International Journal of Pavement Engineering, 2015
http://dx.doi.org/10.1080/10298436.2014.993204

Taylor & Francis
Taylor & Francis Group

Towards an optimised lab procedure for long-term oxidative ageing of asphalt mix specimen

Daniel Steiner[a], Bernhard Hofko[a]*, Markus Hospodka[a], Florian Handle[b], Hinrich Grothe[b], Josef Füssl[c], Lukas Eberhardsteiner[a] and Ronald Blab[a]

[a]*Institute of Transportation, Vienna University of Technology, Gusshausstrasse 28/230/3, Vienna 1040, Austria;* [b]*Institute of Materials Chemistry, Vienna University of Technology, Getreidemarkt 9/BC/01, Vienna 1060, Austria;* [c]*Institute for Mechanics of Materials and Structures, Vienna University of Technology, Karlsplatz 13/202, Vienna 1040, Austria*

(*Received 4 November 2014; accepted 23 November 2014*)

Ageing of bitumen leads to increased stiffness and brittleness. Thus, bituminous bound pavements become more prone to failure by low-temperature and fatigue cracking. Therefore, the ageing behaviour of bitumen has a crucial impact on durability, as well as recyclability of pavements. To assess ageing of bitumen, the rolling thin film oven test and pressure ageing vessel are standardised methods for short-term and long-term ageing in the lab. For lab-ageing of hot mix asphalt (HMA), various methods have been developed in the last decades. This paper presents a study on the potential of employing a highly oxidant gas for simulating the long-term oxidative ageing of asphalt mix specimens in the lab. Based on the results, an optimised lab-ageing procedure (Viennese Ageing Procedure – VAPro) for compacted HMA specimens to assess mix performance of long-term lab-aged specimens is developed. Thus, it is possible to optimise mix design not only for short-term performance but to take into account effects of oxidative ageing during its in-service life. VAPro is based on a triaxial cell with forced flow of a gaseous oxidant agent through the specimen. The oxidant agent is enriched in ozone and nitric oxides to increase the rate of oxidation. It is shown by stiffness tests of unaged and lab-aged specimens, as well as by Dynamic Shear Rheometer tests of recovered binder from aged specimens that asphalt mixes can be long-term aged at moderate temperatures (+60°C) and within 4 days and a flow rate of 1 l/min by applying VAPro. Thus, an ageing procedure is at hand that can simulate long-term ageing at conditions that are representative of conditions that occur in the field within an efficient amount of time.

Keywords: oxidative ageing; ozone; asphalt mix; bitumen; dynamic modulus; dynamic shear modulus; gaseous phase ageing

1. Introduction and background

Bitumen as an organic material is subject to changes in its behaviour throughout its life by thermal and oxidative ageing. In pavement engineering, ageing of bitumen and bituminous bound pavements is divided into short-term ageing (STA) in the process of HMA production and compaction within a few hours and long-term ageing (LTA) of a pavement during its in-service life within years. STA is triggered by fast oxidation due to high temperatures and a high specific surface contacting with oxidant agents at mix production, as well as evaporation of remaining volatile components from the bitumen (thermal ageing) (Petersen *et al.* 1994, Baek *et al.* 2012). LTA is driven by slow oxidation especially of the upper pavement layers by atmospheric oxygen and other highly oxidant gases available in the field (e.g. ozone, nitric oxides) (Morian *et al.* 2011). Bitumen becomes stiffer and more brittle, and thus, pavements are more prone to failure by low-temperature and fatigue cracking with increasing ageing of the binder (Teshale *et al.* 2011). Because bitumen ageing affects durability and recyclability of pavements crucially, it is important to assess ageing behaviour and resistance to ageing of binders and mixes at the stage of mix design optimisation to achieve cost and energy efficient pavements with low maintenance demands, a long service-life and high recycling potential.

To assess bitumen ageing in the lab within an efficient amount of time, the rolling thin film oven test (RTFOT) (CEN 2007a, ASTM 2012) and the pressure ageing vessel (PAV) (Airey 2003, Mallick and Brown 2004, CEN 2012, ASTM 2013) are standardised and widely accepted methods to transfer virgin binders into the state of STA (RTFOT) and LTA (RTFOT + PAV) (Lu and Isacsson 1998, Airey 2003, Mallick and Brown 2004, da Costa *et al.* 2010).

The mineral component and mix design of a pavement can have an impact on ageing of the mix. Thus, it seems important to have a standardised method for LTA of HMA in the lab as well. Therefore, HMA ageing procedures could assist in analysing changes of HMA material behaviour due to ageing from changes of binder behaviour. More than 30 lab-ageing procedure of loose or compacted asphalt mix have been developed in the last decades (Bell

*Corresponding author. Email: bernhard.hofko@tuwien.ac.at

et al. 1994, Çetinkaya 2011, Steiner 2014). Most of these methods have to be seen as critical due to the following reasons:

- For all methods that incorporate ageing of loose HMA before specimen compaction, it is questionable how binder ageing affects compatibility and quality of cohesion and adhesion of the compacted mix. Thus, it cannot be determined in the subsequent mechanical testing of these specimens whether difference in results between unaged and aged specimens are due to oxidative ageing or rather due to imperfect compaction.
- High temperatures (+100°C and higher) that are used in ageing protocols for loose HMA exceed temperatures that usually occur in surface layers of pavements. Additional thermal effects (e.g. vaporisation of further volatile binder components) could be activated that cannot occur in the field. In addition, high temperature could lead to other chemical reactions than in the field, like increased oligomerisation and polymerisation with less decomposition reactions.
- The duration of existing ageing protocols for compacted specimens is quite high for some methods (even up to several weeks). Thus, it is questionable whether these methods could be applied for routine testing in the future efficiently.
- To increase the oxidation rate, some protocols for compacted specimens apply high pressures (comparable to pressures in the PAV). Again, these conditions could lead to other chemical reactions than those occurring in the field.

Thus, the main objective of an on-going research project is to develop a new or optimise an existing procedure for ageing of compacted HMA specimens in the lab. To develop an efficient procedure that produces realistically lab-aged specimens for further mechanical testing, an emphasis was put on the following issues:

- Temperature and pressure should not exceed values that are regularly achieved within a surface layer to prevent chemical reactions in the lab that could not occur in the field.
- Increased oxidation should be achieved by using high concentrations of highly reactant gaseous agents that occur in the field in lower concentrations.
- To be applicable for future routine use in the practice, it is important that the procedure can be carried out within a reasonable amount of time.

The procedure presented within this paper can be seen as an extension of a method used within SHRP-A-383 (Bell et al. 1994). The paper contains

- the principles of the developed ageing procedure,
- an in-depth parameter study on impacts of temperature and duration of ageing on the viscoelastic behaviour of a typical surface layer mix, as well as of bitumen recovered from lab-aged specimens,
- a preliminary analysis of the repeatability of the method and
- a set of parameters for the procedure based on a benchmark of RTFOT + PAV aged bitumen samples.

2. Materials and test methods

2.1. *Materials*

For the presented study, an asphalt concrete with a maximum nominal aggregate size of 11 mm (AC 11) was employed. The coarse aggregates used for the mix is a porphyrite, the filler is powdered limestone. As a binder an unmodified 70/100 pen (PG 58-22) was used. The main characteristics of the binder are listed in Table 1.

The binder content was set to 5.2% by mass with a target void content of 8.0% by volume. The maximum density of the AC 11 70/100 was determined to be 2.282 kg/m^3. The grading curve is shown in Figure 1.

2.2. *Specimen preparation*

The mix was prepared in a laboratory reverse-rotation compulsory mixer, according to EN 12697-35 (CEN 2007c), with a mixing temperature of +165°C. HMA

Table 1. Main characteristics (mean values) of binder.

Parameter	70/100 pen
Penetration (1/10 mm)	91
Softening Point Ring & Ball (°C)	46.8
SHRP Performance Grade (°C)	58–22

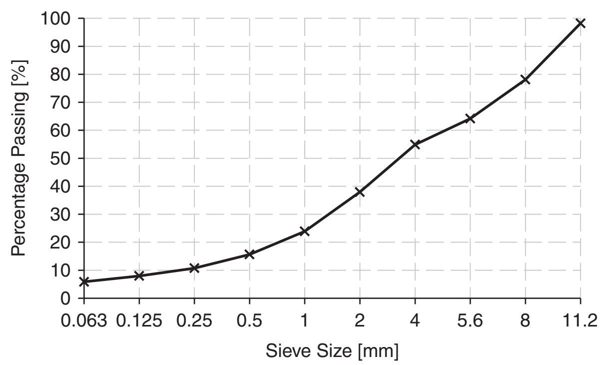

Figure 1. Grading Curve of AC 11.

Slabs ($50 \times 26 \times 4$ cm) were compacted in a roller compactor according to EN 12697-33 (CEN 2007b). The compacter consists of a roller segment for compacting the slabs, which corresponds to the dimensions of a standard roller compactor used in the field. All slabs were compacted with one lift. From the slabs, eight specimens are cored out with a diameter of 100 mm. The air void content of the specimens range from 6.8% to 8.7 % by volume.

For bitumen testing, bitumen was extracted according to EN 12697-3 (CEN 2013) with tetrachloroethylene (C_2Cl_4) as a solvent. The solvent-bitumen solution was distilled according to EN 12697-3 (CEN 2013) to recover the binder samples.

2.3. *Gaseous phase ageing (GPA) – Viennese Ageing Procedure (VAPro)*

Figure 2 shows the setup and equipment which was used for VAPro. Compressed air at ambient temperature was supplied from the local laboratory system and passed a pressure regulator, which ensures a constant flow rate and gas pressure. The subsequent ozone generator, using a dielectric barrier discharge tube (Kogelschatz 2003), enriches the compressed air with ozone and nitrogen oxides. The ozone generator can produce ozone using oxygen in the range of 1.0–1.2 g/h. Using air instead of oxygen helps to reduce the operating costs. Furthermore, the presence of nitrogen and nitrogen oxides enables reaction paths for the formation of ozone molecules, and this corresponds better to what happens in the field, respectively (Stanley 1999). This gas mixture flows through a coiled Cu–Ni tubing, where it is heated up to $T_{\text{liq}} = +70°C$. Therefore, the coil is placed in a beaker glass, filled with vegetable oil and positioned on a heatable magnetic stirrer.

The HMA specimen is assembled within a triaxial cell between two filter stones and is covered by an elastic membrane. A slight overpressure of 80 kPa was applied in the triaxial cell to force the gas mixture to flow through the specimen instead of passing on the outside. The triaxial cell and setup for heating up the gas are located in a heating cabinet, where the temperature T_{air} was varied for the first experimental run.

2.4. *Dynamic modulus testing of HMA*

Cyclic indirect tensile tests (IT-CY) were carried out on all specimens before and after ageing at a temperature of $+10°C$ and frequencies ranging from 0.1 to 20 Hz by applying a sinusoidal load. From test data the dynamic modulus $|E^*|$ and the phase angle φ can be determined to describe the viscoelastic behaviour of the specimen (Di Benedetto et al. 2001).

In a series of pretests, the upper stress level of the sinusoidal load (see Table 2) was determined so that the elastic horizontal strain amplitude of the specimen during testing is lower than 5×10^{-5} m/m. It was shown that repeated tests on the same specimen are possible with these loading conditions (Steiner 2014). This is a necessary precondition before all specimens were tested twice, before and after lab ageing.

2.5. *Dynamic shear modulus of bitumen*

Dynamic Shear Rheometer (DSR) tests were carried out on bitumen samples recovered from all lab-aged HMA specimens. The test conditions were chosen according to

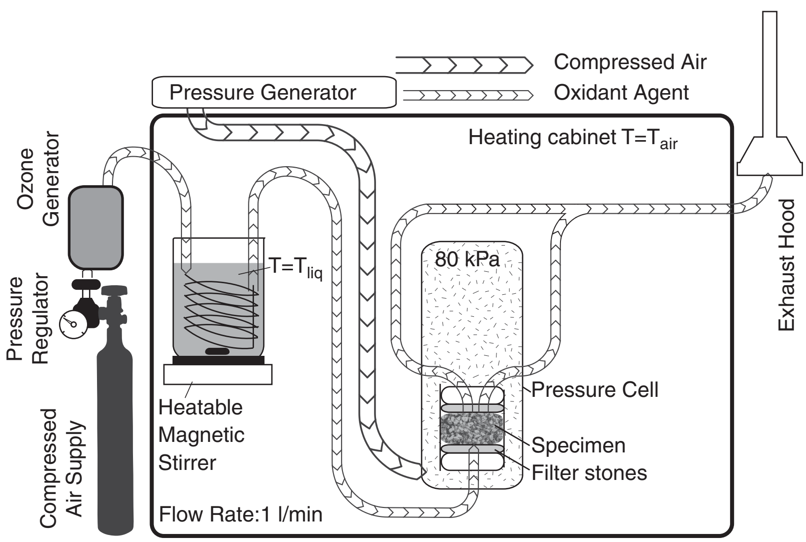

Figure 2. Aging setup for VAPro.

Table 2. Test conditions for IT-CY| + 10°C.

Frequency (Hz)	0.1	1	5	10	20
Lower stress level (kPa)	35	35	35	35	35
Upper stress level (kPa)	210	240	300	360	390
Number of load cycles [−]	9	15	50	100	100

the SHRP procedure (Petersen *et al.* 1994) and EN 14770 (CEN 2005) with a temperature sweep from +46 to +82°C using the small plate (diameter: 8 mm) and a 2-mm gap. A frequency of 1.6 Hz is employed. From test data, the dynamic shear modulus $|G^*|$ and the phase angle φ against frequency are determined.

2.6. *Thermal ageing effects in VAPro*

To validate the ageing procedure presented in this paper, it is necessary to analyse and differentiate the oxidative from the thermal impact on ageing. To investigate whether any thermal ageing occurs in VAPro, an HMA specimen was placed within the triaxial cell. Instead of the oxidant gas, the specimen as well as the triaxial cell was saturated with nitrogen to prevent any oxidation. The triaxial cell was placed in the heating cabinet for 4 days at +75°C. Stiffness tests were carried out on the specimen before and after ageing, as well as DSR tests on the recovered binder from the aged specimen.

3. Experimental programme

Table 3 gives an overview of the test programme on HMA specimens on the left and on recovered binder samples on the right. The first part of the study looked into the impact of temperature T_{air} and ageing time. Therefore, three different temperatures T_{air} = +45/+60/+75°C were combined with ageing times of 1, 2, 3 and 6 days. The results of this first phase should give an overview of the expected increase of dynamic modulus $|E^*|$ and dynamic shear modulus $|G^*|$.

In the second part, the dynamic shear modulus $|G^*|$ of an RTFOT + PAV-aged binder was set as a benchmark to determine which conditions (ageing time, T_{air}) in VAPro match an RTFOT + PAV ageing of bitumen. This benchmark to set ageing conditions for VAPro will be replaced in the future by data from actually field-aged samples that will be taken from a test field in Vienna (Hofko *et al.* 2014). Furthermore, the repeatability of VAPro was examined by a replication of ageing on three specimens.

4. Discussion and laboratory test results

4.1. *Investigation into thermal ageing effects*

4.1.1. *Hot Mix Asphalt*

To check VAPro for any non-oxidative, i.e. thermal ageing effects due to elevated temperatures, a specimen was stored in the triaxial cell under nitrogen atmosphere at +75°C for 4 days. Stiffness tests were run on the specimen before and after the storage to analyse changes in the behaviour due to nitrogen storage. Figure 3 shows the relative change in the dynamic modulus $|E^*|$ after nitrogen storage vs. before storage for 3 frequencies in the left diagram. The right diagram shows the change in the phase lag. Both diagrams indicate that no significant change in the material behaviour occurs due to thermal effects. The dynamic modulus is reduced by 5–10%, the phase angle φ increases by 1.0°–2.2°. Thus, it can be stated that no effect of increasing HMA stiffness due to temperature can be found for VAPro.

Table 3. Test programme for lab ageing of HMA specimen and IT-CY, DSR tests.

		IT-CY +10°C 0.1–20 Hz					DSR SHRP 1.6 Hz 46–82°C		
		Temperature T_{air} [°C]					Temperature T_{air} [°C]		
		45	60	75			45	60	75
Ageing time [d]	1	B/A	B/A	B/A	VAPro	Long-term aged	x	x	x
	2	B/A	B/A	B/A			x	x	x
	3	B/A	B/A	B/A			x	x	x
	4							3x	
	6		2x B/A	B/A				2x	x
	4			B/A	N_2 storage				x
					RTFOT + PAV				
					Control specimen C&R	Short-term aged		x	
								x	
								x	
					RTFOT				
						Virgin		x	

B – Tested before ageing
A – Tested after ageing
C&R – Compacted and recovered

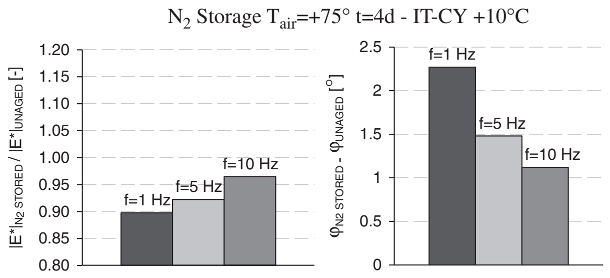

Figure 3. Change in dynamic modulus $|E^*|$ (left) and phase angle φ (right) of N_2 stored HMA specimen relative to unaged condition.

4.1.2. *Bitumen*

A second check on potential effects from temperature was carried out. Bitumen was extracted and recovered from the nitrogen stored specimen, as well as from a control specimen that was just compacted without any further ageing. DSR tests were run on these samples.

The results are presented in Figure 4. It shows the relative change in dynamic shear modulus $|G^*|$ from recovered bitumen samples vs. virgin bitumen. Both, the control specimen and the nitrogen stored specimen deliver very similar results that are close to the RTFOT-aged binder sample. Thus, no thermal effect can be found from the bitumen analysis either.

4.2. *Impact of temperature and duration on ageing*

4.2.1. *Hot Mix Asphalt*

HMA specimens were subjected to VAPro for 1, 2, 3 and 6 days at temperatures ranging from +45 to +75°C at a constant flow rate of the oxidative gas of 1 l/min. The specimens were tested for their viscoelastic behaviour (dynamic modulus $|E^*|$ and phase angle φ) in the IT-CY at +10°C and frequencies ranging from 0.1 to 20.0 Hz. All specimens used for the study were tested before and after ageing to directly compare changes in its behaviour due to VAPro. The results are shown in Figure 5. The diagram shows the relative change in dynamic modulus $|E^*|$ of the

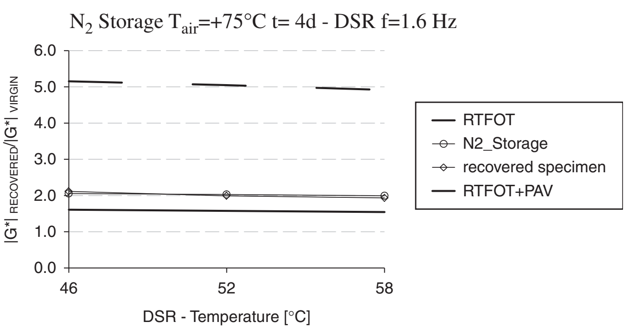

Figure 4. Change in dynamic shear modulus |G*| of bitumen recovered from N_2 stored HMA specimen to virgin bitumen sample.

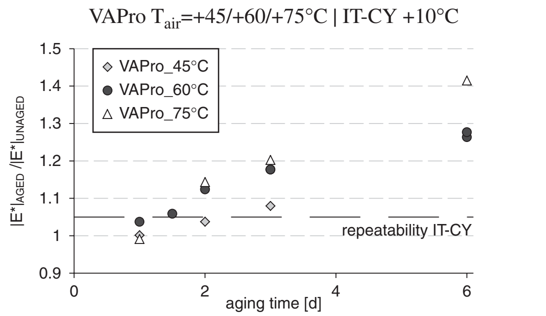

Figure 5. Change in dynamic modulus $|E^*|$ of lab-aged HMA specimen relative to unaged condition.

aged specimen vs. unaged specimen over the duration of ageing. Results for +45, + 60 and +75°C are depicted. The data shown in the diagram are mean values (MV).

At all temperatures a logarithmic increase of stiffness can be observed with increasing ageing time. At +45°C ageing durations between 1 and 3 days show no significant changes in the stiffness of the aged specimens because the data is within the repeatability of the test method. At +60 and +75°C, changes in stiffness become significant after 2 days of ageing, though no difference between +60 and +75°C can be seen between 1 and 3 days of ageing. After 6 days of ageing, results from ageing at +60 and +75°C show clear differences. While the specimens aged at +60°C for 6 days come to an increase in stiffness of around 30%, the increase in stiffness at +75°C and 6 ageing days is over 40%.

4.2.2. *Bitumen*

From all VAPro-aged HMA specimens, bitumen was extracted and recovered for analysis of changes of the viscoelastic behaviour in the DSR. In addition, STA bitumen by RTFOT and LTA bitumen by RTFOT + PAV was tested as well to compare standardised bitumen ageing procedures to VAPro.

Results are presented in Figure 6. Analogue to the test results shown for the HMA specimens in Figure 5, the diagram in Figure 6 shows the relative change in the dynamic shear modulus $|G^*|$ of bitumen extracted from VAPro-aged HMA specimens vs. virgin bitumen over the duration of ageing. Data was obtained from SHRP DSR testing at 1.6 Hz and +46°C. The dotted lines represent data from the RTFOT and RTFOT + PAV aged bitumen. Similar to the data from stiffness tests on HMA specimens, no significant changes in the binder seem to occur for 1–3 days of ageing. Independent from ageing temperature, all recovered bitumen samples show stiffness similar to RTFOT aged binder. After 4 days of VAPro at +60°C, the binder is in the state of RTFOT + PAV aged bitumen. After 6 days of VAPro at +60°C, the extracted binder is 2–3 times stiffer than RTFOT + PAV aged binder. From the results of the +60°C aged samples, it is obvious that different from the evolution of HMA stiffness with

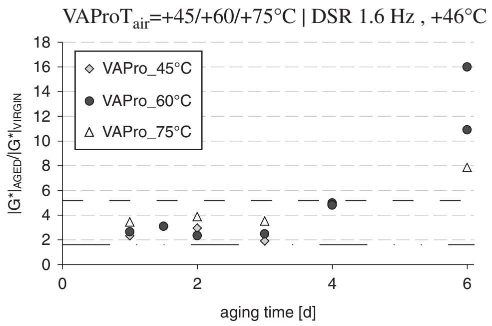

Figure 6. Change in dynamic shear modulus $|G^*|$ of bitumen recovered from lab-aged HMA specimen to virgin bitumen sample.

duration of ageing (Figure 5), the stiffness of the extracted binder samples show a non-linear, exponential increase with increasing duration of ageing.

4.3. *VAPro Repeatability*

For a preliminary analysis of the repeatability of VAPro, three single HMA specimens were aged at +60°C for 4 days and a flow rate of 1 l/min. The reason for this set of ageing parameters is that the preliminary test programme showed that the extracted binder behaves similarly to RTFOT + -PAV aged bitumen at these conditions. The viscoelastic behaviour of RTFOT + PAV aged bitumen is the temporary benchmark for the set of parameters of VAPro. It will be replaced by actual data from field-aged HMA samples taken from a test field that was constructed with the same mix design in summer 2012 (Hofko *et al.* 2014) as soon as data from these field-aged samples become available.

Subsequent to the HMA ageing, binder was extracted and recovered separately for each specimen and SHRP DSR tests were run at 1.6 Hz from +46°C to +82°C. Figure 7 shows the results in two diagrams. The left diagram contains the relative change in dynamic shear modulus $|G^*|$ over the test temperature from +46°C to +58°C. The right diagram shows the change in phase angle φ over the test temperature. The data from the extracted binder is a mean

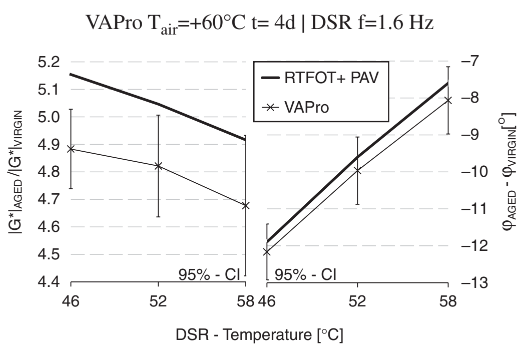

Figure 7. Change in dynamic shear modulus $|G^*|$ (left) and phase angle φ (right) of bitumen recovered from VAPro-aged HMA specimen to virgin bitumen sample.

value from three samples together with the 95% confidence interval. The bold line represents the results of an RTFOT + PAV aged binder. As it can be seen from both diagrams in Figure 7, the chosen ageing conditions (+60°C, 4 d) lead to binder that behaves similar to RTFOT + PAV aged samples. The VAPro binder is 4.7 to 4.9 times stiffer than the virgin bitumen, whereas the RTFOT + PAV sample is 5.05 to 4.9 times stiffer. In terms of change in phase angle φ, both, the VAPro and RTFOT + PAV aged samples show a decrease between 8° and 12°.

5. Conclusions and future research

The main drive for the research project presented within this paper is to develop an efficient lab-ageing method for compacted HMA specimens to assess long-term performance at the stage of mix design optimisation in the future.

The presented ageing procedure, Viennese Ageing Procedure (VAPro), is an extension of a procedure used within SHRP-A-383 (Bell et al. 1994). It is based on a triaxial cell with forced flow of a highly oxidative gaseous agent through the specimen at elevated temperatures. In contrary to existing ageing methods for loose or compacted HMA, VAPro works at temperatures that regularly occur on surface layers in summer (+45°C to +75°C). It seems important to keep the temperatures as close to the field as possible in order not to enable any chemical reactions that cannot occur in the field. The oxidation rate is increased by using a compressed air that is enriched in highly oxidative ozone and nitric oxides. Both gases also occur in the field, just in smaller concentrations.

To investigate changes in the viscoelastic behaviour of the HMA specimens, cyclic indirect tensile tests were run on the unaged and lab-aged samples. Changes in the dynamic modulus $|E^*|$ were compared. In addition bitumen was extracted and recovered from all lab-aged HMA specimens and DSR tests were run to analyse changes in the bitumen behaviour and to compare the changes to virgin bitumen samples.

In a preliminary analysis, it was proven by using nitrogen instead of the oxidant gases that no thermal ageing occurs within VAPro because nitrogen storage at +75°C for 4 days did not result in significant changes of the viscoelastic behaviour of HMA and bitumen.

VAPro was carried out on HMA specimens at +45°C, +60°C and +75°C with ageing time ranging from 1 to 6 days to investigate the impact of temperature and time on the ageing behaviour of the mix. From the stiffness tests carried out on the HMA specimens, it was found that ageing times of 3 days and less lead to no significant ageing at +45°C. At +60°C and +75°C, significant ageing occurs after 2 days of ageing. Significant differences between the two temperatures are only visible after 6 days of ageing. In general, there is a linear trend between increase in stiffness and ageing time in VAPro.

From the extracted binder samples from VAPro-aged HMA samples, it becomes obvious that the stiffness in terms of dynamic shear modulus $|G^*|$ increases in a non-linear, exponential way with ageing time. For ageing times of 3 days and less, no significant impact can be found on the bitumen behaviour for any of the three ageing temperatures applied. Four days of ageing at +60°C is similar to RTFOT + PAV ageing; 6 days of ageing leads to bitumen stiffness that is 2–3 times higher than RTFOT + PAV aged samples.

In a first repeatability study with three HMA specimens aged at +60°C for 4 days, the repeatability was found to be satisfying.

Because the results presented within this paper are encouraging, research on VAPro will be continued:

- A detailed analysis of the oxidant gases with a residual gas analyser (RGA) will be carried out to optimise the gas mix in terms of flow rate and concentration of oxidising gases.
- A test field with the same mix used as for this study, which has been constructed in summer 2012 (Hofko *et al.* 2014), will be sampled regularly to analyse field aged mixes. Therefore, mechanical and chemical investigation of HMA and recovered bitumen samples will be carried out and compared to data from the VAPro-aged samples to check the procedure for compliance with field ageing. The field-aged samples will also be used as a benchmark that is closer to the field than the RTFOT + PAV aged samples that are used as a benchmark at the present stage.
- The ageing device will be adapted for different specimen dimensions to be able to study the low-temperature and fatigue resistance of lab-aged mixes in a future test programme.
- Impact of different mix design parameters, like the void and binder content or the mineral source on ageing behaviour will be studied.
- Also, the impact of various HMA modifiers that should reduce ageing (e.g. (Sustersic *et al.* 2013)) can be studied in the lab thoroughly before being used in a large-scale test field.

Funding

The authors gratefully acknowledge financial support from the Austrian Research Promotion Agency (FFG) and the sponsors Pittel + Brausewetter, Swietelsky and Nievelt through project 'OEKO-PHALT – Physical-chemical fundamentals on bitumen ageing'.

References

Airey, G.D., 2003. State of the art report on ageing test methods for bituminous pavement materials. *International Journal of Pavement Engineering*, 4 (3), 165–176.

ASTM, 2012. ASTM D2872 – 12e1: Standard test method for effect of heat and air on a moving film of asphalt (rolling thin-film oven test), West Conshohocken, PA.

ASTM, 2013. ASTM D 6521-13: Standard practice for accelerated aging of asphalt binder using a pressurized aging vessel (PAV), West Conshohocken, PA.

Baek, C., Underwood, B., and Kim, Y., 2012. Effects of oxidative aging on asphalt mixture properties. *Transportation Research Record: Journal of the Transportation Research Board*, 2296 (-1), 77–85.

Bell, C.A., *et al.*, 1994. *Selection of laboratory ageing procedures for asphalt aggregate mixtures (SHRP-A-383)*. Washington, DC: National Reserach Counil.

CEN, 2005. EN 14770: Bitumen and bituminous binders – determination of complex shear modulus and phase angle – Dynamic Shear Rheometer (DSR), Brussels.

CEN, 2007a. EN 12607-1: Bitumen and bituminous binders – determination of the resistance to hardening under the influence of heat and air – Part 1: RTFOT method, Brussels.

CEN, 2007b. EN 12697-33: Bituminous mixtures – test methods for hot mix asphalt – Part 33: Specimen prepared by roller compactor, Brussels.

CEN, 2007c. EN 12697-35: Bituminous mixtures – test methods for hot mix asphalt – Part 35: Laboratory mixing, Brussels.

CEN, 2012. EN 14769: Bitumen and bituminous binders – accelerated long-term ageing conditioning by a pressure ageing vessel (PAV), Brussels.

CEN, 2013. EN 12697-3: Bituminous mixtures – Test methods for hot mix asphalt – Part 3: Bitumen recovery: Rotary evaporator, Brussels.

Çetinkaya, R., 2011. Bewertung der Einflussgrößen auf die thermisch-oxidative Alterung von Bitumen im Asphalt. Bochum: Europ. Univ.-Verl.

da Costa, M.S., *et al.*, 2010. Chemical and thermal characterization of road bitumen ageing. *Advanced Materials Forum V*, 636–637 (Pt 1 and 2), 273–279.

Di Benedetto, H., *et al.*, 2001. Stiffness testing for bituminous mixtures. *Materials and Structures*, 34 (236), 66–70.

Hofko, B., *et al.*, 2014. Impact of field ageing on low-temperature performance of binder and hot mix asphalt. Proceedings of the 12th ISAP Conference on Asphalt Pavements, Raleigh, NC.

Kogelschatz, U., 2003. Dielectric-barrier discharges: their history, discharge physics, and industrial applications. *Plasma Chemistry and Plasma Processing*, 23 (1), 1–46.

Lu, X.H. and Isacsson, U., 1998. Chemical and rheological evaluation of ageing properties of SBS polymer modified bitumens. *Fuel*, 77 (9–10), 961–972.

Mallick, R.B. and Brown, E.R., 2004. An evaluation of superpave binder aging methods. *International Journal of Pavement Engineering*, 5 (1), 9–18.

Morian, N., *et al.*, 2011. Oxidative aging of asphalt binders in hot-mix asphalt mixtures. *Transportation Research Record: Journal of the Transportation Research Board*, 2207 (-1), 107–116.

Petersen, J.C., *et al.*, 1994. *Binder characterization and evaluation volume 4: Test methods (SHRP-A-370)*. Washington, DC: National Reserach Counil.

Stanley, B.T., 1999. *Feedgas for modern high-performance ozone generators*. Duebendorf: Ozonia Ltd.

Steiner, D., 2014. *Towards an optimized lab aging protocol for asphalt mix specimen to assess long-term aging*. Master Thesis (MSc). Vienna University of Technology.

Sustersic, E., Tusar, M., and Valant, A.Z., 2013. Rheological and mechanical characterization of waste PMMA/ATH modified bitumen. *Construction and Building Materials*, 38, 119–125.

Teshale, E.Z., *et al.*, 2011. Pressure aging vessel and low-temperature properties of asphalt binders. *Transport Research Record: Journal of the Transport Research Board*, 2207, 117–124.