FINAL REPORT

Marshall Plan Scholarship

Aging of Bituminous Materials: Laboratory Simulations

Research Exchange

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Contents

1 Short Description of the Research

Bitumen is of organic nature making it prone to oxidation and other effects that alter its behavior over its lifetime. Aging of bitumen and hot mix asphalt can be divided into short-term aging STA and long-term aging LTA. STA includes effects of bitumen storage, asphalt mix production and compaction. LTA is defined as aging effects that occur during the in-service lives of compacted pavements. LTA is a highly complex field of research, due to the slow and long processes taking place over several years.). For improving the materials at an early stage, i.e. during mix design optimization, engineers must have realistic (equivalent to field aging) and efficient (test duration) methods to simulate field processes.

First studies with the Viennese Aging Procedure that was developed at TU Wien are promising (Frigio, Raschia, Steiner, Hofko, & Canestrari, 2016; Raschia, 2015; Steiner et al., 2015). Still, the method has to improve to ensure a more general applicability. This project aims to study the impact of different parameters on the achieved ageing level by Viennese Aging Procedure. Testing bituminous materials for their aging susceptibility requires a broad knowledge of different fields in the laboratory (e.g. production processes, mechanical test methods, aging parameters, mix design, specimen properties). Therefore, different studies are combined in this report. The report it is structured as follows: an introduction, dealing with basics of aging and the Viennese Aging procedure. Furthermore, each conducted study to analyze one part of the aging procedure is presented in the subsequent chapters.

1.1 General Information

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2 Introduction

Asphalt mixtures play a major role for road infrastructure since more than 90% of the European and US road network consists of asphaltic material (Mollenhauer, De Visscher, Carswell, & Karlsson, 2012). Bitumen acts as the binder component in asphalt mixtures. It exhibits a complex chemical composition and mechanical behavior due to its organic origin as the heavy residue of crude oils. Bitumen is a viscoelastic material with temperature-dependent characteristics. Its organic nature makes it prone to oxidation and other effects which alter the microstructure, chemical composition and mechanical behavior over its life-time (Lesueur, 2009). Bitumen tends to become stiffer and more brittle. Thus, asphalt pavements become more susceptible to low-temperature and fatigue cracking, two major failure modes of road infrastructure. In this way, bitumen aging can be a limiting factor to a pavement's in-service life.

In recent years, awareness increases that most natural resources have to be considered as scarce goods. In addition, strained public budgets lead to cuts for infrastructure maintenance and rehabilitation. Therefore, infrastructure construction, maintenance and rehabilitation need to become more energy and cost efficient. Two strategies have to be combined to achieve this aim: road pavements need to be optimized in terms of their durability and of their recyclability. While asphalt mix optimization in terms of resistance to traffic loading can already be achieved on laboratory-scale by performance-based test methods (established in the EN 12697 series), resistance to climate loading and high recyclability are strongly related to the phenomenon of aging. Aging affects the resistance to low-temperature cracking (Steiner, Hofko, Dimitrov, & Blab, 2016) as well as the recyclability of mixtures (Hofko, Hospodka, et al., 2014).

2.1 Aging of Bituminous Materials

Aging of bitumen and asphalt mixtures can be divided into short-term aging (STA) and long-term aging (LTA). Several physical and chemical reactions like polymerization, volatilization, steric hardening and oxidation (Masson, Collins, & Polomark, 2005; Traxler, 1961) interact in both aging steps. STA includes effects of bitumen storage, asphalt mix production and compaction. STA is mainly triggered by high temperatures and fast oxidation due to a large specific surface during mix production. STA can be simulated in the lab by exposing asphalt mixtures for a short period at production temperatures in a ventilated oven. This procedure has been validated to plant produced asphalt mixtures. LTA is defined as aging effects that occur during the in-service lives of pavements. Research projects, e.g. (Hofko, Eberhardsteiner, et al., 2014), have shown that the current idea of atmospheric oxygen being mainly responsible for LTA of asphalt pavements has to be critically reviewed. The reactivity of atmospheric oxygen at temperatures that occur in surface layers (up to + 65°C in moderate climates) is considered too low to act as the major source for oxidation in pavements. Other gases, like ozone or nitric oxides, which are also available in the atmosphere and which are much more reactive at the temperatures in summer have to be taken into consideration for a realistic simulation of aging in the laboratory.

2.2 Simulation of Aging in the Laboratory

Several methods are available for simulating aging of bituminous materials in the laboratory. Two major procedures are widely used in terms of STA and LTA of binder / bitumen within efficient amount of time. The Pressure aging vessel (PAV) (Airey, 2003; ASTM, 2013; CEN, 2012a; Mallick and Brown, 2004) and the rolling thin film oven test (ASTM, 2012; CEN, 2007a) are standardized and frequently applied methods to transfer virgin binders into the state of STA (RTFOT) and LTA (RTFOT+PAV).

Existing lab-aging methods (>25) for asphalt mixes use only atmospheric air at high temperatures (100°C and above) and/or high pressures to increase oxidation and thus aging rate (Bell, AbWahab, Cristi M.E., & D., 1994; Çetinkaya, 2011; Steiner et al., 2016). From a microstructural and chemical point of view, it is questionable whether the same processes as in the field occur at these conditions that are not available in the field. Therefore, the question as to be raised whether existing aging methods simulate field aging in a realistic way. Widely spread and applied methods simulate the LTA of HMA material in loose condition. Thus, the materials has to be compacted afterwards to produce specimens. Only then, subsequent analyses of the mechanical behavior are possible. It is questionable, whether the change of the mechanical properties are simulated as they occur in the field. HMA is never aged to a state of LTA in loose condition in the field, since LTA includes aging effects starting with opening the road for traffic after compaction.

2.2.1 Viennese Aging Procedure

Based on the findings listed above, a new aging method for compacted asphalt mix specimens was developed, called Viennese Aging Procedure (Steiner, et al., 2015). In principle, an ozone and nitric oxide enriched compressed air flows through a specimen at slightly elevated pressure (20 kPa) and at elevated, yet realistic temperatures (+60°C). The layout of the device is shown in FIGURE 1. With Viennese Aging Procedure, a method is now available that does not exceed temperatures or pressures actually occurring in surface layers of pavements but increase oxidation rates by increasing the concentration of oxidant agents. First studies on lab aged asphalt mix specimens and recovered binder from the specimen show good correlations with long-term field processes. The change of mechanical properties, can only attributed to oxidation processes and does not show effects of thermal aging relating to loss of volatile components at higher temperatures (Steiner, Hofko, Grothe, & Blab, 2018).

By applying Viennese Aging Procedure to lab-produced specimens and assessing the performance (Steiner, Hofko, & Blab, 2016) of these long-term aged specimens in the lab, a better performance prediction can be realized. With an efficient and field-validated aging method for simulating LTA of asphalt mixes in the lab, asphalt mixtures can thus be optimized in terms of durability and recyclability at a very early stage, e.g. during mix design optimization. Viennese Aging Procedure is able to age the binder in an asphalt mix specimen to RTFOT+PAV level within 3 days of aging. In addition, since the temperature is limited to 60°C and highly oxidant gases are used, the method seems to be closer to reality than other procedures. This is mainly because temperature and pressure are similar to field conditions.

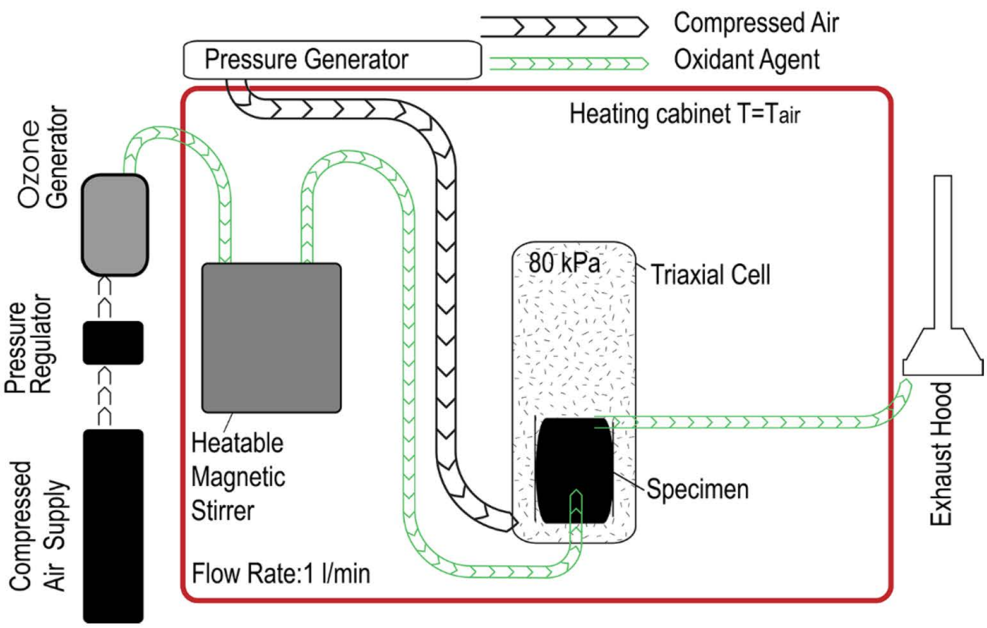

FIGURE 1 : Schematic view of Viennese Aging Procedure

3 Considering Short-Term Aging for Long Term Aging Procedures

Conducting aging studies in the laboratory requires knowing all possible influences on the material behavior. Dealing with bituminous materials means, that properties are steadily changing. With presence of oxidative species, the material is continuously oxidized. Depending on the surrounding parameters, this process is slow or accelerated due to higher pressure, temperature or higher concentrations of oxidative agents. It is important to know, how strong the bitumen was aged in previous stages and to control these effects for subsequent tests, that all samples are pre aged equally.

The viscoelastic behavior of hot mix asphalt (HMA) is influenced by several factors, e.g. by the binder behavior, which has crucial impact on the pavement performance The behavior changes due to aging during its in-service life within years, as well as during HMA production and compaction processes within a few hours. Therefore, aging processes are classified into long-term aging (LTA) and short-term aging (STA). This chapter presents a study, analyzing STA processes at laboratory productions of HMA slabs. Six production cycles, with up to three slabs each were analyzed. The mix of binder and aggregates were prepared at once. This requires storing the hot and prepared mix inside the laboratory mixer while the first and the second slab are compacted, respectively. Bitumen was recovered for each produced slab. $|G^*|$ stiffness tests (DSR) are carried out afterwards at 52-82°C and 1.592 Hz. The results show that mixing times lead to a stiffness increase of 1.5 compared to the virgin bitumen, what is slightly below RTFO aging level. Recovered binder from Slab 2 and Slab 3 show increases of $|G^*|$ of 1.9 and 2.5. Therefore, for the used binder, longer mixing times have a significant impact on STA. The results can be used to back-calculate STA levels for specimens that are used for LTA aging studies. Furthermore, both binder and additional HMA stiffness results can serve as basis for multi-scale stiffness modelling by coupling the results.

3.1 Motivation

As previously mentioned, to assess bitumen aging in the laboratory the rolling thin film oven test (RTFOT) (CEN, 2007) and the pressure aging vessel (PAV) (CEN, 2012, Airey, 2003) are standardized and widely accepted methods. To assess HMA aging in the laboratory, HMA slab production and oven conditioning of the mix are widely used to achieve STA. For simulating LTA of loose or compacted asphalt mix, far more procedures have been developed and published in the last decades.

HMA consists of huge amount of aggregates (~ 95% by mass) and a small proportion of bitumen (~5% by mass). However, the latter is mainly responsible for the material properties of hot mix asphalt with highly temperature depended and aging susceptible behavior. Therefore, several factors (e.g. amount of HMA, temperature, mixing time) can influence the materials properties in the field production process. These effects also contribute in the laboratory at hot mix asphalt slabs / specimens production for hot mix asphalt LTA studies. Therefore, from a practical point of view, it is important to pay attention to some specific issues while carrying out laboratory hot mix asphalt production to minimize STA differences between all produced samples.

On occasion, only small amounts of material is required for a sample production. The lower the total handled material mass is, the more vulnerable is the mix for changes. This is the case for compacting slim slabs. On the one hand, a risk of segregation or stick of bitumen on the walls and stirrer of the mixer exists. Therefore, the actual mix design can deviate significantly from the targeted design. Furthermore, small amounts of materials leads to greater specific surfaces in large mixers. This can cause more severe STA. For these reasons, preparing and mixing of higher amounts of material for more than one slab at a time is preferred. STA due to high specific surface can be reduced in this case. On the other hand, longer mixing times are needed to store the material within the mixer while one slab is compacted. The impact of this additional storage time on STA and thus, the mechanical behavior has to be investigated. Therefore, a study was carried out within this chapter. The results of the study will give a magnitude of the sensitivity of the mixing time on STA for slabs that are produced in series.

3.2 Materials, Test Methods and Experimental Program

The presented study looked into the impact of mixing time of HMA at slab production in the laboratory. Data from six different production days were collected. For all produces slabs, the same bitumen, HMA mix design and mixing temperatures were used. Laboratories usually operate only one roller compactor. Therefore, more than one slab or specimen of the same material can only be produced at the same time by storing the additional material in the mixer until the first slab is compacted. This additional storage time at higher temperatures can lead to changes of the visco elastic properties due to STA. The mix has to kept within the mixer, while the first and the second slab are compacted, respectively. In this case, the amount of slabs varied between 1-3 slabs each production.

An asphalt concrete with maximum nominal aggregate size of 11mm AC11 was mixed within a laboratory reverse-rotation compulsory mixer according to EN 12697-35 (CEN, 2007c). The mix consist of an unmodified 70/100 pen (PG 64-22 / 79 pen) binder with a share of 5.2 % by mass and a porphyrit as aggregate material. The hot mix asphalt slabs (50x26x4 cm) were compacted in a steel roller segment compactor according to EN 12697-33 (CEN, 2007b), after the material was mixed at a temperature of 170°C. The void content was set to a target of 8.0% by volume. Since the mix was prepared at once for all slabs at each production, the mixing time /storage varied. The material for slab 1 was mixed and stored for 5 minutes. The share for slab 2 stayed for 13 minutes within the mixer and slab 3 for 21 minutes.

Eight specimens are cored from each slab with a diameter of 100mm. Bitumen from slab remains was extracted according to EN 12697-3 with tetrachloroethylene (C2Cl4) as a solvent. The solvent-bitumen solution was distilled according to EN 12697-3 (CEN, 2013) to recover the binder samples. (Hospodka, Hofko, & Blab, 2017) showed that no changes occur at binder properties due to temperature at the distillation. Solvent residues can be fully removed of and the initial mechanical behavior fully restores.

The binder was tested for the mechanical behavior by Dynamic Shear Rheometer (DSR) tests. All bitumen samples recovered from all laboratory STA material were analyzed. The test conditions were chosen according to the SHRP procedure (Petersen et al., 1994) and EN 14770 (CEN, 2012b)with a temperature sweep from +52°C to +82°C using the large plate (diameter: 25 mm) and a 1 mm gap. The tests were carried out at a standard frequency of 1.59 Hz. From test data the dynamic shear modulus |G*| and the phase angle δ vs. frequency are determined. Similar trends develop for all testing temperatures. In order to make the figures more legible, the results are only shown for DSR testing temperatures of +64°C. Furthermore, the dynamic shear modulus |G*| of the same virgin and RTFOT -aged binder were determined as benchmarks.

3.3 Influence of Mixing Time in the Laboratory

The results of the DSR testing are presented in FIGURE 2. The relative change of the complex modulus |G*| of all STA binders vs. unaged condition at 64°C testing temperature is shown in the diagram. The illustrated columns represent mean values. Additionally, the scattering of the data is shown by using 95% confidence intervals for all single tests. Furthermore, the green column shows data from RTFOT aged bitumen. Significant Changes due to longer mixing times can be seen by analyzing the mechanical properties. For all first slabs of a mixing cycle of three slabs, the binder samples are 1.5 times stiffer than the unaged binder is. It can be seen, that these slabs are close to a STA level by using RTFOT. The results of the second and third slabs are by far stiffer than RTFOT. The binder of the second slabs are 1.9, the third slabs are 2.5 times stiffer than the virgin binder is.

The stiffness results of the DSR phase angle δ testing are presented in FIGURE 3. The change of the unaged phase angle δ subtracted by phase angle δ of all short term aged binders at 64°C testing temperature is shown in the diagram. HMA lab production this leads to the decrease of the phase angle δ due to STA. Similar Significant Change as for the complex modulus results can be seen due to longer mixing times. Here, the results of the first slabs of a productions cycle of three slabs perfectly match the RTFOT aging level. The phase angle was reduced by 3.0° at high testing temperatures of 64°C. The MV of all second slabs reduces by the value of 4.0°. The phase angle of the third slabs is reduced by 6°.

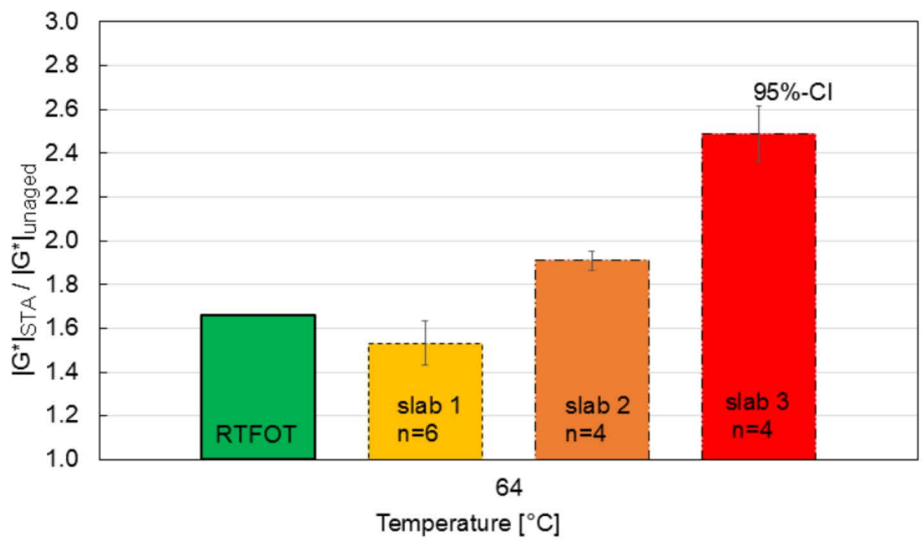

FIGURE 2 : Complex Modulus |G*| @ 64°C, 1.6 Hz. Recovered bitumen from compacted slabs (n=4-6) with different mixing times (5 min, 13 min, 21 min)

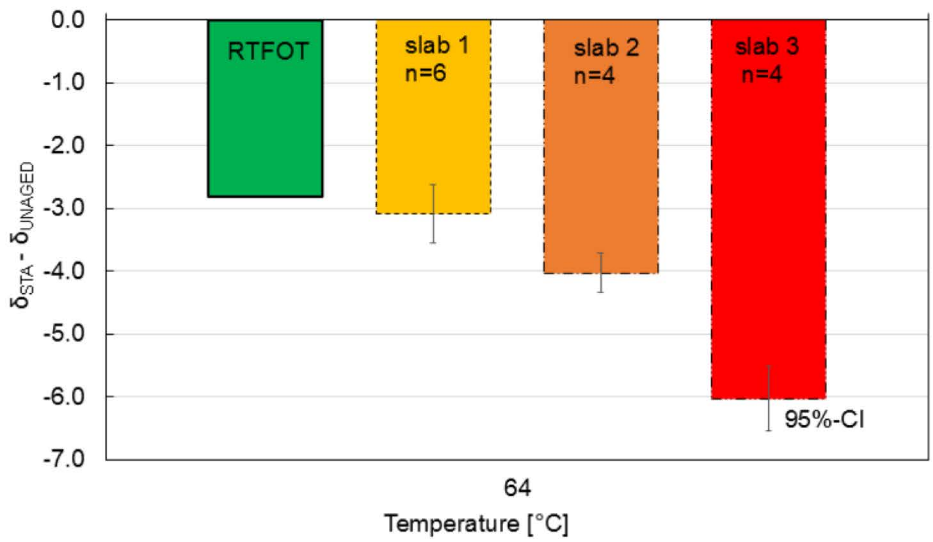

FIGURE 3 : phase angle δ @ 64°C, 1.6 Hz. Recovered bitumen from compacted slabs (n=4-6) with different mixing times (5 min, 13 min, 21 min)

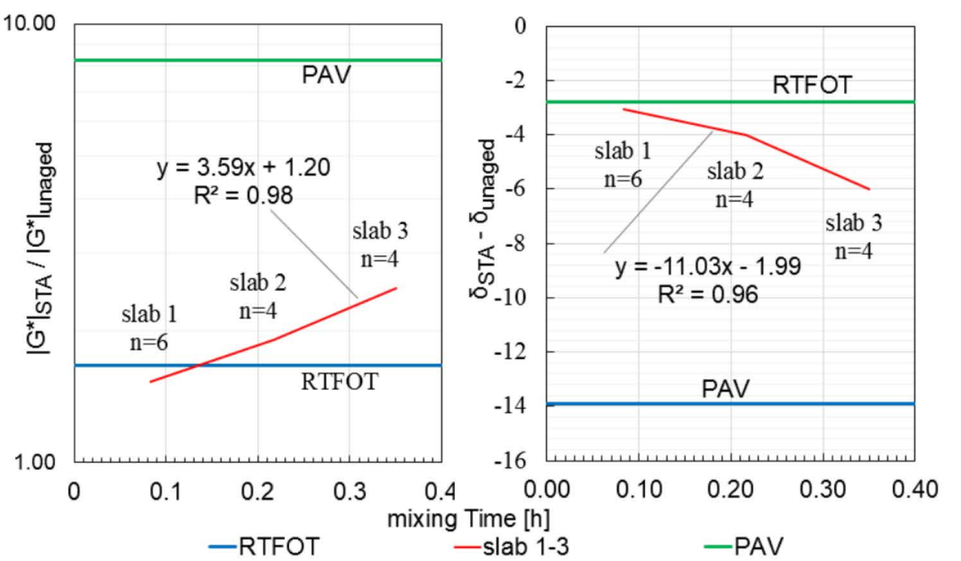

FIGURE 4 : Complex Modulus |G*| and phase angle δ @ 64°C, 1.6 Hz vs mixing time. Recovered bitumen from different mixing times (n=4-6)

All results are summarized and combined in FIGURE 4 using Dynamic Shear Rheometer results (complex modulus |G*| and phase angle δ) at a testing temperature of +64°C. All data obtained from DSR testing (G* and δ) are plotted on the vertical axis vs. the mixing time plotted on the horizontal axis. This allows for a regression analysis vs. time. To get a better overview, where the results are located between a standard RTFOT level and a standard RTFOT + PAV aging level, the results of both aged binder are illustrated. Strong correlation between the mechanical behavior and the mixing time is obvious. Nevertheless, none of the recovered binder comes close to a LTA level (RTFOT + PAV). To give a magnitude correlation, linear regressions are calculated and illustrated within the figures. Looking at the coefficient of determination, R^2 are equal or above 0.95, which indicates an excellent linear correlation of phase angel/dynamic modulus vs. mixing time.

As main motivation, this chapter aims at giving a better understanding of the pre-aged of hot mix asphalt specimens that are intended for further LTA studies. It can be stated and summarized, that with longer mixing / storage times the mechanical behavior of the recovered binder is significantly influenced. Since stiffness changes can be seen more clearly on binder level at elevated testing temperatures, the question is whether significant difference can be seen at intermediate service temperatures and on hot mix aging level as well. For future research, additional studies of the STA on hot mix asphalt level are necessary. Furthermore, both, binder and hot mix asphalt stiffness results could serve as basis for multi-scale stiffness modelling with coupling the results. Additionally, for LTA studies with a limited number of specimens, the STA results can be used as fallback option. If the behavior was already heavily influenced by the specimen production, the LTA will not start at the same level for all specimens and therefore the scattering of the results can be stronger at a LTA level. Recovering and testing the results allows back calculating the results and isolating only the increase in stiffness due to LTA.

Since only one binder was analyzed in this study, the results cannot be seen as a blueprint for all bitumen sources used for pavements. A study of the aging behavior on HMA and binder level with binder from different bitumen grades and provenances should give a better idea of aging in general. Different origins can lead to completely different behaviors. The binder used in this chapter, was already tested with the Viennese Aging Procedure. Results revealed that the binder is more prone to short and LTA than other bitumen form the same variety.

Furthermore, the results can be used in multi-scale modelling studies. Thereby, hot mix asphalt stiffness results are coupled with binder stiffness testing results. Hot mix asphalt stiffness data exist for all produced slabs and are evaluated in combination with binder data in a future study.

4 Applicability of the Viennese Aging Procedure

Asphalt pavement structures consist of several layers, each with certain tasks. Surface layers, in particular are responsible to distribute traffic loads, to ensure road safety by having high skid resistances and to seal the structure from water. Sealing properties of surface layers are significant in terms of aging as well. Hot Mix Asphalt Mix designs with higher air void contents are more prone to aging since all reactive oxygen species (ROS); both in liquid and gaseous phase can easily penetrate into the asphalt structure. Therefore, a procedure to measure the sealing properties in addition to determining the air void content can assist in evaluating the aging durability for bituminous pavements.

4.1 Relevance of Air Void Contents for Long-Term Aging and Long-Term Aging Procedures

As mentioned previously, higher air void contents leads to higher penetration of liquids and gases into the pavements. The material should be designed, that the resistant against oxidative gases or liquids is as high as possible. Therefore, dense asphalts concepts have an advantage over open graded concepts in terms of ageing resistance.

Simulating the LTA of compacted hot mix asphalt by using forced gas flows through the specimen requires a certain amount of gas permeability. For specimens with lower air void contents, the pressure of the gas flow has to be increased to achieve a certain amount of flow volume / time. Some parts of the VAPro ageing device set limit values to the maximum applicable pressure to avoid damages. Furthermore, an air permeability measurement device would ensure a quick test to assess, whether a specimen is sufficiently permeable to be mounted within the Viennese aging procedure.

4.2 Water permeability procedure as basis for a Development of an air Permeability procedure

Hot Mix Asphalt (HMA) mixtures as the main construction material for road pavements are a composite of bitumen as the binder and mineral aggregates with a predefined mix design. Stiffness, quality of the aggregates and the mix design play an important role. Thus, one key element is the bulk density of the compacted layer. Bulk densities or rather air void contents (AVC) have an influence on the mechanical behavior and serviceability of hot mix asphalt layers, as mentioned in the introduction of this chapter. Therefore, conventional design approaches set maxima and minima for air void contents of hot mix asphalt pavements and use it as a sign of quality for the construction process to comply with the criteria of the road administration. Minimum limits for AVC are necessary to prevent the pavements from bleeding effects of the bitumen. Bitumen films due to bleeding will lower the friction on pavement surfaces. Maximum limits for AVC are set to ensure a sufficient layer stiffness that is necessary to distribute stresses from mechanical impacts (Daniel Steiner, Bernhard Hofko, & Ronald Blab, 2016) and to prevent water from entering the pavement structure. Based on experience, AVC limits are given for commonly used pavement designs (Müller and Vasiljevic, 2010). When developing new mix designs using innovative and alternative materials, initial proof of concept studies have to be carried out, since no empirical data are available to ensure that all requirements are fulfilled. Regarding the function as a sealant layer, a water permeability test is helpful. In the U.S., different methods for analyzing the water permeability in the laboratory

(Harris, 2007) or in the field (Allen, Schultz Jr, & Fleckenstein, 2001) were developed.

In this chapter, a method to measure the water permeability is presented using HMA specimen sizes that are commonly used in Austria. With this procedure, coefficient of permeability (COP) *k* can be determined to be used for further calculations (e.g. flow calculations for asphalt sealing elements in hydraulic engineering). Water permeability tests for soils are already standardized in German standards (DIN, 1998) with defined permeability classifications using coefficient of permeability *k*. These classifications are applied on bituminous pavements. Table 1 contains the classes given by (DIN, 1998).

Table 1: Scale of permeability (DIN, 1998)

k [m/s].	**scale**
Below 10-8	Very low permeable
10^{-8} to10^{-6}	low permeable
10^{-6} to 10^{-4}	permeable
10^{-4} to 10^{-2}	very permeable
above 10^{-2}	Extremely permeable

An asphalt concrete with a maximum nominal aggregate size of 11mm (AC 11) was employed. The binder content was set to 5.2% by mass. Five HMA Slabs (50x26x4 cm) were compacted in a roller compactor. From the slabs, 40 specimens (8 each) are cored with a diameter of 100mm and a height of 40mm. The air void content of the specimens range from 4.0 to 9.0 vol%.

Water permeability tests are carried out on all specimens. The method, that was used as described below, is based on the water permeability test for soils, standardized in German Standards (DIN, 1998). The setup and equipment is shown in FIGURE 5. HMA specimens are located within a triaxial cell between filter stones and are covered by an elastic membrane. The triaxial cell is flooded with ambient air. To enable the water flow through the specimens, a confining pressure has to build up in lateral direction. Thus, an overpressure of 150 kPa was applied within the triaxial cell. Due to the higher lateral pressure, the elastic membrane is pressed onto the specimen surface and the water is forced to flow through the specimen, instead of passing on the outside. The tests were carried out three times on each specimen. Starting with a flow pressure of 50 kPa, it was increased to 70 and 100 kPa for the second and third test. The specimens are flown through with water until the system is saturated. To measure the used flow pressure accurately, a pressure gauge was installed directly before the inlet to the triaxial cell. Once the test is started, water will exit the system into a measure vessel. With recording time, flow pressure and water amount, it is possible to calculate the coefficient of permeability k, which is shown in equation 1.

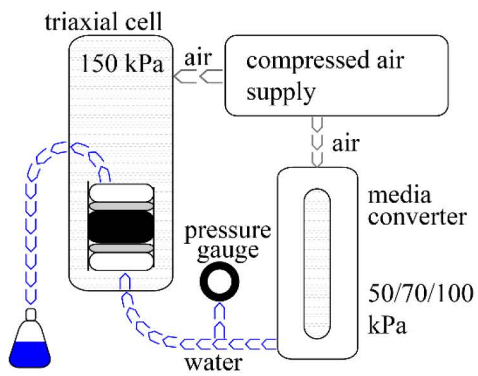

FIGURE 5 : Water permeability set-up

$$k = Q * \frac{L}{h} * \frac{1}{A*t} \qquad (1)$$

k… coefficient of permeability [m/s]
Q…flow volume [m³]
L… specimen height[m]
A… specimen cross section [m²]
t… testing time [s]
h… pressure head [m]

Main results are shown in FIGURE 6. The diagram shows AVC vs. the coefficient of permeability *k* for all 40 specimens. The mean values of three tests, varying the flow pressure were used. Furthermore, the scales of permeability according to (DIN, 1998) are stated. The correlation of both values, AVC and COP *k* is shown by applying a power function. The coefficient of determination R^2 is 0.7, which indicates a fair correlation. Looking at the scales of permeability, it can be seen, that almost every data point can be classified in this areas. Therefore, according to (DIN, 1998), specimens with AVC lower than 5.0 vol% can be classified as “very low permeable”, between 5.0 vol% and 7.1 vol% as “low permeable” and above of 7.1 vol% as “permeable”. Comparing that with classifications according to (Müller and Vasiljevic, 2010) that are based on experience in Austria, the results can be compared. Pavements are as defined as “permeable” with AVC higher than 8.0 vol%. Between 5.0 and 8.0 vol%. It is classified as “low permeable” and in the range from 3.0 to 5.0 vol% “practically impermeable”. It can be stated that no significant influence of varying the flow pressure (50, 70, 100 kPa), can be seen in the results.

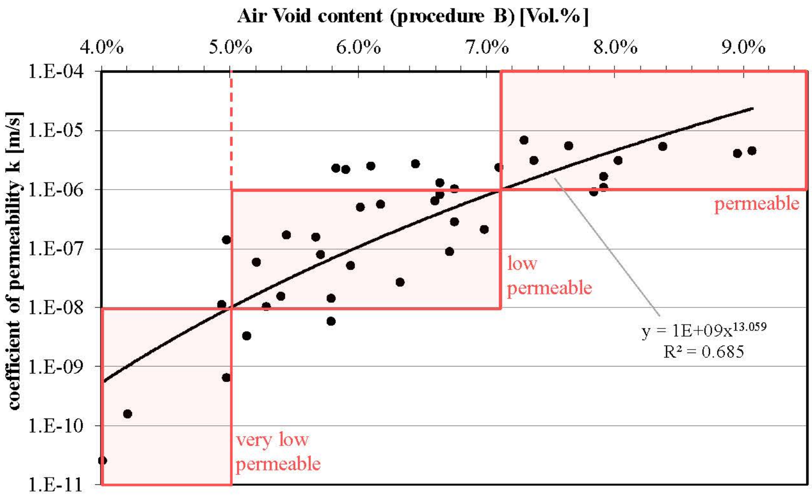

FIGURE 6 : Air Void Content vs. coefficient of permeability k for water

The presented method to measure the water permeability of HMA specimens is suitable to achieve coefficient of permeability *k* for air void ranges of approximately from 3.5 to 10.0 vol%. The correlation of the presented data in the range from 5.0 to 7.5 vol% can be improved by using higher specimen heights. Using slim specimens, air voids can be distributed inhomogeneously and can thus act as drainages.

4.3 Development of an air Permeability procedure to measure the applicability of the Viennese aging procedure

Based on the procedure for measuring the water permeability, a procedure for testing the air permeability was developed. Therefore, equipment from the previously described set up and some parts from the Viennese aging procedure were used. The procedure can be used to measure, whether the air permeability of specimens is high enough without destroying expensive VAPro equipment. Furthermore, the lower boundaries for VAPro in terms of the air void content can be defined. Testing the permeability with gases has certain advantages compared to using water. After specimens were treated or tested with water, it cannot be guaranteed that all remaining water is removed from all voids of the hot mix asphalt. This can have an impact on the results of subsequent mechanical tests (e.g. stiffness tests).

The setup and equipment is shown in FIGURE 7. An HMA specimen is mounted in a triaxial covered by an elastic membrane. The lateral pressure within the air flooded triaxial cell presses the elastic membrane onto the surface of the specimen. Therefore, the gas flow is forced to flow through the specimen. The forced gas flow is controlled with a pressure regulator. Before entering into the triaxial cell, the gas pressure is monitored with a pressure gauge. Since VAPro is carried out with a gas flow of 1 l/min, this value is also used for this method. Therefore, this value is fixed and controls the test. The pressure is adjusted until the airflow reaches the required benchmark. Hence, specimens with higher air void contents need less flow pressure to reach the same flow speed.

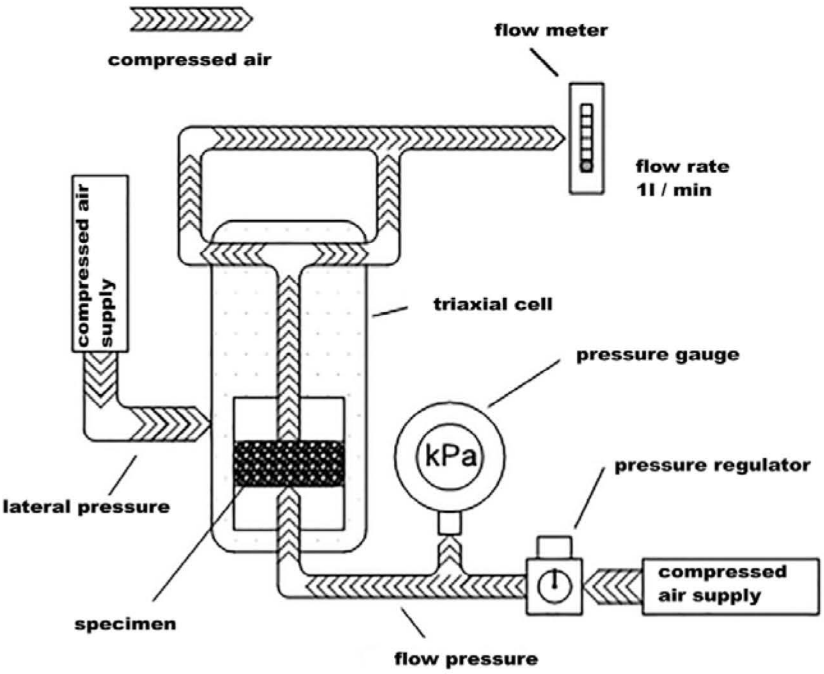

FIGURE 7 : Air permeability set-up

An asphalt concrete with a maximum nominal aggregate size of 11mm (AC 11) was employed. The binder content was set to 5.2% by mass. Three HMA Slabs (50x26x4 cm) were compacted in a roller compactor. From the slabs, 24 specimens (8 each) are cored with a diameter of 100mm and a height of 40mm. The air void content of the specimens range from 4.0 to 9.0 vol%.

All results of the tests are summarized in FIGURE 8. The diagram shows the air void contents AVC vs. flow pressure for all specimens that was needed to reach a flow rate of 1 l/min. All results are classified in AVC classes. The mean values for each class were calculated. Furthermore, the standard deviations for AVC and flow pressure are presented. The correlation of both values, AVC and flow pressure is shown by applying a power function.

To define a lower limit of AVC for specimen that can be tested with VAPro, it can be assumed, that samples from a dense graded AC11 mixture can be aged starting from an AVC of 5.5 – 6.0 % by volume.

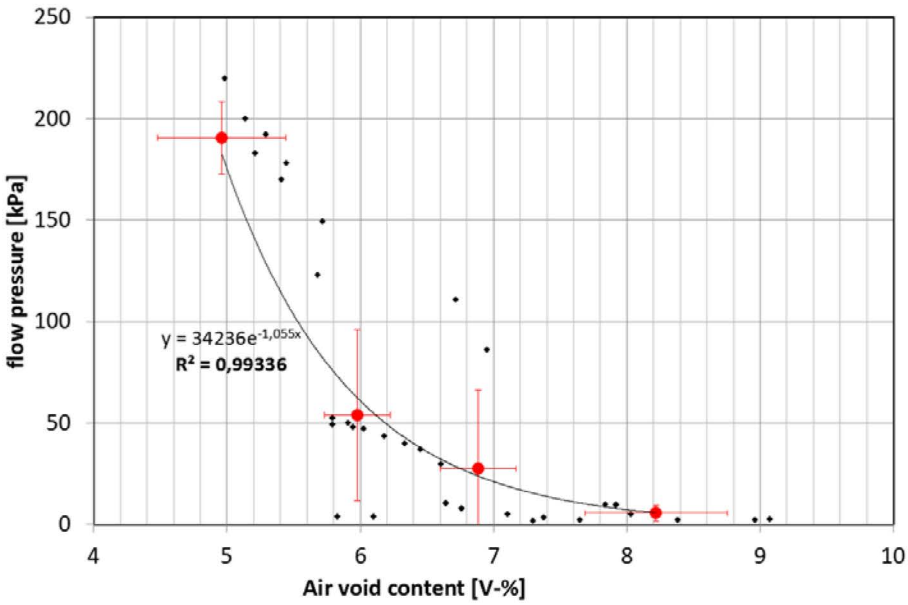

FIGURE 8 : Air Void content vs. flow pressure (flow rate: 1 l/min)

The presented method to measure the air permeability of HMA specimens is as well suitable to achieve air - coefficient of permeability *k_air*. The test is performed quickly and gives a fast estimation of the permeability properties.

5 Choosing the Aging Parameters

Aging of bituminous materials can be seen as a combination of several chemical and physical processes that are triggered by temperature (oxidative aging). As described previously, simulating LTA effects in the laboratory is often carried out by using high temperatures and / or high pressures. One of the main motivation to develop a new aging procedure for LTA aging is to simulate field processes as realistically as possible within an efficient time span. Therefore, it is necessary to be fully aware of the main chemical and physical reactions that can occur in the field. Subsequently, striking a balance between accelerating reactions by increasing temperature, particle concentrations or pressure and reproduce long-term field processes realistically, is needed.

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 a physical effect, the evaporation of remaining volatile components from the bitumen (thermal aging) (Baek, Underwood, & Kim, 2012; Petersen et al., 1994). LTA can be describes as a very slow oxidation at intermediate or high service temperatures (<70°C). Efficient simulations of LTA reactions in the laboratory should bring the material as fast as possible onto the chosen aging level without increasing the temperature that high, that other reactions than in the field can occur.

5.1 Motivation

The main objective of this chapter is to analyze the influence of thermal aging (by separating thermal from oxidative aging effects) at different temperatures that are commonly used for LTA procedures. The results should be used to recommend limit aging temperatures for laboratory LTA of HMA. These temperatures should stay below values where relevant thermal aging effects occur. Similar studies for STA in the laboratory has been carried out with Nitrogen Rolling Thin Film Oven Tests NRTFOT by Parmeggiani (Parmeggiani, 2000).

Preliminary analysis of the thermal effects at LTA procedures were carried out by an aging study at TU Wien (D. Steiner, et al., 2016). The results showed, that no thermal effects could be observed. HMA specimens where placed in nitrogen atmospheres at +60°C for 4 days. The aging level of the extracted bitumen was comparable with bitumen from lab-produced specimens without further aging procedures.

5.2 Approach to separate non oxidative / thermal from oxidative aging effects

Two different setups were used to carry out this study. The equipment and both set ups are based on the Viennese Ageing Procedure (D. Steiner, et al., 2016). In the course of the project, some adaptions had to be made to tackle some issues and improve the procedure. The equipment that was used for BINDER I, basically is based on a triaxial cell as core component, where hot mix asphalt specimens are assembled within it. These samples are located between filter stones and covered by an elastic membrane. The complete system was flooded with nitrogen for a few minutes to expel any ambient air. An overpressure of 50 kPa was applied within the triaxial cell. The specimens were also flowed through with nitrogen for a few minutes to saturate all air voids. The flow pressure was set to 25 kPa. Due to the higher lateral pressure, the elastic membrane is pressed onto the specimen surface and the gas is forced to flow through the specimen, instead of passing on the outside. Afterwards all inlets were closed to retain the nitrogen within the pressure cell and specimen. The triaxial cell is located in a heating cabinet with sealed inlets where temperature T_{air} was varied for the experimental run for an aging duration of four days. After the first aging study of binder I, it turned out that a set-up is to prone for damages. It consists of a glass cylinder that is sealed with metal adapters with a slight pressure in axial direction, which can lead quickly to cracks in the glass cylinder.

To overcome this downside of setup I, an alternative approach was followed up to carry out aging on specimens from binder II. A pipe clamp (DN=100 mm) was used to seal the specimens on the lateral surface. HMA specimens are also assembled in a row between filter stones and two endplates. The pipe clamp can be perfectly fitted to the specimen dimensions. This equipment is more durable for aging tests with forced gas flow at temperatures far higher than +100°C. In this stage, the specimens were continuously flowed through with nitrogen for an aging duration of three days. In general, conditioning under nitrogen atmosphere is supposed to prevent any oxidative aging and thus, only trigger thermal aging effects. In a next step, 3 HMA specimens were placed in the ambient air of the heating cabinet at each selected aging temperature for four (binder I) or three (binder II) days. This time the heating cabinet was equipped with a forced convection. The HMA specimens are exposed to all reactive oxygen species (ROS), which are present in the ambient air. This leads to different oxidation processes at the varied temperatures T_{air}. To maintain its proper shape, the specimens are positioned in Marshall compaction molds and wrapped with silicone foil as a release layer. Ambient air aging is supposed to trigger both, oxidative, as well as thermal aging effects.

By comparing results from specimens and recovered binders subjected to nitrogen and ambient air aging, thermal and oxidative aging effects can be separated.

5.3 Materials, Test Methods and Experimental Program

For the presented study presented in this chapter, an asphalt concrete with a maximum nominal aggregate size of 11mm (AC 11) was employed. The coarse aggregates used for the mix is a porphyrite, the filler is powdered limestone. As a binder two (I / II) unmodified 70/100 pen (PG 58-22 / 91 pen and PG 64-22 / 79 pen) were used. The binder content was set for both mixtures (binder I and II) 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 2.593 kg/m3 (I) and 2.562 kg/m3 (II). The grading curve is within the borders according to national specifications (ONI, 2009).

The mix was prepared in a laboratory reverse-rotation compulsory mixer, according to EN 12697-35, with a mixing temperature of +170°C. HMA slabs (50x26x4 cm) were compacted in a roller compactor according to EN 12697-33. 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 100mm. The air void content of the specimens used for the test program range from 6,6 to 8,3 % by volume for mixture I and from 5,9 to 8,0 % by volume for mixture II.

For bitumen testing, bitumen was extracted according to EN 12697-3 with tetrachloroethylene (C2Cl4) as a solvent. The solvent-bitumen solution was distilled according to EN 12697-3 to recover the binder samples.

Table 1 provides an overview of the test program. The study looked into the impact of temperature T_{air} by comparison of the nitrogen and ambient air stored specimen. Therefore, tests on four and three different temperatures

T_{air}= +60 / (+85) / +110 / +135°C were carried out. The results provide information on how the rheological behavior of the binder (change of |G*| and phase angle) changes due to thermal and oxidative aging effects. Furthermore, the dynamic shear modulus |G*| of the same virgin and RTFOT+PAV-aged binder were determined as benchmarks.

Table 2 provides an overview of the test program. The study looked into the impact of temperature T_{air} by comparison of the nitrogen and ambient air stored specimen. Therefore, tests on four and three different temperatures T_{air}= +60 / (+85) / +110 / +135°C were carried out. The results provide information on how the rheological behavior of the binder (change of |G*| and phase angle) changes due to thermal and oxidative aging effects. Furthermore, the dynamic shear modulus |G*| of the same virgin and RTFOT+PAV-aged binder were determined as benchmarks.

Table 2: Test Program

	BINDER I 70/100 PEN PG 58-22				**BINDER II 70/100 PEN PG 64-22**			
	# of Specimens [-]		# of recovered binder samples and DSR SHRP Tests		# of Specimens [-]		# of recovered binder samples and DSR SHRP Tests	
	Aging duration: 4 days				Aging duration: 3 days			
Temp Tair [° C]	N2	Air	N2	Air	N2	Air	N2	Air
+60	3	3	1	1	3	3	3	3
+85	3	3	1	1	-	-	-	-
+110	3	3	1	1	3	3	3	3
+135	3	3	1	1	3	3	3	3

Dynamic Shear Rheometer (DSR) tests were carried out on bitumen samples recovered from all lab-aged (short-term and long-term nitrogen vs. ambient air) HMA specimens. The test conditions were chosen according to the SHRP procedure (Petersen, et al., 1994) and EN 14770 with a temperature sweep from +46°C to +82°C using the large plate (diameter: 25 mm) and a 1 mm gap. A frequency of 1.592 Hz is employed. From test data the dynamic shear modulus |G*| and the phase angle φ vs. frequency are determined.

5.4 Results and Recommendation for Long Term Aging Parameters

To investigate potential thermal effects from temperature, bitumen from all nitrogen stored and ambient air stored samples were extracted and recovered from the HMA. Analysis of changes of the viscoelastic behavior were carried out. In addition, STA bitumen by RTFOT and LTA bitumen by RTFOT+PAV was tested as well to compare standardized bitumen aging procedures to the presented procedures. The results are presented in FIGURE 9, FIGURE 10, FIGURE 11 and FIGURE 12. It shows the relative change in dynamic shear modulus |G*| from recovered bitumen samples vs. virgin bitumen. The dotted lines represent data from the RTFOT (B I 1.45 / B II 1.66 [-]) and RTFOT+PAV (B I 4.49 / B II 7.17 [-]) aged bitumen. For both binders, the changes in the |G*| vs. the aging temperature of the different procedures are shown. The results of binder I as well as binder II indicate no significant changes in the mechanical behavior for the nitrogen-stored samples below 110°C. At 135°C, |G*| significantly increased for both binders in the nitrogen atmosphere. If it is assumed that the mechanical changes at the temperatures of 110°C and below are only due the STA at the slab production, it can be concluded that aging effects that can be attributed to thermal aging are starting at temperatures between 110°C and 135°C. Between +60°C and +110°C all binders are 1.6 -2.0 times stiffer than the virgin binder, whereas the +135°C nitrogen samples are 2.6 and 3.4 times stiffer.

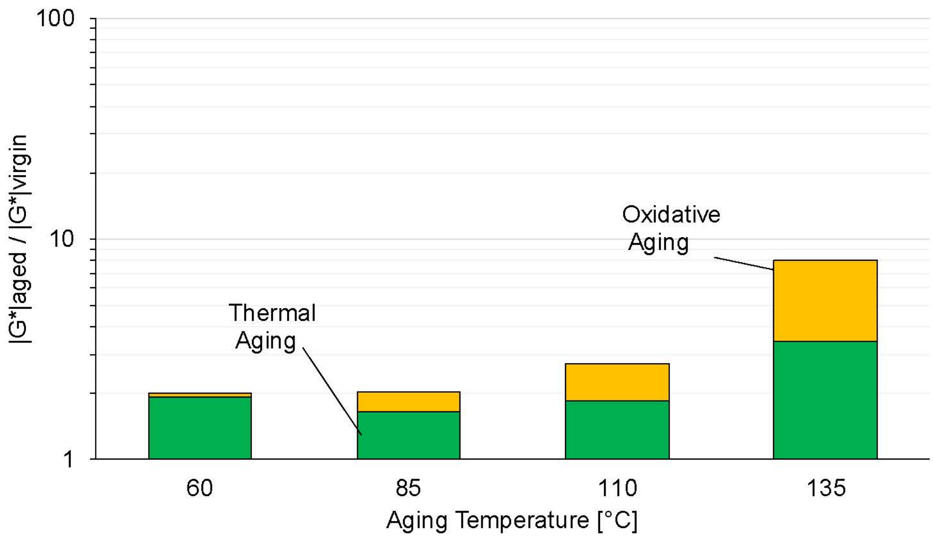

FIGURE 9 : Binder I: Change in dynamic shear modulus |G*| of bitumen recovered from lab-aged HMA specimen (4 days binder I) to virgin bitumen sample

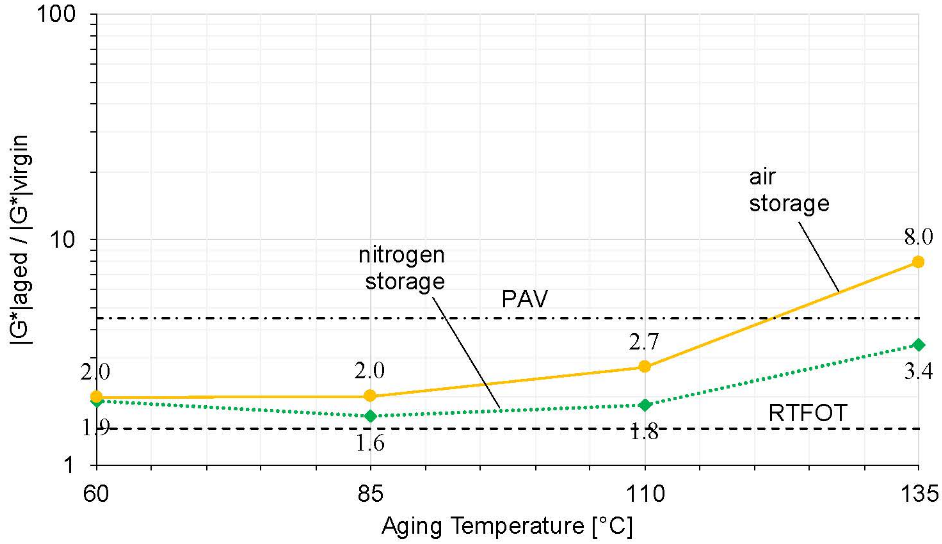

FIGURE 10 : Binder I: Change in dynamic shear modulus |G*| of bitumen recovered from lab-aged HMA specimen (4 days binder I) to virgin bitumen sample

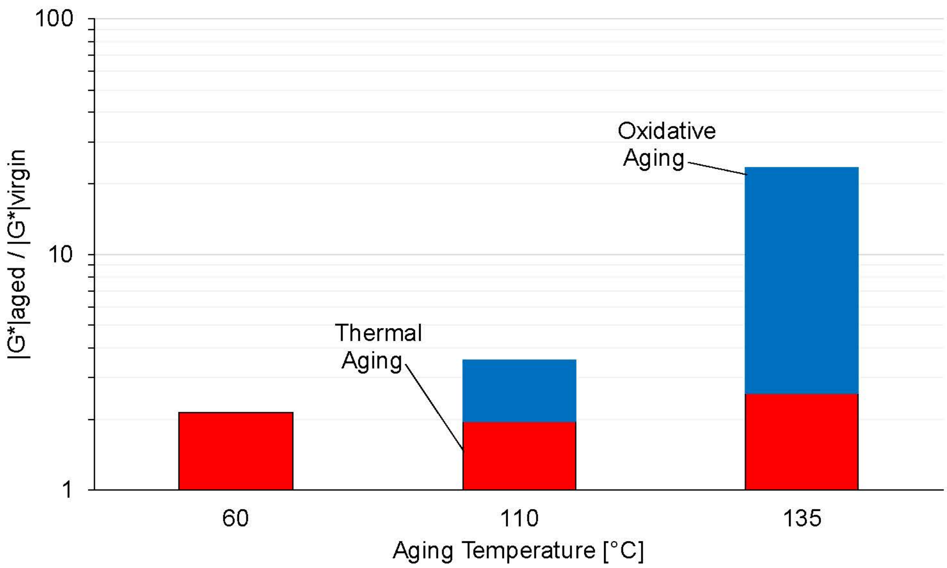

FIGURE 11 : Binder II: Change in dynamic shear modulus |G*| of bitumen recovered from lab-aged HMA specimen (3 days binder II) to virgin bitumen sample

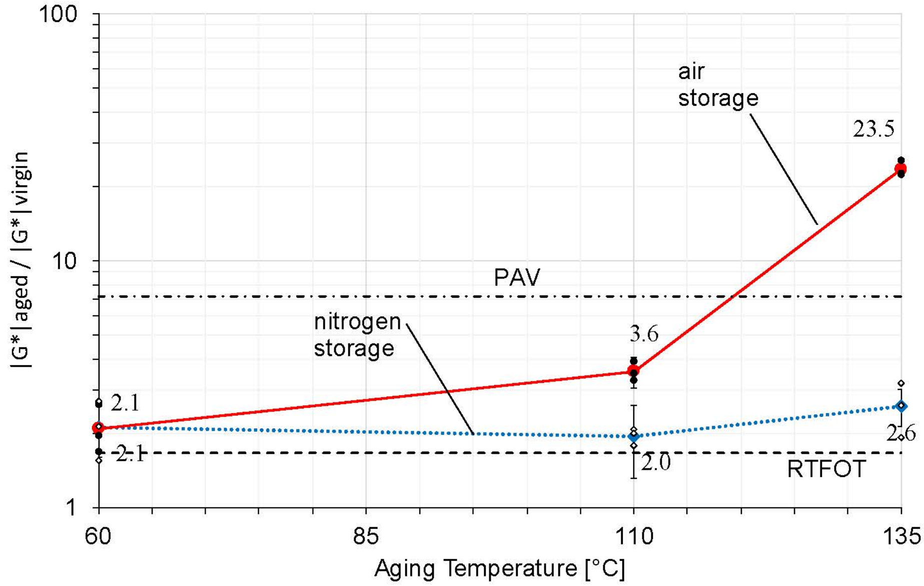

FIGURE 12 : Binder II: Change in dynamic shear modulus |G*| of bitumen recovered from lab-aged HMA specimen (3 days binder II) to virgin bitumen sample

Looking at the data from the ambient air aged samples, almost no effects occur until 85°C. Starting at 110°C significant changes can be seen. The oxidative species of the ambient air are reactive enough to trigger oxidative aging only above 110°C. The difference between nitrogen stored and ambient air stored samples can be attributed to oxidative aging effects. Both binders follow the same trend in relation to the aging temperature. Comparing both binder, it must be stated, that binder I was conditioned for 4 days, while binder II was aged for 3 days. Despite this, binder II is more prone to aging. The increase of stiffness is higher for RTFOT+PAV aged samples and for samples aged as HMA in ambient air in comparison to samples from binder I.

The main drive for the research project presented within this paper is to investigate the potential of ambient air conditioning as realistic LTA procedures and to isolate thermal aging effects from oxidative aging effects. Considering realistic field conditions (max. 65°C on asphalt surface), thermal effects should not be triggered by laboratory LTA procedures since they are not expected to occur in the field. To prevent thermal aging effects, conditioning temperatures must kept below 110°C as it is shown in this paper. At the same time, ambient air conditioning for HMA specimens is only suitable as an LTA procedure for temperatures above 110°C. Below this threshold, the conditioning hardly triggers any oxidative effects in a short amount of time and is therefore inefficient. Additionally, at temperatures above 110°C, the viscosity of some binder grades is so low that permanent changes in the structure of compacted HMA specimens are likely to appear upon conditioning for an extended period of time. Results from mechanical tests of such samples are therefore biased.

Summarizing, to achieve both, realistic and efficient LTA procedures for HMA, temperatures have to stay well below 110°C and oxidative aging effects have to be increased by other means, e.g. the use of more reactive gases that are present in the atmosphere as well.

The data presented in this chapter will be extended in publications in the future. Additional Stiffness data are available from recovered binder from control HMA specimens that were not subjected to any aging after production. By using these data, any STA effects from mixing and slab production can be taken into account and separated from the analysis of LTA effects.

6 Relevance of the Research

The project described here is of great relevance for further developments and an application-oriented use of the Viennese Aging Procedure. Already, a significant number of different laboratory and field-aged samples provide the basis for a validation of the procedure. A broad database with samples from different aging methods and approaches are necessary for the set up parameters of Viennese Aging Procedure and make it applicable in a more general way. Every new aging project broadens the view on aging general and allows to enhance Viennese Aging Procedure. Mechanical analysis of the aged and unaged samples are essential. In combination with classifications from standardized aging procedures, benchmarks can be set for the aging levels. In outlook of editing the current European Technical Specification CEN/TS 12697-52:2017 Procedure B for getting closer to field aging, parameters can be then chosen individually and Viennese Aging Procedure can then be used universally.

Roads and asphalt pavements are built worldwide in a relative similar way. Bitumen as a binder and aggregates form the basis. Nevertheless, due to the highly temperature dependent behavior and dependencies of local resources, each region has to optimize the material to their own needs. In order to characterize the mechanical or chemical behavior of the material, a large quantity of test methods are available for researchers worldwide. The approach of characterizing, designing and choosing the right parameters for the local materials. Major failure modes of bituminous pavements are rutting, fatigue cracking and (low) temperature cracking (transverse or longitudinal). First and last are mainly influenced by the bitumen stiffness. The softer the binder is the more rutting can occur. Transverse or longitudinal Cracks can occur, if the material, even if it is only for a short period, is too brittle to relax all of the temperature induced tensions. At a first sight, thermal or low temperature cracks should only be relevant for regions with moderate and cold climates. (Alavi, Hajj, & Sebaaly, 2016)showed, that transverse cracks, that are often called low temperature cracks, can as well occur in hot climates. Fast temperature changes, for example in the night, can increase the stresses that tensile strengths are exceeded and the cracks occur. FIGURE 13 and FIGURE 14 show pictures of thermal cracks that were taken at a parking lot next to the Theodore Roosevelt Lake, Az and in Tuscon, Az. These failures can be found all over Arizona.

FIGURE 13 : Thermal Cracks in Roosevelt, Arizona

FIGURE 14 : Thermal Cracks in Tucson, Arizona

With LTA, the binder behavior shifts to a stiffer and more brittle behavior and asphalt pavements are more prone to cracking. Therefore, characterizing the aging behavior for all used materials can help to predict the life span.

Due to the warmer climate, hardly any snow falls in phoenix metropolitan area (“Valley of the Sun”), and therefore no need of preventive winter maintenance by the use of salt is given. This allows using open graded or gap graded asphalt mix designs. As previously described, one disadvantage of using these mix designs is, that oxidative agents can enter easily in the asphalt pavements and increase the aging.

All of these aspects need to be taken in consideration in terms of aging. Parameters of aging procedures have to be chosen accurately. Therefore, a major part of the study conducted at ASU was to, get familiar with the concept of designing asphalt pavements and how aging is treated in that context. Results are given in chapter 5 “Choosing the aging parameters”. In the future, it is considered to use the Viennese Aging Procedure for asphalt rubber.

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