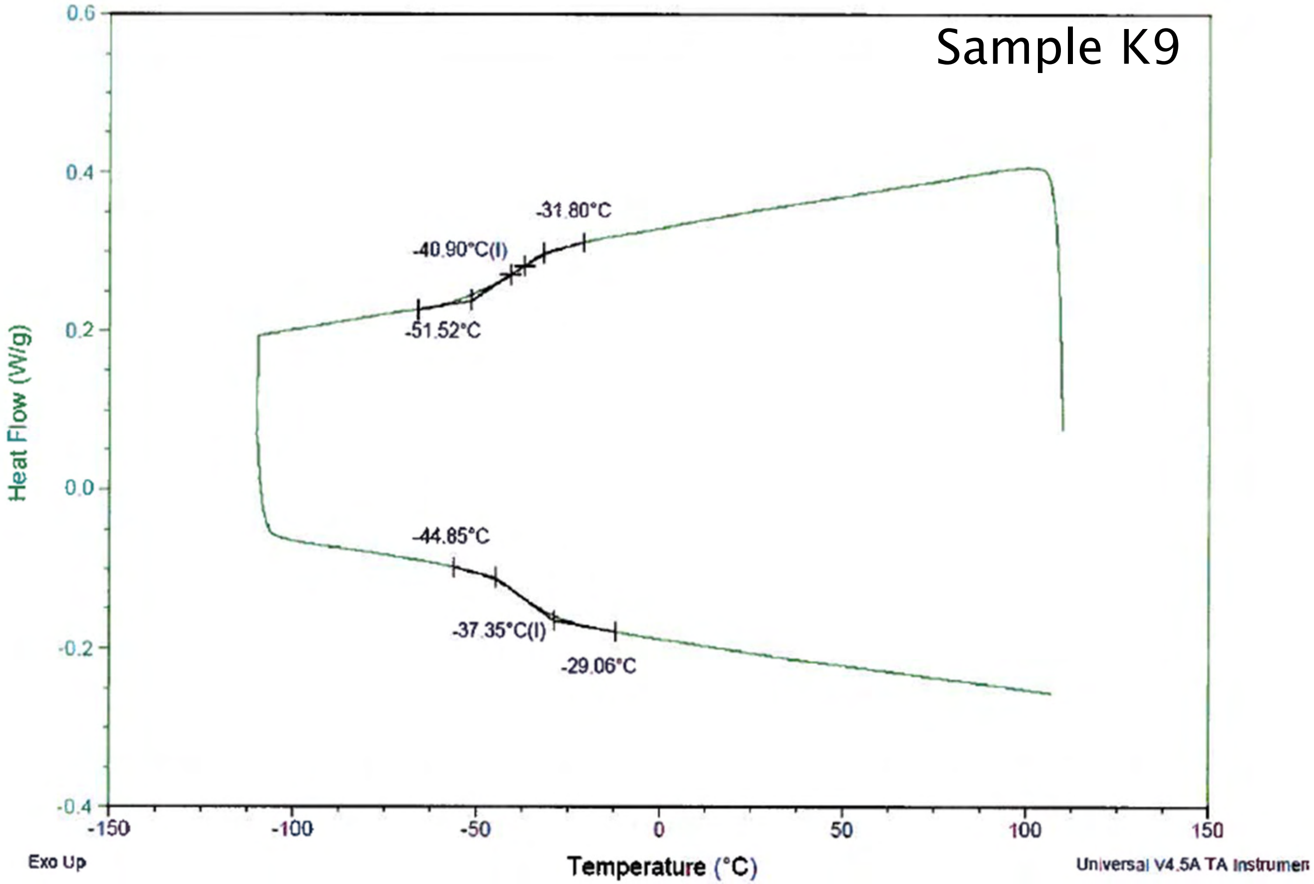
Differential Scanning Calorimetry DSC



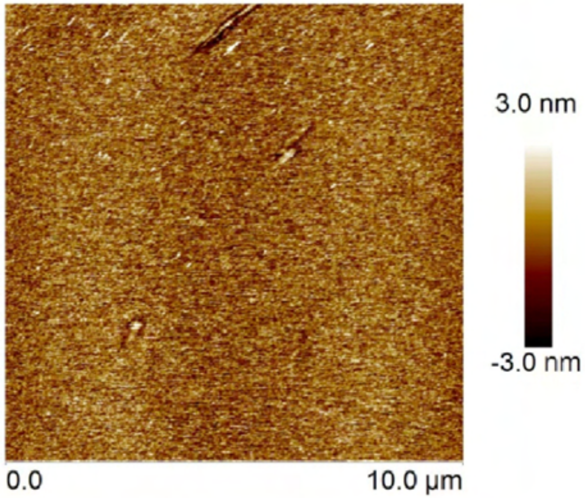
Differential Scanning Calorimetry DSC



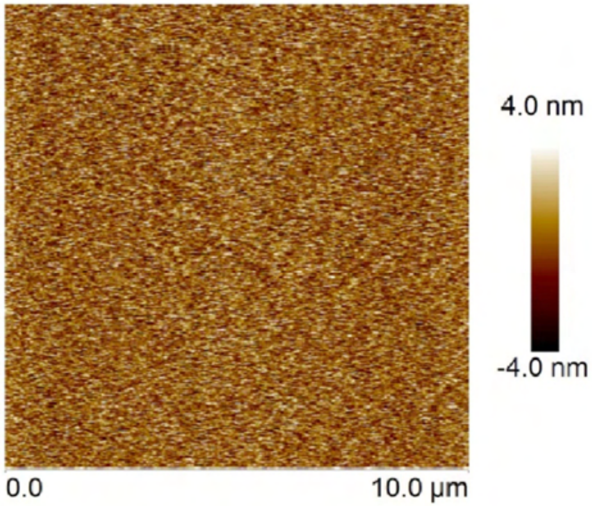
Differential Scanning Calorimetry DSC



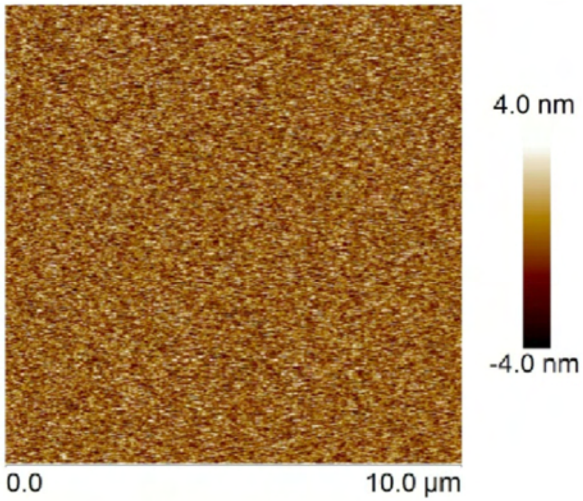
Atomic Force Microscopy AFM
Surface Topography



Bitumen N

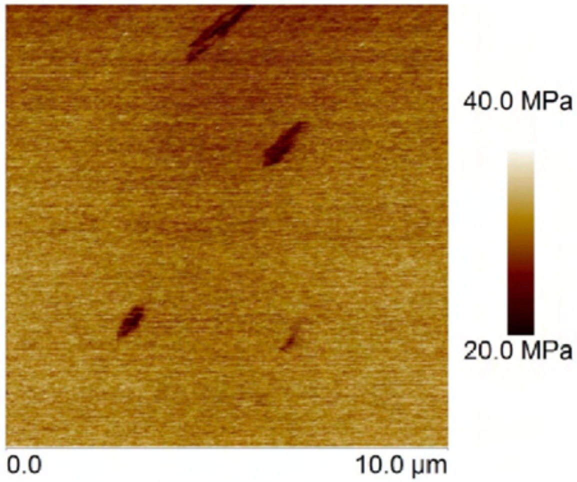


Bitumen K8

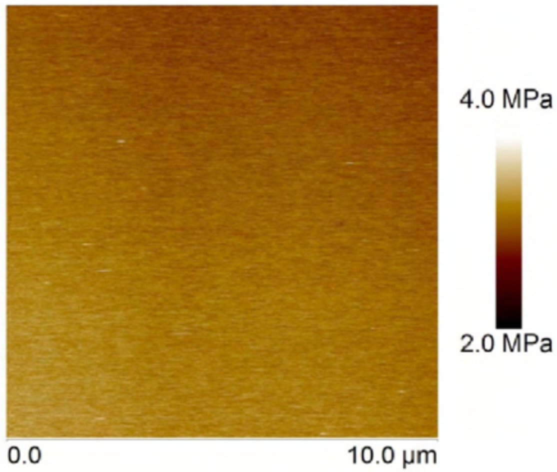


Bitumen K9

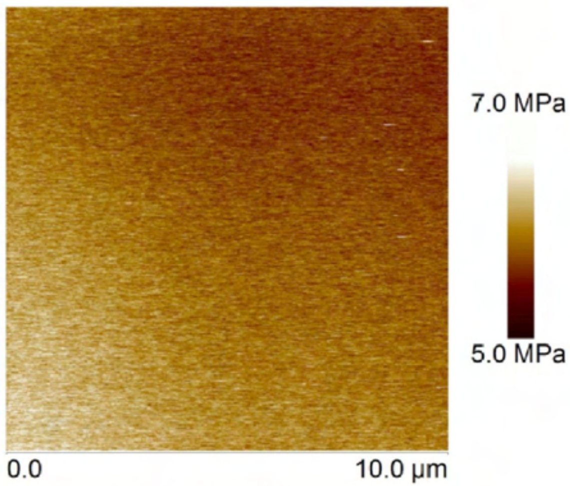
Atomic Force Microscopy AFM Surface Modulus



Bitumen N

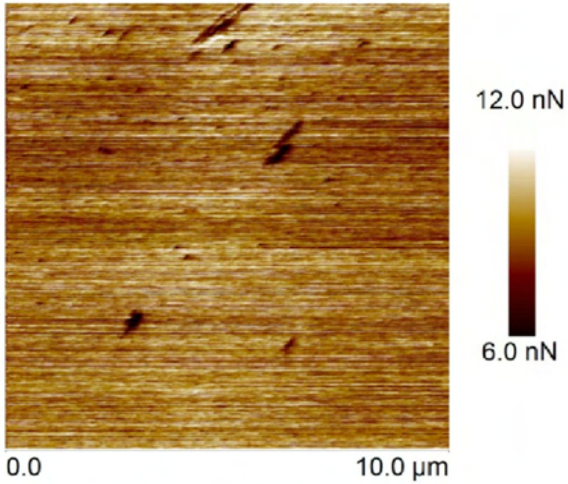


Bitumen K8

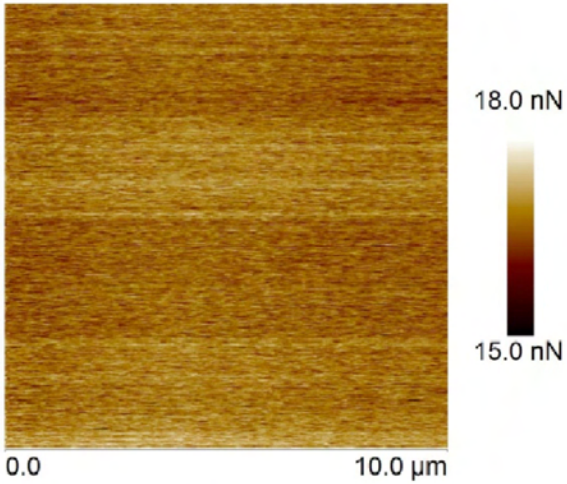


Bitumen K9

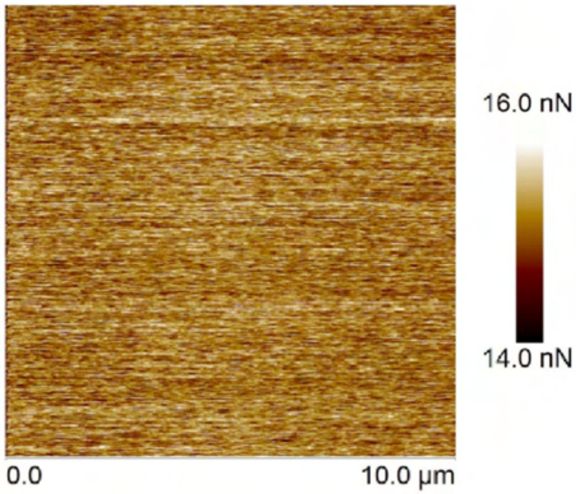
Atomic Force Microscopy AFM Surface Adhesion



Bitumen N

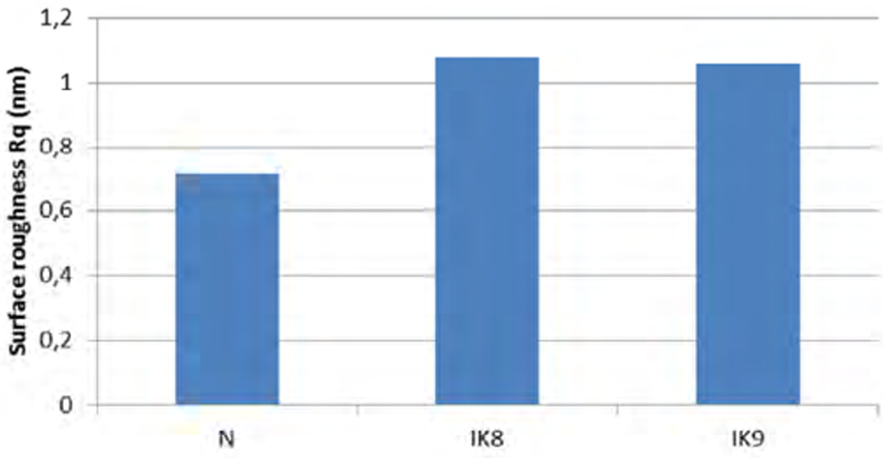


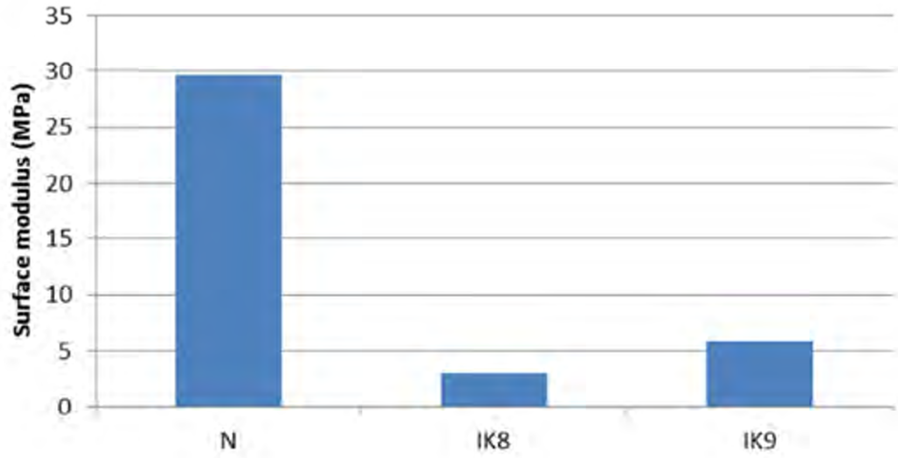
Bitumen K8

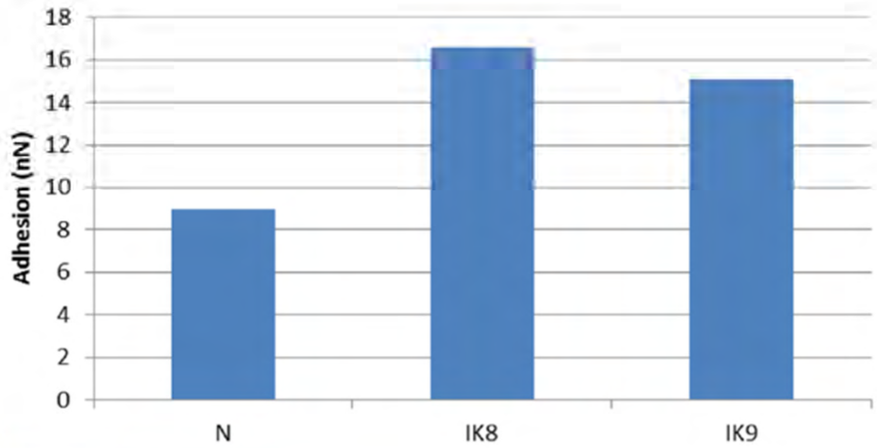


Bitumen K9

AFM Surface Micromechanical Properties







Surface Free Energy (Sessile drop)

Probe liquids used and their surface tensions

Probe Liquids	Total Liquid IFT (l) [mN/m]	Dispersive Part (d) [mN/m]	Polar Part (p) [mN/m]
Diiodo-Methane	50.80	48.50	2.30
Formamide	58.20	39.50	18.70
Water	72.80	21.80	51.00

Surface energy contributions of bitumen 160/220

Bitumen (160/220)	Total IFT [mN/m]	Dispersive [mN/m]	Polar [mN/m]
Tangent-1 Method	41.9 ± 1.27	41.9 ± 0.74	0.0 ± 0.53
Circle Method	43.5 ± 1.19	43,4 ± 0.84	0.1 ± 0.35
Height-Width Method	44.5 ± 0.9	44.5 ± 0.53	0.0 ± 0.37

Mean contact angles of probe liquids on the surface of bitumen 160/220

Bitumen Substrate	Mean contact Angle [deg.] (Tangent-1 Method)	Mean contact Angle [deg.] (Circle Method)	Mean contact Angle [deg.] (Height-Width Method)	Probe Liquids
160/220	31.2	26.0	24.3	Diiodo-Methane
	74.5	71.1	71.9	Formamide
	93.4	90.2	91.7	Water

Surface Free Energy (Sessile drop)

Mean contact angles of probe liquids on the surface of K8 binder

K8 Substrate	Mean contact Angle [deg.] (Laplace-Young Method)	Mean contact Angle [deg.] (Circle Method)	Mean contact Angle [deg.] (Height-Width Method)	Probe Liquids
K8	34.4	28.0	28.8	Diiodo-Methane
	76.5	63.5	63.8	Formamide
	70.4	65.8	63.6	Water

Surface energy contributions of K8 binder

K8	Total IFT [mN/m]	Dispersive [mN/m]	Polar [mN/m]
Laplace-Young Method	36.4 ± 3.2	31.5 ± 1.93	4.8 ± 1.27
Circle Method	41.7 ± 2.0	34.6 ± 1.32	7.1 ± 0.67
Height-Width Method	41.7 ± 3.03	33.5 ± 1.82	8.2 ± 1.21

Surface Free Energy (Sessile drop)

Mean contact angles of probe liquids on the surface of K9 binder

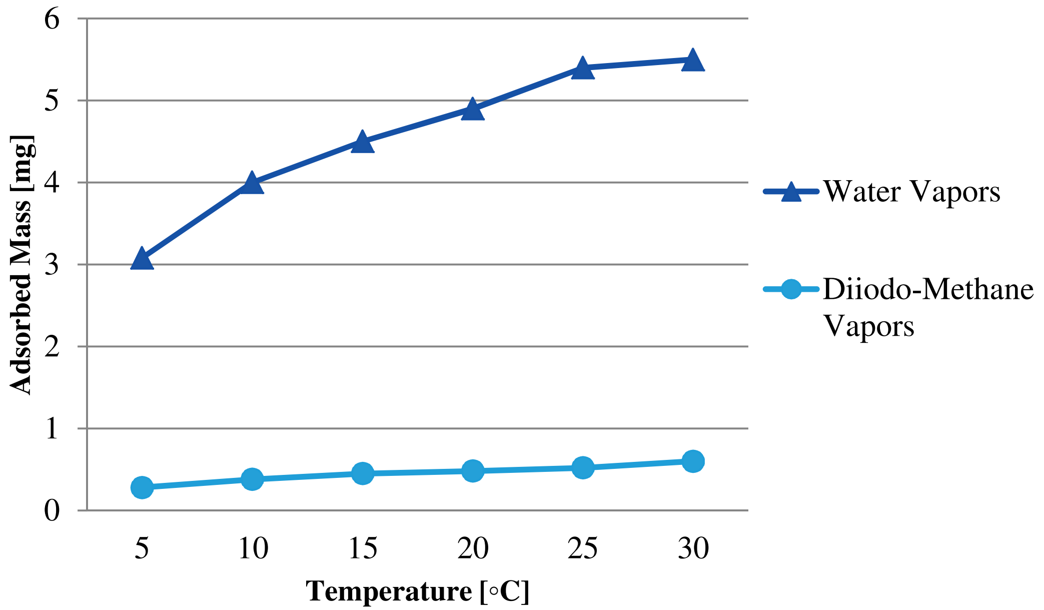
K9 Substrate	Mean contact Angle [deg.] (Laplace-Young Method)	Mean contact Angle [deg.] (Circle Method)	Mean contact Angle [deg.] (Height-Width Method)	Probe Liquids
	30.6	29.9	30.0	Diiodo-Methane
K9	62.0	62.4	62.1	Formamide
	67.2	62.1	61.9	Water

Surface energy contributions of K9 binder

K9	Total IFT [mN/m]	Dispersive [mN/m]	Polar [mN/m]
Laplace-Young Method	41.4 ± 1.62	34.3 ± 0.96	7.1 ± 0.66
Circle Method	42.1 ± 4.72	32.8 ± 2.51	9.2 ± 2.21
Height-Width Method	42.2 ± 4.84	32.8 ± 2.42	9.4 ± 2.41

The surface of pure binder is dispersive or non-polar whereas modified binders showed high polarity. Possible reasons could be solubility issue of ethyl ester from fish and rape seed oil in bitumen. It appears at the surface as a cream due to light molecular weight. This is why, stone surfaces were covered with oil as both stones and ethyl esters were polar. As a result, there was poor adhesion

Universal Sorption Device USD



An increment of temperature or P/Po (ratio between partial pressure and equilibrium pressure), leads to an increase in mass adsorbed at the stone surface. In case of water vapors, this mass increase was dominant as compared to the diiodo-methane vapors.

Universal sorption testing confirmed that stone surface is polar and it has high affinity toward polar liquid such as water as compared to dispersive liquid such as diiodo-methane. Hence, stone surface was covered with polar oil phase rather than bitumen which led to debonding.

Conclusions

Binder rheology

Strictly speaking, the N binder does not meet the penetration grades requirements to be classified as 160/220; however, it could also be a 160/220 originally, which due to several causes has become slightly stiffer. Binders are a lot stiffer after recovery (mixing, laying, field aging, laboratory recovery); probably fairly close to the unaged original bitumen (Binder N).

The binder N was classified as Superpave PG 52-28; conversely, the modified binders, K8 and K9, did not meet the Superpave binder specifications. Binders that do not comply with Superpave requirements may have negative effects on the road performance such as reduced binder-aggregate adhesive strength, bleeding and/or reduced lifetime of the surface layer.

Conclusions

Binder Chemistry

The chemical characterization of the binders revealed enhanced surface polarity due to the solubility issues of ethyl ester from fishoil with the original bitumen (binder N). The density difference between ethyl ester and bitumen popped the polar oil phase at the surface as cream.

Furthermore, universal sorption studies confirmed the polar surface of stones because of their high affinity toward polar liquid such as water as compared to dispersive liquid such as diiodo-methane. Hence, stone surface was covered with polar oil phase rather than bitumen; which led to debonding of asphalt binder from aggregate surface. This problem can be substantially aggravated when the surface dressings are exposed to moisture or water.

Conclusions

X-Ray Computed Tomography

Two major problems: debonding between aggregates and binder, as well as significant binder bleeding were clearly identified and appear to play a key role diminishing the structural capacity of the surface dressings. Other concerns are related with the use of rounded and/or relatively light/weak aggregate particles in the surface dressings.

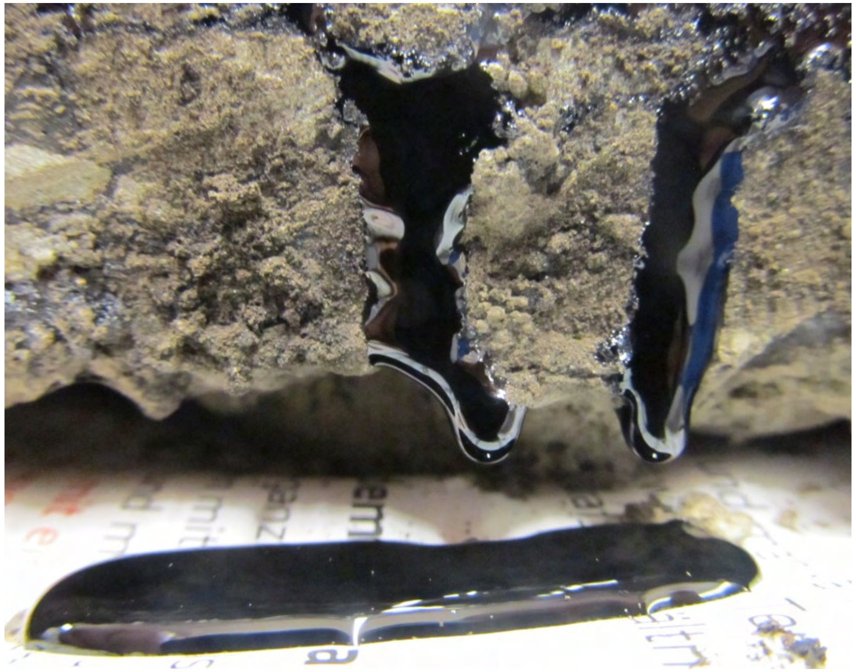
Recommendations

In order to prevent future damage of surface dressing, the following recommendations should be followed:

- Any modified asphalt binder to be used for pavements must meet the respective binder specifications for construction of roads. For instance, both of the modified binders evaluated in this work, K8 and K9, did not meet the Superpave binder requirements.

- Aggregates must meet the respective guidelines for road construction; especially toughness/abrasion resistance.

- The used of rounded particles for roads construction must be minimized.



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Nov-8-2013