

# Comparing field aging to artificial laboratorial aging of bituminous binders for porous asphalt concrete using black space graph analysis

Diederik Quirinus van Lent<sup>1, a</sup>, Steven Dirk Mookhoek<sup>1</sup>, Dave van Vliet<sup>1</sup>, Cecile Giezen<sup>1</sup>,  
Greet Aletta Leegwater<sup>1</sup>

<sup>1</sup> Netherlands' Organisation for Applied Scientific Research (TNO), Delft, Netherlands

<sup>a</sup> [diederik.vanlent@tno.nl](mailto:diederik.vanlent@tno.nl)

Digital Object Identifier (DOI): [dx.doi.org/10.14311/EE.2016.070](https://dx.doi.org/10.14311/EE.2016.070)

## ABSTRACT

*The majority of the Dutch highway network (approx. 95%) is paved with a wearing course of Porous Asphalt Concrete (PAC). Aging behaviour of the bituminous binder in the PAC is a key parameter for the service life of the pavement. In this paper a review is described concerning the development of field aging and artificial laboratorial aging in terms of the rheological properties (complex modulus and phase angle) of the bituminous binder. For this an extensive database with the development of the rheological properties of the bituminous binder over years from three different pavement trial sections are compared to a large set of artificial laboratorial aging results found in literature. The results are analysed and compared by plotting the data together in black space graphs as function of their aging. From these black space graphs conclusions can be drawn concerning the suitability and predictability of the artificial laboratorial aging methods for the aging of the bituminous binder in the field. On basis of the black space graphs it is demonstrated which laboratorial aging technique appears most representative for simulating ageing in PAC.*

**Keywords:** Ageing, Complex Modulus, Rheology

## 1. INTRODUCTION

The majority of the Dutch highway network is paved with a top layer of Porous Asphaltic Concrete (PA). PA top layers have advantages over dense top layers in the areas of noise reduction, splash and spray of water and permanent deformation. A disadvantage of PA top layers is their limited service life compared to dense asphalt top layers. The service life of PA is mainly limited due to a mechanism called ravelling; the loss of aggregates from the surface [1].

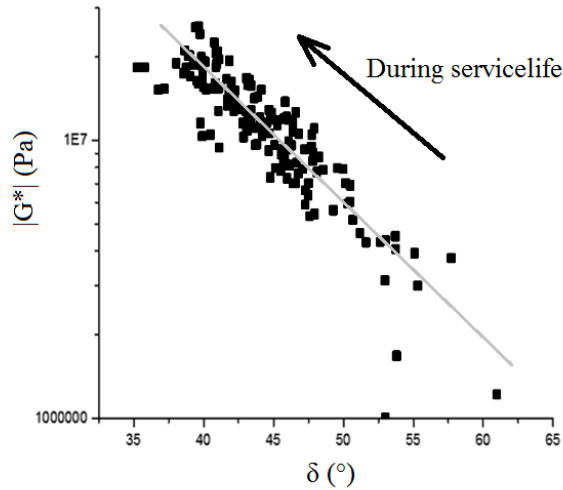
It is generally known in road engineering that the properties of an asphalt top layer will change over time due to a phenomenon known as asphalt ageing. More specifically the material undergoes potential physical/chemical (irreversible) changes due to cycles in temperature, the ingress of moisture, air (oxygen) and irradiation of UV that change the material performance. Generally such changes reduce the durability of materials, when subjected to continuous loading. This phenomenon for asphalt pavement is for instance commonly observed by changes in the (viscoelastic) mechanical properties of the bituminous binder [2, 3, 4, 5]. This is specifically true for PA wearing courses as the open structure allows the environment to influence a large fraction of the binder throughout the wearing course. Although bitumen is only one constituent in such asphalt mixtures, the overall performance of asphalt pavements is greatly influenced by the changes in the binder's properties. As the binder ages the magnitudes of the complex modulus of bituminous binders increases and the phase angle of the complex modulus reduces. This leads to embrittlement of the binder. As a result the PA loses its intrinsic designed property and is more prone to damage, which exhibits for PA in ravelling [6, 7].

In this work the progression of ageing of the bituminous binders is investigated by means of their rheological properties (magnitude and phase angle of the complex modulus) measured by means of Dynamic Shear Rheometry (DSR). Using the rheological properties of the binders, field ageing and different laboratory ageing techniques are compared by representing the rheological properties in time in a so called black space graphs. A black space graph presents the magnitude of the complex modulus as function of the phase angle of the complex modulus [8]. Based on this representation in black space graphs the most appropriate laboratory ageing technique with respect to the field exposure is proposed.

## 2. FIELD AGEING OF BITUMINOUS BINDERS

### 2.1 Polymer modified bitumen trial sections

To investigate field ageing of bituminous binders in PA on the basis of a black space graph, rheological properties are measured from material from trial sections of 3 PA mixtures (3 different binders) at 4 different locations in the Netherlands (A28, A30, A15, A59). These trial sections are all constructed from PA with a polymer modified bituminous binder (PMB). From these trial sections cores were drilled at specific moments in time (annually over a period of 10 years). The pure binder was extracted from the asphalt cores using solvent extraction and its recovered properties were subsequently measured in the DSR. In Figure 1 the magnitude and phase angle of the complex modulus at a temperature of 20°C and strain rate of 10 rad/s are plotted in a black space graph for all recovered binders. This figure shows that there is a clear trend of increasing  $|G^*|$  and decreasing  $\delta$  over time in the pavements and that the slope of change for the two parameters stays relatively constant. More specifically, it is observed that all the data points from an individual specific binder show very little variation from a linear dependency between  $\log |G^*|$  and  $\delta$  over time. The rate of ageing (increase of  $|G^*|$  and decrease of  $\delta$  in time) for the three binders varies slightly since different binders from different locations were studied. That is why there is variation in the graph of Figure 1. However, for each individual binder at each location, a similar slope (coefficient approx. -0.120) can be defined.

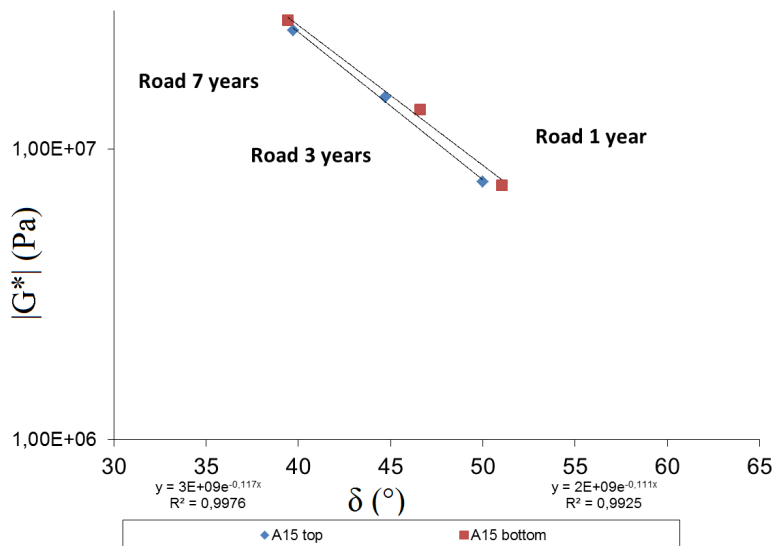


**Figure 1: Black space graph with duration in the pavement**

Based on this observation it is hypothesized that, although individual binders may differ in their sensitivity to ageing, the development of field ageing for bituminous binders in PA follows a constant slope in black space. In order to challenge or confirm this hypothesis a similar investigation was done for recorded rheological data obtained from other trial sections of PA.

### 2.2 Trial section with unmodified Q8 70/100 bitumen

A PA trial section in the Netherlands at the A15 highway with an unmodified Q8 70/100 binder was described by Hagos [7]. Also here cores were drilled at specific moments in time. The pure binder was extracted from the asphalt cores with solvent and subsequently measured in the DSR. The rheological properties of the binder (20°C and at 10 rad/s) are plotted over time in a black space graph (Figure 2).



**Figure 2: Black space graph of unmodified bitumen from the trial section at the A15**

Figure 2 presents two sets of data acquired from the A15 highway. The first set of results was obtained from measurements on the binder taken from the top section of a PA wearing course, the second set was obtained from the bottom section. In agreement with Figure 1, Figure 2 shows the increase of the stiffness and the decrease of  $\delta$  over the service life of the recovered unmodified binder. Moreover, the slope of the development of ageing in the black space graph shows that the ratio of  $\log |G^*|$  and  $\delta$  is comparable

to what was found for the aged binders from the field sections. The exponents of the ageing line for the two sets of data are -0.111 and -0.117 respectively. These values are very similar to the exponent of the field ageing line of the recovered modified binders (-0.120). As these binders were completely different from the ones investigated in the sections, the agreement between the slopes of the different ageing data supports the hypothesis.

### **2.3 Comparing field ageing to laboratory ageing**

From the data in the previous paragraphs it was found that the slopes of the field ageing data in the black space graph for the PMB and the unmodified Q8 70/100 binder are very similar. This suggests that the slope for field ageing of binders is rather constant for bituminous materials and therefore appears independent of the type of bituminous binder investigated. Though similar slopes are found, a prediction of the binder properties in time is still difficult because a constant slope between  $|G^*|$  and  $\delta$  does not mean that the sensitivity of ageing (advancement of  $|G^*|$  and  $\delta$  with time) of the binders is constant. Nevertheless, the observation of the constant slope for naturally field aged binders is interesting, since it allows comparison between field ageing and laboratory ageing of the same binders in order to establish whether the ageing mechanism under laboratory conditions yields comparable trends and ageing characteristics. Furthermore, it allows assessment of different laboratory ageing techniques in terms of their representativity and acceleration factor.

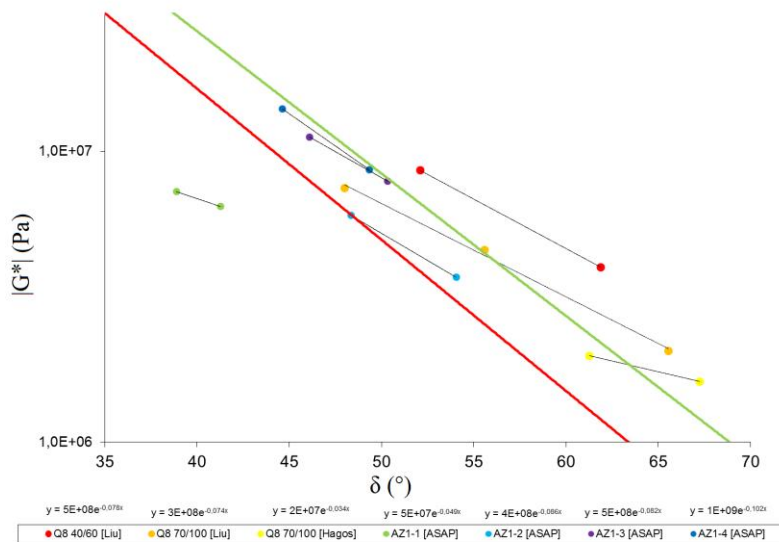
## **3. LABORATORY AGED BITUMINOUS BINDERS**

There are different standardised techniques for artificial ageing of bituminous binders. Every technique has its own conditions and procedures, which will influence the final properties of the aged binder. In this research the rheological data of aged binders using the following ageing methods are evaluated: Rolling Thin Film Oven Test (RTFOT), temperature ageing and the Pressurised Ageing Vessel (PAV).

### **3.1 Rolling thin film oven test**

The RTFOT ageing technique is a method to artificially age a bituminous binder at higher temperatures in order to simulate ageing during asphalt production and laying of the pavement. This sort of ageing is called short term ageing. Rheological data from different researches are collected [9, 7, 10].

Liu [9] and Hagos [7] have performed RTOFT ageing according to the European standard EN 12607-1 [11]. According to this standard bituminous binder (50 g) in cylindrical containers in the instrument is heated to 163°C for 75 minutes. During this time fresh air is injected into the containers with a fixed flow of  $4000 \pm 200$  ml/min. In the other project [10] an equivalent standard, AASHTO T-240 [12] was used with only small differences in dimensions of the glass cylinders compared to the European standard. The rheological data of the virgin and the RTFOT aged bituminous binders as collected from different data sets are shown in the black space graph in Figure 3.



**Figure 3: Different bituminous binders before and after RTFOT ageing compared to the positions and slopes of the PMB (red line) and Q8 70/100 (green line) field ageing relations**

Figure 3 presents the development of the magnitude and phase angle of the complex modulus with RTFOT ageing for seven different binders. The figure demonstrates that similar behaviour is found for the whole dataset. Here also, with ageing, the ratio between  $\log |G^*|$  and  $\delta$  of the bituminous binders, due to the RTFOT ageing, nearly stays constant. As reference, the position and slope of the field aged binders (PMB and Q8 70/100) is also given in the black space graph (red line and green line respectively). A comparison between the slopes shows that although RTOFT ageing of the binders presents a linear increase of the  $|G^*|$  and  $\delta$ , the slope for the RTFOT aged binders is different from the slope of the field aged binders. For clarification all the determined black space exponents (slope coefficients) are listed in Table 1.

**Table 1: Exponents in the black space graph for different RTOFT aged binders**  
**Material Exponent**

Material	Exponent
Field aged binder (PMB)	-0.120
Field aged binder (Q8 70/100)	-0.114
Q8 40/60 [9]	-0.078
Q8 70/100 [9]	-0.074
Q8 70/100 [7]	-0.034
AZ1-1 [10]	-0.049
AZ1-2 [10]	-0.086
AZ1-3 [10]	-0.082
AZ1-4 [10]	-0.102

As can be seen from the table, the coefficients for the RTFOT aged binders are significantly lower than those for the field aged binders. Although based on the slopes of only two data points for each binder, the coefficients from the whole dataset suggest that the progression of the binder properties with RTFOT ageing advances unlike under field ageing conditions and thus may imply that a different ageing mechanisms take place. It is hypothesized that this discrepancy might be an effect of more forced ageing at higher temperatures in RTFOT (through which unwanted side-effects take place), a difference in sample geometry (binder film thicknesses) or perhaps due to the absence of other pavement constituent such as filler, sand and aggregates.

In order to look into these three effects two other temperature accelerated ageing methods and also ageing of complete asphalt mixes (PA) are evaluated.

### 3.2 Temperature ageing

Temperature ageing is an ageing technique in the laboratory in which, by means of elevated temperatures, a simulation is made for the ageing that binders undergo during asphalt production, laying of the pavement and/or for the ageing during the service life of the pavement. By looking into asphalt mixtures, the binders are aged within a representative geometry as compared to field materials. Additionally, the binders are aged in the presence of filler, sand and aggregates, which may influence the ageing characteristics. Hagos [7] therefore used three ageing protocols on laboratory compacted PA mixtures with a Q8 70/100 binder:

1. 60°C for 1000 hours and 40°C
2. 60°C for 1000 hours and UV (60 W/m<sup>2</sup>)
3. 60°C for 1000 hours and UV (60 W/m<sup>2</sup>) and 70% rH.

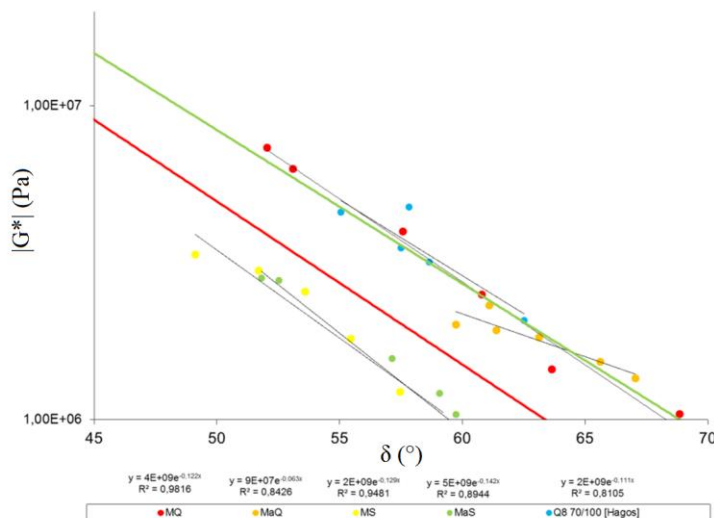
In the project of Mookhoek et al. (2014) four different PA mixtures were investigated:

1. Uncompacted PA mixture with a Q8 70/100 bituminous binder (MQ)
2. Uncompacted PA mixture with an SBS polymer modified 160/220 binder (MS)
3. PA mastic of the Q8 70/100 binder : Wigro 60K filler = 1:1 (MaQ)
4. PA mastic of the SBS polymer modified 160/220 binder : Wigro 60K filler = 1:1 (MaS)

The four different mixtures were first heated to 135°C for 4 hours to simulate short term asphalt ageing during the production process. Afterwards the mixtures were put in an oven at a temperature of 85°C for a maximum of 9 days. After 1, 2, 6 and 9 days samples were taken for binder recovery, as shown in Table 2. The recovered binders were subsequently characterized using dynamic shear rheometry (DSR). The change of magnitude and phase angle of the complex modulus due to temperature ageing for the different recovered binders are shown in Figure 4.

**Table 2: Temperature ageing protocol of Mookhoek et al. [13]**

Ageing	Production simulation	Service life simulation
Amount	9 kg	
Duration	240 minutes	9 days
Ventilation	Air	Air
Ventilation amount (l/min)	unknown	unknown
Temperature (°C)	135	85
Pressure (MPa)	0.1 (standard pressure)	
Estimated mixture thickness (mm)	40 - 60	
Sampling (about 50 - 100 g)	After 240 minutes	After 1, 2, 6, 9 days



**Figure 4: Changes of the magnitude and phase angle of the complex modulus due to temperature ageing compared to the positions and slopes of the PMB (red line)**

### and Q8 70/100 (green line) field ageing relations

Figure 4 shows the datasets for the collected data from Hagos [7] and Mookhoek [13]. It can be seen in the figure that the data describes a comparable trend for the  $|G^*|$  vs.  $\delta$  along with ageing as was observed earlier from the field aged and RTFOT aged materials. When looking more closely at the data, it can be observed that the fitted lines for the temperature ageing data in the black space graph are not all parallel. Proof for this is shown in Table 3, in which the exponents of the ageing relation are described.

**Table 3: Exponents in the black space graph for different temperature aged binders**

Material	Exponent
MQ (Mookhoek et al., 2014)	-0.122
MaQ (Mookhoek et al., 2014)	-0.063
MS (Mookhoek et al., 2014)	-0.129
MaS (Mookhoek et al., 2014)	-0.142
Q8 70/100 (Hagos, 2008)	-0.111

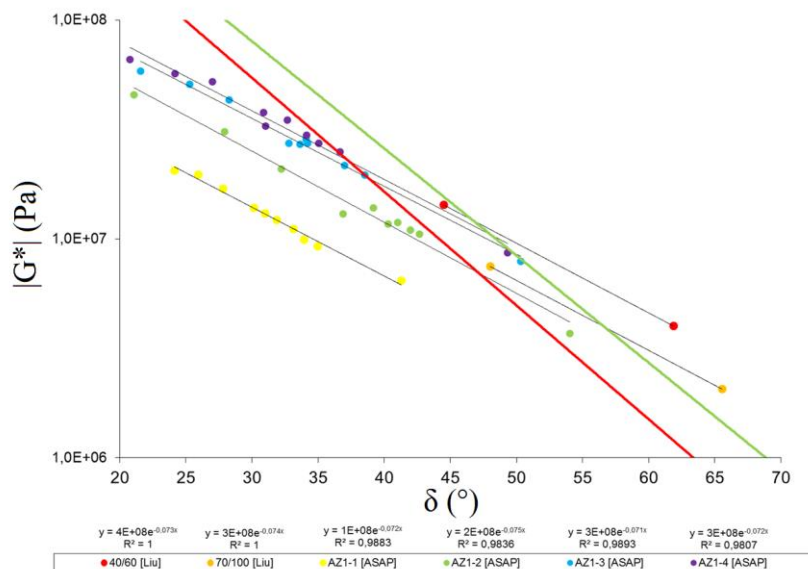
Based on this analysis it is found that the ageing relation of the recovered MQ binder from a PA mixture and the recovered MS binder from a PA mixture are very similar [13]. Moreover, the value of their coefficient is equal to the coefficients found earlier for field aged materials (unmodified Q8 70/100 and PMB). For the temperature aged mastics however lower agreement is found with either field or RTFOT aged materials. Finally, the coefficient for another Q8 70/100 binder recovered from a PA [7] is again very similar to the coefficient of both recovered binders from PA mixtures (MQ and MS) from Mookhoek et al. [13]. These results show that a moderate temperature ageing procedure (60 - 85°C) of asphalt mixtures appears to be relatively consistent in reproducing field aged characteristics, whereas the artificial ageing of only mastics appears not.

### 3.3 Pressure ageing vessel

The PAV ageing method was designed to simulate the ageing of binder during asphalt production, the construction of the pavement and the ageing during service life of the pavement. Liu [9] performed PAV ageing according to the European standard EN 14769 [14]. A binder amount of  $50 \pm 0.5$  g was poured in a PAV pan with a diameter of 140 mm. The thickness of the binder is about 3.2 mm. The pans with the binders were placed in the PAV for 20 hours at a temperature of 100°C and a pressure of  $2.1 \pm 0.1$  MPa.

In the ASAP project [10] an equivalent standard was used for PAV ageing (AASHTO R28) [15]. In this project binders were aged with the PAV for different durations and at different temperatures at  $2.1 \pm 0.1$  MPa. Binders were aged at 60°C and 80°C with durations of 96, 192, 336 and 504 hours. At 100°C binders were only aged for a duration of 20 hours, analogous to the ageing time performed by Liu [9]. The results of both investigations are shown in the black space graph in Figure 5.





**Figure 5: Changes in magnitude and phase angle of the complex modulus by PAV ageing compared to the positions and slopes of the PMB (red line) and Q8 70/100 (green line) field ageing relations**

From Figure 5 it can be observed that PAV ageing has a different effect on the ratio between  $\log |G^*|$  and  $\delta$  when compared to field and temperature ageing. The slopes of the lines through all the PAV data are surprisingly similar, hence all slopes are parallel to each other. The coefficient of the slopes in the black space graph for PAV however are clearly lower than those obtained for field ageing and temperature ageing (Table 4). This means that with PAV ageing  $\delta$  decreases, but that  $|G^*|$  does not increase correspondingly.

**Table 4: Exponents in the black space graph for different PAV aged binders**

Material	Exponent
Q8 40/60 (Liu, 2011)	-0.073
Q8 70/100 (Liu, 2011)	-0.074
AZ1-1 (ASAP, 2010)	-0.072
AZ1-2 (ASAP, 2010)	-0.075
AZ1-3 (ASAP, 2010)	-0.071
AZ1-4 (ASAP, 2010)	-0.072

An interesting observation is that the value for the coefficients found for the PAV ageing of binders is comparable to those found for the RTFOT ageing. Both artificial ageing procedures appear to yield lower coefficients; resulting in less advancement in  $|G^*|$  as  $\delta$  decreases. Fact is that both RTFOT and PAV deal with relative high temperature or elevated pressure ageing and deal with pure binder only, thereby possibly introduction unwanted side effects and omitting effects of the other asphalt constituents. Since it was found that temperature ageing of asphalt mixtures (PA) was much more representative to ageing field ageing of PA, it is discussed here whether ageing techniques should be less accelerated, be performed at lower temperatures and pressures or that ageing in the field can only be simulated when the effects of filler, sand, aggregates and the correct sample geometry are taken into account. Based on the results presented here, it should be investigated further whether for instance PAV ageing on whole asphalt mixtures (PA) is able to provide similar characteristics of ageing comparable to the field ageing in black space or whether moderate temperature ageing of pure binder (at atmospheric pressure) is able to do the same. This might conclude if either moderate temperatures, or taking sample geometry and/or the presence of filler, sand and aggregate more into account has the best impact on improving the standard ageing procedures for PA.

#### 4. CONCLUSIONS



From the results presented in this paper it is concluded that there is a clear trend of increasing  $|G^*|$  and decreasing  $\delta$  over time in the field for PAC and that the slope of change for the two parameters stays relatively constant. For all PAC roads and binders, a similar ageing relation and corresponding slope is found in the black space graph. The ageing relations in the black space graph found for the PAV and RTFOT laboratory ageing deviates from the field ageing relation. In comparison to the ageing relation for the laboratorial oven ageing at moderate temperatures is in good agreement with the ageing relation of the PAC binder in the field. It is believed that this shows that more forced ageing because of higher temperatures in for example RTFOT and more forced ageing because of higher pressures in for example PAV, a difference in sample geometry (binder film thicknesses) and due to the absence of other pavement constituent such as filler, sand and aggregates unwanted ageing side-effects may take place in the binder. At this moment it is therefore proposed for PAC to perform laboratory binder ageing at moderate temperatures and pressures in an asphalt concrete mixture consisting of filler, sand and aggregates.

## REFERENCES

- [1] Unravelling Porous Asphalt Concrete towards a Mechanistic Material Design Tool, Huurman, R.M., Mo, L. & Woldekidan, M.F., Road Materials and Pavement Design, 11:3, 583-612, 2010
- [2] Asphalt Oxidation Mechanisms and the Role of Oxidation Products on Age Hardening Revisited, Petersen, J.C. & Glaser, R., Road Materials and Pavement Design, 12:4, 795-819
- [3] Characterisation of ageing processes on the asphalt mixture surface, Lopes, M., Zhao, D., Chailleux, E., Kane, M., Gabet, T., Petiteau, C. & Soares, J., Road Materials and Pavement Design, 15:3, 477-487, 2014
- [4] Development of a new method for assessing asphalt binder durability with field evaluation, Glover, C.J., Davidson, R.R., Domke, C.H. Ruan, Y. Juristyarini, P., Knorr, D.B. Jung, S.H., Federal Highway Administration and Texas Department of Transportation, Report # FHWA/TX-05/1872-2, 2005
- [5] UV and Thermal Aging of Pure Bitumen-comparison Between Laboratory Simulation and Natural Exposure Aging, Wu, S., Pang L., Mo, L., Qiu, J., Zhu, G. & Xiao, Y., Road Materials and Pavement Design, 9:sup1, 103-113, 2008
- [6] Response Modelling of Bitumen, Bituminous Mastic and Mortar, Woldekidan, M.F., Ph.D. dissertation, Delft University of Technology, 2011
- [7] The Effect of Aging on Binder Properties of Porous Asphalt Concrete, Hagos, E.T., Ph.D. dissertation. Delft University of Technology, 2008
- [8] Using black space diagrams to predict age-induced cracking, King, G., Anderson, M., Hanson, D. & Blankenship, P., Proceedings of the 7th RILEM International Conference on Cracking in Pavements, 2012
- [9] Characterization and Identification of Bituminous Materials Modified with Montmorillonite Nanoclay, Liu, G., Ph.D. dissertation, Delft University of Technology, 2011
- [10] Asphalt Surface Aging Prediction (ASAP) System, Western Research Institute, Final Report, September 2010
- [11] Bitumen and bituminous binders - Determination of the resistance to hardening under influence of heat and air - Part 1: RTFOT method, EN 12607-1, 2007
- [12] Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test). Standard Specifications for Transportation Materials and Methods of Sampling and Testing, American Association of State Highway and Transportation Officials AASHTO T-240, 27th Ed. Washington, D.C
- [13] Accelerated ageing protocols for (polymer modified) PA to obtain representative (rheological) properties, mimicking field aged materials, Mookhoek, S.D., Liu, G., Erkens, S.M.J.G., Giezen, C. & J.L.M. Voskuilen, Proceedings of the International Conference on Ageing of Materials & Structures, Delft 26-28 May 2014, the Netherlands
- [14] Bitumen and bituminous binders - Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV), EN 14769, 2005
- [15] Accelerated Aging of Asphalt binder Using a Pressurized Aging Vessel (PAV). Standard Specifications for Transportation Materials and Methods of Sampling and Testing, American Association of State Highway and Transportation Officials AASHTO R28, 27th Ed. Washington, D.C.

