Laboratory aging of asphalt mixtures: simulation of reclaimed asphalt and application as test method for durability

Konrad Mollenhauer, Virginie Mouillet, Nathalie Pierard, Marjan Tusar, Thomas Gabet

LABORATORY AGING OF ASPHALT MIXTURES – SIMULATION OF RECLAIMED ASPHALT AND APPLICATION AS TEST METHOD FOR DURABILITY

Konrad Mollenhauer[1], *Virginie Moulliet*[2], *Nathalie Piérard*[3], *Marjan Tusar*[4], *Thomas Gabet*[5]
[1]*University of Kassel, Institute of Transportation, Kassel, Germany*
[2]*Laboratoire Régional des Ponts et Chaussées, d'Aix-en-Provence, France*
[3]*Belgium Road Research Centre (BRRC), Brussels, Belgium*
[4]*Slovenian National Building and Civil Engineering Institute (ZAG), Ljubljana, Slovenia*
[5]*Division Matériaux (IFSTTAR), Nantes, France*

ABSTRACT

Surface asphalt courses reach their end of service life after a time span between 10 and 30 years, depending on their durability. Afterwards, the surface layer is usually milled and reused in asphalt mixtures as reclaimed asphalt. In order to enable the analysis of durability and recyclability of a new asphalt mixture, four laboratory aging procedures were designed and comparatively applied on twoasphalt mixes. Besides aging of loose asphalt mix in heating cabinets at varied temperatures and durations, the additional application of UV radiation and pressure were analyzed. To evaluate the aging effects standard as well as performance bitumen tests were conducted. The aging effects on chemical properties of recovered binders were evaluated as well as properties of the aged asphalt mix. The paper presents the differences obtained in aging properties between the applied aging procedure in order to give a indication for the applicability of these procedures in order to analyse the asphalt mix durability and recyclability.

Keywords: laboratory aging, asphalt properties, binder properties, reclaimed asphalt, durability

1. INTRODUCTION

Surface asphalt courses reach their end of service life after a time span between 10 and 30 years, depending on its durability. Afterwards, the surface layer is usually milled and reused in asphalt mixtures as reclaimed asphalt. During service life, the road surface is subjected to aging due to oxidation as well as UV radiation. This long-term aging can reduce the flexibility of the surface course which may lead to distresses such as top-down cracking of the pavement or raveling. In case of severe aging, the reuse of the surface asphalt in new asphalt mix as reclaimed asphalt may be limited.

Whereas the aging properties of binders are commonly investigated (e. g. by conducting short-term aging by means of Rolling Thin Film Oven Test according to EN 12607-1 and long-term aging by means of Pressure Aging Vessel according to EN 14769), the evaluation of aging properties of asphalt mix is still uncommon. Despite the aging of the asphalt mix is mainly influenced by the aging properties of the asphalt binder, the aggregates may influence the aging effects. Therefore, an aging procedure for analyzing the effect of aging on asphalt properties taking the whole asphalt mix into account is needed. Additionally, due to the change of mechanical properties during service time caused by aging the asphalt properties at the end of the service life of a pavement are of interest as well. In case of serviceability contracts, the material performance during service life shall be considered already during the mix design. Therefore, in order to evaluate the durability of its performance properties, an appropriate laboratory ageing method for conditioning asphalt mixes is needed. Such an evaluation may for example demonstrate the need to reactivate the aged binder during the recycling process.

In order to develop a laboratory aging method for asphalt mix to evaluate its recyclability as well as conditioning methods to be used to analyse durability properties, four aging procedures were applied on two surface asphalt mixes in the frame of the research project Re-Road [1].

As indicated by several researches, the aging of surface asphalt courses on site is influenced by several material properties and external parameters such as climate:

- The evolution of the binder properties upon field ageing (e. g. increase of softening point, decrease of penetration value) depends on the type of binder used [2, 3, 4, 5, 8] as well as type of modification [9, 10, 11].
- The higher the air void content of the surface layer, the deeper the oxidation in the exposed layer progresses. While aging occurs over the full thickness of the layer in porous asphalt mixes [8], it is almost limited to the upper 0.5 to 1 centimeter of the layer in case of dense bituminous layers (voids <5 %) [6, 7].
- The location of the road influences the aging effects due to weather conditions and sun exposure [6].

To simulate long-term aging of asphalt mix several procedures were developed based on following procedures:

- Aging of compacted specimens in oven with air ventilation [12]. Though, as the aging occurs presumably on the surface of the specimens, the conditioning causes inhomogeneous material properties.
- Aging of loose asphalt mix in heating cabinets with air ventilation results in thoroughly aged mix properties [13, 14, 15]. By compacting asphalt specimens from the asphalt mix before and after accelerated laboratory aging, the influence of the binder hardening on the asphalt mix performance can be evaluated [13]. The found effect of loose mix aging procedure on asphalt and binder properties were similar to the long-term aging effects observed on surface pavements [16].

This contribution presents results of 4 laboratory aging procedures conducted on 3 asphalt mixes in 5 laboratories. The objective is to analyse the applicability of these laboratory procedures as conditioning procedure to simulate the aging of asphalt mixtures during service life.

2. EXPERIMENTAL STUDY

2.1 Materials investigated

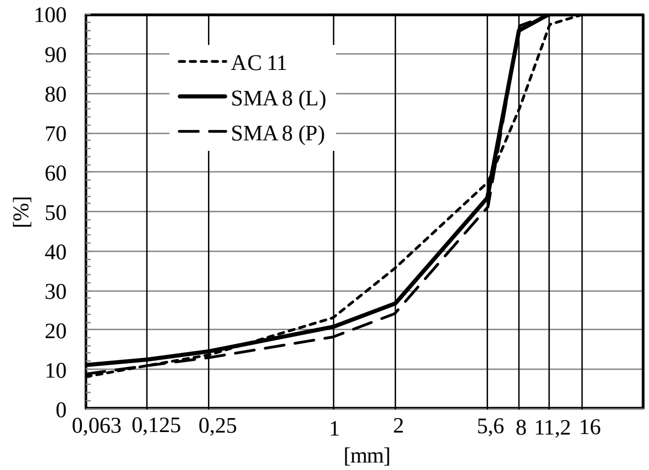
The laboratory aging procedures were conducted on 3 asphalt mixes produced in laboratory:

- AC 11 with binder 35/50,
- SMA 8 (P) with binder 25/55-55 A, mixed in plant,
- SMA 8 (L) with binder 25/55-55 A, mixed in Lab.

In the following, these mixes will be titled AC 11, SMA 8 (L) and SMA 8 (P). The compositions of the mixes are summarised in table 1. Whereas the plant-produced mix SMA 8 (P) was used to check the applicability of the aging procedures, the lab-produced mixes AC 11 and SMA 8 (L) were analysed in more than one laboratory each in order to derive indicators for the expected repeatability and reproducibility of these procedures. The mixes were produced in each laboratory separately according to a common mixing procedure. Directly after mixing, samples were taken for binder recovery. Asphalt mix samples were filled in buckets which were closed for further analyses. These samples were marked as “Fresh”.

For simulating the short-term aging occurring during production, hot storage at the asphalt plant, transport and site construction, the hot mix was stored on a tray in a heating cabinet at T = 135 °C for 4 hours based on previously study described in [15]. These sampled were marked as “STA” (Short-term aged).

Table 1 : Asphalt mix properties

Property	SMA 8 (P)	AC 11	SMA 8 (L)
Binder content [%]	7,0	5,6	7,4
Binder type [-]	25/55-55 A	35/50	25/55-55 A
Grading [%]			
Mixing procedure:	Plant-mixed	Lab-mixed, T = 165 °C, mixing time: 120 s	

2.2 Mix aging procedures

Four aging procedures for loose asphalt mixes were selected:

- BRRC-method: Aging of loose mix in a heating cabinet at a ambient temperature of 60 °C (comp. [15]),
- RILEM-method: Aging of loose mix in a heating cabinet at an ambient temperature of 85 °C (comp. [14]),
- PAV-method: Aging of loose mix spread on trays of a PAV device according to EN 14769,
- UV method: Aging of loose mix in heating cabinet at 60°C with additional vertical UV radiation.

Commonly for all aging procedures, the loose asphalt mix was spread on metal trays. The four procedures varied according the aging conditions temperature T, pressure p and additional UV radiation as indicated in table 2. To indicate suitable aging durations, primarily tests were conducted on sample SMA 8 (P) with the 4 aging procedures. The chosen aging times applied on mixes AC 11 and SMA 8 (L) are indicated in brackets. For procedures BRRC and RILEM, samples of asphalt mixes were taken after different aging durations in order to analyse the aging process.

To evaluate the effect of the aging procedures on additional mixes as well as to obtain information on the reproducibility of the aging procedures, the 4 aging methods were applied on the laboratory produced mixes AC 11 and SMA 8 (L).

Table 2 : Applied aging procedures

Name of Aging Procedure	Mixing temperature T [°C]	Short-term aging		Long-term aging			
		Temperature T [°C]	Duration [h]	Temperature T [°C]	Pressure p [MPa]	UV-Radi-ation	Aging duration t [days]
BRRC	165	135	4	60	-	-	0; 1; 3; 9; [14]
RILEM			1,5	85	-	-	0; 3; 6; [9]
PAV			1,5	90; 100	2,1	-	[1]; 5
UV			4	60	-	x	[14]

2.3 Binder tests

In order to evaluate the aging effects on the properties of recovered binders, following binder tests were conducted on samples recovered from aged asphalt mixes as well as the freshly mixed material and on the virgin binder. For binder recovery each laboratory standard procedure and solvent was used.

- Penetration according to EN 1426
- Softening temperature TR&B according to EN 1427
- Force ductility tests according to EN 13589.

Additionally, the binder’s chemical characteristics are evaluated by the maximum heights of the oxidation peaks around 1,700 and 1,030 cm^{-1} observed in IR spectrometry [17] and characteristic for the presence of carbonyl functions and sulfoxide functions in the binder respectively. Both are indicators of binder ageing. The presence of SBS polymer in the bitumen and its quantity are also evaluated by making use of the height of the peaks at 700 and 968 cm^{-1}. Each spectrum is beforehand normalized by following the procedure described in [17].

2.4 Asphalt tests
The freshly mixed asphalt as well as samples from the short-term aged and RILEM-aged asphalt were used to compact asphalt slabs according to EN 12697-33 resulting in similar void content. From these slabs asphalt specimens were cut in order to conduct TSRST (Thermal Stress Restrained Specimen Tests) for analysing the materials resistance against low-temperature cracking according EN 12697-46.

3. RESULTS

3.1 Results of primarily study to indicate suitable aging conditions.

The results of the binder tests conducted on samples recovered from asphalt mix SMA 8 (P) after several aging durations (for RILEM and BRRC-method) as well as after UV and PAV methods are shown in figure 1. The first value shown “-1” was measured on the virgin binder. The aging duration of 0 day represents the asphalt mix properties after mixing before the aging procedures were applied.
As shown for the BRRC and RILEM aging method, the mix aging results for the tested asphalt mix in a widely linear decrease of penetration as well as an increase of softening temperature. The change of binder properties is faster for the RILEM-aging method with an aging temperature of 85 °C compared to BRRC-method where a temperature of 60 °C was applied. At this temperature, additional UV radiation will decrease the penetration after 14 days of aging for additional 2 1/10 mm. The UV effect of softening temperature is about 2 °C if the difference in the initial value is taken into account.
With an aging temperature of T = 85 °C in nine days the penetration is reduced from 27 to 19 1/10 mm, whereas the softening temperature is increased from 69 to 73 °C.
Similar change of penetration and softening point is reached during 1 day (i. e. 20 h) of PAV aging at a temperature of 90 °C and an ambient air pressure of 2,1 MPa. If the temperature and duration is increased for the PAV aging method, the asphalt mix ages severely resulting in a penetration of only 2 1/10 mm and a softening point of 122 °C.

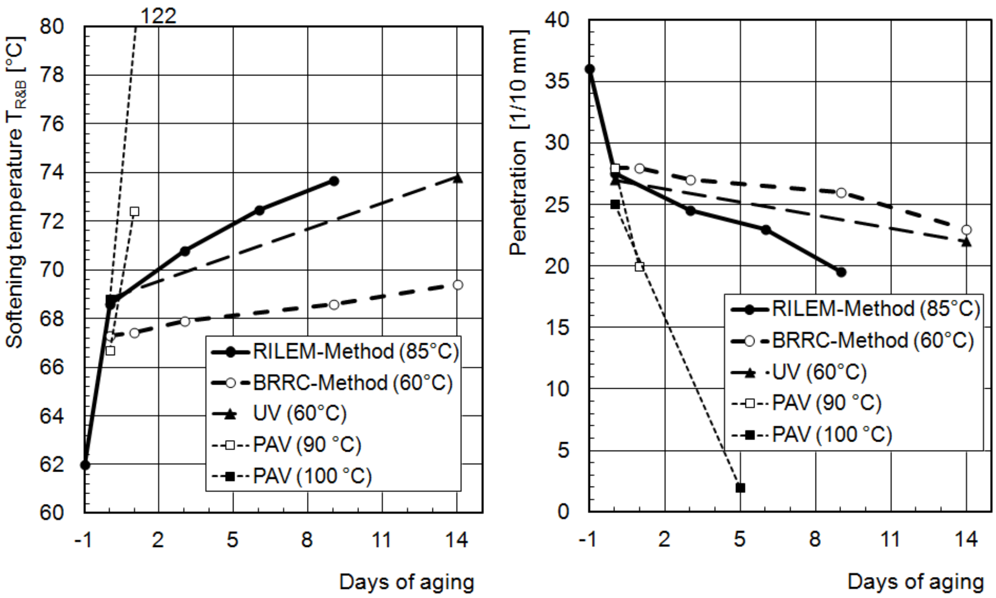


Figure 1 : Effect of laboratory aging on the properties penetration and softening point $T_{R\&B}$ of binders recovered from SMA 8 (P)

Figure 2 shows the results of IR spectroscopy on binder samples after different aging times in BRRC and RILEM-method. The increase of the oxidation peaks (carbonyl and sulfoxide functions) shown on the left is consistent to the evolution of the binder properties pen and $T_{R\&B}$. The oxidation peak reached after 14 day aging at 60°C (BRRC) is reached during 85°C-aging (RILEM) already after two days. During the laboratory aging procedures, the value of polymer peaks indicated in Figure 2 (right) seems slightly decrease for the butadiene peak whereas the styrene peak seems going down slowly during RILEM aging. But these changes are within the test accuracy and therefore not significant.
The results of the force ductility tests are shown in figure 3. For the RILEM aging procedure (85 °C), the aging results in higher force values for the initial peak, whereas the plateau indicating the influence of polymers is shortened. According to EN 13703 the deformation energy was calculated for the deflection 0 m to 0,2 m $E'_{0,2}$, representing the deformation energy controlled by the raw bitumen, and for the deflection 0,2 to 0,4 m $E'_{0,2\text{-}0,4}$, representing the deformation energy of the polymer plateau. A shown in figure 3, the deformation energy $E'_{0,2}$ increases due to proceeding aging. This is caused by higher force values generated due to higher stiffness/viscosity of the bitumen. For the deformation energy $E'_{0,2\text{-}0,4}$ the aging causes an increase first, also caused by higher force levels. With proceeding aging, the polymer plateau is reduced in length which reduces the deformation energy $E'_{0,2\text{-}0,4}$. In order to separate the aging of the polymer which causes a destruction of the polymer chains, the deformation energy ratio $E'_{0,4\text{-}0,2}$ / $E'_{0,2}$ was

calculated and plotted in figure 3. The higher the ratio, the higher is the deformation energy caused by the polymer compared to the deformation energy caused by the pure bitumen. As shown in figure 3, the deformation energy ratio is nearly the same for the pure binder and the sample recovered from the freshly mixed asphalt. The laboratory aging causes a decrease of the deformation energy ratio from 0.85 to 0.35 during 9 days of aging. Therefore the initial beneficial impact of polymer in the binder has decreased substantially after aging process.

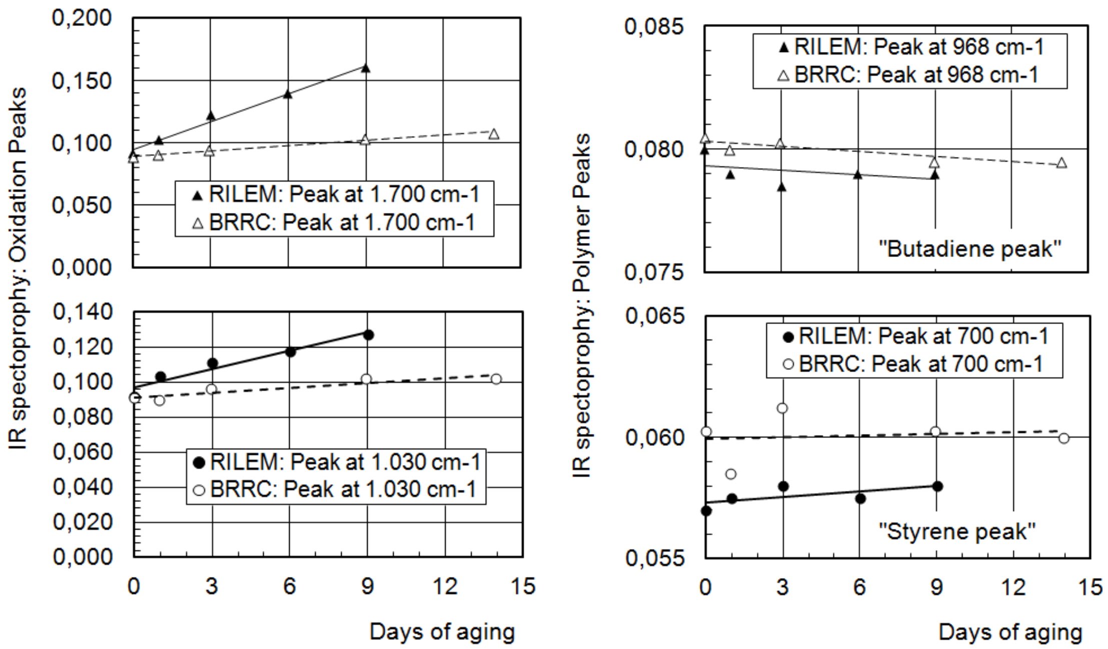


Figure 2: Effect of laboratory aging on the results of IR spectroscopy on binder samples recovered from SMA 8 (P)

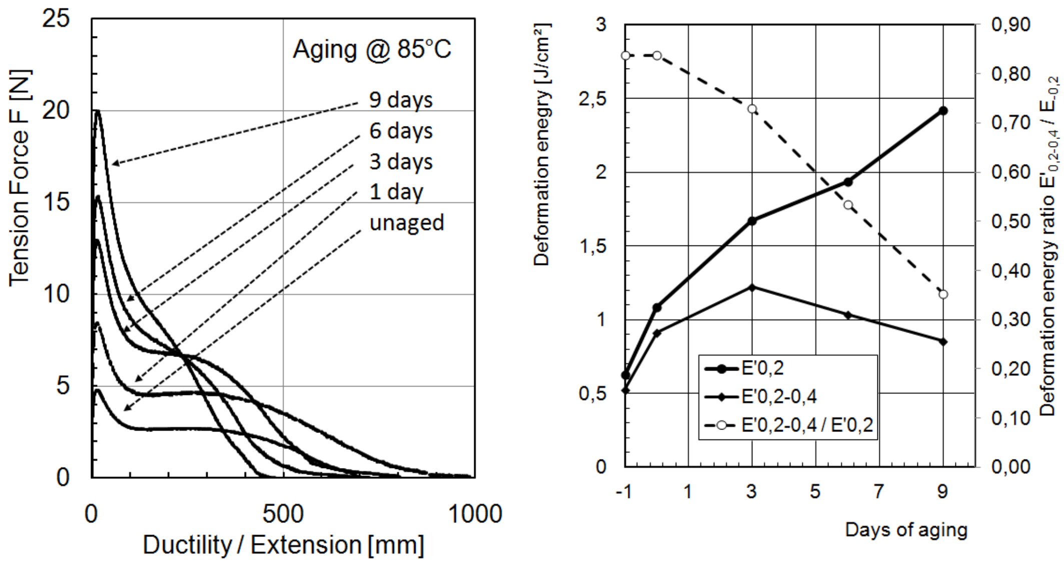


Figure 3 : Effect of RILEM aging procedure on the results of force ductility tests of binders recovered from asphalt sample SMA 8 (P)

3.2 Precision of applied laboratory aging methods

Based on the results of the primarily study (compare section 3.1) common laboratory aging procedures were selected for indicating their repeatability and reproducibility. The aging conditions selected are typed in bold figures in table 1. This study was conducted by using asphalt mixtures AC 11 and SMA 8 (L) both prepared according a prefixed mixing scheme at the collaborating laboratories.

The results of the binder tests are summarised in figure 4. The plotted values indicate the mean values of the measured properties (1 – 4 samples per aging step were tested). The error bars indicate the calculated standard deviation of the single values.

For the samples SMA 8 (L) the results of the force ductility tests (Maximum force F_{Max} and deformation energy $E'_{0,2}$) as well as the results for penetration and softening temperature indicate increased aging from freshly mixed material,

short-term aged, BRRC, RILEM and finally PAV aged material. For the sample AC 11 the same rankings were found except that the PAV aged sample results indicate a viscosity between that after BRRC and RILEM aging.
The deformation energy $E'_{0,2\text{-}0,4}$ indicating the polymer plateau during force ductility tests indicating the polymer modification of sample SMA 8 (L) binder, whereas the binder extracted from sample AC 11 doesn't show a polymer peak. If the deformation energy ratio $E'_{0,4\text{-}0,2} / E'_{0,2}$ is used as an indicator for the overall polymer activity, freshly mixing and short-term as well as BRRC long-term aging (65 °C) doesn't seem to effect the polymer activity. Though, aging at a higher temperature (85 °C) will reduce the polymer activity indicator from 0.65 to 0.5 for RILEM and PAV aging protocol.

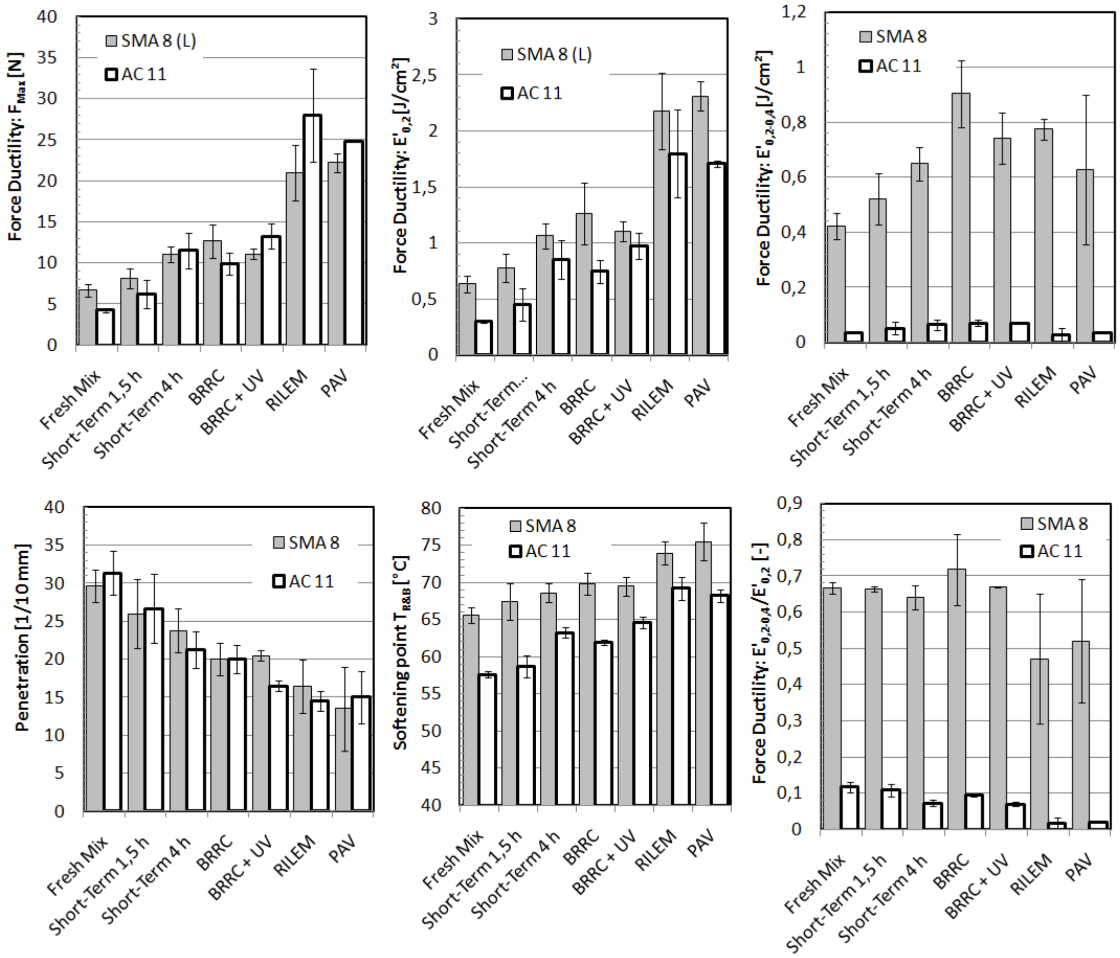


Figure 4 : Effect of laboratory aging procedures on properties of binders recovered from loose mix

3.3 Effect of mix aging on low-temperature cracking resistance of asphalt mix

For evaluating repeatability and / or reproducibility limits for the applied aging procedures the number of conducted aging trials is not sufficient so far. Nevertheless, the standard deviations shown as arrow bars in figure 4 indicate feasible test precision.
In figure 5 the coefficients of variation as the quotient of the standard deviation and the mean result are plotted for the binder properties pen, $T_{R\&B}$ and E'0,2 obtained from the force ductility test. Please note that each coefficient of variation is obtained from four single test results obtained in 2 laboratories (except of BRRC + UV, which was conducted only in one laboratory). The comparison of the coefficients of variation obtained after aging with the variation obtained after mixing gives an indicator for the precision of the aging procedure. Of course the overall scatter is built not only from the aging procedure but also from originates from differences in mixing, recovery and testing between the participating laboratories. These differences are also valid for the freshly mixed material and therefore can be used to evaluate the precision of the aging method only.
For the pen result, the values obtained from the aged mixes indicate generally higher coefficients of variation compared to the freshly mixed samples for the SMA 8 (L) mix whereas the AC 11 mix shows no increase of scattering due to most aging procedures. For the results of softening point $T_{R\&B}$ as well as the deformation energy obtained in force ductility test $E'_{0,2}$ the opposite result is observed , though the overall coefficient of variation measured on SMA 8 samples is higher compared to AC 11.
BRRC aging method result in a lower scatter compared to RILEM method and PAV method for pen and $T_{R\&B}$ results.

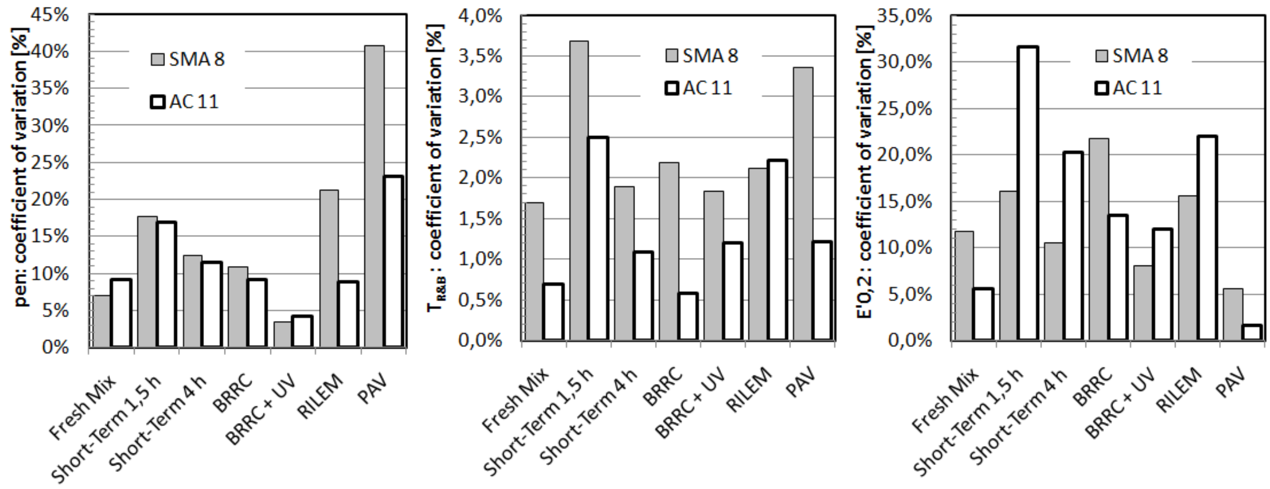


Figure 4 : Effect of laboratory aging procedures on properties of binders recovered from loose mix

3.3 Effect of mix aging on low-temperature cracking resistance of asphalt mix

The resulting graphs of cryogenic stress from TSRST are shown in figure 5 for the three asphalt samples analysed. For all asphalt samples, the aging results in higher cryogenic stress values due to higher viscosity of the aged asphalt samples. For samples SMA 8 (P) and AC 11, the aged asphalt mixes show higher failure temperatures T_F and lower failures stresses σ_F compared to the freshly mixed materials. Despite the course of cryogenic stress measured for the RILEM-aged asphalt SMA 8 (L) indicate higher stiffness, the failure temperature is lower compared to the freshly mixed material and short-term aged (STA) samples.

The observed effect of aging on the results of TSRST was also observed on site-aged material [16].

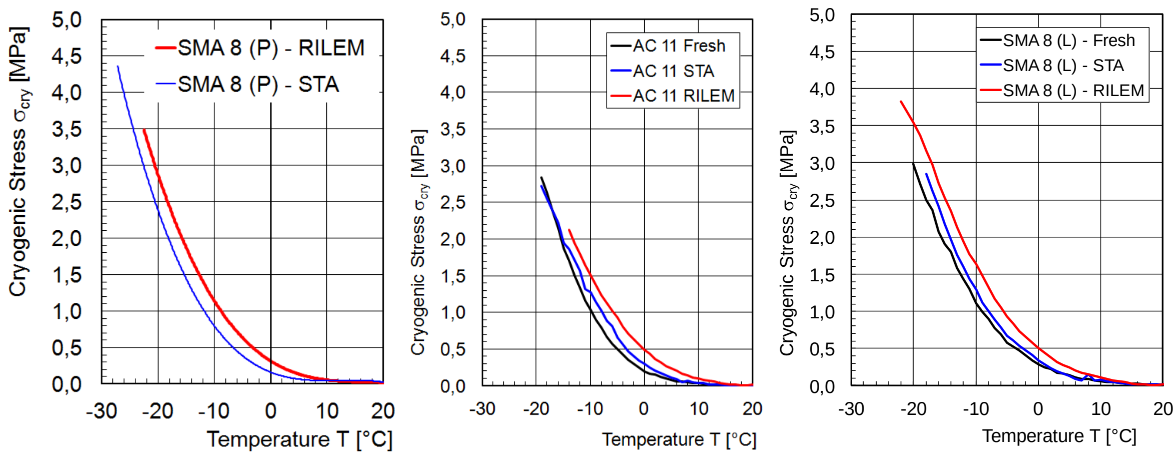


Figure 4: Effect of laboratory aging procedures on results of TSRST of specimens, produced from aged asphalt mixtures

4. CONCLUSIONS

Various laboratory aging procedures for simulating the long-term aging of asphalt mix were analysed during an comparative study in order to develop a suitable conditioning method for aging. Such a conditioning method should provide a basic for assessment of durability of asphalt performance as well as its end-of-life recyclability. It was demonstrated that the aging of asphalt mixes could be carried out successfully by applying the conditioning procedures as described in this paper. Moreover, such methods allow for a characterisation of the recovered binder as well as for performance testing on compacted aged asphalt mixes. In particular, the following observations are made:

- The aging of loose asphalt mix in heating cabinets is a feasible method in order to simulate the long-term aging of asphalt mixture.
- The higher the ambient temperature during the aging, the higher is the aging impact. At a temperature of 85 °C as applied for the RILEM aging method the aging of the binder after 9 days is more severe than at an aging temperature of 60 C and 14 days of aging (BRRC aging method).
- Additional UV radiation leads only to slightly increased aging (shown for aging temperature of 60 °C).
- To reach an aging comparable to the RILEM-method in a short time, the aging of loose mix for 20 hours at a temperature of 90 °C in PAV was found suitable. Though, if higher temperature and aging time is applied, the asphalt mix can age unrealistically hard.

- If the deformation energy ratio $E'_{0,4-0,2} / E'_{0,2}$ derived from force ductility test results is used as an indicator for the polymer activity, it can be concluded that RILEM and PAV aging protocol effects both bitumen and polymer molecules in the SMA 8 (L) mix whereas the polymers are not affected by the BRRC aging procedure.
- The test scatter obtained from the binder properties of binder samples extracted from aged asphalt mixes are slightly higher compared to those extracted from freshly mixed material.
- BRRC aging protocol results in lower coefficients of variation compared to RILEM or PAV protocol.

The first results of a study to analyse the precision of the applied aging procedures indicate that the laboratory aging of asphalt mixes results in feasible repeatability and reproducibility levels. The results presented also indicate, that the relative effect of aging in the applied methods is different from mix to mix. While the PAV aging results in the highest aging for mix SMA 8 (L), the RILEM-method is the most severe method for asphalt mix AC 11.
From this work the “RILEM” and “BRRC” aging methods seems to be more suitable methods to condition large amounts of asphalt mixtures in order to produce specimens for performance asphalt tests for analysing the effect of aging on durability. Whereas BRRC protocol results in a higher precision compared to RILEM method its aging condition has a lower effect on the polymer degradation of modified mixes.

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REFERENCES

[1] Kalman, B. Re-Road - Knowledge & Innovative Technologies for Enhanced End-of-life Strategies, Supporting Asphalt Road Infrastructures. ECTP-E2BA Conference 24 & 25 November 2009. Brussels.
[2] Strickland, D., Evaluation of the performance of Multiphalte 35/50 on the A38 during the first 10 years service, Shell Bitumen Technical Brochure, UK, 1998
[3] Desmoulin, D., Giguet P. et al., Entretien autoroutier : essais comparatifs de liants et de bitumes spéciaux sur enrobés minces anti-orniérants, RGRA, 842, septembre 2005, 79-84
[4] Farkas, F., Etude d’une méthode de simulation du vieillissement des bitumes sur route, Chaussées-CR21, Laboratoire centrale des Ponts et Chaussées, Paris, 1998.
[5] Dreessen, S., Ponsardin, M., et al., Durability study: field aging of conventional and polymer modified binders, Proceeding of TRB 2010 annual meeting, 14p
[6] Choquet, F S., The search for an ageing test based on changes in the generic composition of bitumens, International Symposium “Chemistry of Bitumens”, Vol. II, Rome, 1991, 787-812
[7] Piérard, N., Vanelstraete, A., Développement d’une méthode d’essai pour vieillir de manière accélérée les enrobés bitumineux en laboratoire, XXIème Congrès Belge de la Route, Gent, 22-25 septembre 2009, paper I.1.9
[8] Renken P., Optimierung und Qualitätssicherung Offenporiger Asphaltdeckschichten Teil II Verändeung der Eigenschaften nach längerer Liegezeit. Schriftenreihe "Forschung Straßenbau und Straßenverkehrstechnik", Bundesministerium für Verkehr, Bau- und Stadtentwicklung, Heft 951. 2007.
[9] Lenk, Loibl, Mocker. Long term aging properties of polymer modified binders, Theory & Experience, 3rd Eurasphalt & Eurobitume Congress, Vienna 2004, Paper 083
[10] Angst, C.: Der Pressure Aging Vessel (PAV), Strasse und Verkehr, Nr. 2, 1997
[11] Francken, L., Vanelstraete, A., Verhasselt, A. Long term ageing of pure and modified bitumen: influence on the rheological properties and relation with the mechanical performance of asphalt mixtures, 8th Int. Conference on Asphalt Pavements, Seatttle, Washington, 1997
[12] Bell C. A., Ab Wahab, Y., Cristi, M.E., Sosnovske, D. Selection of Laboratotry Aging Procedures for Asphalt-Aggregate Mixtures. SHRP-A383. ISBN 0-309-05762-0. Washington D.C. 1994
[13] Büchler, S., Mollenhauer, K., Wistuba, M., Renken, P. Ageing of stone mastic asphalt and evaluation of cracking resistance. Proc., 8th int. Conference on Bearing Capacity of Roads, Railways and Airfields BR²AC, Champaign, 2009; pp. 339-348
[14] De la Roche C., Van de Ven M., Van den Bergh W., Gabet T., Dubois V., Grenell J., Porot, L. Development of a laboratory bituminous mixtures aging protocol. Procs. Int. Conf. on Advanced Testing and Characterisation of Bituminous Materials, 2009.
[15] Piérard, N., Vanelstraete, A., Developing a test method for the accelerated ageing of bituminous mixtures in the laboratory, Advanced Testing and Characterization of Bituminous Materials, Loizos, Partl, Scarpas & Al-Qadi, Taylor & Francis Group, London, 2009, 163-171
[16] Mollenhauer, K., Piérard, N., Tusar, M., Moulliet, V., and Gabet, T. 2010. Development and validation of a laboratory aging method for the accelerated simulation of reclaimed asphalt. Proc., 1st Int. Conf. on Sustainable Construction Materials, Design, Performance and Application, Aug. 10-12, 2010, Wuhan, China.
[17] Piérard N., Vanelstraete A., The Impact of moisture on the ageing of bituminous binders and asphalt mixtures, 4th Congress Eurasphalt & Eurobitume, Copenhague, 21 - 23 May 2008, paper 406-11