Laboratory simulated asphalt ageing: Myth or reality?

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ABSTRACT

The oxidation of the asphalt binder is thought to be one of the most important aspects of the durability of paving. For more than 40 years the simulation of oxidation is tested in laboratory by binder ageing tests like RTFOT, TFOT, RCAT, PAV and other methods used. Unfortunately simulation of real asphalt cores or plates is still under investigation and no standardised test has been approved. At the time of writing prEN 12697-52 is launched for comment and suggests two conditioning methods. The first is a loose mix ageing and the other asphalt core ageing.

The research reported here started as a search for a representative simulation of open graded asphalt ageing.

Several asphalt aging conditionings have been tested and analysing binder recovered showed the influence of the ageing conditioning. Binders were analysed on mechanical (empirical and rheological) and chemical characteristics. The results are compared with previous reported research on binder ageing and field aged material.

The results showed that the general idea of simulating ageing by high temperature oxidation as most important aspect should be taken into consideration.

It is found that high temperature laboratory conditioning of open graded asphalt cannot simulate asphalt field ageing. All tests results ranked the different laboratory ageing conditioning more or less in the same order. But mechanical results were always less than field aged material while chemical characterisation clearly showed that certain ageing conditions were beyond the field aged material.

It is also found that a small amount of extraction liquid influences the results of analyses and therefore a headspace test should be introduced at research working with recovered binder. This should also be taken into account when contractors work with RAP material and calculate the amount of new binder based on the test results of extracted RAP binder.

Keywords: Ageing, Chemical properties, Physical properties, Porous asphalt

1. INTRODUCTION

The bitumen used in asphalt road structures and other applications is organic material and mainly consists of Carbon (C) and Hydrogen (H). It is still difficult to identify all molecules in bitumen. Speculatively the molecule count in bitumen goes up to a million, but research showed that several groups of active molecules are present [1-4]. Active molecules are considered here as active for reaction with oxygen. Therefore ageing due to reaction with oxygen takes place over time. The degree of oxidation depends on the construction, the pavement structure, the surroundings and environment and the origin of the material [5].
Until now, standardised conditioning methods are available to simulate ageing of bitumen in laboratory environment. For years the RTFOT (Rolling Thin Film Oven Test) is considered to reflect the ageing of bitumen [6 - 8] as result of asphalt production. The combined conditioning of RTFOT and sequentially PAV (Pressure Ageing Vessel) has been considered to reflect the long term ageing aspect of road deterioration in service over the years. In 2012 work was presented at the E&E congress in Istanbul identifying the limitations of these conditioning methods with respect to different asphalt pavement structures [9].
A new project was started exploring a conditioning method to realistically simulate ageing of asphalt in the laboratory based on the data available from previous work [9, 10]. In [10] a loose porous asphalt mix was aged in an oven at 165ºC for two hours followed by 7 days of PAV ageing at 90ºC. This conditioning method is believed to simulate ageing of the top half of a 10 year old porous asphalt layer. As indicated by the test results after 42 and 46,5 hours, ageing induced by the RTFOT/PAV conditioning method is similar to conditioning for 44 hours at 135°C.Performing asphalt tests on a loose mix is not representative and therefore an ageing conditioning for asphalt slabs should be developed. The new prEN 12697-52 *Conditioning to address oxidative ageing* [11] was recently introduced with two major routes for conditioning: the loose mix method or the compacted mix method.

2. MATERIALS AND EXPERIMENTAL DESIGN

The experimental design of this research study was made in continuation of [9]. In this way the results from the new ageing protocol can be accurately ranked relative to other protocols already examined. Particularly, in [9] measurements on bitumen extracted from a porous asphalt layer, which was loaded for 10 years by traffic in practice, is included. These measurements will serve as a reference.

The main purpose of this research was the simulation of asphalt ageing. No further bitumen conditioning was performed and therefore new bitumen conditioning [12] was not taken into account.

It was decided to design a conditioning protocol that thermally induces oxidative ageing of a compacted asphalt mixture. The asphalt mixture chosen is a PA 8. Porous asphalt is surface course that contains a relatively high amount of voids, ±20%. Because of these reasons, among others, the binder in this mixture is highly susceptible to ageing. Any changes in bitumen properties as a result of ageing are therefore most likely to be detected in porous asphalt. The mixture constituents of the used PA 8 can be found in table 1. It should be noted that the same bitumen 70/100 is used as in [9]. This bitumen originates from one single production site and can therefore be considered highly consistent in composition. Any distortion in bitumen property changes and subsequently the (mis)interpretations of results is hence excluded.

Table 1: Mixture constituents PA 8

Mixtures constituents Porous Asphalt (PA 8)			
Binder	**Mineral aggregate**	**Filler**	**Sand**
Bitumen 70/100	Bestone	Wigro 60K	Breaker sand

The porous asphalt mixture was produced and compacted into slabs of 500 mm x 500 mm x 50 mm at one lab according to a fixed procedure. The concerning slab production protocol is designed to mimic practice as much as possible. The laboratory mixing unit for example, this is designed to mix batches of 50 kg according to the same forced mixing procedure used in asphalt mills. Next the hot asphalt mixture is placed within a wooden mould and compacted immediately with a water cooled double steel roller. After the slabs had cooled down they were transported to the second participating laboratory. Here, the slabs were thermally aged in an oven with forced ventilation on perforated shelves with continuous temperature registration. Taking into account lessons learned from previous research on oxidative ageing, five ageing protocols were considered. The conditions of these protocols are summarized in table 2. Also included in table 2 are the accompanying tags and colours from which the individual ageing protocols can be recognized in the following section 3, *Results and discussions*.

In the same laboratory, after the slabs had been subjected to their assigned ageing protocol the bitumen binder was recovered by means of rotary evaporation according to EN 12697-03 [13]. The rotary evaporation method is used with a closed loop system to prevent evaporation of dichloromethane solvent to the environment. The recovered binder was subsequently transported to the third and final participating laboratory, where the same experiments were conducted as in [9] according to the same, fixed procedure and equipment. In addition, bitumen from a reference unaged slab was also extracted and tested. The combination of test procedures is focuses on detecting the change in bitumen properties, chemically and mechanically, as a result of the different ageing protocols. An overview of the executed tests can be found in table 3.

Table 2: Ageing protocols and accompanying identification tags

Tag	Colour	Temperature [°C]	Duration [hr]
BAM_AA135	red lines	135	44
BAM_AA0_0	white	-	-
BAM_AA90_336	red	90	336
BAM_AA90_504	red	90	504
BAM_AA100_504	blue lines	100	504
BAM_AA100_504_2	blue	100	504
BAM_AA100_840	blue	100	840

Table 3 Bitumen properties measured to identify conditioning influence

Property	Standard	Brief description
Penetration	EN 1426	Hardness by needle penetration at 25.0ºC
Softening Point	EN 1427	Softening point measurement in stirred water bath
Dynamic Viscosity	EN 12596	Vacuum Capillary viscometer at 60ºC
Kinematic viscosity	EN 12595	Kinematic viscometer at 135ºC
Fraass	EN 12593	Brittleness of binders at low temperature
BBR	EN 14771	Flexural creep stiffness of binders at low temperature
DSR	EN 14770	Determining the complex modulus and phase angle of binders
FTIR	Variation method	Infrared adsorption of bitumen
Solvent detection	Head Space analysis	'Headspace' is the gas space above the sample in a chromatography vial for detecting volatile components in liquids or solids.
Components	GC	Gas Chromatography is a separation method to analyse gas components.

The FTIR values are calculated by the variation method. The peak height around 1700 cm^{-1} for C=O respectively the peak height around 1030 cm^{-1} for S=O are divided by the peak height at 2600 cm^{-1} of CH.

In the following section the results of the performed tests are shown and discussed. Because the newly obtained results will be ranked with respect to the results found in [9] table 4 contains the tags and colours of the protocols in [9].

Table 4 Conditioning variations used in [9]

Unaged	Unaged bitumen, pen 88	
Unaged_d	Duplicate unaged bitumen, pen 78	
Prod	Extracted bitumen direct after plant production	colour: yellow
Calc ST	Short term aged, Q8 data base information	
PAV90	RTFOT(163) + PAV(90)	
Calc LT	Long term aged, Q8 data base information	
RCAT90	RCAT(163) + RCAT(90)	
PAV100	RTFOT(163) + PAV(100)	
E_PAV100	RTFOT(151) + PAV(100)	
6 wk	Extracted bitumen after 6 wks in road service	colour: black
E_RCAT100	RCAT(151) + RCAT(100)	
E_RCAT100_d	RCAT(151) + RCAT(100), duplicate with pen 78	
RCAT100	RCAT(163) + RCAT(100)	
RCAT100_d	RCAT(163) + RCAT(100), duplicate with pen 78	
RCAT100_1.5	RCAT(163) + RCAT(100) @ 210h	
RCAT100_2x	RCAT(163) + RCAT(100) @ 280h	
10 year	Extracted bitumen after 10 years in road service	colour: black

3. RESULTS AND DISCUSSIONS

All results are presented in one graph sorted from low to high values at the x-axis. The penetration values are presented as percentage of the originated penetration value. All other values are presented as the measured values.
The objective of this presentation is to evaluate a ranking in the conditioning method. Different colours are used to simplify the evaluation of results presented.

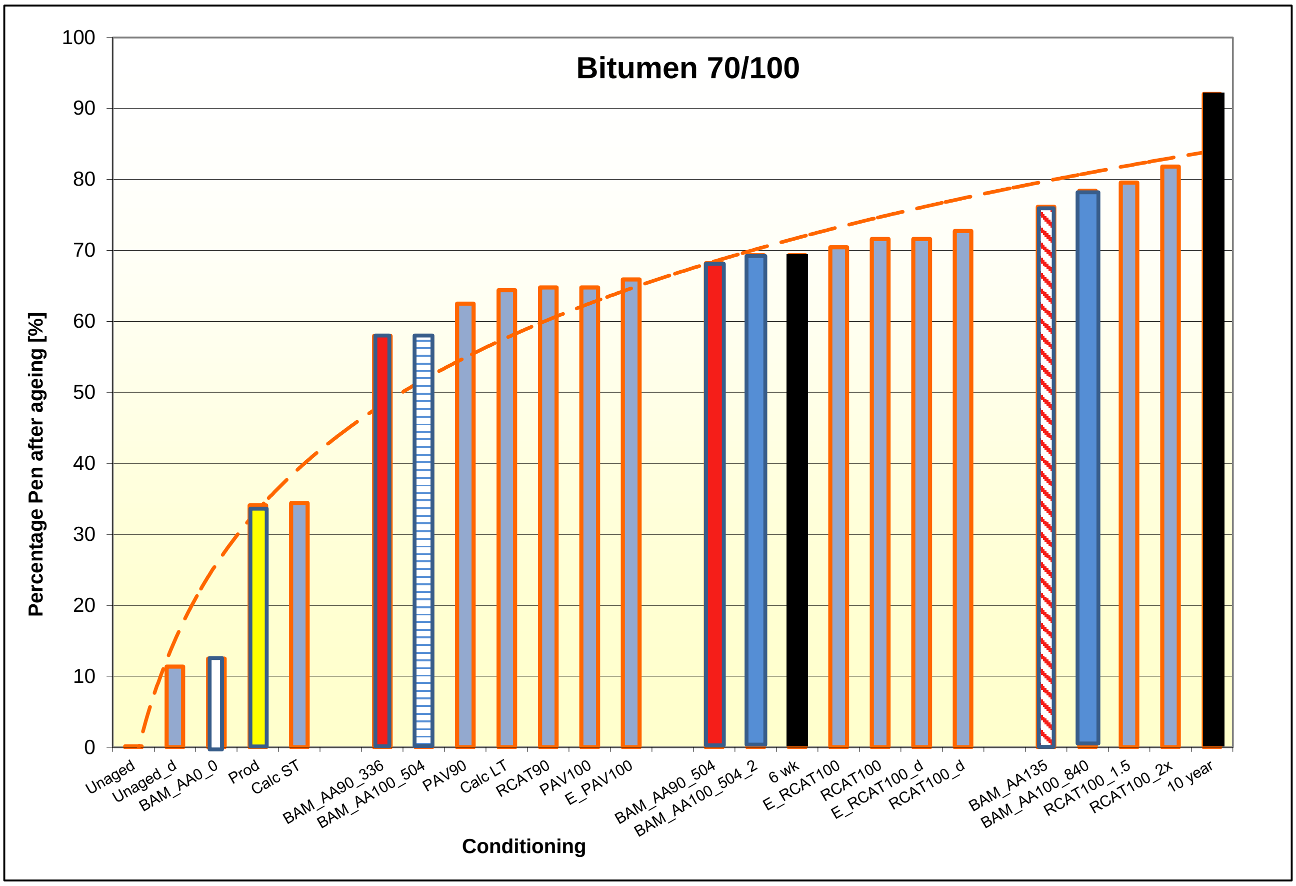

Figure 1: Ranking of Penetration results

The results of the penetration measurements according to EN 1426 show a distinct ranking.
The white bar is the result from tests on unaged bitumen and is clearly below the production ageing of porous asphalt (=yellow bar). Normally, porous asphalt is produced in an asphalt plant while in this paper the slab is prepared in the laboratory. The Unaged_d sample is a duplicate 70/100 sample with a small deviation in pen value from the original and is used to identify differences in ageing based on the pen value. The original bitumen has a pen value of 88 while the duplicate has a pen value of 78, as shown in the graph by 11% difference from the original.
The BAM_AA135 sample (red/white striped bar) is the sample aged for 44 h at 135°C as suggested in the introduction.
The BAM_AA90_336 sample (red bar) and BAM_AA90_504 sample are aged for respectively 2 weeks (336 h) and 3 weeks (504 h) at 90ºC.
The BAM_AA100_504 (white/blue stripes) is a sample for which, after evaluating all the results, an extra test has been introduced: the Gas Chromatography headspace. Comparing all the results showed that this sample was “diluted” with solvent, used for the extraction of the binder. GC Headspace confirmed these findings. An extra sample was taken from the slabs available. The difference was that this sample was already used for an asphalt scuffing test, referred to as the BAM_AA100_504_2 sample.
The BAM_AA100_504_2 sample (blue bar) and BAM_AA100_840 sample (blue bar) are aged for respectively 3 weeks (504 h) and 5 weeks (840 h) at 100ºC.
From the penetration results, the ranking shows that 1 week extra time (33%) has a larger effect on ageing than the increase of 10°C. The engineering rule of thumb, “10°C increase in temperature doubles the ageing time” could not be confirmed by penetration values.
All conditioning methods evaluated do not result in ageing as observed in a 10 year old open grade asphalt core (the black data).

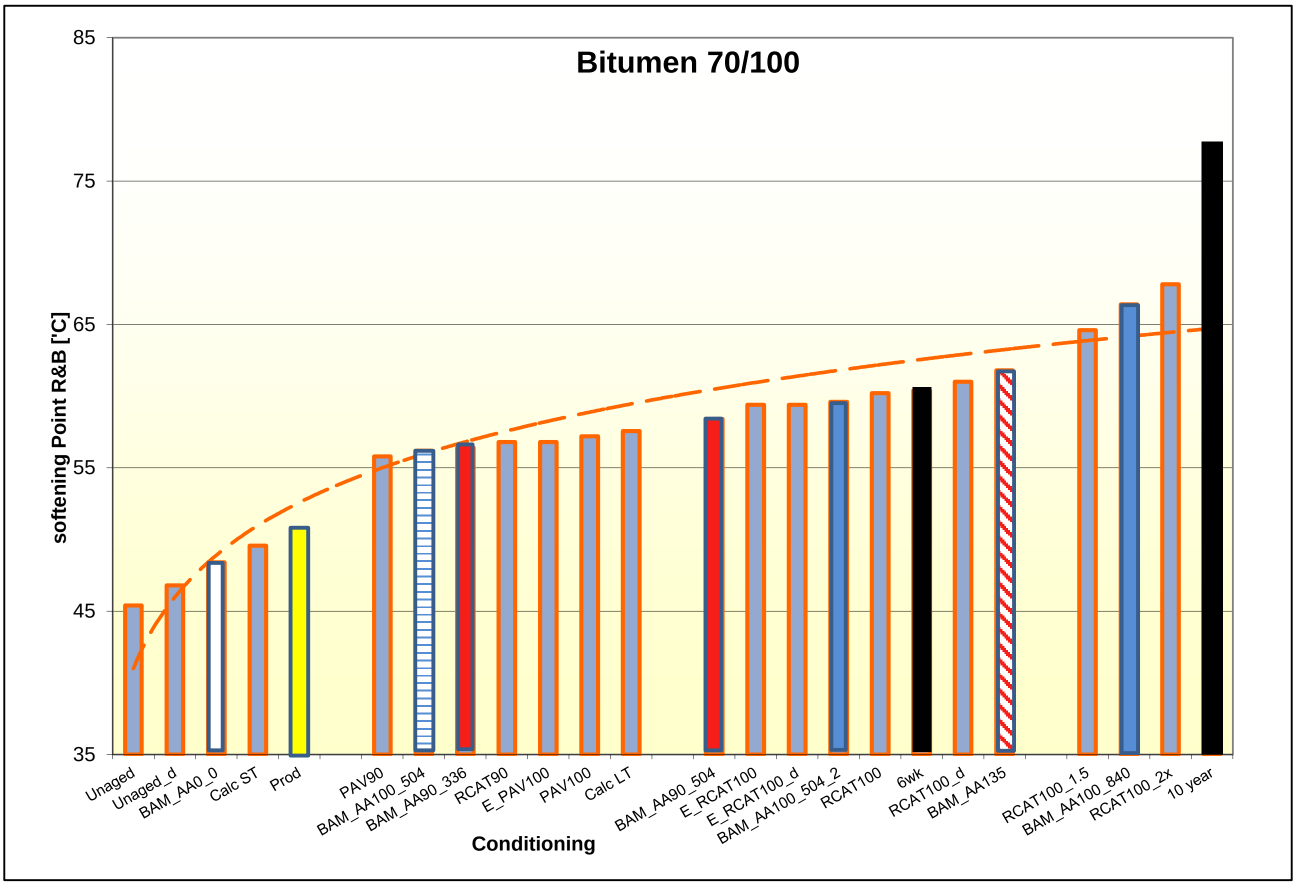

Figure 2: Ranking of Softening point results

The group ranking of the softening point results are similar with the ranking of the penetration results.
The BAM_AA135 sample for softening point seems a little less ranked but the sequence of the aged slabs did not change. Again the influence of the solvent pollution on the softening point (white/blue striped bar) is significant.

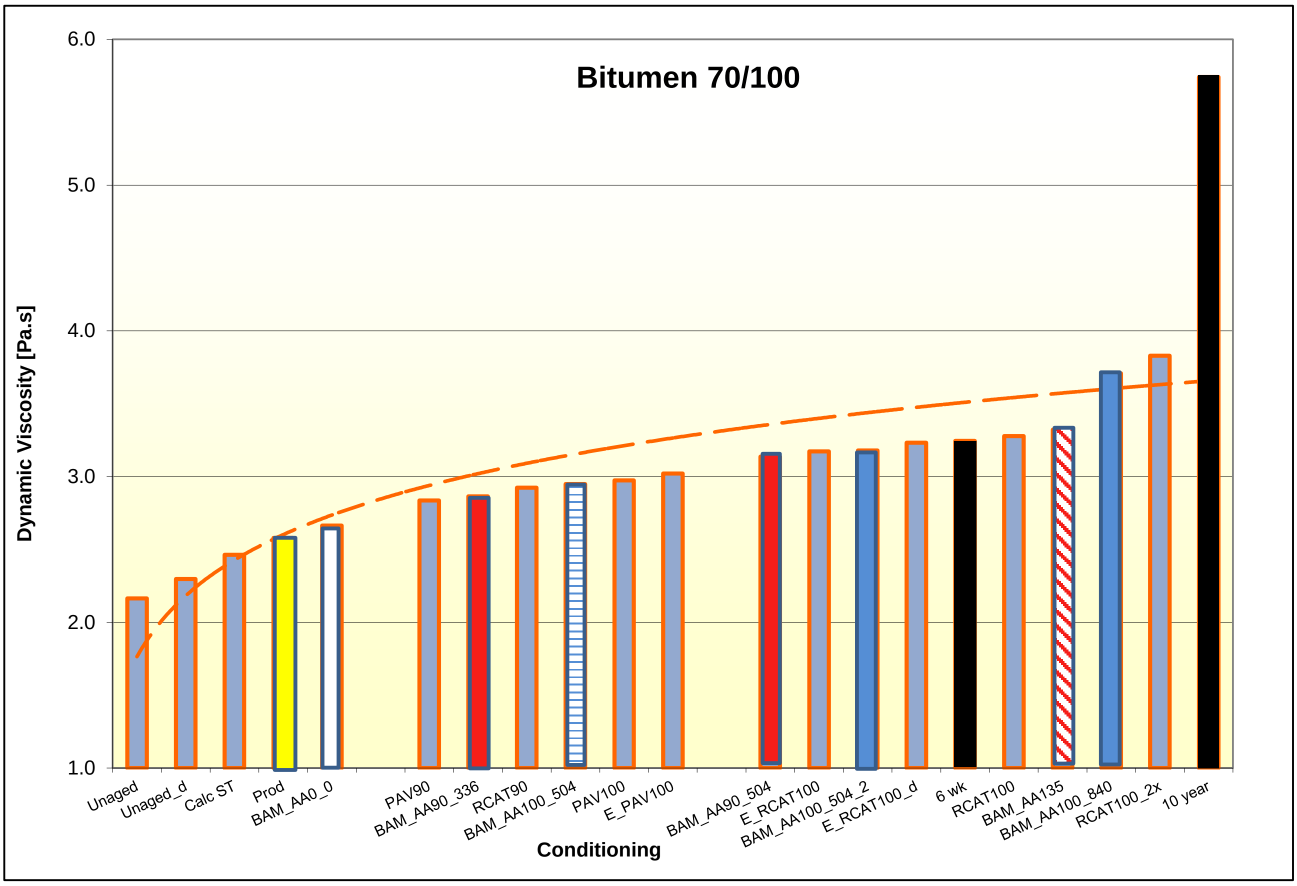

Figure 3a: Ranking of Dynamic Viscosity results

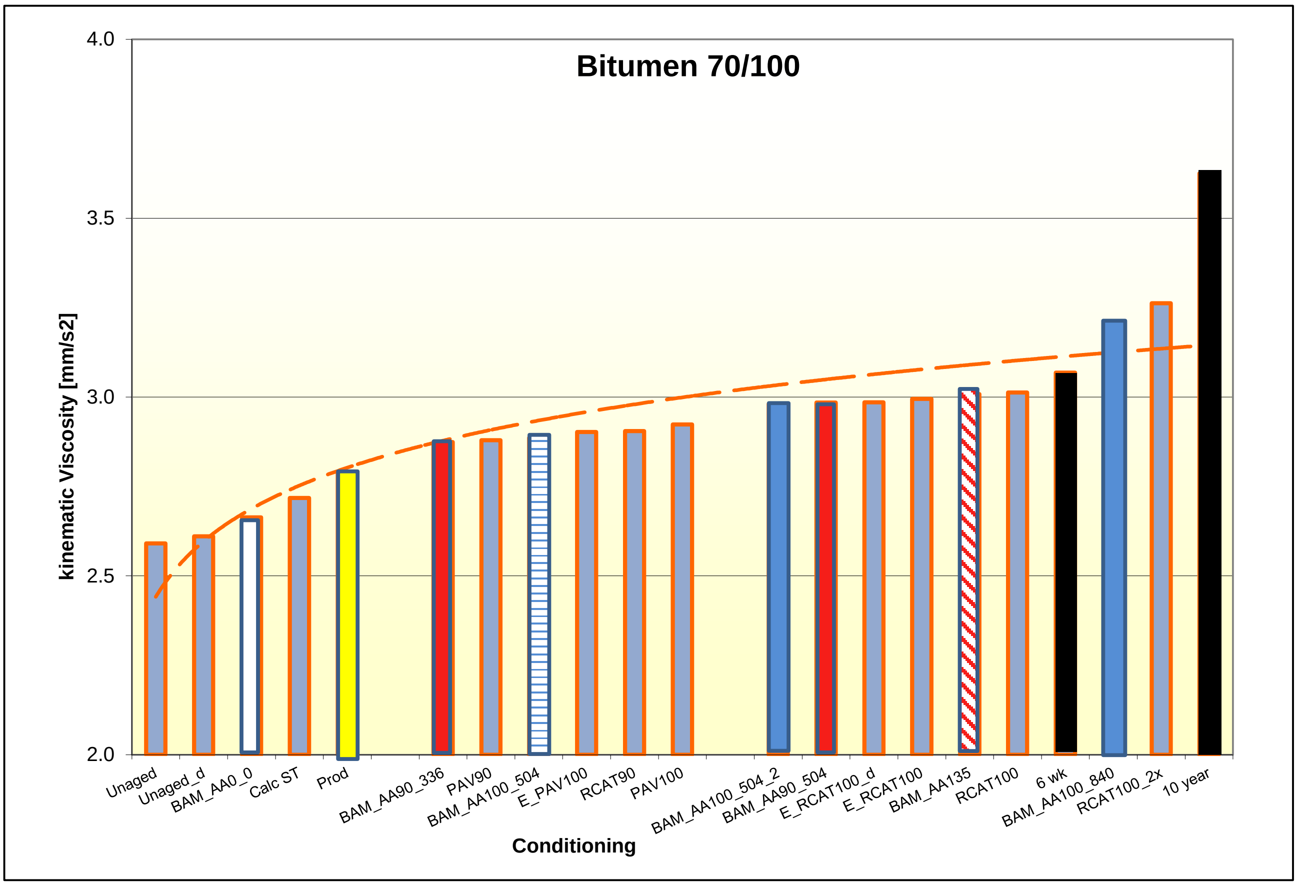

Figure 3b: Ranking of Kinematic Viscosity results

The ranking of the kinematic and dynamic viscosity is almost equal and in line with the ranking of penetration and softening point. Due to small differences, sometimes two results switch places but they are always in the same area. Again, the engineering “rule of thumb” could not be confirmed.

In some research, the ratio of viscosity is used to identify ageing index. Asphalt construction is the key factor for change in binder properties. The understanding and introduction in asphalt durability models of the differences in top-layer oxidation and bulk oxidation of dense asphalt [14] will help to evaluate asphalt properties in a less generalised way, e.g. open grade asphalt behaves different than dense asphalt. An asphalt core of an old dense asphalt road will have a heavily aged top layer but the rest of the core is far less aged. By extracting the complete core, reclaimed binder will have an average value and not reflecting the visual condition of the road inspected.

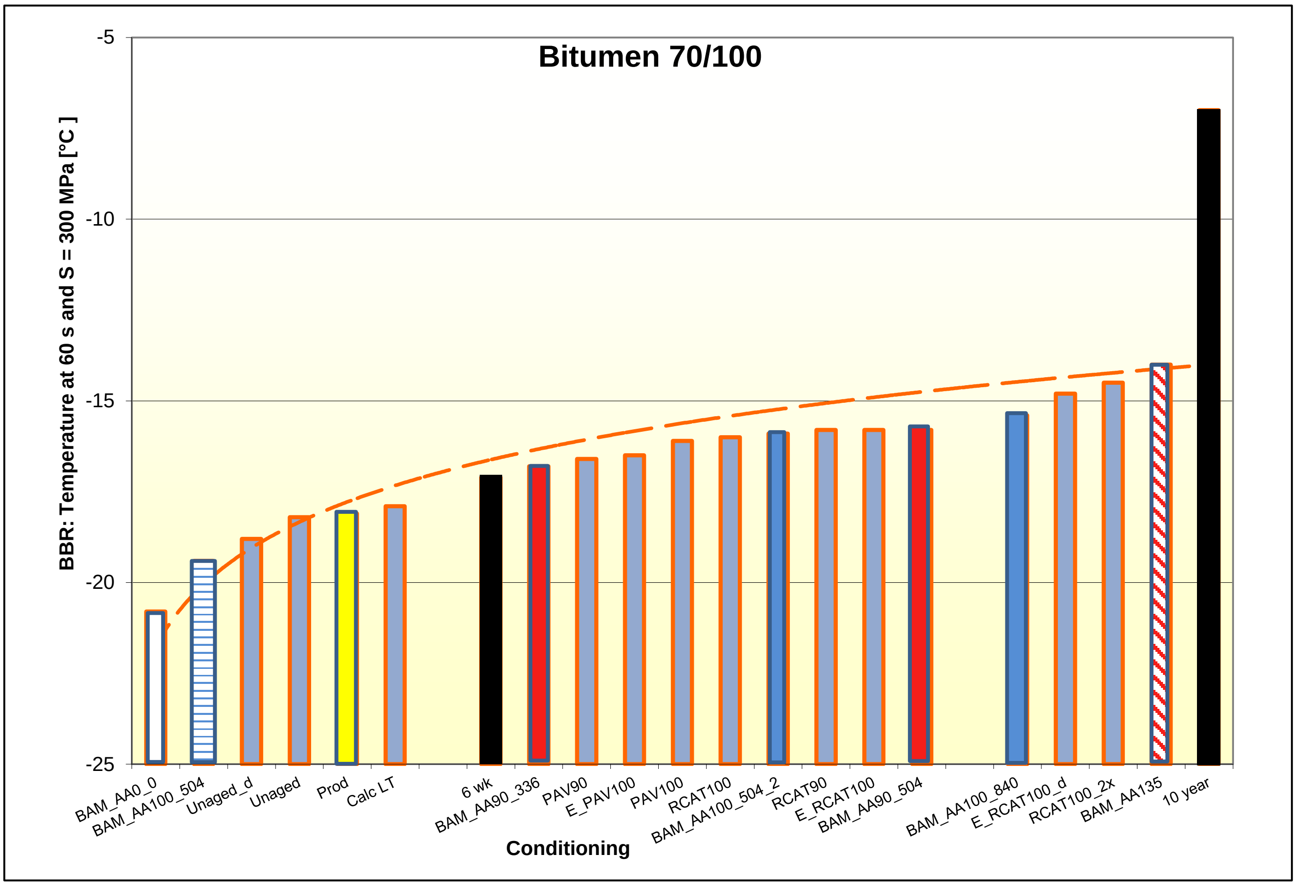

Figure 4a: Ranking of BBR results, Creep Stiffness

The same discussion as previous results is relevant for the results of BBR in Figure 4a and 4b, respectively for the temperature where stiffness equals 300 MPa and for the temperature where the m-value equals 0,3 [-].
The only value worth mentioning is the white/blue striped bar. The sample BAM_AA100_504 is now almost equal to the measurements on unaged bitumen. Again the influence of the solvent is significant in the BBR results. This is already reported in earlier reports [15] showing that BBR test results can be highly influenced by small amounts of solvents.

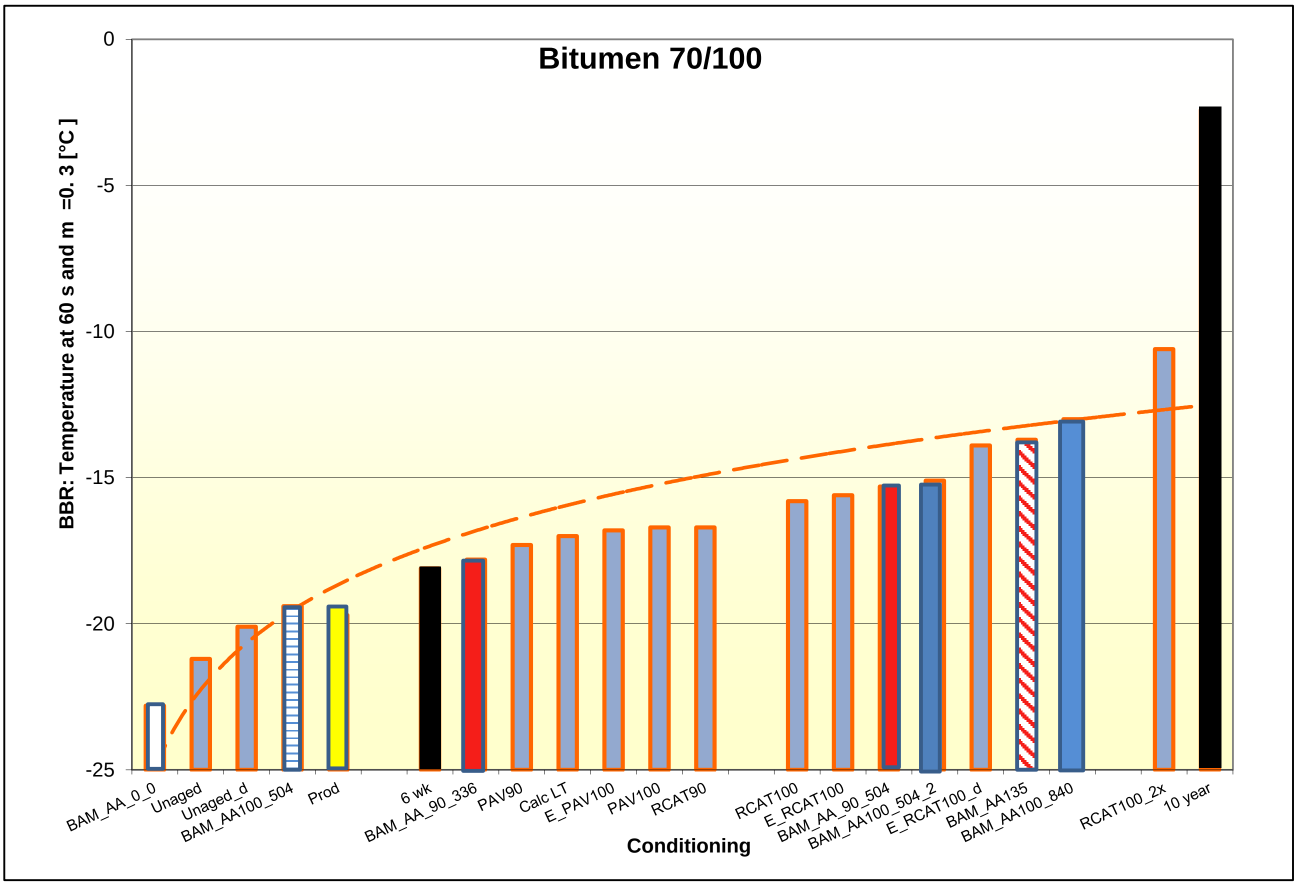

Figure 4b: Ranking of BBR results, m-value

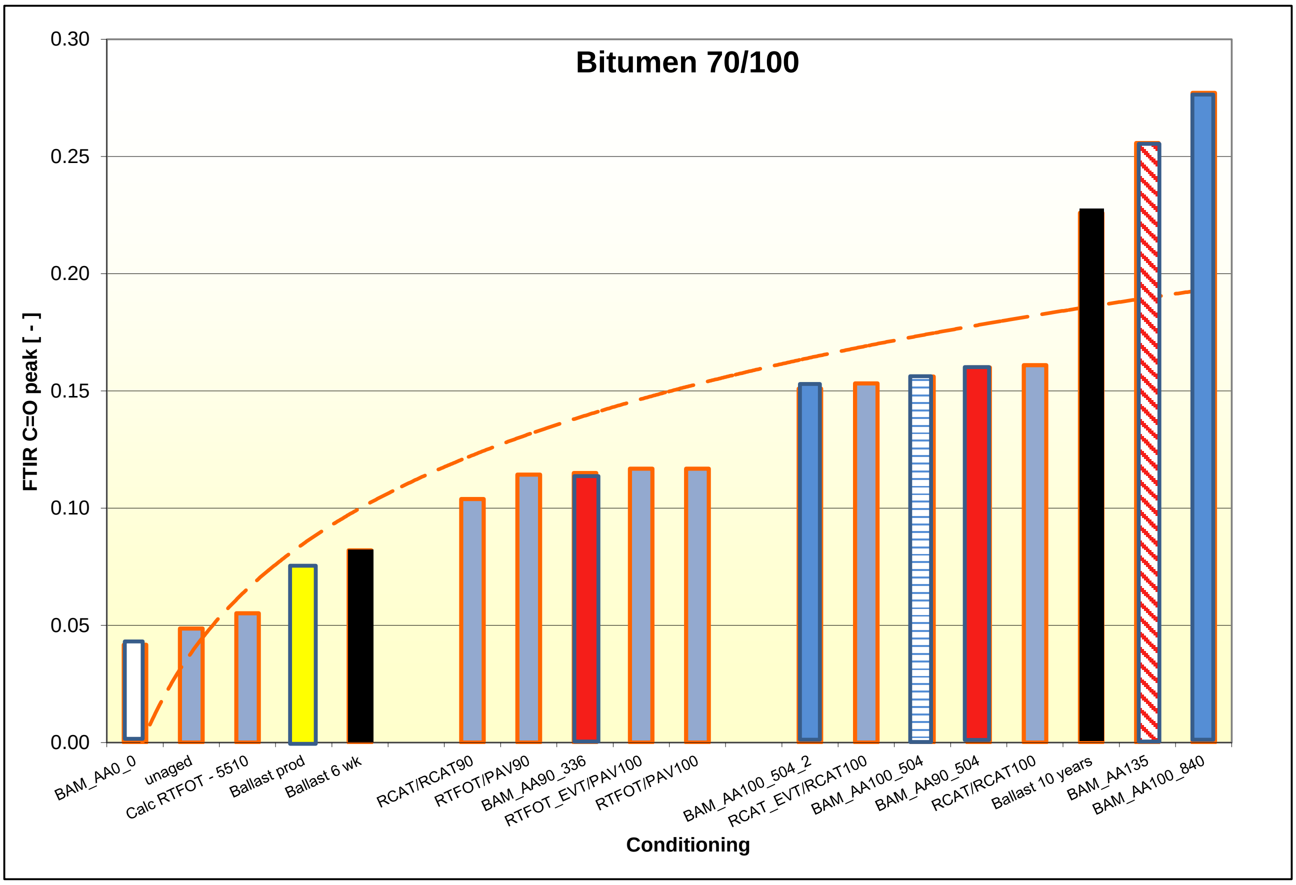

Figure 5: Ranking of FTIR C=O results

The results of the oxidation peaks in Figure 5 show a different ranking.
The C=O results of the binder after 6 weeks in a road pavement are almost equal with the results of asphalt plant production. This is in contrast with the mechanical test results where the six weeks service was always in line with the RTFOT + PAV conditioning in the lab.
The results of BAM_AA135 and BAM_A100_840 show a higher oxidation value than the 10 years old asphalt cores. Both conditioning regimes create a more pronounced oxidation than 10 years in service, in contrast with the results of the mechanical tests.
These results indicate that oxidation alone is not enough to simulate ageing of asphalt roads in service.

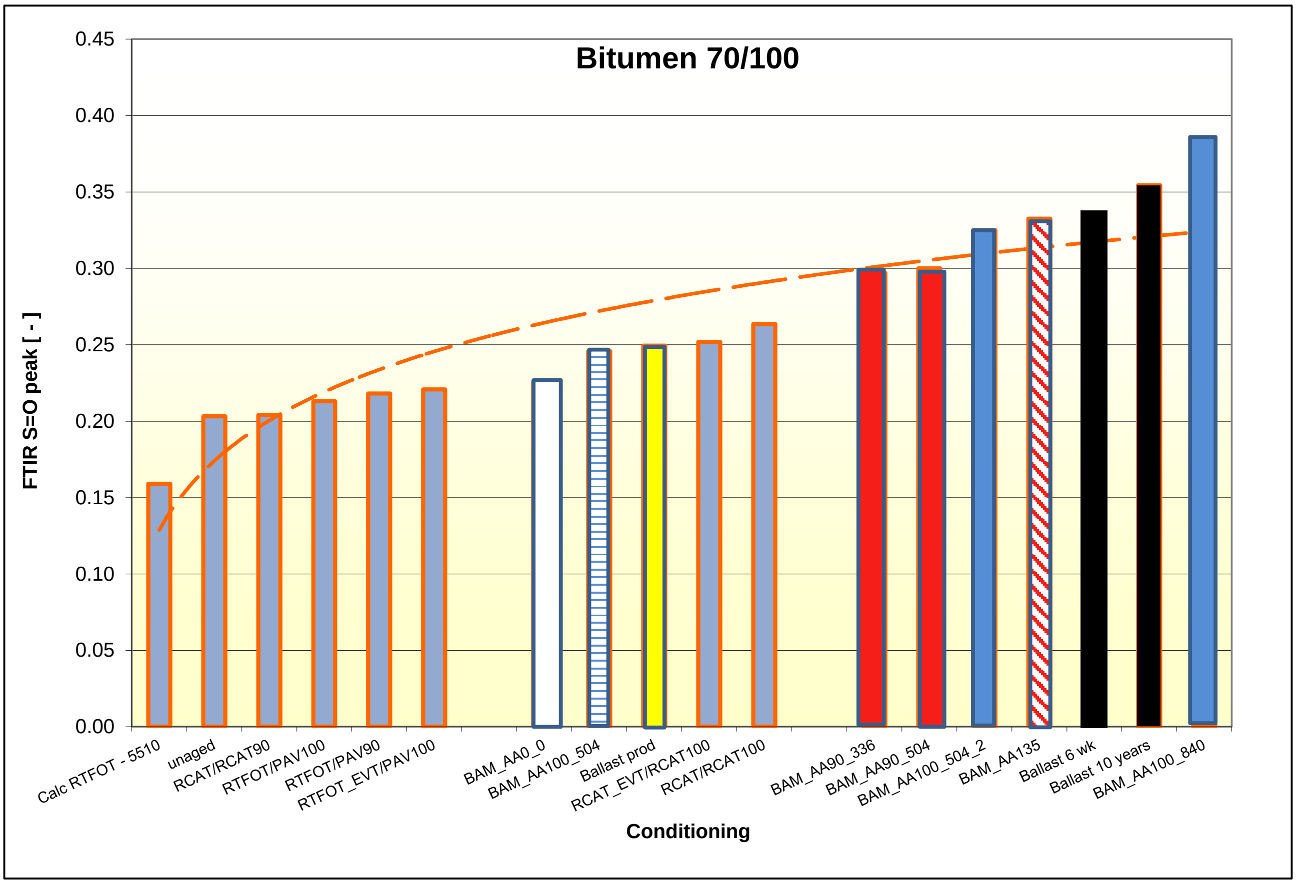

Figure 6: Ranking of FTIR S-O results

In Figure 6 almost all aged bitumen from slabs are ranked high (on the right side) while almost all laboratory conditioned bitumen are ranked low (on the left side) except for the RCAT conditioned samples at 100°C. This might indicate that there is a difference in oxidation kinetics at 100°C compared to oxidation kinetics at 90°C. But most probably this indicates the more severe conditioning of RCAT over PAV conditioning.
The results are again, different from the rest of the results, even different from the C=O FTIR results. This might indicate that the kinetics of Sulphur oxidation in the presence of filler and/or aggregates is different from the kinetics of Carbon oxidation.

The lower ranking of the BAM_A100_504 (white/blue striped bar) and the higher ranking of BAM_A100_504_2 (blue bar) might indicate that some sulphur oxidation has been introduced at the asphalt scuffing test of this slab.

The results of the chemical analyses clearly show that conclusions from only chemical analysis are not sufficient to indicate the ageing of binders, despite literature published [16].

The overall discussion on the test results indicate that oxidative ageing in the lab, as asphalt slabs, will not simulate ageing in practice in pavements. This is indicated by several other researchers [17 - 20] discussing the possible influence of UV, rain, radiation, traffic loads, oil and grease spillage, salt and physical hardening which are not taken into account with oxidative conditioning.

4. CONCLUSIONS

The outcome of this research program clearly indicates that oxidative conditioning for a standard 70/100 pen bitumen from Kuwait export crude is not enough to simulate asphalt road ageing for open grade asphalt.

The influence of a small amount of solvent in the investigated bitumen will result in complete different ranking of the binder.

The oxidation peaks of carbonyl and sulphur oxides of this bitumen show different results when combined with filler and aggregates. It seems that sulphur oxidation kinetics is more influenced by the presence of filler and aggregate than the kinetics of formation of carbonyl groups.

5. RECOMMENDATIONS

Research on asphalt cores should take into account the surface effect of oxidation. This indicates that (even for PA) the oxidation of the binder depends on the location of the bitumen in the PA-layer.

Extraction to recover the binder from the asphalt mix should be considered with care. Small amount of solvent has a substantial influence on the chemical test results. The GC headspace is an easy and simple technique to identify possible solvent pollution.

Most probably the difference between oxidative ageing and road pavement ageing in practice will also be the case for other types of asphalt mixes. The influence of different asphalt pavements are not investigated and should be acknowledged by further research.

Bitumen from different sources and different bitumen grades will show variation in oxidative response. Therefore generalisation of “binder” research should be avoided as much as possible unless test results confirm conclusions.

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