PRACTICAL LABORATORY AGEING METHOD FOR POROUS ASPHALT

Martin F.C. van de Ven[1], Jan L.M. Voskuilen[2] and Maarten M.J. Jacobs[3]

[1]Martin F.C. van de Ven, Delft University of Technology, Faculty of CiTG, the Netherlands, m.f.c.vandeven@tudelft.nl;
[2] Jan L.M. Voskuilen, Rijkswaterstaat, Centre for Transport and Navigation, the Netherlands, jan.voskuilen@rws.nl;
[3] Maarten M.J. Jacobs, BAM Wegen, the Netherlands, m.jacobs@bamwegen.nl.

ABSTRACT

During the mix design phase of porous asphalt (PA) pavements, it is imperative to know the PA properties after field ageing. This will help to optimize pavement performance and to increase the confidence level of the pavement design. A study was initiated to develop a laboratory ageing procedure for PA mixtures. This procedure should be able to mimic 10 years of field ageing within 10 days in a laboratory using only standard lab equipment. Conventional empirical and sophisticated fundamental rheological tests (penetration, softening point ring and ball, DSR), chemical tests (FTIR) and adhesion tests (Wilhelmy plate) were conducted on binders recovered from field cores and on binders recovered from specimens that were subjected to laboratory ageing.
Finally two ageing procedures were developed: one for the mortar in PA and one for the loose PA mix. To accomplish the same ageing of the bitumen as in 10 year period in the field, the mortar has to be heated in an oven for 2 hours at 165°C and then be placed in a PAV at 90°C for 7 days under air pressure of 2.1 MPa. The loose PA mix has to be heated for 44 hours at 135ºC to accomplish the same degree of ageing.
In this paper the research program is discussed and the findings are presented.

Keywords : ***ageing, porous asphalt, rheology,***

1. INTRODUCTION

Since 1990 the single-layer Porous Asphalt (thickness 50 mm) is applied on Dutch motorways as a standard wearing course for noise reduction reasons. Generally PA has a lower service life in comparison with dense asphalt mixtures. Loss of aggregates from the pavement surface, called raveling, is the most common damage of PA. There are several factors contributing to the accelerated failure or raveling of PA. The ageing of the mortar is believed to be one of the main reasons for raveling. It increases the chance of damage development because of applied traffic loading and thermal stresses at low temperatures.
During the design phase of pavement, it is imperative to know beforehand what the properties of the pavement will be after field ageing. This will help to minimize maintenance costs and to increase the confidence level of the design of the pavement. It is evident; therefore, that there is a need for an accelerated laboratory ageing process that can simulate binder properties similar to that of field aged binder. As a result, this study was initiated and the objective was to develop a laboratory ageing procedure which is able to mimic binder properties of 10 years field aged PA pavement within 10 days in a lab using standard laboratory equipment.

In this paper first the research program will be discussed [1]. Then the characteristics of the reference material will be discussed. In the last part of this paper the results of the various ageing procedure on the bitumen, mortar and mix properties will be discussed en finally the conclusions and recommendations will be formulated.

2. RESEARCH PLAN

To achieve the objective, three different ageing protocols were proposed and investigated in the first phase of the research program. Then, based on the results, two ageing protocols were further refined and examined:

1. The first protocol was ageing of mortar by using a PAV.
   First, materials (bitumen, filler and sand) are mixed at a temperature of 155 ºC for 3 minutes. This is just to make the mortar ready for short term ageing. After preparation of the mortar, 50 grams has been poured on to a circular steel plate of diameter 140 mm. The sample was subjected to a short term ageing (STA) at a temperature of 165ºC in an oven for a duration of two hours. This period has been chosen arbitrarily. Subsequent to a short term ageing, a pressure ageing vessel PAV with a temperature of 80ºC and a pressure of 2.1 MPa (300 psi) has been used for 7 days for long term ageing (LTA) of the specimens. After the LTA the specimens have been heated in an oven at a temperature of 150ºC for 30 minutes to ease the removal of the aged samples from the circular plates;
2. The second protocol was ageing of a compacted PA mixture in a PAV.
   This ageing protocol concerns with ageing of a compacted mixture by using a pressure ageing vessel. The loose asphalt mixture has been placed in an oven with a temperature of 135 ºC for 4 hours (short term ageing). Every hour the mixture has been turned and stirred to get a more homogeneous mixture. This procedure is also used in the SHRP studies. Then the short term aged mix has been heated up to a temperature of 155ºC and compacted with gyratory compaction to produce a cylindrical specimen of 100 mm diameter and a height of 50 mm. These compacted specimens are placed in a PAV with a pressure of 2.1 MPa for duration of 7 days. During this period, the temperature is increased step by step (70ºC for the first 3 days and 80ºC for the last 4 days);
3. The third ageing protocol was ageing of a loose asphalt mixture in an air forced ventilated oven.
   In this protocol, the ageing procedure from RILEM TC-ATB-TG5 [2] has been adopted. The only deviation from this technical group procedure is the mixture used for ageing was 5 kg instead of 15 kg in a steel box with a dimension of 50*30*8 cm. The weight is reduced to minimize the thickness of the loose mixture so that it gives stronger ageing. In the ageing procedure, the loose mix is placed in an air-draft ventilated oven for 4 hours at 135ºC to simulate short term ageing. Each hour the material is stirred for 1 minute and placed back into the oven. The stirring action is only for homogenization. Then the loose mixture has been placed in an air ventilated oven at a temperature of 85ºC for duration of 7 days (long term ageing).

These three ageing procedure are carried out and after that the results were evaluated. Based on the experiences some new ageing procedures are defined and tested.

In choosing the ageing protocols, the ageing influence of aggregate, sand and filler or at least sand and filler was aimed to be included. It is for instance known [3] that hydrated lime,$Ca(OH)_2$, in the PA mixture has an influence on the rheological behaviour of the bitumen itself. In the Netherlands the filler content has to contain at least 25% of hydrated lime. That is the reason why mortar and mixture and not only the bitumen was aged.

In order to compare field and laboratory ageing, rheological, chemical and adhesion tests were conducted on binders recovered from field cores and on binders recovered from specimens that were subjected to laboratory ageing. These tests were:

1. Conventional empirical rheological tests: penetration (EN 1426) and softening point ring and ball (EN 1427);

2. Fundamental mechanical test: the complex shear modulus and phase angle determination of the binders at different temperatures using the Dynamic Shear Rheometer (EN 14470).
   The test is conducted at seven temperatures (-10, 0, 10, 20, 30, 40 and 50ºC). Every test is carried out at frequencies ranging between 0.01-400 rad/s. For lower temperature (less than and including 20ºC), an 8 mm diameter plate has been used and for high temperatures (greater than 20ºC) a plate with a diameter of 25 mm has been used. The thickness of bitumen specimens tested was 1 mm and 2 mm for a respectively plate diameter of 25 mm and 8 mm. Before starting the test and during a change in test temperature, the samples were left for a minimum period of 10 minute after reaching a test temperature for conditioning purpose.
   The results of the DSR-tests in this paper are presented in the form of the mastercurve of the stiffness using the shift factors of the WLF-approach;
3. The chemical characterization of binders was conducted using Fourier Transform Infrared spectroscopy (FTIR).The main reason for using FTIR is the fact that it is believed that ageing affects the chemical composition of the mortar. Infrared spectroscopy is a widely used technique to identify functional groups in organic compounds. It is a valuable tool to identify the chemical composition of materials at molecular level. FTIR spectroscopy makes use of the Infrared part of the electromagnetic spectrum. Absorption of this lower energy radiation causes vibrational and rotational excitation of groups of atoms within the molecule. In analysing the FTIR-results, the procedure of De la Roche [4] is used;
4. The surface energy determined by means of Wilhelmy plate test has been used as an indicator for the adhesion property of bitumen.
   The Wilhelmy plate test method [5] is based on kinetic force equilibrium when a thin plate, suspended from a highly accurate balance, is immersed or withdrawn from a liquid (water, glycerol and diiodomethane) at a very slow and constant speed. The dynamic contact angles that develop between the bitumen coated glass plates and liquids are obtained. The dynamic contact angle measured during the immersion process is called the advancing contact angle (a wetting process), while the dynamic contact angle measured during the withdrawal process is called the receding contact angle.

The possibility that the use of a standard bitumen recovery process like soxhlett extraction may have influence on the recovered bitumen was also investigated. The same rheological and chemical tests as mentioned before were carried out on bitumen sample which passed through the standard recovery process and on bitumen sample which is not exposed to the recovery process.

3. PROPERTIES OF THE REFERENCE AND LABORATORY MATERIALS

In order to compare field ageing with accelerated laboratory ageing, cores were taken from a PA pavement taking into account the age or service period of the pavement. Specimens from a 10 year old PA pavement were cored and used as reference material. The cores were taken from a motorway in order to ensure that they were all exposed to similar conditions in the field. Motorway A4 near Burgerveen in the Netherlands was chosen for sampling of the specimens. All the cores were taken from the emergency lane of the pavement in order to avoid the effect of traffic load and noxious waste. The effect of clogging due to the absence of traffic of the emergency lane on the aging of the mortar is negligible: in the Netherlands the emergency lane is kept clean during its life span using vacuum cleaners.

The field PA specimens were horizontally cut into two segments: an upper and a lower part. The binder recovery process was conducted separately for these two parts of the specimens. In this research the upper part of the PA layer is given special attention since raveling takes place at the surface of the pavement.

From the 10 years old PA that was available on the A4 motorway, the mix components are known. The same constituent materials are used again in this research program. In table 1 the an overview of the mix composition and constituent materials is given.

Table 1: Mix composition of the PA on the A4 motorway near Burgerveen

Mix component	Mass percentage in the mix [%]	Density [kg/m^3]
Crushed gravel river Rhein 11/16	17.8	2650
Crushed gravel river Rhein 8/11	41.3	2659
Crushed gravel river Rhein 4/8	22.6	2665
Granite crusher sand	10.1	2650
Wigro 60K filler containing 25% $Ca(OH)_2$	3.9	2570
Pen grade bitumen 70/100	4.3	1035
Total	100.0	

A penetration grade bitumen 70/100 was used in this study. This bitumen is commonly used in the Netherlands in a PA mix. The properties of this bitumen are presented in table 2.

Table 2: Properties of the used bitumen

Bitumen	Penetration [0.1 mm]	Softening Point [ºC]	Penetration Index [-]	Density [kg/m^3]
70/100	70	44.6	-1,9	1025

The material preparation can be broadly categorized into two main groups based on the type of specimens to be aged. The first group dealt with ageing of the mortar (protocol 1) and the second one was dealing with ageing of the PA mixture (protocol 2 and protocol 3).

The mortar used in this study consisted of bitumen, filler and fine sand, mixed at a mass ratio shown in table 3. This mass ratio is equivalent to the mass ratio in the PA mix from the A4 motorway as given in table 1. The fine sand used to prepare the mortar is smaller than 0.5 mm. The reason for this is that according to previous research [6], it was found that the aggregate skeleton of PA only consists of aggregates larger than 0.5 mm. This means that the mortar in PA can be defined as a mixture of bitumen and any of aggregates smaller than 0.5 mm. The sand percentage (m/m) in the mortar has been determined by using the total sand percentage in the asphalt mixture composition and by multiplying this value with the percentage of sand fraction less than 0.5 mm from the sand fraction. The final mix composition for the mortar is shown in table 3.

Table 3: Mortar composition of the Motorway A4 PA mixture.

Mortar components	Mass percentage in the mortar [%]
Granite crushed sand (between 0.5 mm and 0.063 mm)	28.4
Filler (Wigro 60K) containing 25% $Ca(OH)_2$	34.1
Pen grade bitumen 70/100	37.5
Total	100.0

4. TEST RESULTS

This chapter discusses the results and analysis of test data obtained from the first three preliminary ageing protocols examined in this study. The data analysis will compare recovered bitumen test results for the three laboratory ageing methods with field aged bitumen properties. But first the effect of the binder recovery procedure on the properties of the binder will be discussed.

4.1 Effect of the binder recovery procedure on the properties of the binder

There was a concern that a standard binder recovery procedure can have a significant influence on the properties of the recovered bitumen. For that reason, a test program has been deployed to verify the effects of binder extraction and recovery on the recovered binder before using the standard binder recovery procedure in this research.

The virgin 70/100 bitumen used in this study has been short term aged by using RTFOT in accordance with EN 12607-1. The short term aged bitumen sample was divided into two halves. The first half has been used for the standard extraction and recovery process (mixing with methylene chloride and recover the bitumen using a rotary evaporator distillation apparatus) and the other half was kept untouched. The effect of binder recovery can easily be seen (if any) from the comparison of the test results of the two samples i.e. the short term aged sample (STA) and the short term aged and then recovered sample (STA + recovered). Empirical and fundamental rheological tests as well as chemical tests have been conducted on these two samples and the results are shown in table 4 and figures 1 and 2.

Table 4: Test results to determine the influence of binder recovery procedure on binder properties

Properties	Virgin	STA	STA+ recovery
Empirical tests			
Penetration [0.1 mm]	70	50	48
Softening Point [ºC]	44.6	48.6	49.0
Penetration Index [-]	-1.9	-1.6	-1.6
WLF parameters DSR-test			
C1	n.a.		13.550
C2	n.a.	104.655	108.046
FTIR tests			
Ico		0.0000	0.0000
Iso		0.0321	0.0335
1700 peak height		0.0000	0.0000

1030 peak height		0.0058	0.0058

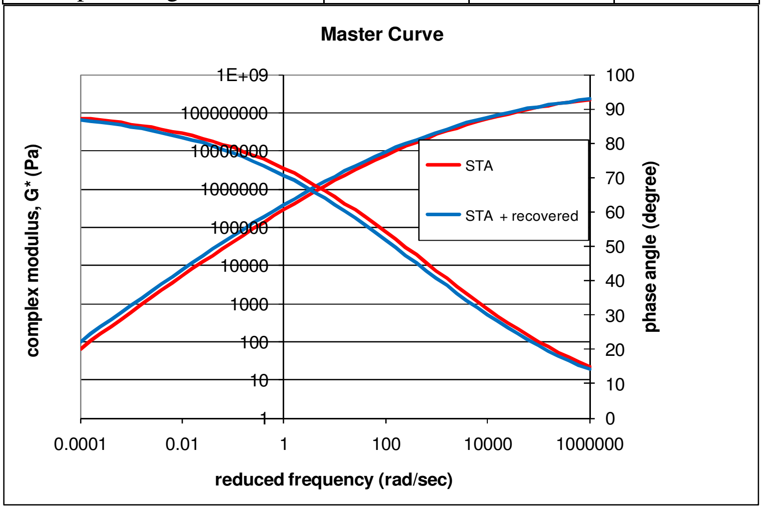

Figure 1: Master curves for STA and STA + recovered bitumen specimens at a reference temperature of 20 °C

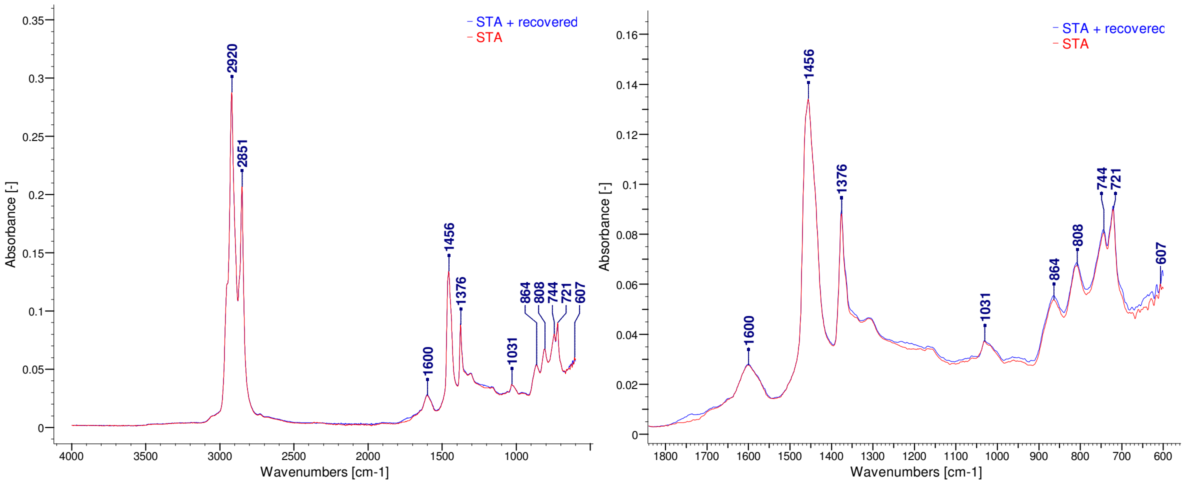

Figure 2: Infrared spectra of STA and STA+recovered bitumen in total and in 'finger-print' region

All the test results demonstrate that the effect of binder recovery on the properties of the bitumen is not significant. Therefore, the standard binder recovery method using dichloromethane is deployed in this research.

4.2 Comparison of preliminary ageing protocols with field result

The field specimens were horizontally cut into two segments both approximately 2,5 cm thick. The upper and lower parts and the binder recovery process was conducted separately for these two parts of the specimens. In this research the upper part of the PA layer was given special attention since raveling takes place at the surface of the pavement.
The recovery of the bitumen from specimens aged under laboratory ageing protocols was conducted in the same manner as the recovery of bitumen from field materials. So, the correlation of the binder properties will be based on the same approach which avoid any bias with respect to the effects of recovery procedure on the binder property.
The effect of the 3 ageing protocols have been examined. From the three preliminary proposed ageing protocols, protocol 2 failed the competition at the early stage due to the fact that the specimens were highly deformed/damaged and bitumen was drained to the bottom of the plate after 7 days. Recovery of bitumen was not possible anymore so comparison with field data was not an option. Therefore it was decided to delete protocol 2 as candidate for a ageing protocol. Consequently, only the results from protocol 1 and protocol 3 are discussed in the subsequent paragraphs.
In table 5 the results of the ageing protocols 1 and 3 are compared to the in field ageing specimens with a life span of 10 years.

Table 5: Test results field cores and ageing protocols 1 and 3

Property	Protocol 1	Protocol 3	Field aged upper part	Field aged lower part
		Emperical tests		
Penetration [0.1 mm]	19	27	12	17
Softening Point [ºC]	60.0	55.2	67.6	63.0
Penetration Index [-]	-1.0	-1.3	-0.5	-0.6

Property	Protocol 1	Protocol 3	Field aged upper part	Field aged lower part
WLF parameters DSR-test				
C1	11.6	17.8	28.7	20.4
C2	96.9	137.0	206.1	169.2
Wilhelmy Plate tests				
ã^{total} (advancing)	26.79	29.86	33.33	38.50
ã^{LW} (advancing)	24.89	29.86	33.33	37.04
ã$^{+}$ (advancing)	6.90	4.79	3.75	1.13
ã$^{-}$ (advancing)	0.13	0.00	0.00	0.47
ã^{total} (receding)	41.40	42.28	38.39	40.90
ã^{LW} (receding)	39.01	42.28	38.39	40.90
ã$^{+}$ (receding)	0.04	0.00	0.00	0.00
ã$^{-}$ (receding)	33.22	29.70	48.93	26.21
FTIR tests				
Ico	0.0382	0.0170	0.0462	0.0206
Iso	0.1307	0.1063	0.1867	0.1307
1700 peak height	0.0078	0.0045	0.0099	0.0048
1030 peak height	0.0139	0.0136	0.0189	0.0141

From the test results the following observations can be done:

1. The penetration and softening point result for binder recovered from the upper and the lower part of the field PA specimens show differences in viscosity characteristics. The upper part shows a higher degree of ageing compared to the lower part of PA;
2. The penetration and softening point reveal that the laboratory ageing protocol 1 and 3 were not severe enough to simulate the ageing of the top part of PA layers in the field. However, results from ageing protocol 1 are close to the results from the lower part of PA from the field. The results from protocol 3 are far away from both the lower and upper part of PA from field;
3. The DSR results shown in figure 3 are compatible with the penetration and softening point test results. Bitumen recovered from the upper and lower part of field aged specimens exhibits a higher complex modulus values and lower phase angle values compared to the two laboratory aged bitumen samples;
   The master curves of protocol 1 and protocol 3 shows very similar results. The complex modulus for protocol 1 is positioned slightly above the curve for protocol 3 and the reverse is found for phase angle curves. The comparison of complex modulus and phase angle test results of laboratory and field aged specimens confirm the fact that the laboratory ageing protocol 1 and 3 were not severe enough to simulate field ageing of PA;
4. As it can be observed from the spectrum in Figure 4 and the computed indices and peak heights for C=O and S=O, the material from the upper part of the 10 year old pavement seems to have relatively higher peaks both at C=O and S=O band. Protocol 1 has the second highest peak and area at C=O band next to the upper part of field aged bitumen sample. The specimen from the lower part of the PA pavement have comparable peak at the S=O band with Protocol 1. The binder from Protocol 3 seems to have the lowest peak at 1030 cm^{-1} and 1700 cm^{-1} wave numbers next to the virgin bitumen. From the FTIR result, the change in C−C band (1600 cm^{-1}) is also apparent and field aged PA samples have the highest peaks. In general the spectrum line for field aged samples stays above the laboratory aged samples for the whole range of the finger print region except at 1700 cm^{-1} where Protocol 1 lies above the lower part of field PA. These results are in full agreement with the empirical and fundamental rheological test results from the other tests;
5. It is clear from the above results that the total surface energy computed by using receding angles gives a higher value compared to the result from advancing angles. The same trend has been reported by other researchers [7,8]. The results also show that the LW component (free energy of Lifshitz-Van der Waals forces) is the most significant contributor to the total surface energy of the asphalt binders, while the magnitudes of acid and base components are very small. This is in harmony with the fact that most asphalt binders are weakly polar materials.
   From the above test results it was not possible to give a general trend for the surface energy in relation to the ageing of the bitumen with all the test samples. Since ageing leads to the formation of weak bases and acids such as ketones and sulfoxides, the net effect on the surface energy components due to ageing will depend on the initial chemical nature of the asphalt binder and the dynamics of various functional groups during the ageing process. Other possible reasons for this may be a difference in bitumen type (difference between bitumen used in the laboratory ageing protocols and the one used to build the pavement from which the field samples were cored). The purity of the chemicals (liquids) used for the test and the higher sensitivity of the test for the thickness and width measurement of the glass plate coated with bitumen might be additional reasons.

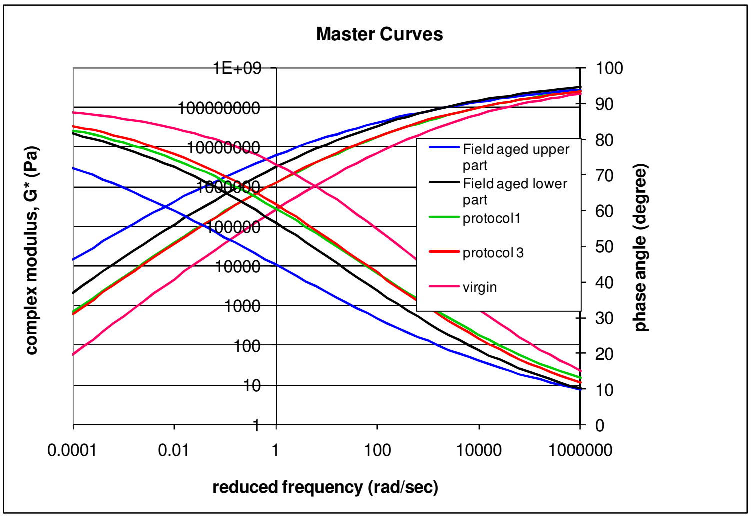

Figure 3: Master curves for the recovered bitumen from field cores and specimens after ageing protocol 1 and 3 at a reference temperature of 20 °C

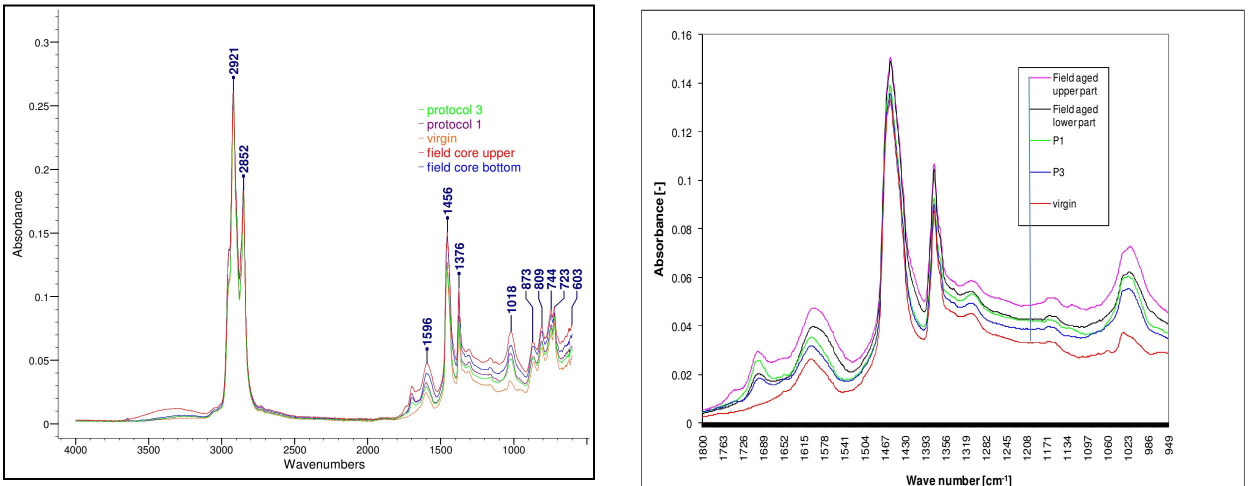

Figure 4: Infrared spectra of field, protocol 1 and protocol 3 aged specimens in total and in 'finger-print' region

In general the penetration, softening point, DSR and the FTIR test results for all the bitumen samples are in good agreement with each other. The analysis of the test results have shown clear differences in the rheological and chemical properties of the laboratory aged and field materials. It can be seen from the above result that protocol 1 and 3 are not severe enough to simulate 10 years of field ageing. Yet, both protocol 1 and 3 have a potential to be improved for the next trial to give a better result.

4.3 Revision of the ageing protocols 1 and 3

It was concluded that both ageing protocols were not severe enough to get similar ageing in the lab as 10 years of ageing in the field. For this reason both protocols were changed:

- In protocol 1 the temperature for the long term ageing was raised from 80 to 90 (protocol 1B) or from 80 to 100ºC (protocol 1A);
- In protocol 3 the temperature of the long term ageing was raised to 135ºC and the period was shortened to 42 (protocol 3A) and 46,5 hours (protocol 3B);

In table 6 the results of the changed protocol on the various parameters are given.

Table 6: Test results field cores and ageing protocols 1A, 1B, 3A and 3B

Name	Protocol 1A (80→100ºC)	Protocol 1B (80→90ºC)	Protocol 3A (85→135ºC for 42 hours)	Protocol 3B (85→135ºC for 46,5hours)	Field aged upper part
Emperical tests					
Penetration [0.1 mm]	9	14	19	10	12
Softening Point [ºC]	73.6	65.4	63.4	76.4	67.6
Penetration Index [-]	0.0	-0.6	-0.4	0.5	-0.5

Name	Protocol 1A (80→100ºC)	Protocol 1B (80→90ºC)	Protocol 3A (85→135ºC for 42 hours)	Protocol 3B (85→135ºC for 46,5hours)	Field aged upper part
WLF parameters DSR					
C1	28.0	28.0	31.0	28.0	28.7
C2	213.0	205.0	220.0	219.0	206.1
Wilhelmy Plate tests					
ã^{total} (advancing)	37.75	32.70	n.a.	n.a.	33.33
ã^{LW} (advancing)	34.99	32.70	n.a.	n.a.	33.33
ã$^{+}$ (advancing)	3.52	4.05	n.a.	n.a.	3.75
ã$^{-}$ (advancing)	0.54	0.00	n.a.	n.a.	0.00
ã^{total} (receding)	43.06	45.48	n.a.	n.a.	38.39
ã^{LW} (receding)	39.58	42.85	n.a.	n.a.	38.39
ã$^{+}$ (receding)	0.15	0.17	n.a.	n.a.	0.00
ã$^{-}$ (receding)	19.65	13.00	n.a.	n.a.	48.93
FTIR tests					
Ico	0.096	0.064	0.0351	0.0646	0.0462
Iso	0.1370	0.1892	0.1285	0.1598	0.1867
1700 peak height	0.0175	0.0107	0.0069	0.0106	0.0099
1030 peak height	0.0147	0.0182	0.0137	0.0152	0.0189

Based on these results, the following conclusions can be drawn:

1. The bitumen from the mortar in the upper part of field aged PA specimen and bitumen from laboratory aged mortar using protocol 1B shows pretty well similar results. The penetration, softening point, DSR as well as the FTIR test results of protocol 1B and the upper part of field aged PA cores show comparable results. In other words, the bitumen recovered from protocol 1B exhibit similar rheological (both empirical and fundamental) and chemical characteristics compared to bitumen recovered from the upper part of 10 years field aged PA. Therefore, it can be concluded that ageing protocol 1B can reasonably mimic binder properties from 10 years field aged PA;
2. Based on the chemical test results it can be concluded that when compared to the upper part of field aged PA, protocol 3A with ageing period of 46.5 hours gives higher ageing while the same protocol with ageing duration of 42 hours gives less ageing. Which means, in order to get an ageing result comparable to field ageing, the ageing period for protocol 3 should be 44 hours. hours. However, this procedure is only validated for a 70/100 bitumen. For different kinds of bitumen the aging procedure can be different.

5. CONCLUSIONS AND RECOMMENDATION

Based on the findings of this research project, the following conclusions and recommendations can be formulated:

1. The effect of binder recovery using methylene chloride as a solvent on recovered bitumen property is insignificant;
2. The field cored PA specimens shows differences in the ageing properties of the binder in the upper and lower part. The top part showed higher ageing compared to the lower part. This can be due to the fact that the top part is exposed to UV light and other environmental factors. As raveling is a surface phenomenon, the laboratory simulation of ageing in this research has targeted the properties of the upper part of the PA specimen;
3. In this study it was not possible to find a general trend for the adhesive properties of bitumen with change in ageing. The surface energy results determined with the Wilhelmy plate test for bitumen samples at different level of ageing show no consistent trend. The reasons are not know at this time however it might be due to the sensitivity of the Wilhelmy plate test to the thickness measurement of the bitumen samples and/or may be due to the integrity of the test liquids used;
4. It is shown that ageing protocol 1B on mortar (2 hours at 165ºC and 7 days in a PAV at 90ºC and 2.1 MPa pressure) and ageing protocol 3 on mortar (44 hours at 135ºC; during the first 4 hours the mortar is stirred manually each hour) can very well mimic 10 years of field ageing of PA. The penetration, softening point, DSR, FTIR and Wilhelmy plate tests on recovered bitumen from the test and field aged PA specimens show comparable results. These ageing protocols uses standard ageing laboratory equipment.
   It should be emphasized that in the ageing protocol only one porous asphalt mixture from the road has been used as a reference. The validity of the ageing protocol has to be verified using samples from various pavements with porous asphalt top layers;
5. The ageing protocols were developed for a PA with 4,3 % standard bitumen 70/100. In case more bitumen is used or a different type of bitumen (e.g. a PMB), the protocols must be reconsidered;
6. Further studies need to be done to investigate the possible change in adhesive property of bitumen due to change in ageing. Surface energy using the Wilhelmy plate test has been used in this research to assess and compare the adhesive property of field and laboratory aged bitumen samples. As already mentioned, it was not possible to fully use the test results of the surface energy to compare laboratory and field aged samples. Maybe, for future studies it is

highly recommended to determine the surface energy with the use of sessile drop test method instead or in addition to Wilhelmy plate test;

7. In this study the Infrared spectrum characteristic peak heights and peak areas were used as chemical test. In addition to this method, however, it is highly recommended in future studies to perform SARA (saturates, aromatics, resins and asphaltenes) classification by using elemental analysis of bitumen samples for a comprehensive qualitative and quantitative chemical investigation.

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