Assessment of performance specification tests for rheologically simple bitumens

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ABSTRACT

The current edition of the European standard for specifying paving grade bitumens (EN 12591) is based on empirical test methods, such as penetration and softening point. Since the USA and Canada successfully adopted the rheology based performance specification system, Superpave, the discussion whether to introduce rheology tests and develop performance specification has moved to Europe.

Recently, a Comité Européen de Normalisation (CEN) proposal to start collecting rheology data for bitumens has been drafted. For low temperature performance assessment, an approach nearly identical to Superpave was selected; low temperature testing in a Bending Beam Rheometer (BBR) performed on long term aged bitumen. For high temperature performance assessment Superpave equiviscous testing was not adopted. An isothermal dynamic shear rheometer tests at 15, 40 or 60 °C were selected to be performed on short term aged bitumen instead.

This paper offers a comparison between empirical and rheology test methods. It highlights the advantages and disadvantages of each testing system. It explains the philosophy in Superpave specification and the process by which the rheology test results are related to bitumen performance in asphalt. Current CEN proposal for rheology data collection is reviewed and suggestions to development of a performance based specification system are provided.

Keywords: Comité Européen de Normalisation, Complex Modulus, Performance testing, Rheology, Temperature susceptibility

1. INTRODUCTION

Understanding rheological properties of bitumens is a critical task in selecting appropriate materials for asphalt road construction. Traditional empirical tests such as penetration and softening point have been used in Europe for many decades to determine bitumen consistency and suitability for a given application. The advantage of the empirical tests is in their general availability, easy execution, and historical experience. Although the traditional specification system has worked reasonably well in most situations, the introduction of modified bitumens, diversification of bitumen refinery feedstocks, and demand for higher durability materials has raised increased challenges for the existing specification system. A viable upgrade to the current specification system and harmonization across Europe has been sought for approximately the last two decades.

The current harmonized specification proposal by Comité Européen de Normalisation (CEN) for simple bitumens (i.e. unmodified paving grades, prEN 12591 [1]) builds on the previous national penetration-softening point specifications. There is still an opportunity to harmonize penetration ranges and additional regional specifications in order to allow European suppliers to take a full benefit of the European marketspace. In Annex C of prEN 12591, there is nevertheless a proposal for rheology data collection for information purposes. The rheological data can later be used for development of bitumen performance specification system for simple as well as for complex (e.g. polymer modified) bitumens.

The objective of this paper is to review the latest CEN proposal of the new rheological tests at the time of paper writing (August 2015). The proposed low temperature test and the selected test limits are nearly identical to the Superpave Bending Beam Rheometer (BBR) test (AASHTO T313). The scientific merit of the proposed high temperature and intermediate temperature tests, their relation to existing traditional tests and their potential to predict bitumen performance in paving applications are also reviewed. Comparisons are made to the North American Superpave specification system.

2. FROM EMPIRICAL TESTS TO FUNDAMENTAL RHEOLOGY

The rheological properties of bitumen have been traditionally studied by empirical tests such as penetration and softening point. These tests are called empirical, because results are difficult to interpret in common physical or engineering terms such as modulus, and an empirical knowledge is needed to interpret the test results. Nevertheless, empirical bitumen tests are indeed rheological tests. Penetration is an isothermal, constant-stress experiment, with a very complex deformation shape and with the test result being impacted by needle shape (combination of a cone and a cylinder), adhesion of needle to bitumen, bitumen surface depression, bitumen surface skin formation etc. With all that, penetration was surprisingly well related to modulus for a variety of bitumens [2] [3] [4]. Softening point is also a constant stress experiment with a complex shape of deformation, however performed in non-steady thermodynamic conditions of gradually increasing sample temperature. It can be very vaguely considered to be an equiviscous test. Attempts to relate softening point to modulus or dynamic viscosity has also been completed in the past [2].

Although empirical tests work reasonably well to determine binder consistency, application of empirical results to mix design, mix modulus, relaxation properties etc. in standard physics terms is cumbersome. Therefore since the 1960s more fundamental rheological studies have been performed [5] [6] [7] [8] [9]. Well-defined rheological tests were introduced or developed for bitumen, namely within Strategic Highway Research Program (SHRP). Isothermal dynamic oscillatory and creep tests were performed at small (linear) deformations to determine stress/strain relations and their temperature dependency. Analysis of experimental data indicated that many of the rheological features observed in other materials such as polymers apply to bitumen. The major advantage of using well defined tests is that the rheology of bitumen can be represented by well-understood and well-defined physical properties. These properties can be readily used to model or predict mechanical properties of asphalt and asphalt pavement.

3. EXPERIMENTAL DATA

1) ExxonMobil Asphalt Assay Database (EMAAD) contains specification properties of a great variety of bitumens that were prepared by laboratory vacuum distillation of crude oils sourced globally. The purpose of this dataset is to demonstrate general relationships among bitumen rheological properties. Not all properties were available for all bitumen, maximum possible number of properties was used in correlations and outliers were not removed. These data were collected according to relevant ASTM, EN and AASHTO standards by Imperial Oil Limited Asphalt Laboratory.

2) Eurobitume Data Collection Database (EDCD) contains properties of 82 Paving, 17 Hard, 63 Polymer modified, and 5 Special bitumen grades. Not all properties were available for all bitumens, maximum possible number was used in correlations and outliers were not removed. A large variety of properties including Dynamic Shear Rheometer (DSR) data were collected by multiple European laboratories and compiled by Eurobitume [10]. 2014 version of EDCD available to Eurobitume members is used here.

3) For select EMAAD bitumens a Thermal Analysis (TA) AR 2000ex rheometer was used to generate experimental data for Mastercurves of dynamic material functions. Typical geometry selection is listed in Table 1. A strain sweep was performed at maximum testing frequency of 100 rad/s for each test temperature to determine the linear viscoelastic region and appropriate strain level for subsequent frequency sweeps. Isothermal frequency sweeps were subsequently performed at 10 °C increments with test frequency ranging from 0.1 to 100 rad/s. Mastercurves of dynamic material functions were merged graphically in the TA Data Analysis software. The reference temperature was 30 °C.

Table 1: Geometry, temperature ranges and gap settings for paving bitumens.

Geometry	Shear Modulus	Gap
40 mm Parallel or cone & plate	<1 kPa	0.5 mm
25 mm Parallel or cone & plate	1 to 100 kPa	1 mm
8 mm Parallel plate	100 kPa to 10 MPa	2 mm
Torsional Bar (25x12x3 mm)	>10 MPa	25 mm

4. DYNAMIC EXPERIMENTS IN SIMPLE SHEAR

Both, Superpave specification (AASHTO T315) and the current European proposal for paving bitumens for high and intermediate temperature specification test utilizes dynamic shear rheometer with a parallel plate configuration. The scientific background of dynamic or periodic experiment in simple shear is reviewed here first.

Rheological experiments are typically made by observing external forces and changes in external dimensions of a specimen with a certain shape. The state of deformation at a given point is specified by strain tensor which represents the relative changes in dimensions. The state of stress is specified by a stress tensor which represents the forces acting on the specimen from different directions. For homogenous deformation the stress and strain do not vary with position. Simple shear is an example of experimental geometry where this assumption holds true. Simple shear provides an obvious advantage over penetration or softening point deformation patterns. The strain, $\boldsymbol{\gamma}$, and stress, $\boldsymbol{\sigma}$, tensors are functions of time and for small deformations they are related by a constitutive equation for linear viscoelasticity:

$$\boldsymbol{\sigma}(t) = \int_{-\infty}^{t} G(t-t')\dot{\boldsymbol{\gamma}}(t')dt' \qquad 1$$

where $\dot{\boldsymbol{\gamma}} = \partial\gamma/\partial t$ is the shear rate tensor, $G(t)$ is called the relaxation modulus and the integration is carried out over all past times t' up to the current time t. For a simple shear in positive direction of Cartesian x-axis and after subtracting isotropic pressure, only the deviatoric stress tensor components remains, $\sigma_{12} = \sigma_{21} \neq 0$. The tensors in the Equation 1 are thus simplified to component 12 and for simplicity the subscript 12 is omitted here for σ and $\dot{\gamma}$.

When very short deformation times are required, the stress can be varied sinusoidally at frequency ω in radians per second. If the viscoelastic behavior is linear, than the strain also alternates sinusoidally, but it is out of phase with the stress. The strain time function can be written as:

$$\gamma = \gamma^0 \sin \omega t \qquad 2$$

where γ^0 is the strain amplitude. The strain rate is derived as:

$$\dot{\gamma} = \omega\gamma^0 \cos \omega t \qquad 3$$

Substitution of Equation 3 into Equation 1 with $t - t' = s$ yields:

$$\sigma(t) = \int_0^\infty G(s)\omega\gamma^0 \cos[\omega(t-s)]ds = \gamma^0\left[\omega\int_0^\infty G(s)\sin\omega s\, ds\right]\sin\omega t + \gamma^0\left[\omega\int_0^\infty G(s)\cos\omega s\, ds\right]\cos\omega t \qquad 4$$

The quantities in square brackets are independent of elapsed time t and Equation 4 can be written as:

$$\sigma = \gamma^0(G' \sin\omega t + G'' \cos\omega t) \qquad 5$$

thereby defining two test frequency dependent functions: shear storage modulus, $G'(\omega)$, and shear loss modulus, $G''(\omega)$. Shear stress can alternatively be expressed by using stress amplitude, σ^0, and the phase angle, δ, between stress and strain functions:

$$\sigma = \sigma^0 \sin(\omega t + \delta) = \sigma^0 \cos\delta \sin\omega t + \sigma^0 \sin\delta \cos\omega t \qquad 6$$

by comparison of equation 5 and 6:

$$G' = (\sigma^0/\gamma^0)\cos\delta \qquad G'' = (\sigma^0/\gamma^0)\sin\delta \qquad G''/G' = \tan\delta \qquad 7$$

It is common to express sinusoidal stress as a complex quantity. The time invariant complex shear modulus, G^*, is:

$$G^* = \sigma^*/\gamma^* = G' + iG'' \text{ and its magnitude as } |G^*| = \sigma^0/\gamma^0 = \sqrt{G'^2 + G''^2} \qquad 8$$

The function G' then represents the ratio of the stress in phase with strain to the strain and reflects the elastic response of the viscoelastic material or energy stored during the deformation cycle, and the function G'' represents the ratio of the stress 90° out of phase with strain to the strain and reflects the viscous response of the viscoelastic material or energy dissipated as heat during the deformation cycle. A dynamic experiment at a given frequency provides simultaneously two quantities G' and G'' or else $\tan\delta$ and $|G^*|$. It is not possible to describe material viscoelastic properties with just one of these quantities [11].

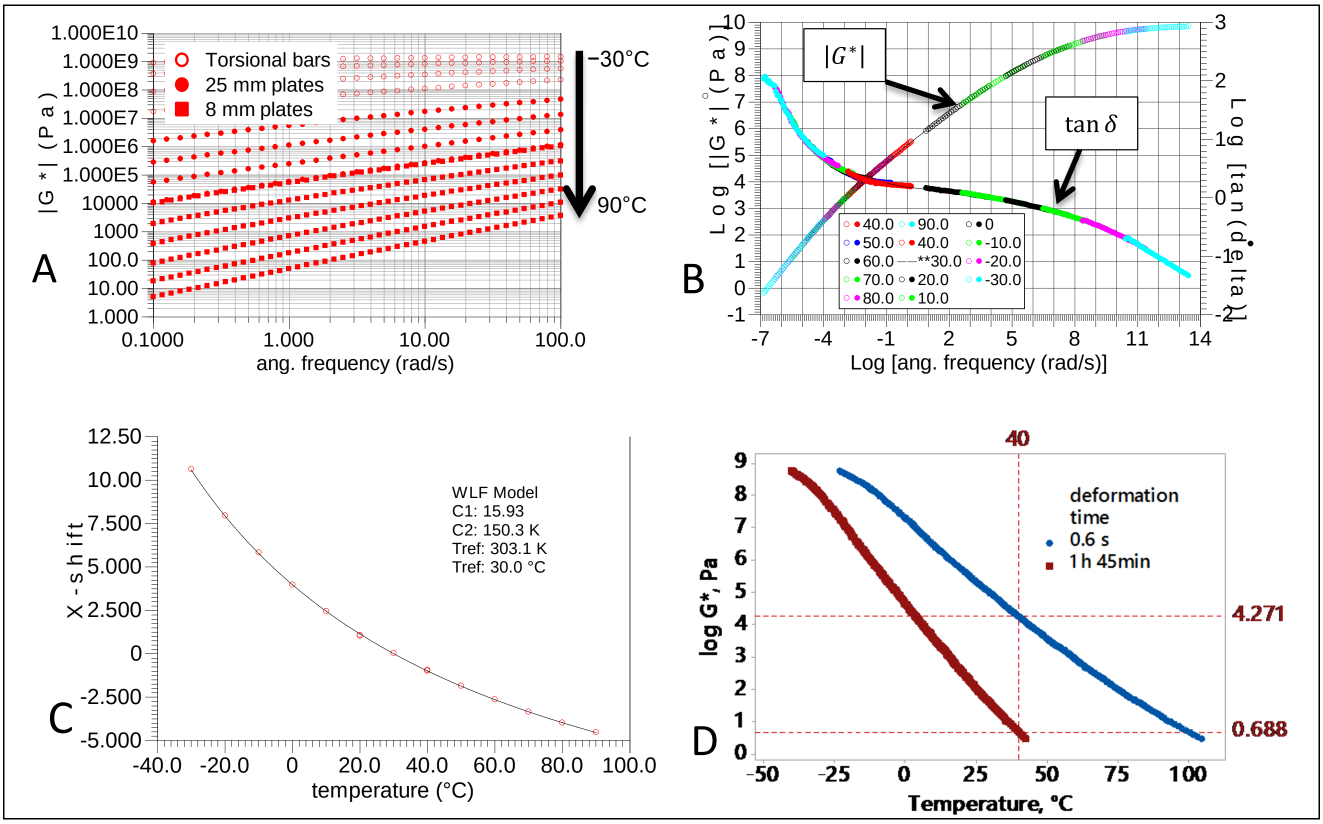

Figure 1: A - Experimental data ($|G^*|$) from isothermal frequency sweeps collected at constant temperatures between -30 and 90 °C at 10 °C increments. 1B - Isotherms shifted horizontally alongside the frequency axis ($T_0 = 30$ °C), 1C - logarithm of horizontal shift factor versus temperature fitted with WLF model. 1D – application of WLF to obtain $|G^*|(T)$ function at two different test frequencies.

In order to obtain material dynamic functions over the large range of temperatures or test frequencies it is convenient to perform dynamic test at a range of frequencies isothermally at several temperatures and then merge the isotherms into a Mastercurve of a given dynamic function. The isotherms can be simply shifted horizontally alongside the test frequency axis with respect to arbitrarily selected fixed or reference temperature, T_0, dataset and merged into a single Mastercurve. Material then obeys time-temperature superposition or thermorheological simplicity and reduced variables can be introduced as [5]:

$$\sigma(T, \dot{\gamma}, \omega, t) = \sigma(T_0, a_T\dot{\gamma}, a_T\omega, t/a_T) \qquad 9$$

Where a_T is the horizontal factor which represents a shift distance alongside the horizontal axis and essentially depends only on $(T - T_0)$. Practical advantage is in obtaining dynamic material properties at loading frequencies which are too low or too high to be obtained experimentally. The temperature and time (of frequency in the case of dynamic experiments) are interchangeable when the temperature dependency of a_T is determined. For temperatures above the glass transition temperature, T_g, the Williams-Landel-Ferry (WLF) model [12] typically provides a good fit of the experimental data:

$$a_T(T, T_0) = -\frac{C_1(T - T_0)}{C_2 + T - T_0} \qquad 10$$

where C_1, C_2 re the empirical fitting parameters. Now the frequency domain, ω, of the Mastercurve can be converted to the temperature domain for an arbitrarily selected testing frequency, ω_t:

$$T = T_0 + \frac{C_2 \log(\omega_t/\omega)}{C_1 + \log(\omega/\omega_t)} \qquad 11$$

Development of Mastercurve of dynamic material functions for arbitrarily selected straight run bitumen is presented in Figure 1(A-D). Straight run bitumen typically obeys time-temperature superposition principle and a smooth Mastercurve is obtained. Utilization of various geometries did not cause any discontinuity in the data (Figure 1B). It is important to perform strain sweeps to identify linear viscoelastic range of strains for each subsequent frequency sweeps. Also all experiments should be performed well within the instrument transducer capability, corrected for instrument compliance and test frequency extremes should be avoided. WLF equation was subsequently utilized to transform the reduced frequency scale to temperature scale (Figure 1C). This is a convenient approach when material property dependence is studied at varying deformation times. Practical example is presented in Figure 1D. Two curves for $|G^*|(T)$ are presented. One was obtained at testing or deformation time ($t = 2\pi/\omega$) of 1 h 45 min and the other at 0.6 s. For illustration, the former may represent a bus parked at a station for 1 h 45 min the latter a bus passing by at a speed of 60 km/h. If the same stress would be applied in both cases, the same deformation caused by the standing bus in 1 h 45 min would be achieved by nearly 4000 passes of the bus at 60 km/h. In reality, the comparison may be nontrivial because the experimental data presented here are for bitumen and not for asphalt but it demonstrates that time and temperature are interchangeable when deformation is considered.

5. SUPERPAVE SPECIFICATION

A review of the current North American bitumen specification system, Superpave, is provided here to offer comparison to European CEN proposal which is largely based on the Superpave test methods. Superpave was developed between late-1980s to mid-1990s and has been used since mid-1990s by the USA and Canada. Implementation was gradual with Western Canada [13] and California being one of the last jurisdictions moving to Superpave in recent years.

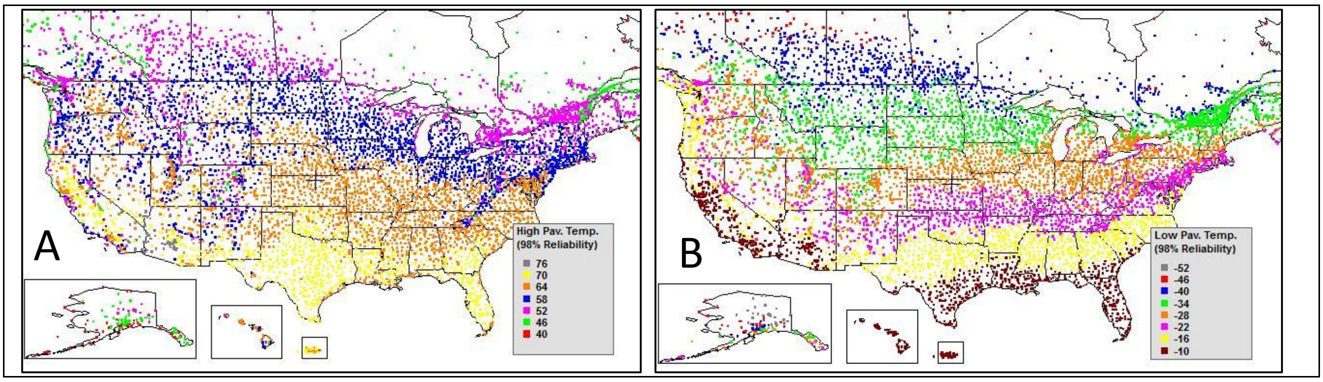

Figure 2: Performance Grade recommendation for North America at high temperature (2A) and at low temperature (2B). Outcome from LTTPBind Software at 98% reliability interval.

Following assumptions were made in Strategic Highway Research Program (SHRP) during Superpave development. The highest risk of bitumen being too soft to induce pavement rutting exists at highest temperature pavement can experience when the bitumen is new. The highest risk of bitumen being too stiff or brittle to induce pavement cracking exists at the lowest pavement temperature when the bitumen is aged. The climate where the future pavement will be built has to be examined first and the maximum and minimum in-service temperatures have to be determined. These temperatures represent a temperature range where bitumen consistency must meet the requirements, i.e. stiff enough at high and ductile enough at low temperature. For practical reasons, the high and low temperatures are divided by 6 °C increments. This is based on the assumption that the bitumen stiffness approximately doubles/halves by every 6 °C. This approach provides lower number of grades. For example Performance Grade (PG) 64−22 represents bitumens which are expected to perform well at pavement temperature between 64 and −22 °C. All bitumens fitting in this grade are considered to perform comparably. If high or low PG is exceeded, e.g. pavement temperature drops below −22 °C, this bitumen may no longer be able to relax thermal stresses and a transverse low temperature cracking can occur. In this case, climate is too cold for PG 64−22, and e.g. PG 64−28, should be used to mitigate low temperature failures.
For the USA and Canada, LTPPBind (Long-Term Pavement Performance) software [14] was developed to determine pavement high and low temperature extremes in a given region. By using the climatic data, an average 7-day maximum and 1-day minimum pavement design temperatures in degrees Celsius were determined for each region [15]. Based on these temperatures the high and low PGs are recommended for each region (Figure 2).
The accelerated aging tests are performed first to simulate binder aging in a hot mix plant (Rolling Thin Film Oven, RTFO) and in-service (Pressure Aging Vessel, PAV). Three bitumen samples, original, RTFO and PAV[1], are prepared and tested. At high PG, the magnitude of the complex shear modulus divided by the sine of the phase angle, $|G^*|/\sin\delta$, is determined on the original and the RTFO aged binder. The test is performed at a fixed test frequency of 10 rad/s and fixed strain of 12 % for original and 10 % for RTFO bitumen, respectively. The frequency simulates deformation rate caused by a car passing at 60 km/h and the strain is expected to be within the linear viscoelastic range. The minimum limit for $|G^*|/\sin\delta$ is 1.0 kPa for original bitumen and 2.2 kPa for RTFO bitumen. The limits ensure the stiffness is high enough for the new pavement to resist rutting during the hottest climate it will experience in-service.

[1] The PAV refers to bitumen aged first in RTFO and subsequently in PAV unless noted otherwise.

An intermediate test temperature is determined as (High PG+Low PG)/2+4, e.g. for PG 64−22, the intermediate temperature is (64−22)/2+4 = 25 °C. For intermediate temperature test, the specification limit is $|G^*| \cdot \sin\delta \leq 5000$ kPa. Equations 7 and 8 directly imply $|G^*| \cdot \sin\delta = G''$ which is the imaginary part of the complex function. Note that at intermediate temperatures the phase angle may vary significantly among bitumens and describing only G'' does not provide a complete picture.

To assess low temperature properties, PAV bitumen is subjected to flexural creep test in BBR. For practical reason, the test temperature is set to 10 °C higher temperature than the low PG. Due to time-temperature equivalency a test which would take 2 h at low PG can be done in 60 s at PG + 10 °C. The test results are evaluated at creep time of 60 s. The limits for flexural stiffness modulus and m-value are ≤ 300 MPa and ≥ 0.3 MPa/s, respectively. The m-value is defined as an absolute value of the slope of the logarithm of the stiffness versus the logarithm of time, and it is related to stress relaxation properties of a bitumen. The faster the stiffness decreases with the creep time the higher the absolute slope (m-value) and the more ductile the bitumen at the test temperature. The relationship between flexural creep and dynamic simple shear is not straightforward. One of the challenges is the time dependence of the beam Poisson ratio. Several assumptions are typically made to convert creep experimental results to dynamic simple shear and vice versa [16]. Nevertheless, the relationship between $|G^*|/\sin\delta\,(T)$ function and the flexural creep stiffness is presented in Figure 3A. A temperature satisfying $|G^*|/\sin\delta\,(T) = 300$MPa at testing frequency of 0.105 rad/s[2] is plotted versus the temperature satisfying $S = 300$ MPa, or BBRST (cf.). Very good parity was obtained, even though the BBR test was performed on PAV bitumen and the $|G^*|/\sin\delta$ function was obtained by dynamic testing on original bitumen. This suggests that bitumen aging may not impact relative ranking in the low temperature test significantly.

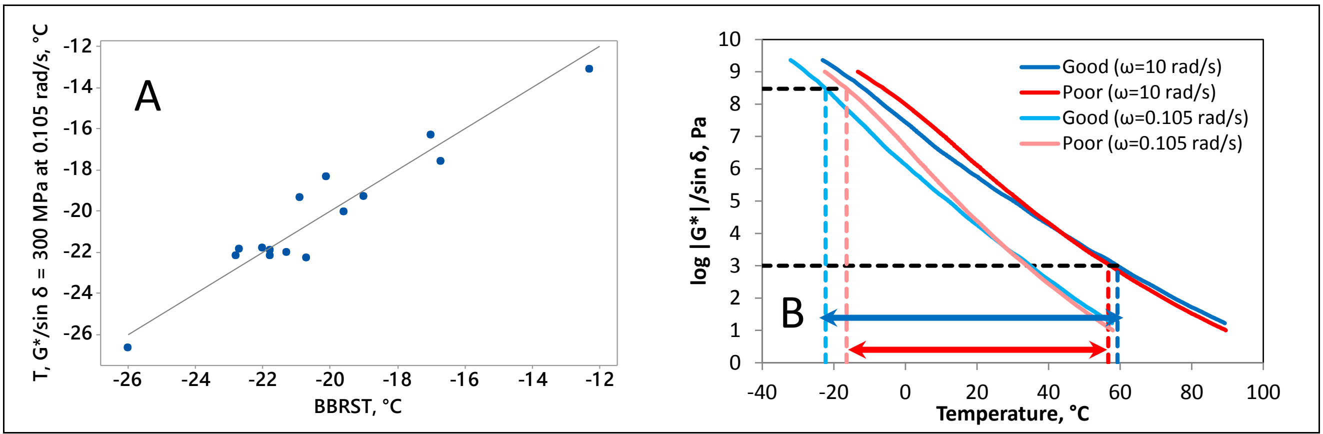

Figure 3: A - Comparison between temperatures at $|G^*|/\sin\delta = 300$ MPa at 0.105 rad/s test frequency (original bitumen) and at $S(60) = 300$ MPa (PAV bitumen). Results for 15 straight-run bitumens, parity line indicated. 3B - Temperature susceptibility in Superpave. Steeper slope of $|G^*|/\sin\delta\,(T)$ function represents higher temperature susceptibility(right). Continuous HT and LT PG indicated on the chart at 1000 Pa (10 rad/s curves) and at 300 MPa (0.105 rad/s curves), respectively. Horizontal arrows indicate 'PG' temperature range.

It is practical to assume linear behavior of $\log(|G^*|/\sin\delta)$ with temperature, especially within a narrow range of temperatures. The limiting temperature then can be calculated from test results at 'pass' temperature where $|G^*|/\sin\delta > 1.0$ kPa, e.g. at 58 °C, and at temperature by 6 °C higher or 'fail' temperature where $|G^*|/\sin\delta < 1.0$ kPa, e.g. at 64 °C. By plotting $\log(|G^*|/\sin\delta)$ versus T a temperature where $|G^*|/\sin\delta = 1$ kPa is found by interpolation. This temperature is referred to as DSROT where "O" stands for original bitumen and "T" for temperature. The same can be repeated for measurements performed on RTFO bitumen, and temperature where $|G^*|/\sin\delta = 2.2$ kPa is found, DSRRT. The lower of the two interpolated temperatures is the High Temperature PG (HTPG), an approximated highest temperature where Superpave limits are met. The same logic can be used to find Intermediate Temperature PG or ITPG as temperature where $|G^*| \cdot \sin\delta = 5000$ kPa and Low Temperature PG or LTPG as a higher temperature of the temperatures where $S = 300$ MPa (BBRST) or $m - value = 0.300$ MPa/s (BBRMT).

Figure 3B demonstrates how temperature susceptibility is evaluated in Superpave. Two straight-run bitumens are compared. One is good paving bitumen originated from Western Canadian heavy crude oil with low temperature susceptibility, the other is poor paving bitumen, with high wax content and high temperature susceptibility. The faster the $|G^*|/\sin\delta$ changes with temperature the more temperature susceptible the bitumen is. It is evident that the slope is efficiently captured by simultaneous use of HTPG with LTPG (width of the horizontal arrow in Figure 3B). The same concept can be visualized on so called Superpave plot, where HTPG is plotted versus LTPG (Figure 4A). The bituminous quality of the crude oil defines the position of the distillation line (typical slope 1.3 – 1.5). The bitumen made by distillation would follow the distillation line and move diagonally from left bottom corner (soft bitumen) to top right (hard bitumen) by distillation [13]. The larger the span between HTPG and LTPG the less thermally susceptible

[2] 300 MPa target for $|G^*|/\sin\delta$ was chosen to match the BBR results. Testing frequency of 0.105 rad/s equals to deformation time of 60s which is a deformation time in BBR test.

paving bitumen. Thus the distillation line for poor PG bitumens are in the lower right section while distillation lines of good PG bitumens are in the top left corner in Figure 4A.

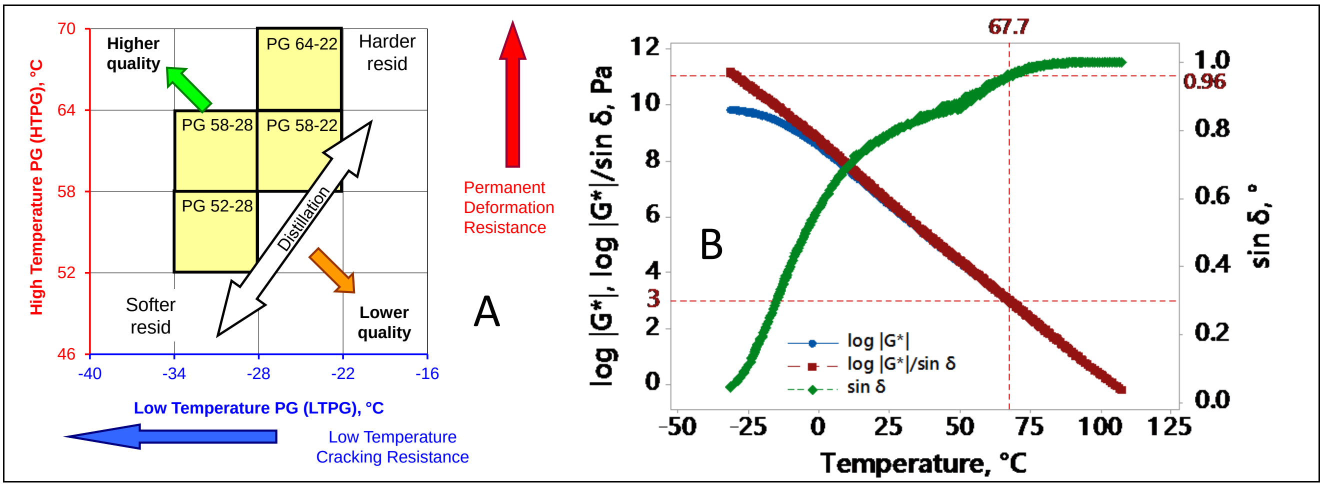

Figure 4: A - Bitumen quality as represented in Superpave system. 4B - Mastercurve of dynamic material functions; sin $\delta \cong 1$ at $|G^*| = 1$ kPa indicated on the chart.

In summary, the major advantages of Superpave are:

1. System is verified on a number of test tracks and by two decades of industry use
2. Specification limits are directly derived from the pavement performance and the rheological limits are universal for every grade while the test temperature is dictated by the climate
3. Properties are measured in common engineering properties
4. Fundamental rheology offers a platform for further research, e.g. Multi Stress Creep and Recovery [17], Linear Amplitude Sweep [18], and Glover-Rowe parameter for fatigue [19].

There are also some opportunities for improvement:

1. Long-term aging (PAV) is a time consuming test and may underestimate aging in the pavement especially with modified binders [20] [21]. New long term aging protocols are being developed [22] [23]
2. Intermediate temperature test (DSR test on PAV bitumen) suffers from high variability (d2s% = 40.2) [24]
3. All measurements are done in a linear viscoelastic domain (i.e. small deformations) although pavements fail in non-linear domain (i.e. large deformations) [24]
4. No strength, cracking or fatigue tests are routinely performed on a bitumen[3]
5. Superpave may not well represent properties of some modified asphalts [20].

6. RELATION BETWEEN EMPIRICAL SPECIFICATIONS AND DYNAMIC MATERIAL FUNCTIONS

Sometimes the term rheological specification (meaning the dynamic testing) is used interchangeably with the term performance specification (meaning specification reflects performance). The type of rheology test (empirical or dynamic) used to assess the consistency of the bitumen, is in this case, of limited importance. The objective here is to demonstrate this fact and not to develop a robust model for property correlation.

6.1 Correlation between High PG and Empirical Tests

DSROT was first correlated to the logarithm of dynamic viscosity at 60 °C (ASTM D2171) in Figure 5. A better correlation was found for the EMAAD dataset. This can possibly be attributed to higher data precision since all EMAAD data were collected by a single lab versus multiple labs for EDCD data. Also all bitumens in EMAAD are lab distilled versus refinery blends in EDCD which may not be pure vacuum residua in some cases. Also some extrapolation instead of interpolation was done for EDCD data as pass/fail temperature results were not always available. Lastly, data were only available in 10 °C increments instead of 6 °C as done in Superpave. For viscoelastic liquids it is convenient to express phase relationship by a complex viscosity $\eta^* = \eta' - i\eta''$, where η' is the ratio of stress in phase with rate of strain to the rate of strain and η'' is the ratio of stress 90° out of phase with the rate of strain divided by the rate of strain. These relations are the opposite of those for G^* and are related to G' and G'' as $\eta' = G''/\omega$ and $\eta'' = G'/\omega$. For a viscoelastic liquid η' approaches steady-flow viscosity η_0 as the frequency ω approaches zero. At $|G^*|/\sin\delta = 1$ kPa, it can be assumed that $\delta \approx 90°$ or $\sin\delta \approx 1$ or $|G^*| \approx G'' = 1$ kPa for most simple bitumens (Figure 4B). Bitumen therefore can be considered a viscous liquid with $\eta_0 \approx \eta' = G''/\omega = \frac{1000}{10} = 100$ Pa.s. Now, for comparison to dynamic viscosity at 60 °C, viscosity of bitumen with HTPG of 60 °C can be derived from Figure 5. Its viscosity is $\eta = 10^{(60-26.05)/16.90} = 102.06$ Pa.s which is within 2% difference from viscosity derived from dynamic

[3] Direct tension test (AASHTO T314) is part of Superpave however it is not mandatory in majority of the cases.

test. DSRRT was also correlated to dynamic viscosity at 60 °C measured on RTFO bitumen (R^2 = 98.6%, data not presented). Dynamic viscosity at 60 °C at DSRRT of 60 °C was $\eta = 230.4\ \mathrm{Pa.s}$ which compared well to $\eta' = 220\ \mathrm{Pa.s}$ from dynamic test (~5% difference). This is a satisfactory result given that these are two very different tests and that some approximations were made.

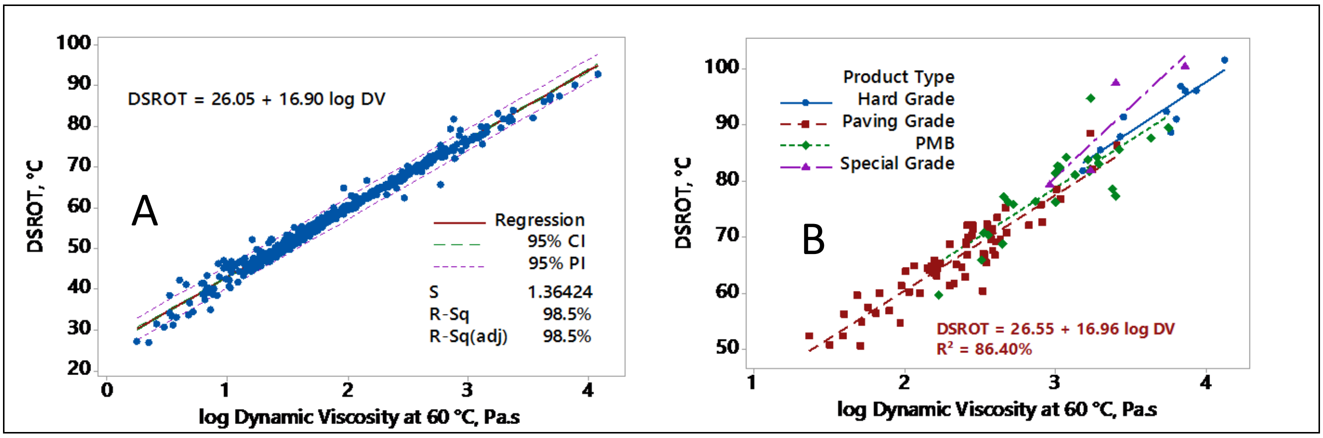

Figure 5: A - Correlation between DSROT and log Dynamic Viscosity at 60 °C, 619 lab distilled bitumen from EMAAD and 5B - 103 bitumen from EDCD, regression equation displayed for Paving Grade only (right).

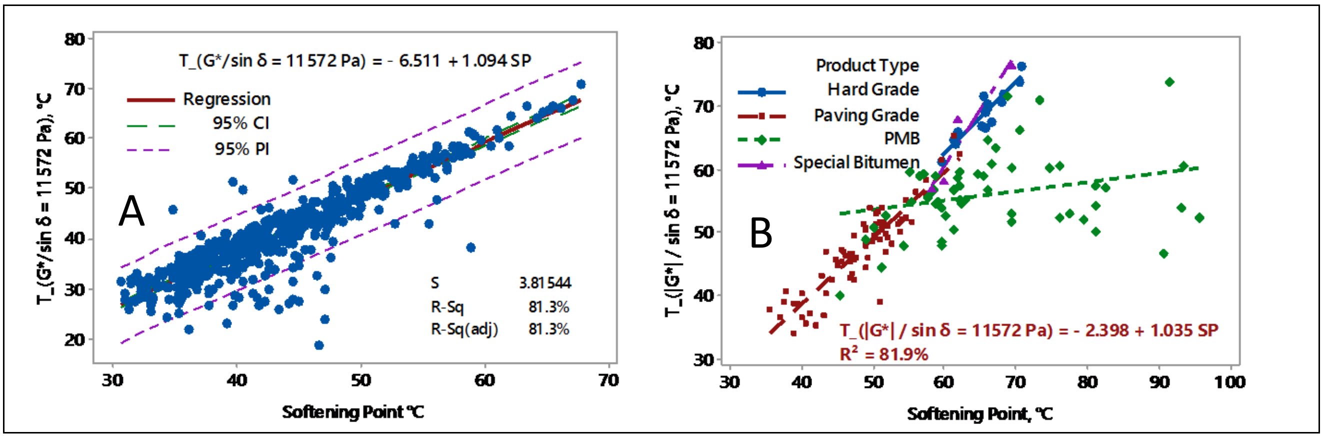

Figure 6: A - Correlation between equiviscous temperature ($|G^*|/\sin\delta = 11\ 572$ Pa) and SP, 578 lab distilled bitumen from EMAAD and 6B - 146 bitumen from EDCD, regression equation displayed for Paving Grade only.

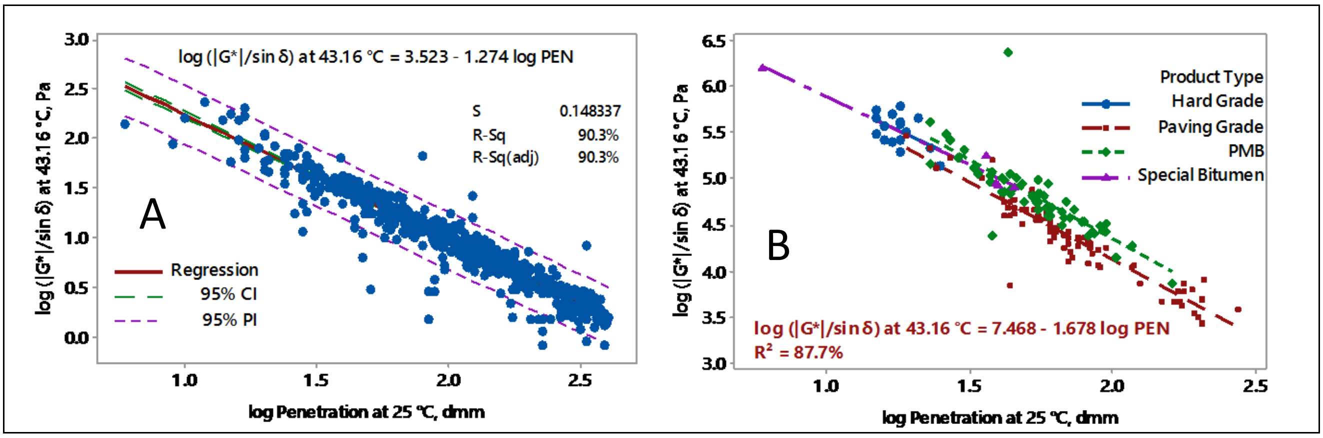

Figure 7: A - Correlation between log $|G^*|/\sin\delta$ at 43.16 °C and log penetration, 578 lab distilled bitumen from EMAAD (left) and 7B - 158 bitumen from EDCD, regression equation displayed for Paving Grade only (right).

As it was mentioned previously, softening point, SP (ASTM D36), can vaguely be considered an equiviscous test. Although this is an over simplification the following comparison was made. An equiviscous temperature for was found at $|G^*|/\sin\delta = 11572\ \mathrm{Pa}$ where the mean of residuals, softening point − equiviscous temperature, approximately equals zero for Eurobitume dataset. Equiviscous temperature was plotted versus SP and results are presented in Figure 6. Correlation is not as strong as with the dynamic viscosity. The complexity and non-equilibrium character of the SP test likely play a significant role in this comparison. Development of a more robust correlation was beyond the scope of this paper. Also note that sensitivity of polymer modified bitumen to $|G^*|/\sin\delta$ is much less than to SP.

A good correlation between penetration at 25 °C, 5 s/100 g, PEN (ASTM D5), and $|G^*|$ was found at testing time 0.4 s by others [2] [3]. For EMAAD, dynamic data were only available at 10 rad/s. With the help of Equation 10, a temperature at 10 rad/s test frequency, equivalent to 25 °C at test frequency of 0.4 rad/s (≈ 0.4 s testing time) was found as 43.16 °C[4]. Logarithm of ($|G^*|/\sin\delta$) at 43.16 °C was calculated and plotted versus penetration (Figure 7).

6.2 Correlation between Intermediate Temperature PG and Empirical Tests

ITPG measured on PAV bitumen according to AASHTO T315 was available only for EMAAD data. However, no empirical properties were measured on PAV bitumen in EMAAD. It is difficult to compare rheological properties between original and PAV bitumen since all bitumen may not age at the same rate. Nevertheless, a general trend was observed between ITPG and log(PEN) (Figure 8A). For EDCD dataset dynamic data were available for original and RTFO bitumen. A temperature where $|G^*|\cdot\sin\delta = 5000$ kPa was therefore interpolated from dynamic data at 10 rad/s available for original bitumen and plotted versus log(PEN) (Figure 8B). Correlation was poor and perhaps high test variability for multi laboratory data is a significant factor here.

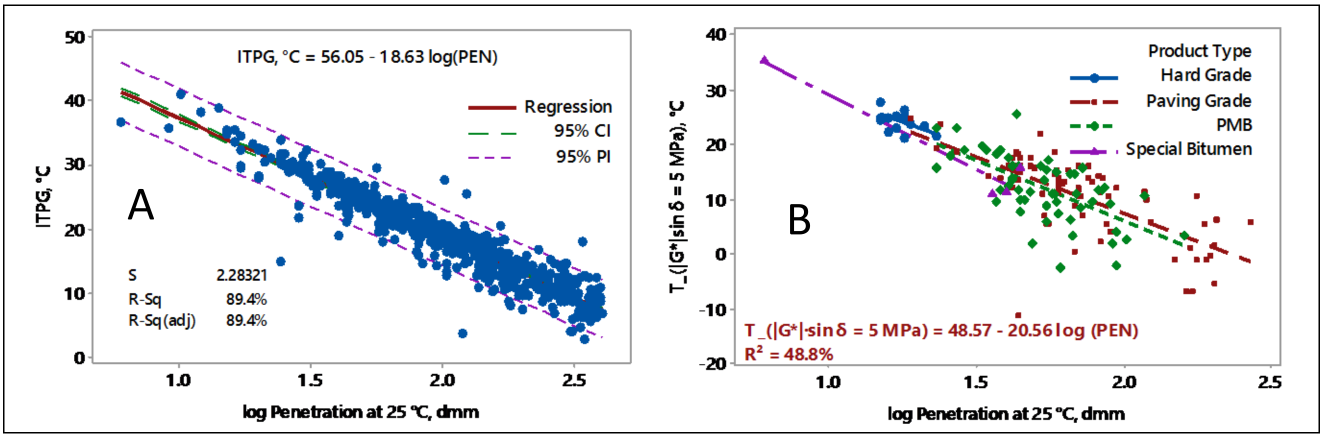

Figure 8: A - Correlation between ITPG and log(PEN) for 578 lab distilled bitumens from EMAAD and 8B - temperature at $\log G^* \cdot \sin\delta = 5$ MPa and log(PEN) for 158 bitumens from EDCD, regression equation displayed for Paving Grade only.

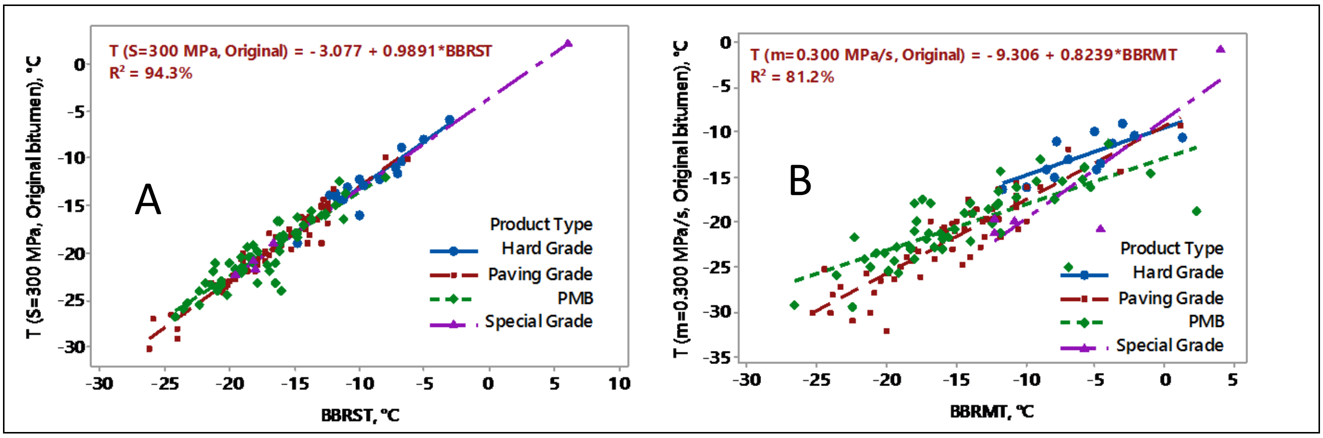

Figure 9: A - Correlation between temperature at $S = 300$ MPa, original bitumen vs PAV bitumen (BBRST) and 9B - between temperature at $m - value = 0.300$ MPa/s, original bitumen versus PAV bitumen (BBRMT) for 109 bitumen from EDCD, regression equation displayed for Paving Grade only.

6.3 Correlation between LTPG and Empirical Tests

Similar challenge as with the ITPG data was observed for BBRST and BBRMT correlations. No empirical properties were measured for PAV bitumen in either dataset. Interestingly, BBR data were available for both original and PAV bitumen in EDCD. Good correlation was observed for temperature at which $S = 300$ MPa measured on original versus

[4] This temperature depends on bitumen temperature susceptibility or $\log a_T(T - T_0)$ function. An average value for Eurobitume dataset was calculated here and also used for EMAAD dataset.

PAV bitumen. Poorer correlation was observed for temperatures at which $m - value = 0.300$ MPa/s (Figure 9). This is consistent with the fact that m-value is much more sensitive to oxidation [25] [26].
For the EDCD dataset, BBRST was found to be weakly correlated to Fraass breaking point ($R^2 = 62.1$ %, Paving grade, data not presented). A multi-linear regression model including Fraass breaking point, penetration at 25 °C and dynamic viscosity at 60 °C on RTFO bitumen yielded better correlation with $R^2 = 90.8\%$ for Paving grade, data not presented. BBRMT was weakly correlated to log penetration at 25 °C ($R^2 = 78.6\%$, Paving grade, data not presented). Similarly, somewhat better correlation was obtained in multi-linear regression using penetration at 25 °C (RTFO), kinematic viscosity at 135 °C and dynamic viscosity at 60 °C (RTFO) as variables ($R^2 = 80.5\%$, Paving grade, data not presented).

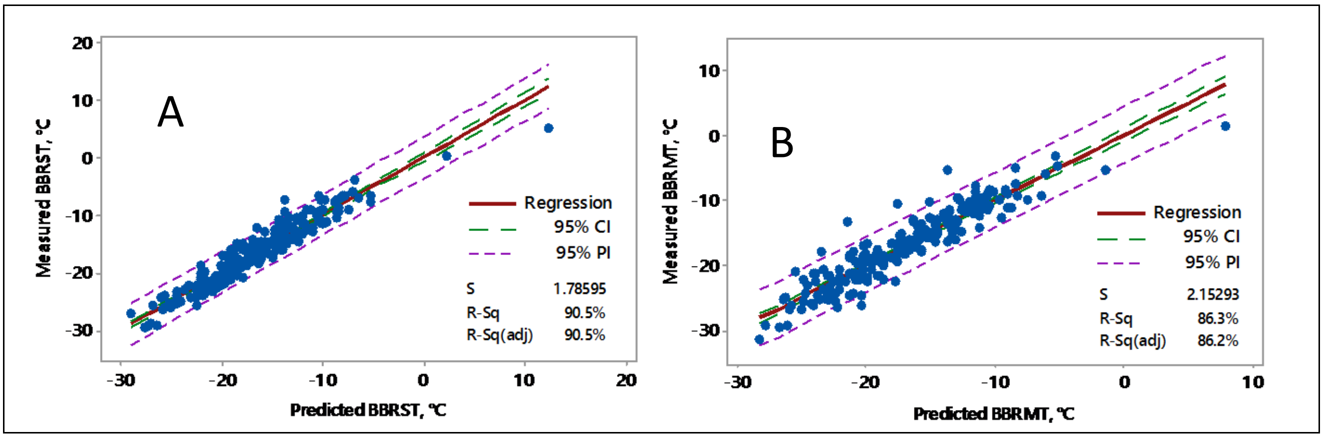

Figure 10: A - Multi-linear regression model prediction of BBRST and 10B - BBRMT, 204 lab distilled bitumen from EMAAD.

For the EMAAD dataset, BBRST was found to be weakly correlated to penetration at 10 °C ($R^2 = 83.3$ %, data not presented). A multi-linear regression model including penetration at 10 °C, mass loss in thin film oven test (TFOT) and viscosity ratio after TFOT yielded somewhat better correlation at $R^2 = 90.5\%$ (Figure 10A). BBRMT was weakly correlated to log penetration at 10 °C ($R^2 = 83.4\%$, data not presented). Similarly, somewhat better correlation was obtained in multi-linear regression using penetration at 10 °C, penetration at 25 °C (TFOT) and dynamic viscosity at 60 °C (TFOT) as variables ($R^2 = 86.3\%$, Figure 10B). Poor correlation is not surprising as m-value is related to relaxation properties which are not captured by empirical stiffness tests. Fraass breaking point perhaps can better capture differences in relaxation properties of the bitumen; however, varying bitumen strength and high testing variability of the Fraass breaking point disallowed any meaningful correlation between Fraass and BBRMT.

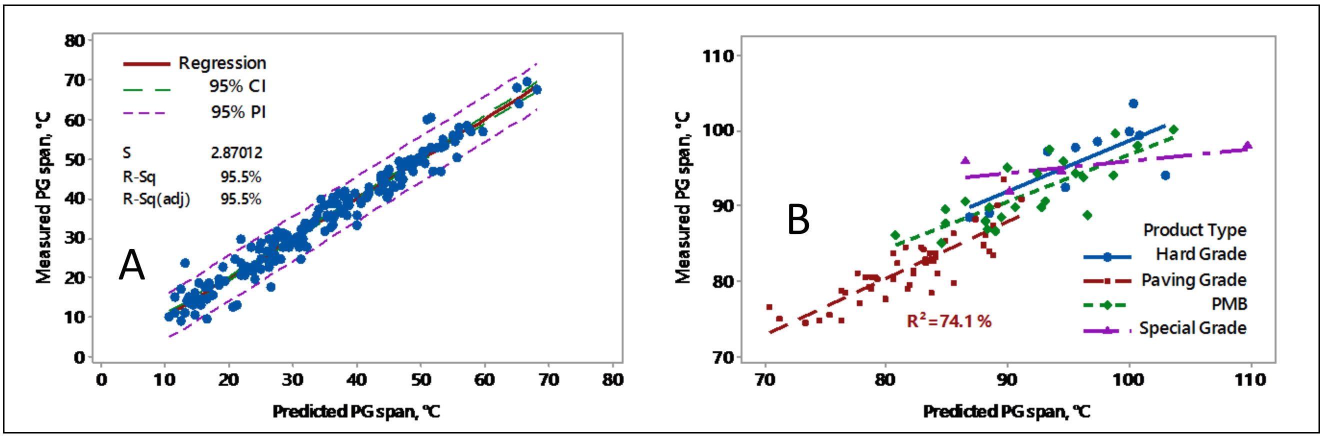

Figure 11: A - Multi-linear regression model prediction of PG span for 196 lab distilled bitumen from EMAAD, and 11B – for 109 bitumen from EDCD, R^2 displayed for Paving Grade only.

6.4 Temperature susceptibility

Temperature susceptibility is a very important parameter for bitumen. As discussed earlier, a span between the HTPG and LTPG can be used as an indicator of temperature susceptibility. In empirical specification, a slope of logarithm of penetration versus temperature is used as penetration index (PI) to do the same. In order to avoid measuring penetration at several temperatures Pfeiffer and van Doormaal [27] proposed, that for most bitumen the penetration at softening point can be assumed to be 800 dmm. This allowed for calculation of PI "ring and ball or R&B" from penetration measured at 25 °C and softening point. No correlation was found in either single or multi-variable models between PI

R&B and PG span (EDCD and EMAAD) or slope of $\log G^*/\sin\delta$ vs. T taken between 20 and 60 °C (EDCD data only).
For EMAAD samples, penetration data at 4, 10 and 25 °C were available. Slope and intercept of logarithm of penetration vs temperature were calculated and both used as predictors of PG span (R^2 = 95.5%, Figure 11). This indicates that penetration, if measured at multiple temperatures, can be a good indicator of temperature susceptibility. This is consistent with the observation that penetration is well correlated to $|G^*|$. To understand the inability of PI R&B to predict PG span, penetration at softening point temperature was extrapolated from logarithm of penetration versus temperature fits assuming linear behavior on log-linear plot. The variation of penetration at softening point was too large to assume it equal to 800 dmm even for straight run bitumen. A mean of 1049 dmm with a standard deviation of 525 dmm was observed. This was already noted by van der Poel [2]. This verification was not possible to perform with the EDCD dataset as only penetration at 25 °C was available. A multi-linear regression with dynamic viscosity at 60 °C and penetration at 25 °C (RTFO) predicted the PG span with R^2 = 74.1% (Figure 11).

6.5 Aging susceptibility

Oxidative aging and evaporation are important parameters in specifying paving bitumen. There are two fundamentally different aging stages. Neglecting a minor aging in storage tanks, the first significant aging (short term aging) takes place in the asphalt plant. During aggregate coating, a thin layer of bitumen is exposed to heat and air for a relatively short time. This results in an increase in modulus which approximately doubles. The second stage of aging refers to long term asphalt exposure to environment. The aging process is slow and difficult to describe due to complexity of bitumen chemistry, fluctuating temperature, oxygen diffusivity, asphalt porosity, moisture, UV light, additives, etc. During the second stage, the stiffness of bitumen increases further, while its ability to relax stresses decreases. Embrittlement or loss of ability to relax stresses imposed by traffic, subgrade movement, freeze-thaw cycles, temperature changes, etc. leads to cracking.

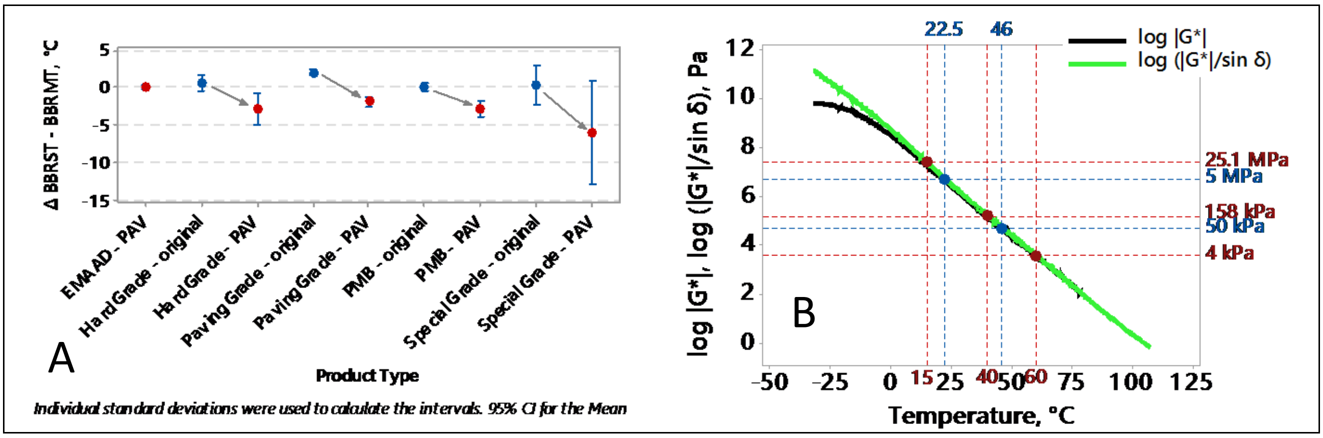

Figure 12: A - Average of delta (BBRST – BBRMT) for original (blue) and PAV (red) bitumen for EMAAD and EDCD samples, arrows indicate change after PAV. 12B – Comparison between isothermal (red reference lines) and equiviscous (blue reference lines) testing, interpolated results indicated, example for 148 dmm PEN bitumen.

The purpose of the specifications is to simulate these two aging stages and evaluate the bitumen rheology after each aging step. Short term aging step is performed in RTFO for both EN 12591 and Superpave. RTFO test somewhat realistically mimics aging in an asphalt plant as it is done at approximately the same temperature (163 °C), although the film thickness and aging time differs. The long term aging is not currently a mandated part of the CEN specification. In Superpave, the long term aging is PAV test. Although it was shown to mimic well 7-10 years of asphalt aging, it was also shown to fail to predict aging in some bitumen [20] [22]. Reasons can be related to accelerated conditions as temperature, pressure and film thickness are significantly higher than in the field.
As previously mentioned, oxidation predominantly impacts the m-value. In Figure 12A, the difference between BBRST and BBRMT is presented for original and PAV bitumen. The delta between BBRST and BBRMT increases in absolute values after PAV. This is because relaxation properties are predominantly impacted by oxidation [25]. For a typical straight run bitumen, the delta between BBRST and BBRMT (PAV bitumen) is approximately zero (Figure 12A). On average the EDCD data after PAV show that BBRMT is larger than BBRST (negative delta in Figure 12A). This can, for example, indicate air blown or other types of modified bitumens.

7. CEN SPECIFICATION PROPOSAL

The latest CEN proposal [1] continues with the use of empirical tests to specify paving grade bitumen. The objective here is to review the proposal for rheological data collection as presented in Annex C of CEN proposal. The supplier declared values are required to be available and updated after a major change in product quality or manufacturing scheme. The requirements are specified in Table 2. The low temperature properties will be measured in BBR after

RTFO + PAV aging. The requirement is almost identical to current Superpave specification. EN 14771 does not provide standard test temperatures or the required temperature increments. Ideally, test results at pass and fail temperatures should be available. As logarithm of stiffness versus temperature cannot be considered linear over the larger span of temperatures, the test temperature intervals should be specified, e.g. as X, X+Y, X+2Y, etc. Also, the method of interpolation should be clarified to avoid discrepancies in BBRST and BBRMT determination. Nevertheless, BBR test and the limits were shown to be relevant to pavement performance, and thus these data can be considered bitumen performance data.

Table 2: Proposal for Supplier Declared Values Collection in prEN 12591, Annex C [1].

Property		Test method	Unit
Low temperature behavior after long term aging	Temperature at which S = 300 MPa	EN 12607-1 + EN 14769 + EN 14771	°C
	Temperature at which m = 0.300		°C
G^* and δ @ 1.59 Hz (10 rad/s) after short term aging	@ 15 °C	EN 12607-1 + EN 14770	MPa
	@ 40 °C or @ 60 °C		°

The situation is somewhat different for the high temperature test. Here, the Superpave philosophy of equiviscous testing is not followed. Isothermal tests are prescribed at 15 and 40 or 60 °C. The challenge is that for a variety of bitumen grades, a variety of testing conditions would have to be used. First, appropriate measurement geometry would have to be selected (e.g. like in Table 1). Next, strain sweeps would have to be performed to find an appropriate test strain at 10 rad/s testing frequency. The strain threshold should be high enough to maximize the signal-to-noise ratio without violating the conditions of linear viscoelasticity. The equiviscous testing as it is done in Superpave or suggested by Eckmann [28] allows for using the same instrument setup (test geometry, test strain) for any bitumen as they would have approximately the same stiffness at the grade test temperature. One additional comment is that EN 14770 differs significantly from AASHTO T315, mainly in sample preparation. Since EN 14770 does not list any precision limits, it is important to verify that these test method differences do not contribute to test variability.
Mastercurves show that comparable results can be obtained from any two measurements performed at any two different temperatures as long as the logarithm of the property changes linearly with temperature (Figure 12B). Therefore, nearly identical results can be obtained from tests performed at 15, 40 and. 60 °C [1], or at 50 kPa and 5 MPa [28]. The difference can perhaps be in precision as $|G^*|/\sin\delta(T)$ function may not be perfectly linear on log-linear scale for some bitumen; however, for EDCD data $\log|G^*|/\sin\delta$ was well correlated to temperature between 20 and 60 °C for all paving, hard, polymer modified and special bitumens – lowest R^2 = 97.7 %, average R^2 = 99.4%. Interpolation of equiviscous temperature from $|G^*|(T)$ function alone is not recommended because the assumption of $\sin\delta \cong 1$ is invalid at intermediate test temperatures (Figure 12B) and $\log|G^*|(T)$ is no longer linear. At this point, it is also unclear how the EN testing proposal develops into performance specification to relate the high and intermediate isothermal test results to pavement temperature and traffic load.

8. CONCLUSIONS

It is shown on hundreds of paving grade bitumens that empirical testing can provide comparable information on bitumen rheology as modern rheological tests such as DSR and BBR. Therefore, adoption of rheological tests cannot by itself be considered a performance specification and it would not provide any added value as such. The advantage in using rheology tests, however, is that these tests provide data in standard engineering properties such as modulus. These properties can subsequently be used in asphalt and road design or in performance predictive models. At this point the current CEN proposal for bitumen rheological testing seems to be very close to the one adopted or considered elsewhere – North America, China, Russia, and Middle East. The simplest approach may be simply adopting Superpave DSR tests and not to invest time in re-developing virtually the same testing methods.
Despite the proposed tests, the key to any future European performance specification is to properly set the stiffness and temperature limits and to field validate these limits to provide satisfactory performance of a real road. To avoid extensive and costly field validation, the Superpave grading system should be considered; however, the temperature ranges for potential performance grades would have to suit the local climate and may differ from those used in North America.

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