Viennese Aging Procedure (VAPro): adaption for low-temperature testing

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Abstract. As for all organic material, bitumen ages due to anthropogenic and natural influences. The aging process changes the properties of the material. It becomes stiffer and more brittle. This leads to a deterioration of low-temperature and fatigue performance and limits the life span of road pavements.

A distinction is made between short-term aging (STA) during production and long-term aging (LTA) during service-life. For simulating aging in the laboratory on bitumen scale, RTFOT for STA and PAV for LTA are used. On asphalt mix scale, various methods have been developed in the past for either loose material or compacted specimens. Many of these methods use unrealistic boundary conditions concerning temperature and pressure, which do not occur in the field.

A lately developed method, Viennese Aging Procedure (VAPro), conditions specimens at close-to-field conditions, i.e. a temperature of $+60^{\circ}$ C and a pressure of about 0.5 bar. To accelerate aging, VAPro perfuses specimens with a gas mixture consisting of compressed air and traces of reactive oxygen species (ROS). These ROS, such as ozone (O₃) or nitrogen oxides (NO_X) can be found in the atmosphere as well, just in lower concentration. Due to the current layout of VAPro, only cylindrical specimens could be conditioned up until now.

However, for investigating the effects of LTA on the low-temperature performance of asphalt mixtures, prismatic specimens are needed for TSRST and UTST. This paper presents the development of a new VAPro version to obtain prismatic specimens and it shows a first comparison of unaged and long-term VAPro aged specimens tested in TSRST with respective DSR results of the extracted binder. With the VAPro method at hand, asphalt mixture can be optimized regarding not only their short-term performance but also their long-term performance including relevant impacts of aging on the performance.

Keywords: VAPro, Long Term Aging, Ozone, NOx, Hot Mix Asphalt, TSRST, UTST

1 Introduction

The change in material behavior of bitumen over time is due to the organic composition of the material. Natural and anthropogenic impacts increase stiffness and brittleness, which leads to a deterioration in the performance properties of asphalt pavements, especially, resulting in more likely low-temperature cracks and decreasing fatigue resistance [1, 2].

Considering the time span, aging of bituminous material is divided in short-term aging (STA) during production and paving and long-term aging (LTA) during its service life on the road. Fast chemical oxidation due to high temperatures and a high specific surface contacting with oxidant agents at mix production, as well as the vaporization of remaining volatile components characterizes STA [3]. Slow oxidation, particularly in the upper layers of the asphalt, by traces of reactive oxygen species (ROS) found in the atmosphere like ozone or nitrogen oxides causes LTA [4, 5].

For prediction of the long-term behavior of asphalt mixtures, it is important to simulate aging of the material in the laboratory in order to choose the most suitable bitumen source and to build long-lasting and thus, more sustainable roads. The standardized methods, Rolling Thin Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) are commonly used for STA and LTA on bitumen scale [6]. On asphalt scale, many procedures were developed in the last decades. Most of these protocols use high temperatures (above +100°C) and/or high pressure, which do not occur in the field. Consequently, additional effects could be induced by these methods, such as evaporation of further volatile binder components that are not triggered in the field. Some of the protocols age loose material and produce specimens afterwards. This raises the question of the influence of the already aged material on compatibility [7].

At TU Wien, a procedure called Viennese Aging Procedure (VAPro) has been developed for aging compacted specimens in field-like conditions concerning temperature (+60°C), pressure (~0.5 bar) and aging medium [8]. To achieve a change in material behavior, VAPro perfuses cylindrical specimens with highly oxidant gases that are present in the atmosphere, just in lower concentrations [5]. These gases are known as ozone and nitrogen oxides.

The original design of VAPro conditions cylindrical specimens. Cyclic indirect tensile tests (IT-CY) can be conducted to gain information about change in stiffness or fatigue behavior due to aging. To investigate the low-temperature behavior of asphalt mixes with the Thermal Stress Restrained Specimen Test (TSRST) and Uniaxial Tension Stress Test (UTST) prismatic specimens are required. This paper shows the improvement of VAPro by developing a cell for aging prismatic specimens. First TSRST results of aged specimens show the proof of principle. The extracted binders are tested in the DSR to assess the aging condition of the samples. With this method at hand, the long-term performance of asphalt mixes can be taken into account in mix design and optimization with regard to realistic aging mechanisms.

2 Material and Methods

2.1 Material

For this paper, a stone mastic asphalt with a maximum nominal grain size of 11 mm (SMA 11) was used. Porphyrite is used as coarse mineral aggregate and powdered limestone as filler. The binder used is a PmB 45/80-65 with a penetration of 67 dmm and a softening point of 70.5°C. According to AASHTO M 320-10, the performance grade is 76-22. The binder content is set to 5.6% by mass.

The mixes are produced at $+160^{\circ}$ C in a roller compactor according to EN 12697-35. The measured air void content of the tested specimens is between 8-9% by volume with the dimensions of 225x50x50 mm.

2.2 Methods

Viennese Aging Procedure (VAPro). The initial setup of VAPro consists of a triaxial cell in which cylindrical specimens are placed to be perfused by the ROS enriched air. The specimens must have sufficient permeability to ensure flow through it. The device has been continuously optimized, leading to a conditioning duration of three days as well as proving its functionality [9-11]. The gas mixture, containing traces of nitrogen oxides (NO_x) and ozone (O₃) is produced by leading compressed air through an ozone generator. The mixture is heated to increase its reactivity. The heating device as well as the triaxial cell are placed in a heating chamber at $+60^{\circ}$ C. Afterwards the gases are securely degraded and removed by a fume hood. In Fig. 1 shows a photo and schematic of VAPro.



Fig. 1. A schematic diagram (left) and Photo (right) of the initial setup [11]

Further Development of VAPro. The limitation of the initial setup is the shape of the specimens, allowing only certain experiments like IT-CY to be performed. The aim is to condition prismatic specimens for conducting low-temperature tests like TSRST or UTST. The new cell ages a whole asphalt mix slab in order to have the possibility to produce various types of test specimens after aging. The dimensions of the slab are 320x260x70 mm. Besides the difficulty of material selection due to the highly corrosive gases, the challenging part is to ensure flow through the specimen,

not around it. An aluminum box, made of three pieces was designed in CAD and manufactured by a mechanical workshop. The slab fits exactly into the box and elastomer seals are used to seal the box and cut off the gas flow around the asphalt slab (Fig. 2).



Fig. 2. Photo of the new prototype inside the heating chamber (left) and the open cell with asphalt slab inside (right)

Reference Aging. Virgin binders are aged in RTFOT according to EN 12607-1 as well as RTFOT+PAV according to EN 14769 for comparison.

Asphalt Mix Testing. TSRST is used to assess the low-temperature performance of the asphalt mix according to EN 12697-46. Cooling down starts at +10° C at a cooling rate of 10 K/h. Stress, strain and temperature are recorded during the test. Cracking temperature (T_{crack}), cracking stress (σ_{crack}) and the transition point (T_t) of visco-elastic (stress relaxing) to elastic (brittle) behavior are derived from the data.

Binder Extraction. After TSRST, the bitumen is extracted with tetrachloroethylene as solvent and distilled according to EN 12697-3.

Binder Testing. The extracted binders are tested in a DSR according to EN 14770 with a 25 mm plate and a gap of 1 mm. A frequency sweep is carried out from 0.1 Hz to 40 Hz at temperatures between $+40^{\circ}$ C to $+82^{\circ}$ C in 6°K steps. From these measurements, Dynamic Shear Modulus |G*| and phase angle δ are calculated.

3 Results and Discussion

The duration of conditioning in the new cell was raised to six days in the first place, because there is more material to age. Temperature (+60°C) and flow rate (1.0 l/min) are the same as for the triaxial setup of VAPro. The duration was raised to 12 days based on the recovered bitumen and its DSR measurements respectively. Fig. 3 illustrates $|G|^*$ of the recovered samples and the reference samples. Lab mixing shows that



it is close to RTFOT. Six days in the new cell achieves some aging, but 12 days inside the new cell corresponds to the binder stiffness of a RTFOT+PAV-aged bitumen.

Fig. 3. Comparison of |G*| of the recovered binders

Resulting from this, a first test program was conducted, consisting of three unaged specimens and two 12-day VAPro-aged specimens. Fig. 4 shows the results of these tests. T_{crack} is hardly affected by aging: The unaged specimens exhibit a mean T_{crack} of -34.7°C and the aged ones of -33.5°C. This increase of 3.3% shows a trend but is not significant. However, the tensile stress is built up more quickly and the point where the curve merges into a straight line (T_t) occurs earlier for the aged material. This shows that the aged material has less stress-relaxation capability. The transition point between dominating viscoelastic and elastic is defined by aligning a tangent to the linear part of the curve starting from T_{crack} . T_t is the point when the coefficient of determination R^2 of tangent used to approximate the test data drops below 0.9999. T_t is -32.5°C for the unaged material and -29.6°C for the aged material. The difference of 2.9°C stands for the significant reduction of the stress relaxation capability of the aged material.



Fig. 4. Comparison of the TSRST Results

4 Summary and Outlook

This paper shows the proof of principle of the new VAPro cell that ages a whole slab in order to cut aged, prismatic specimens for low-temperature tests. First TSRST results show faster increase in tensile stress during cooling, which means that the material has less stress-relaxation capability. Transition from viscoelastic to elastic behavior occurs earlier compared to unaged samples. T_{crack} is hardly affected by the aging process. The achieved $|G^*|$ of the bitumen is comparable to $|G^*|$ of PAV-aged bitumen.

The next steps include optimizing tightness of the cell, flow through, and aging duration. After improving the prototype, a study with a larger number of specimens is planned.

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