Aging of Bitumen and Asphalt Concrete

Comparing State of the Practice and Ongoing Developments in the United States and Europe

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REVIEW OF ASPHALT (CONCRETE) AGING TESTS IN THE US AND EUROPE

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ABSTRACT

Aging is a crucial factor in pavement performance and being able to determine its effect on a mixture is necessary to link its initial properties to the properties over time in order to ensure the intended service life. This is becoming more important now that climate change leads to increased variation in weather conditions, while environmental considerations cause changes in the constituent materials that are used. As a result, past experience is becoming less reliable. In this paper, the USA and EU approaches to aging are compared, showing that those contain the same test equipment and almost identical conditions for aging. This allows the exchange of data and experience.

The current tests are suitable for binders and give an indication of the sensitivity to aging. For short term aging RTFOT conditioning gives a reasonable indication of bitumen aging during asphalt concrete production and construction. This only holds for penetration grade binders during hot mix production and construction.

For long term aging, because of the many variables involved, developing a single test method to characterize aging sensitivity, seems impossible. However, using more elaborate protocols in existing, practical tests, can provide more information and the necessary input for kinetic aging expressions. A PAV protocol for testing at two temperatures and time intervals, specifically at 90 and 100 degrees Celsius and for 20 and 40 hours respectively, is suggested. Using the same conditioning in characterizing materials for pavement construction and research will facilitate the exchange of data and enable faster developments.

Keywords: aging tests review, oxidation, PAV protocol, kinetic expressions

INTRODUCTION

Aging of asphalt concrete is an important aspect of pavement performance, because most pavement damage in well-constructed pavements occurs only after a considerable service life. In the Netherlands and most other west European countries, service life ranges from 10 to 20 years for surface layers to considerably longer times for binder and base layers. Aging causes the material properties to change during this time, especially for surface layers which are exposed to moisture, large temperature changes, oxygen and UV light. This means that to assess the suitability of a material for a given application, not just its original properties, but also some indication of how these properties change over time is needed. Unfortunately, aging is a complex process that is not only influenced by the material characteristics, but also by the conditions during production and construction and the local environmental conditions. This makes it difficult to define a test that covers aging for all materials and climatic conditions.
This is especially true in the current situation, where various changes occur simultaneously. On the one hand environmental and financial considerations lead to changes in the constituent materials that are used. Examples are the increase in recycling and the use of alternative materials like bio-binders, RAS and different additives. On the other hand, climate change causes changes in environmental conditions, which affect the way asphalt concrete properties change over time. These developments lead to an increased variation in material properties and pavement performance.
At the same time decreasing maintenance budgets result in an increased use of asset management systems. For most road authorities, pavement maintenance is a large part of their yearly costs, so a reliable prediction of the average life span of a pavement is crucial and this requires some method of determining the properties of pavement materials over time.

In Europe the Centre European de Normalisation (CEN, European centre for normalization/standards) technical committee on Asphalt Concrete in looking into the possibility to include requirements for aged asphalt concrete in the standards. In order to provide input for that attempt, the Dutch road authority (Rijkswaterstaat) and the Delft University of Technology organized a symposium to obtain an overview of the current practice regarding aging of asphalt concrete as well as the developments in research. This contribution is based on the results from that symposium *(1)* and aims to provide both an overview of the current practice in the USA and Europe and propose a next step that will give more fundamental insight in aging and allow the exchange of aging data. The first part of this paper summarizes the current approach to aging in the standards in the USA and Europe. The second part summarizes the discussion during the symposium, which results in a recommendation for a testing protocol that can be carried out with existing equipment but will provide an overall indication of aging sensitivity as well as input for fundamental aging research.

SUMMARY CURRENT AGING PROCEDURES IN USA AND EU STANDARDS

Aging tests: bitumen

Short term aging: RTFOT

Aging tests can be separated into tests on bitumen and tests on the asphalt mixture. For bitumen a common test to represent the short term aging of bitumen that takes place during mixing, transport and placement is the Rolling Thin Film Oven Test (RTFOT, AASHTO T240 *(2)*, ASTM D2872 *(3)* and EN12607-1 *(4)*). Besides in the actual standards, descriptions of this test can be found in *(5)* and *(6)*.

In this test bitumen is placed in glass bottles in a circular rack in a strictly specified oven. The rack contains eight bottles in total with 35 grams of bitumen per bottle. The oven is heated to 163^{o}C before placing the bottles in the rack and they are left in the oven for 75 *(4)* or 85 (*2, 3*) minutes of testing. The rack rotates the bottles at a rate of 15 revolutions per minute while the oven is kept at 163^{o}C. During the test air is being blown into the oven at 4000 ml/minute. After testing the mass loss, or more specifically the mass change (since some bitumen may increase in density due to oxidation), is determined. In the USA the material from the other bottles is used for DSR testing (T315 *(8)*) to obtain the $G^*/\sin\delta$ after short term aging which is used in AASHTO M320-10 *(7)*, as part of the requirements for binders. Alternatively, the material can also be aged further using the pressure aging vessel. In Europe the remaining material is used to determine the change in penetration, ring and ball temperature and viscosity at 60^{o}C. The standards for penetration bitumen (EN12591), polymer modified bitumen (EN14023) and hard paving grade bitumen (EN13924) specify the allowed changes in mass, penetration and/or ring and ball temperature.

Long term aging: PAV

The pressure aging vessel (PAV, AASHTO R28 *(9)*, EN 14769 *(10)*) is meant to simulate long term aging, the aging that occurs during the pavement service life. The current PAV test was developed during the SHRP program in the USA. In the test, previously RTFOT aged bitumen is aged further in a pressure vessel which is placed in an oven, both increased temperature and increased pressure to accelerate aging. The aim is to achieve an amount of aging that is comparable to several years of service life in a pavement. In developing the test, bitumen reclaimed from field cores was used as a reference, using the bitumen from the whole core. More recent results indicate that the top part of field cores is aged much more than lower parts *(11, 12)*. This indicates that assessing the aging effect based on bitumen reclaimed from whole cores rather than only the top 1 or 2 centimetres underestimates the aging effect. As such, PAV conditions are now thought to represent only limited aging times for the material at the top of a pavement.

USA In the USA, the PAV procedure uses samples of 50 g of bitumen in a 140 mm diameter container (giving a binder film that is approximately 3,2 mm thick) within the heated vessel. The pressure is 2,07 MPa for 20 hours at temperatures between 90 °C and 110 °C. Testing of the PAV (and RTFOT) aged bitumen in the DSR, bending beam rheometer and, in some states, the direct tension test is required for performance grading of bitumen.

Europe In Europe the suggested sample size is the same as in the USA (50 grams in 140 mm containers, but different sizes containers are allowed as well. In case of a different size, the amount of binder must be adjusted to ensure a layer thickness of approximately 3,2 mm. The pressures and temperatures used overlap with those used in the USA, but there are small differences, in Europe the pressure is 2,1 MPa (versus 2,07 in de USA) and the temperature range is 80^{o}C to 115^{o}C (versus 90 °C and 110 °C). More importantly, the current European bitumen standards do not require PAV aging or testing of PAV aged binder to assess the sensitivity to long term aging.
The European standards also allow using the Rotating Cylinder Aging Test (RCAT, EN15323) for aging of bitumen, the RCAT can be used for both short and long term aging but despite its versatility RTFOT and PAV set-ups are more widely available and as such have become more or less the standard procedure for bitumen aging in Europe.

Asphalt concrete

USA

In the MEPDG *(13)*, the effect of aging on the bitumen properties is determined using bitumen aging tests and this is related to the effect on the stiffness of the mixture through regression relations that take the mix composition into account (FIGURE 1).

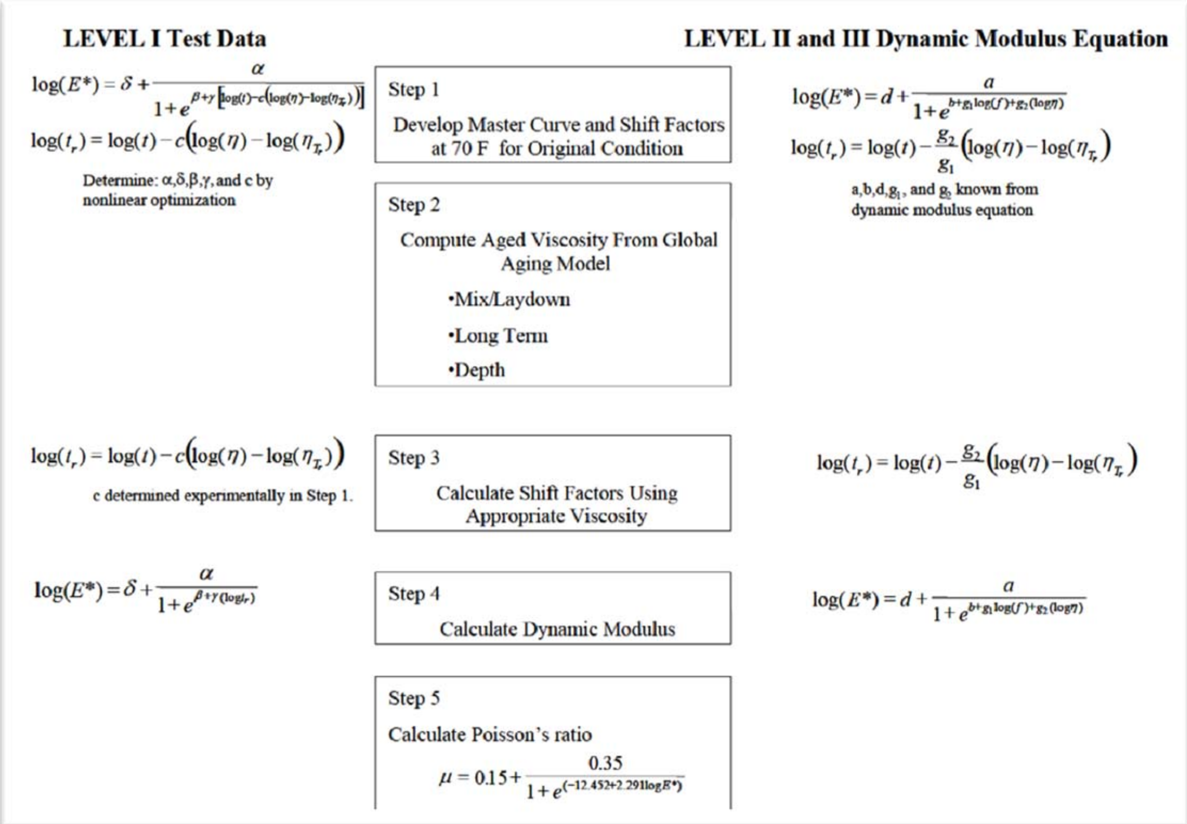

FIGURE 1 Aging of AC properties in the MEPDG works through regression based on bitumen aging (copy of fig 2.2.3 (*13)*)

Europe

Although the current standards for Asphalt Concrete do not require aging of the asphalt concrete itself, the CEN standards do provide tests for aging of AC. There is a test standard for hot mix asphalt saturation aging (SATS *(15)*). This standard aims to assess the durability of adhesion in base and binder courses by aging specimens in the presence of water. The test is currently limited to mixtures with a binder content between 3,5 and 5,5% of 10/20 hard paving grade binder and air voids between 6% and 10%. In this tests five AC cores are first partially saturated (≤80%) by putting them in a vacuum desiccator covered with distilled water for half an hour at a pressure of 40-70 kPa. After this, the specimens are placed on different levels in the SATS set-up. The set-up is partially filled with water, causing one specimen to be under water and the other four at various heights above the water level. The specimens are left in the set-up at a pressure of 2,1 MPa and a temperature of 85^{o}C for 65 hours. The dynamic stiffness (using the indirect tension test, EN12697-26 Annex C) is determined before and after conditioning and the average of the stiffness ratios of the four specimens that were placed above water level is used to obtain the mixture stiffness ratio. Currently, this test is used in the United Kingdom. Experience with this test in other countries is very limited.

CEN TC227 is currently working on a draft standard which allows the assessment of the effect of oxidative aging of asphalt mixtures (prEN 12697-52:2014, *(16)*). This standard aims to provide methods for laboratory aging of both lose (pre-compaction) asphalt concrete and AC cores, either produced in in the laboratory or obtained from the field. The aged material can be used to make specimens and assess the effect of aging on the mixture properties or binder can be extracted from the aged AC to assess the effect of aging in the presence of filler and aggregates on binder properties.

SUMMARY OF SOME RECENT AND ONGOING RESEARCH

The importance of the topic is illustrated by the amount of research on this topic. As a result, this section cannot possibly cover all work going on in this area. Instead, it focusses on some trends that various projects have in common regarding the relation between laboratory and field aging in order to arrive at a protocol to further develop this relation. Projects regarding the relation between the laboratory aging methods and field aging aim to establish a match between the chemical and physical (changes in) properties between both for long and/or short term aging.

Short term aging

Typically, it is found that the RTFOT test provides a good indicator of bitumen aging during production. The type of plant and the composition of the asphalt mixture do not seem to have a large influence on the field aging *(17).* The test does not predict the aging due production, its fixed temperature and duration does not account for variations in production temperature, storage and transport time and weather conditions, but it does provide a reliable indication of the binder sensitivity due to the production process of hot mix asphalt (HMA). For penetration binders the test shows the effect of bitumen source and grade on the aging susceptibility *(6)*. When using two different bitumens in exactly the same mix and using exactly the same production conditions, the bitumen that showed the most aging in the RTFOT will age most during actual production and construction *(1).* As such, the test is a good sensitivity indicator. It does not seem to be representative for hard grade, polymer modified and warm mix binders. For hard grade binders and polymers, this is probably because these materials do not mix as well as penetration binders. For warm mix binders, the test temperature is probably unrealistically high *(17)*. So for those materials and production methods, other tests or test conditions may be needed.

Long term aging

The most common test for long term aging is the pressure aging vessel (PAV, *(9), (10)*). Although in Europe there is also good experience with another method (Rotating Cylinder Method or RCAT), that equipment is much less wide spread. An important consideration in long term aging testing is the temperature. The high temperatures used in short term aging are not useful for long term aging tests, because they introduce secondary reactions. This has led to tests at lower temperatures and longer aging times. However, none of those tests can simulate the actual field aging *(6)*, since that depends on local weather conditions (temperature and water/moisture *(19)*) and mix composition properties such as the void content and/or bitumen film thickness (*6, 20*) and the type of minerals (especially filler *(21)*) used. The effect of mix composition was also found in a study on aging of Porous Asphalt with penetration 70/100 bitumen, where the relative importance of aging due to production was found to be considerably less important than predicted by the Shell bitumen handbook (FIGURE 2, *13*).

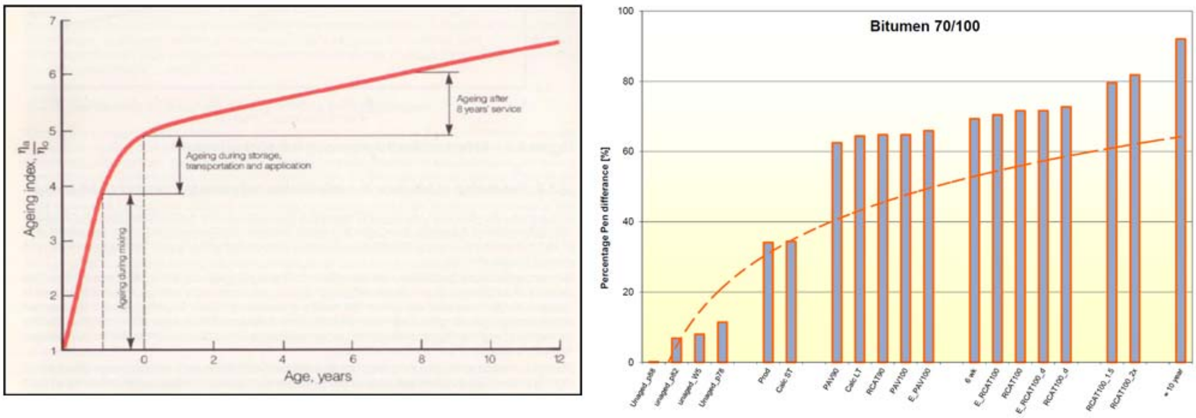

FIGURE 2 Aging effect graph from Shell (left) versus effect aging and various lab tests from Besamusca et al *(13)* (right)

As a result, although there is general agreement that aging is important for AC, especially for (low temperature) cracking, ravelling and fatigue resistance, it seems unlikely that a single test can reliably capture the phenomenon. In order to address the variables that play a role in field aging, a testing protocol should at least involve two temperatures in order to get an indication of aging sensitivity. However, this still doesn't address the effect of mix composition and microstructure. Attempting to age asphalt concrete specimens will have the drawback that the aging gradients that occur will not be the same as those in field applications, making it difficult if not impossible to relate the two.

A useful alternative approach to trying to get a single test that represents all variables in practice seems to be using the test to capture the aging sensitivity of the bitumen. This would require doing the test at two temperatures and two time intervals per temperature (i.e. four tests to characterize a bitumen) in order to be able to determine kinetic information. This information could then be used in models that take into account local climate conditions and ultimately mix composition and structure in predicting pavement aging (FIGURE 3). There is a long history of research into mathematical expressions and relations to describe aging *(22)*, because researchers have always been aware of the complexity of the phenomenon,. In the past decades, many researchers have successfully used a kinetic description of aging *(23, 24, 25, 26* and *27)*. In such descriptions, both rheological (viscosity, complex modulus phase angle, cross-over modulus) and chemical characteristics (change in C=O and/or S=O peak area in FTIR) can serve as reaction indicators for this approach.

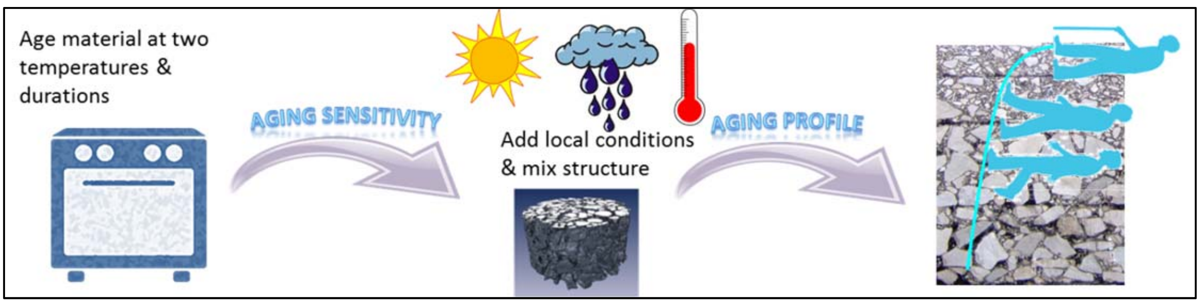

FIGURE 3 Two step approach to determining the aging of Asphalt Concrete

Based on the current standards and the discussions during the symposium *(1)*., PAV tests at 90 and 100 degrees Celsius and 20 and 40 hours, respectively, are suggested to provide the necessary information about aging sensitivity. The low values for temperature and duration are based on the current standards and fit both the USA and CEN procedure, while research using repeated PAV aging cycles at 100°C shows that after 40 hours at 100 degrees, but without previous RTFOT aging, the chemical (ICO from FTIR) and rheological (cross-over modulus from DSR) properties of laboratory aged and field samples were similar *(*FIGURE 4*, (28)).* At 100°C the temperature is low enough so that the effect of secondary reactions is negligible. As such, these conditions are appropriate for kinetic expressions for in service pavement performance. For high temperature processes and possibly also for repeated recycling (very long term) more sophisticated methods are needed.

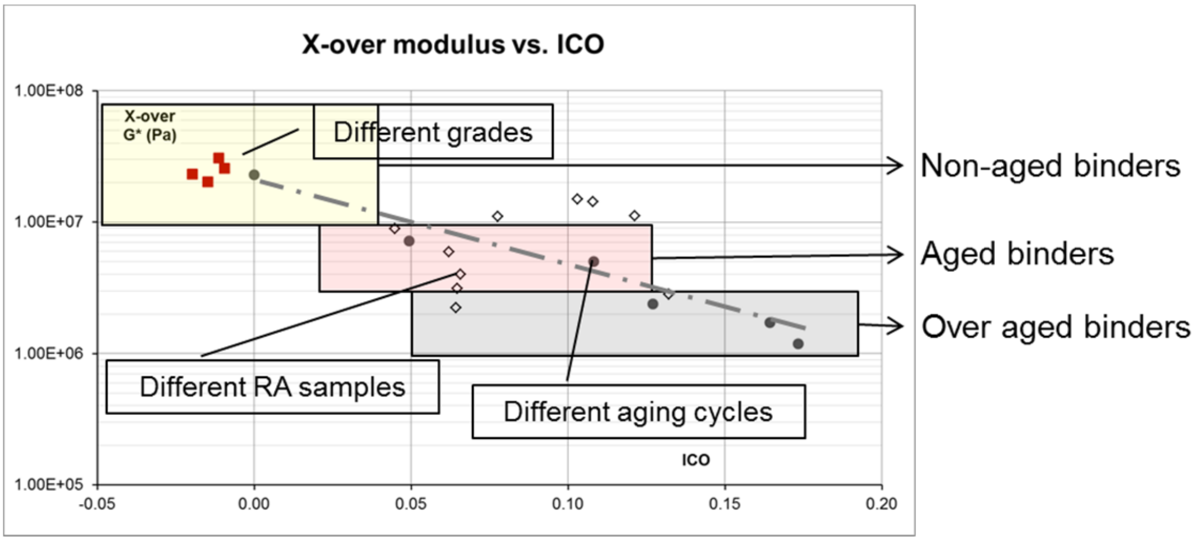

FIGURE 4: Chemical (carbonyl index) and rheological (cross-over modulus) properties of laboratory aged and field samples *(28)*

Additional relations to account for mix composition and micro structure (i.e. the chemo-mechanical aspects of aging) will need to be developed to take this information to the level of pavement aging. This will require a considerable research effort in multi-scale testing and modelling (FIGURE 5). However, in the mean time for practical applications, the requirements for the maximum changes in rheological properties can continue to be used. These requirements can be augmented by adding chemical requirements and/or by developing differentiated requirements for groups of materials (i.e. porous and dense mixture, mixtures with chemically active and inert fillers) or climate zones. Input for such adapted requirements should come from consistent monitoring of field aging, which will also provide the means to validate the models and laboratory test data.

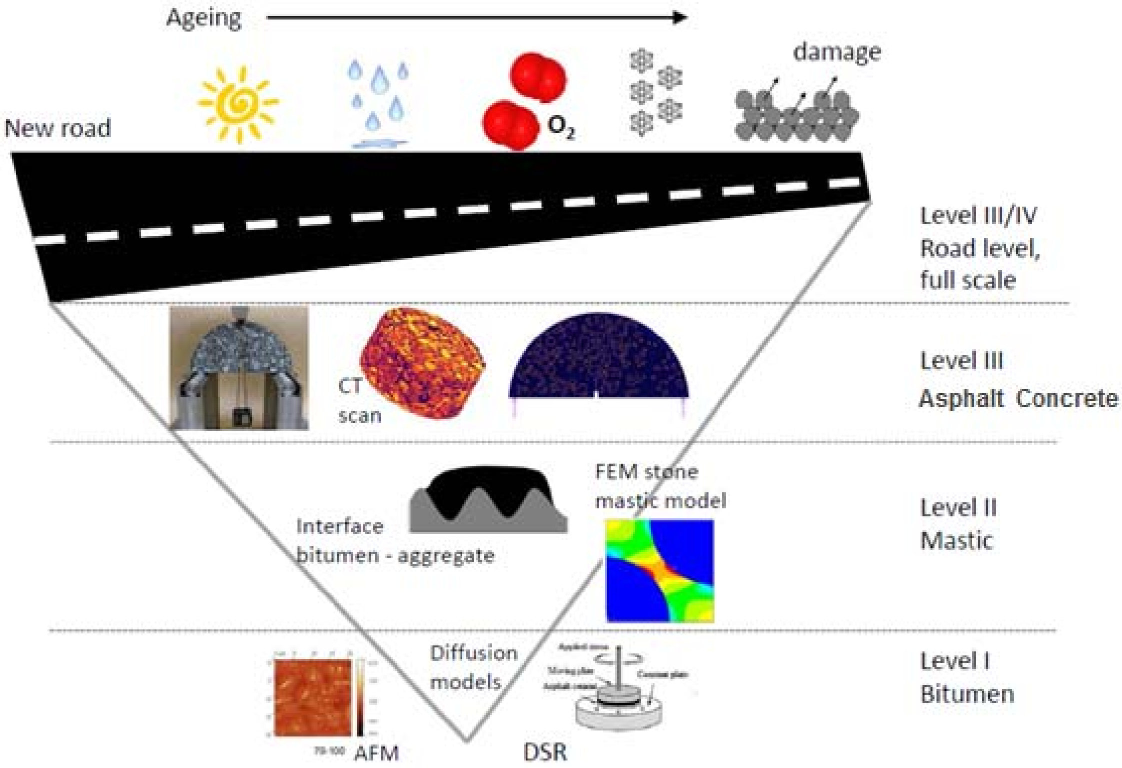

FIGURE 5: Example of an testing and modelling program aiming to account for local climate and mixture composition effects in aging (*29*)

SUMMARY AND CONCLUSIONS

In this paper, the USA and EU approaches to aging are compared, showing that those contain the same test equipment and almost identical conditions for aging. This allows the exchange of data and experience.

These tests are found to be most suitable for binders (not asphalt concrete) and to give only an indication of the sensitivity to aging. For short term aging RTFOT conditioning gives a reasonable indication of bitumen aging during asphalt concrete production and construction (*1*,*6*). But this only holds for penetration grade binders during hot mix production and construction. In its current form it doesn't work for hard grades, PMB's or warm mixes (*17*).

For long term aging, because of the many variables involved, developing a single test method to characterise aging sensitivity of bitumen, let alone asphalt concrete, seems impossible. However, using more elaborate protocols in existing, practical tests, can provide more information and be used to determine the kinetic properties. A PAV protocol for testing at two temperatures and time intervals, for example, could provide additional aging information for the short term and enable model development and validation on the long term.

RECOMMENDATIONS

Extend PAV conditioning to cover two temperatures and two conditioning periods, Based on the current standards and research, PAV tests at 90 and 100 degrees Celsius and 20 and 40 hours, respectively, are suggested. The low values for temperature and duration are based on the current standards and fit both the USA and CEN procedure, while research shows that after 40 hours of PAV at 100 degrees, without previous RTFOT, the chemical (FTIR) and rheological (DSR) properties of laboratory aged and field samples were similar (28, *1*). At 100°C the temperature is low enough so that the effect of secondary reactions is negligible. As such, these conditions are appropriate for kinetic expressions for in service pavement performance. For high temperature processes and possibly also for repeated recycling (very long term) more sophisticated methods are needed.

To provide the necessary background for requirements that take into account the effects of local climate and mix composition on aging, consistent field monitoring of temperature and UV radiation in various climate zones, as well as regular sampling over time to monitor aging over time is needed. Also, sampling at various pavement depths is needed to determine the aging gradient with depth. Such monitoring projects will provide the input for more specific requirements and model validation and ensure the applicability for pavement performance prediction.

In setting up such monitoring projects, it is important to get the properties and/or composition of both the virgin bitumen and the bitumen after mixing, transport and placement in the pavement. These provide the starting points for both the material and pavement structure point of view and can be used to assess the development of aging products over time.

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