Relationships between solubility and chromatographically defined bitumen fractions and physical properties

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ABSTRACT

An understanding of how bitumen chemical composition influences mechanical behavior is critical to addressing a number of practical issues concerning bitumen utilization. Using simple chemical tests to assess bitumen quality is of practical value to the purchaser, but other applications exist as well. Blending to achieve material design objectives is obviously of huge industrial and commercial value, as is designing and selecting better additives. This is also key to understanding physical changes related to bitumen oxidation and predicting performance.

Western Research Institute (WRI), in partnership with Eurovia, has examined several paving grade bitumens using an automated asphaltene solubility fractionation method developed by WRI under contract with the United States Federal Highway Administration (FHWA) that is an expansion of the traditional SARA method (SAR-AD™). The results of these chemical characterization studies were then correlated to a wide range of bitumen properties (Penetration, Ring and Ball softening point, Dynamic Shear Rheometry, and others) using WRI's multivariable significance search algorithms (ExpliFit™). In general, most properties can be explained with a high coefficient of correlation by considering a balance between mobile and relatively immobile constituents, as well as interactions induced by polarity and polyaromaticity. This paper focuses on the micro-structural explanation of the significant parameters affecting the storage shear modulus over the range of temperatures investigated. The correlation results suggest that as temperature changes, the amount of the mobile fractions in the bitumen dominates low temperature behavior, while at high temperatures, multiple fractions must be considered.

Keywords: Ageing, Asphalt, Chemical properties, Compatibility, Mechanical Properties

1. INTRODUCTION

In recent years, a significant evolution on the European market has been observed. European refining, French in particular, is currently in a phase of rationalization and search for maximum flexibility in crude supplies.

The European standard EN 12591 appears as insufficient to ensure satisfactory performance of the finished products, particularly in case of specialty products such as high modulus asphalt regarding stiffness modulus and fatigue resistance, polymer modified bitumen, and bitumen emulsions with respect to settling tendency and viscosity.

The search and validation of performance-related bituminous binder properties continues to be a key issue for the paving industry in Europe, as well as in the US and the rest of the world. With the Superpave system implementation in the US, important progress has been achieved and is still on-going. In Europe, the development of 2nd generation product standards appears to be more necessary than ever.

In this context, Eurovia and the Western Research Institute (WRI) launched a research program, in 2013, to search for correlations between bitumen properties and the performance of the finished asphalt product.

In the short term, the identification of robust correlations between bitumen composition and mechanical properties has obvious practical value in material evaluation and blending to meet given specifications. The information obtained from these correlations can also be applied to testing and improving the understanding of the fundamental concepts of how bitumen composition gives rise to the observed physical properties. Bitumen has long been considered to behave similarly to colloidal systems, and the idea of a colloid-like microstructure has existed at least as long as the turn of the century when asphaltenes were first identified [1]. Since that time, a wide range of conceptual models of the bitumen microstructure have been proposed, at various levels of detail. Although not comprehensive, several references are provided to illustrate some of the work done in this area historically [2-37]. Nearly all of these propose that bitumen is not homogenous at some scale above molecular dimensions. It is also generally conceded there exists some relationship between solubility defined fractions and the resulting micro-structure. This study is an effort to quantify these relationships. The micro-structure, in turn, is primarily responsible for the mechanical properties of interest to the design of a number of bitumen containing products. This work correlating solubility defined fractions to rheological properties suggests, as expected, that the important fractions defining the mechanical behavior change with temperature. At low temperatures, much of the material exists as a relative immobile glass or associated gel-like material, with the content of saturates, the last fraction to solidify into a glass, being the most significant one in defining the mechanical behavior. As the material warms, the portioning of mobile and immobile phases, along with a change from gel-like to sol-like behavior changes according to temperature dependant solubility characteristics. Consequently, empirical correlations of solubility defined fractions with mechanical properties will not show a consistent set of fractions primarily defining the mechanical properties. At low temperatures the most mobile fractions are the most significant where gel-behavior is observed. At high temperatures where sol behavior is observed, multiple fractions are required to define the system, with the suspension defining fraction, the asphaltenes, being the most significant. In rheological terms, low phase angle can be described with a few parameters, while higher phase angle properties depend more strongly on a range of solubility fractions.

2. RESEARCH PROGRAM

This research program was launched by Eurovia in collaboration with the Western Research Institute (Wyoming/ USA). 8 bitumens (all unmodified) were selected: B1 to B8. With these bitumens, 12 asphalts were manufactured (8 with a diorite and 4 with limestone aggregates). For each asphalt, the bitumen content was 4.9 %. Table 1 presents the main characteristics of these bitumens and the different asphalt designs.

Table 1: Bitumen characteristics and asphalt designs

	B1	B2	B3	B4	B5	B6	B7	B8
Penetration (1/10mm): NR EN 1426	40	37	40	22	26	28	55	57
Ring and Ball Temperature (°C) : NF EN 1427	53.4	53	52	59	57.2	61	49	49.2
Superpave Performance Grading (PG)	70-22	70-16	70-16	76-16	76-16	76-10	64-22	64-16
HMA with diorite	X	X	X	X	X	X	X	X
HMA with limestone	X		X	X			X	

The analysis program for the bitumens (neat, after RTFO, recovered, after RTFO + PAV) :

1- Chemical analysis : infrared, SAR-AD : Saturates, Aromatics, Resins and Asphaltene Determinator [38], SEC : Size exclusion chromatography, DSC : Differential Scanning Calorimetry (to assess glass transition, wax content)

2- SHRP tests: Bending Beam Rheometer (BBR), DSR: to determine master curves, crossover, R-parameter… [43],[44]

3- Advanced rheological tests (LAS tests…) [42]

4- Asphalt Binder Cracking Device (ABCD) test [39],

5- Conventional European tests: penetration, ring and ball temperature, Fraass breaking point,…

This paragraph presents the overall research program launched in 2013, but this article will present only some chemometric results [40], the correlation between bitumen rheological properties, specifically the storage modulus, over a range of temperature (10° to 60°C). Other articles will be published in the future to present more results in detail.

3. BITUMEN SELECTION

The key point in a chemometric correlation is based on determination of the quality of bitumen selection. The first step of the program before launching the analyses was to verify that the chemical composition and rheological performances of these bitumens were significantly different.

3.1 Chemical composition

The SAR-AD [38] test is a novel approach, developed by the Western Research Institute, which combines the Automated Asphaltene Determinator (AD) separation with an automated SAR (saturates, aromatics and resins) separation to provide a fully integrated rapid automated SARA (saturated, aromatics, resins and asphaltenes) separation using milligram sample quantities. The combined SAR/AD separation utilizes high performance liquid chromatography (HPLC) equipment with multiple columns and solvent switching valves to conduct the highly complex automated separation. Figure 1 presents the chemical compositions of the 8 bitumens. The sample set represents considerable variation in the solubility defined fractions.

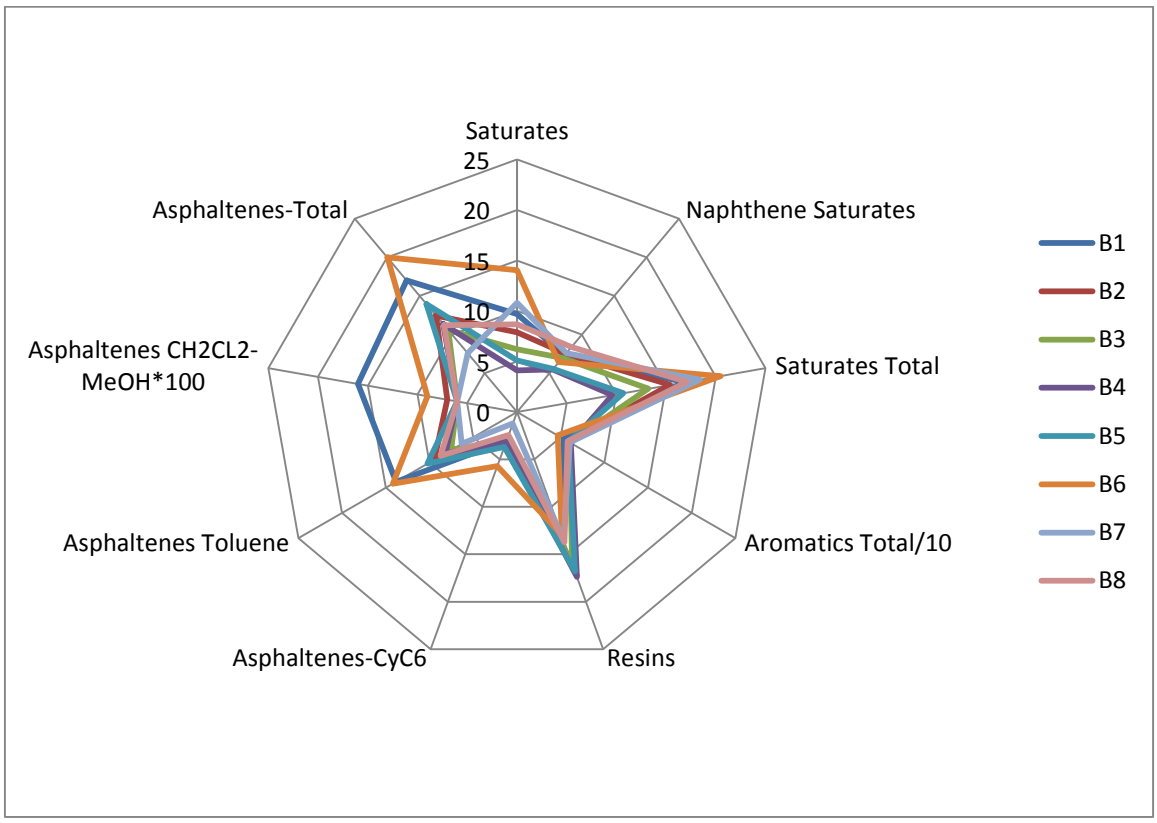

Figure 1: Bitumen composition B1 to B8

3.2 Rheological properties

The bitumen samples selected show a wide range of rheological behavior. Figure 2 illustrates the variation in the complex modulus isotherms at 15 °C from 1 to 30 Hz.

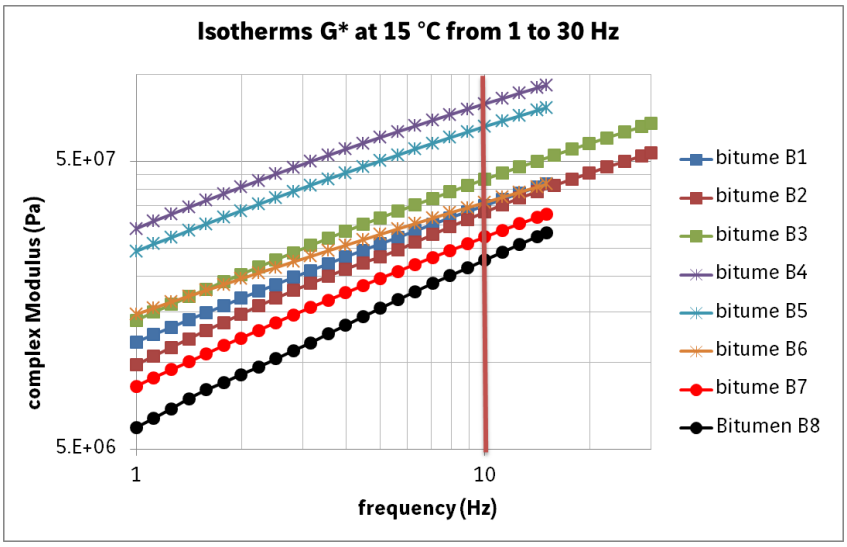

Figure 2: Isotherms G* at 15°C from 1 to 30 Hz

4. CHEMOMETRIC CORRELATIONS

4.1 Explifit® Software

Explifit ® [40] is a software program designed to investigate relationships between independent and dependent variables using standard multivariable linear regression algorithms adapted for under determined problems. An under determined problem is a situation where the count of possibly significant independent variables exceeds the number of observations. For example, measuring 24 chemical properties to correlate with 8 bitumen's is under determined and not tractable with traditional methods. This software was developed at Western Research Institute.

The dependent variables data are all analyses performed on bitumen or asphalt.

The independent variables data are typically measured to predict the dependent variables data. In this research program, the independent data are: infrared (IR) spectra measurements, SAR-AD compositions and distribution of the particle sizes by SEC.

Example: If we try to correlate bitumen IR measurements with the bitumen penetration, in a first step, the software will find out which of the wavenumbers are significant when combined additively with other significant wavenumbers. This step will enable a reduction in the number of relevant wavenumbers. In a second step, the software will propose an equation such as:

Bitumen penetration (1/10 mm) = $c_0 + c_1 [Abs_1] + c_2 [Abs_2] + + \ldots . c_n [Abs_n]$

c_1= Fit coefficient 1, Abs_1 = Absorbance to the wavenumber 1 …

In addition to the actual measurements, a precision file for each independent and dependent variables data set must also be prepared. The program requires these files to create the data clouds needed for computation.

4.2 Results

The research program carried out at Eurovia consists of an enormous collection of measurements for the bitumen samples selected. Only a subset of that data will be studied in detail here, addressing the temperature dependant role of solubility fractions measured by SAR-AD. Seven solubility fractions are measured using 2 detectors, Evaporative Light Scattering Detector (ELSD) to measure mass fractions and a 500 nm UV detector to study polycondensed aromatics. The following ELSD measurements are used in this discussion (Table 2):

Table 2: SAR-AD Measurements correlated with rheology.

Saturates
Naphthene saturates (ring structures)
Aromatics
Resins
Cyclohexane soluble asphaltenes (least polar)
Toluene soluble asphaltenes (moderately polar)
Methylene chloride-methanol soluble asphaltenes (highly polar)
Total asphaltenes

To find the significant bitumen fractions responsible for the observed rheological properties, and the effect of temperature on their relative importance, multivariable correlations of the ELSD measurements were fit against the complex, storage and loss shear moduli measured at 10°C, 15°C, 20°C, 30°C, 40°C, 50°C and 60°C using a dynamic

shear rheometer. Due to space limitations, only the Shear storage modulus (G') at 1Hz frequency will be described in detail here. Similar relationships exist for the complex (G*) and loss (G") moduli and for measurements at other frequencies.

Initially, 4-parameter fits at each temperature range were completed using the eight ELSD measurements. The qualities of the fits are shown in Table 3:

Table 3: Initial ELS detector fits for G'

Temperature °C	Pearson's r^2
10	0.870
15	0.864
20	0.879
30	0.898
40	0.947
50	0.976
60	0.986

The significance of the parameters compared using the F test, which is a ratio of the fit residuals without the parameter of interest divided by the residuals with the parameter of interest. Large F test values are more significant than small ones. An examination of the significance of the parameters appearing in 4-parameter fits suggest that 3 parameters may be sufficient for a robust correlation across the temperature range studied here. (note: F-tests for negative fit coefficients are shown negative)

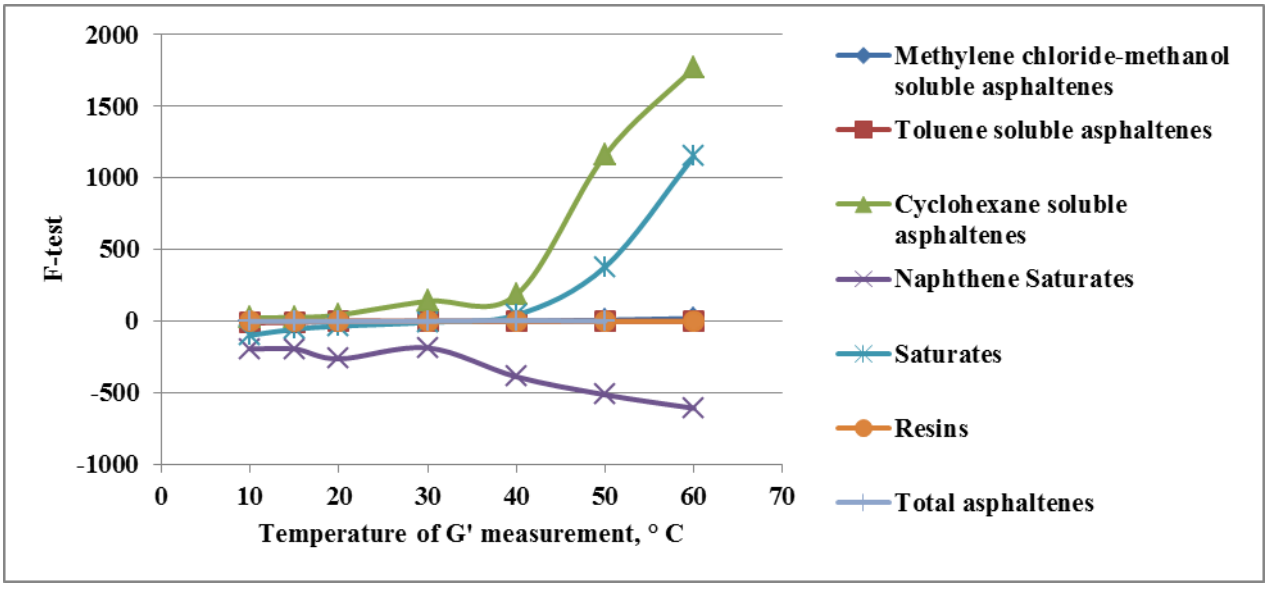

Figure 4 : F-test significance from ELSD fits

Based upon the results depicted in figure 4, the regressions were repeated using only cyclohexane-soluble asphaltenes, saturates, and naphthene saturates values. The fits shown in table 4 are similar in quality to those obtained using the entire ELSD measurement list to generate a 4-parameter model.

Table 4: Correlation fit quality of 4 parameter and 3 consistent parameter models.

Temperature °C	4 parameter Pearson's r^2	3 consistent parameter Pearson's r^2
10	0.870	0.871
15	0.864	0.849
20	0.879	0.843
30	0.898	0.894
40	0.947	0.951
50	0.976	0.973
60	0.986	0.984

Now that a simple consistent set of independent SAR-AD variables is established for all seven correlations at each temperature, it is possible to determine the temperature dependency of the coefficients and produce one equation that describes G' in SAR-AD compositional terms over the entire temperature range. The same approach can be used with the complex and loss moduli. Newtonian materials follow an Arrhenius form,

$$\eta = Ae^{\frac{E_a}{RT}}$$

η is viscosity

E_a is the activation energy

R is the universal gas constant

T is the absolute temperature

A plot of $\mathrm{Ln}(|a_n|)$ vs $1/T$ should yield a straight line with a slope of E_a / R and an intercept of $Ln(A)$.

The constants are nearly Arrhenius, although a slight curvature can be seen in the plots, not unlike the similar master curve shift function shapes observed for bitumens, suggesting that a more sophisticated temperature function such as WLF may produce a higher precision calibration.

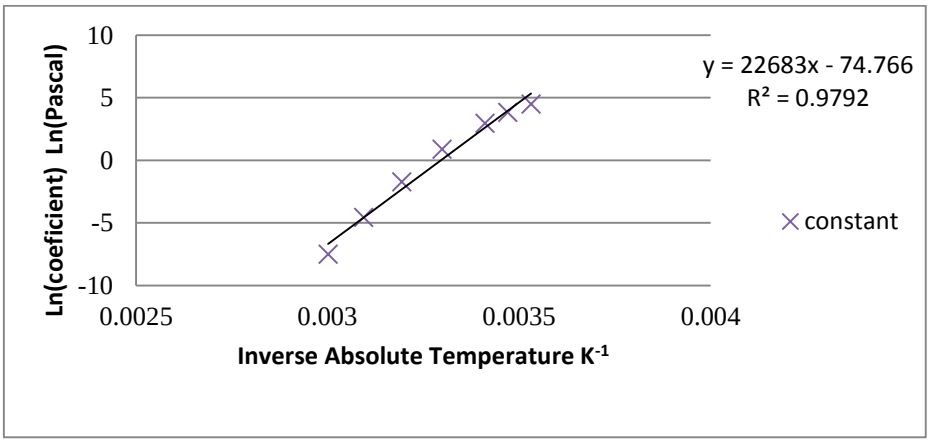

Figure 5: Intercept (*Ln(A)*) Arrhenius plot

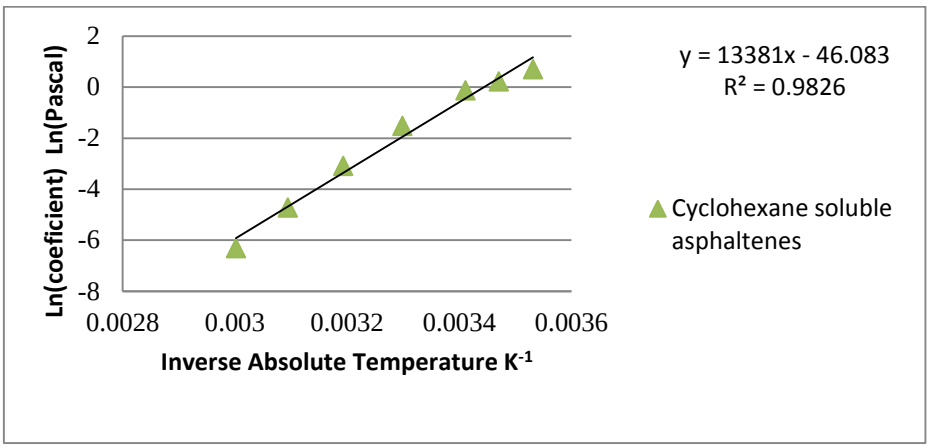

Figure 6 : Cyclohexane soluble asphaltenes fit constant Arrhenius plot

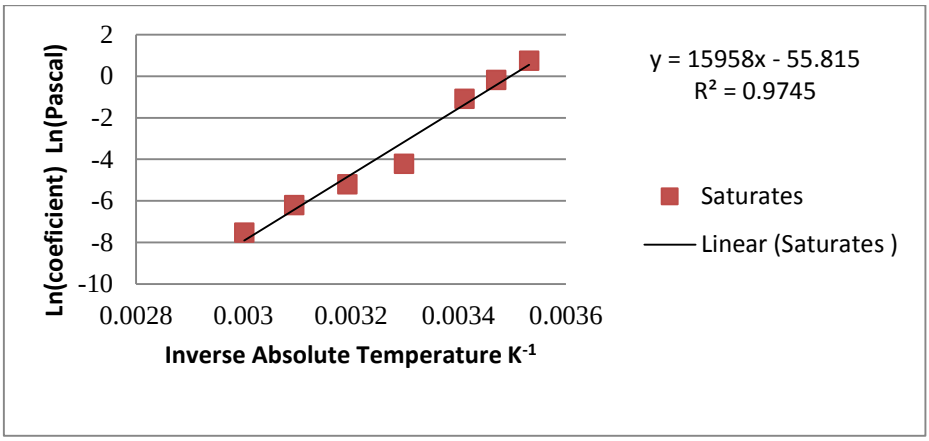

Figure 7 : Saturates fit constant Arrhenius plot

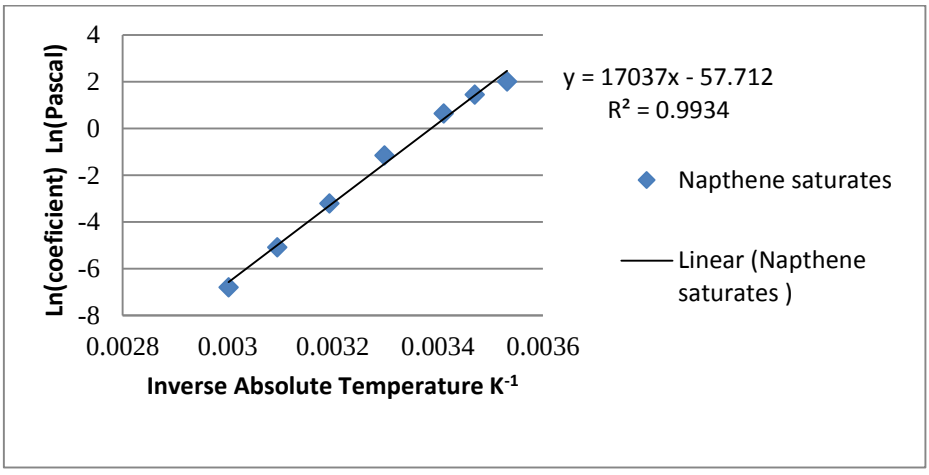

Figure 8: Naphthene Saturates fit constant Arrhenius plot

The resulting correlation over the entire temperature range is:

$$G'(T)_{1Hz} = A_0 e^{\left(\frac{E_a}{R}\right)_0 \frac{1}{T}} + A_1 e^{\left(\frac{E_a}{R}\right)_1 \frac{1}{T}} (\%\ \text{Cyclohexane Asphaltenes})$$
$$+ A_2 e^{\left(\frac{E_a}{R}\right)_2 \frac{1}{T}} (\%\ \text{Saturates}) - A_3 e^{\left(\frac{E_a}{R}\right)_3 \frac{1}{T}} (\%\ \text{Naphthene Saturates})$$

where the fit constants are listed in table 5.

Table 5: Temperature SAR-AD function for G' fit constants

A_0	3.41E-33	$(E_a/R)_0$	22683
A_1	9.72E-21	$(E_a/R)_1$	13381
A_2	5.78E-25	$(E_a/R)_2$	15958
A_3	8.65E-26	$(E_a/R)_3$	17037

4.3 Discussion

The results of the investigation of the SAR-AD fractions needed to estimate the storage modulus (G') over the temperature range examined suggest that at higher temperatures where a sol-like structure is presumed to exist, several fractions exist in the mobile phase and are significant to the estimate. However, at lower temperatures where many fractions exist in an associated gel-like phase, very good estimates of the storage modulus can be obtained using the mobile naphthene saturate fraction alone (see Figure 4 where the naphthene saturates is the only fraction with a significant F-Test value at low temperature). The data and resulting correlations also suggest, that for the eight bitumens studied, the associated materials have similar mechanical characteristics, and since this variance is small, the variance in the storage modulus can be explained at all temperatures with a knowledge of composition of the mobile fraction This is consistent with differential scanning calorimetry results that show the mobile fraction anchors the low end of the glass transition region regardless of asphaltene content [41].

5. CONCLUSION

The collaboration work by Eurovia/Western Research Institute enabled the evaluation of applying chemometric correlations to predict bitumen or asphalt properties from the infrared spectrum or /and the SAR-AD composition. Using multivariable techniques, quite good correlations are often obtained with coefficient-squared values often 0.9 or above.

The correlation of chemical composition with rheological properties generally requires a knowledge of several components in the bitumen, as these components interact to define the structure and ultimately, the mechanical properties. Understanding the relationships between the solubility defined fractions can lead better quality control methods and bitumen composition adjustments to produced desired mechanical properties.

REFERENCES

[1] *Mémoires de J.-B. Boussingault*, Boussingault, Jean-Baptiste (1892–1903), Paris: impr. de Chamerot et Renouard), 1892

[2] Direct observation of the asphaltene structure in paving-grade bitumen using confocal laser-scanning microscopy, Bearsley, S., Forbes, A. and Haverkamp, R. G., *Journal of Microscopy*, Vol. 215, Issue 2, pp. 149-155, 2004

[3] The glass transition temperature and the mechanical properties of asphalt, Breen, J. J. and Stephens, J. E. , *Proceedings of The Annual Conference of the Canadian Technical Asphalt Association*, Report No. JHR-67-16, 1967

[4] A molecular interpretation of the toughness of glassy polymers, Brown, H. R., *Macromolecules*, Vol. 24, pp. 2752-2756, 1991

[5] A new interpretation of time dependent physical hardening in asphalt based on DSC and optical thermoanalysis, Claudy, P., Létoffé, J. M., Rondelez, F., Germanaud, L., King, G. N. and Planche, J. P., *ACS Symposium on Chemistry and Characterisation of Asphalts, Washington, D.C.* pp. 1408-1426, 1992

[6] Electron microscopic investigations on the nature of petroleum asphaltics, Dickie, J. P., Haller, M. N. and Yen, T. F., *Journal of Colloid and Interface Science*, Vol. 29, pp. 475-484, (1969)

[7] A kinetic investigation of association in asphalt, Ensley, E. K., *Journal of Colloid and Interface Science*, Vol. 53, No. 3, pp. 452-460, 1975

[8] *Flory-Huggins Theory*, Franzen, S., North Carolina State University: Department of Chemistry http://chsfpc5.chem.ncsu.edu/~franzen/CH795I/lectures/fht/sld001.htm, Date accessed 7 September 2009, 2004

[9] Asphaltenic aggregates are polydisperse oblate cylinders, Gawrys, K. L. and Kilpatrick, P. K., *Journal of Colloid and Interface Science*, Vol. 288, pp. 325-334, 2005
[10] Influence of composition of paving asphalts on viscosity, viscosity-temperature susceptibility, and durability, Griffen, R. L., Simpson, W. C. and Miles, T. K., *Journal of Chemistry and Engineering Data*, Vol. 4, pp. 349-354, 1959
[11] *Hansen solubility parameters: a user's handbook*, Hansen, C. M. Boca Raton: CRC Press, pp. 1-7, (2007)
[12] The asphalt model: results of the SHRP asphalt research program, Jones, D. R. and Kennedy, T. W., *Proceedings of the Conference, Strategic Highways Research Program and Traffic Safety on Two Continents, Part Four*, Gothenberg, 1991
[13] Models of an asphaltene aggregate and a micelle of the petroleum colloid system, Jovanović, . A., *Chemistry and Industry*, Vol. 54, No. 6, pp. 270-275, 2000
[14] Nature of Asphaltic Substances, Katz, D. L. and Beu, K. E., *Industrial and Engineering Chemistry*, Vol. 37, No. 2, pp.195-200, 1945
[15] Identification of chemical species and molecular organisation of bitumens, Le Guern, M., Chailleux, E., Farcas, F., Dreessen, S., Planche, J.-P. and Débarre, D., *Proceedings of the RILEM Technical Committee TC 231 workshop on micro- and nano-characterisation and modelling of bituminous materials*, Duebendorf, 2011
[16] A structure-related model to describe asphalt linear viscoelasticity, Lesueur, D., Gerard, J.-F., Claudy, P., Letoffe, J.-M., Planche, J.-P. and Martin, D., *The Journal of Rheology*, Vol. 40, pp. 813-836, 1996
[17] Colloid chemistry of asphalts, Mack, C. J., *Journal of the Institute of Petroleum Technologists*, Vol. 36, pp. 2901-2914, 1932
[18] Modelling of asphaltene and other heavy organic deposits, Mansoori, G. A., *Journal of Petroleum Science and Engineering*, Vol. 17, pp. 101-111, 1997
[19] Bitumen microstructure by modulated differential scanning calorimetry, Masson, J.-F. and Polomark, G.M., *Thermochimica Acta*, Vol. 374, No. 2, pp. 105-114, 2001
[20] The colloidal structure of crude oil and the structure of oil reservoirs, Mullins, O. C., Betancourt, S. S., Cribbs, M. E., Dubost, F. X., Creek, J. L., Andrews, A. B. and Venkataramanan, L., *Energy and Fuels*, Vol. 21, pp. 2785-2794, 2007
[21] The constitution of asphalt, Nellensteyn, F. J., *Journal of the Institute of Petroleum Technology*, Vol. 10, pp. 311-325, 1924
[22] The effect of asphaltenes, naphthenic acids and polymeric inhibitors on the pour point of paraffin solids, Oliveira, G. E., Mansur, C. R. E., Lucas, E. F., González, G. and de Souza, W. F., *Journal of Dispersion Science and Technology*, Vol. 28, pp. 349-356, 2007
[23] SANS study of asphaltene aggregation, Overfield, R. E., Sheu, E. Y., Sinha, S. K. and Liang, K. S., *Fuel Science and Technology International*, Vol. 7, Issue 5-6, pp. 611-624, 1989
[24] Asphalt solidification theory, Pauli, T., Beemer, A. and Miller, J., *Proceedings of the 43rd Peterson Asphalt Research Conference*, Laramie, 2005
[24] Chemo-mechanics of bituminous materials, Pauli, T., Grimes, W., Boysen, R. and Kringos, N., *Proceedings of the RILEM Technical Committee TC 231 workshop on micro- and nano-characterisation and modelling of bituminous materials*, Duebendorf, 2011
[25] Chemical composition of asphalt as related to asphalt durability, Peterson, J. C. In T. F. Yen and G. V. Chilingar (Eds.), *Asphaltenes and asphalts*. London: Elsevier Science B.V., pp. 363-399, 2000
[26] Asphaltic bitumen as colloid system, Pfeiffer, J.P.H. and Saal, R.N.J., *The Journal of Physical Chemistry*, Vol. 44, No. 2, pp. 139-149, 1939
[27] Asphaltene macrostructure by small angle neutron scattering, Ravey, J. C., Ducouret, G. and Espinat, D., *Fuel*, Volume 67, pp. 1560-1567, 1988
[28] The structure of asphaltenes in bitumen, Redelius, P., *International Journal of Road Materials and Pavement Design*, Special Issue: EATA 2006, pp. 143-162, 2006
[29] Hansen solubility parameters of asphalt, bitumen and crude oils, Redelius, P., *Hansen solubility parameters: a user's handbook*. Boca Raton: CRC Press, pp. 151-158, 2007
[30] *Relation between bitumen chemistry and performance*, Redelius, P., *Proceedings of the RILEM Technical Committee TC 231 workshop on micro- and nano-characterisation and modelling of bituminous materials*, Duebendorf, 2011
[31] Correlation between bitumen polarity and rheology, Redelius, P. and Soenen, H., *International Journal of Road Materials and Pavement Design*, Vol. 6, No. 3, pp. 385-405, 2005
[32] *Chemical properties of asphalts and their relationship to pavement performance*, Robertson, R. E., Washington, D.C.: Western Research Institute, Strategic Highways Research Program, National Research Council, 1991
[33] Development of a performance related chemical model of petroleum asphalt for SHRP, Robertson, R. E., Branthaver, J. F. and Peterson, J. C., *American Chemical Society Division of Fuel Chemistry Preprints*, Vol. 37, Issue 3, pp. 1272-1278, 1992
[34] Relationship of asphalt properties to chemical constitution, Simpson, W. C., Griffin, R. L. and Miles, T. K., *Journal of Chemical and Engineering Data*, Vol. 6, No. 3, pp. 426-429, 1961
[35] Microstructure-based identification of bitumen performance, Stangl, K., Jäger, A. and Lackner, R., *International Journal of Road Materials and Pavement Design*, Vol. 5, pp. 111-142, 2004

[36] Observation of bitumen microstructure changes using scanning electron microscopy, Stulirova, J. and Pospisil, K., *International Journal of Road Materials and Pavement Design*, Vol. 9, No. 4, pp 745-754, 2008
[37] The colloidal nature of asphalt as shown by its flow properties, Traxler, R. N. and Coombs, C.E., *The Journal of Physical Chemistry*, Vol. 44, pp. 149-165, 1935
[38] Automated HPLC SAR-AD Separation, Fundamental properties of asphalts and modified asphalts product, J. Schabron, R. Boysen, March 2015
[39] Asphalt Binder cracking device to reduce low temperature Asphalt pavement cracking: Final report, Federal Highway administration, July 2010
[40] Chemo- mechanical software, Fundamental properties of asphalts and modified asphalts product, R. Glaser, A. Beemer, T.F. Turner, March 2015
[41] DSC Studies of Asphalts and Asphalt Components, Turner T.F. and Branthaver, J.F., *Asphalt Science and Technology*. Usmani A.M. ed., New York: Marcel Dekker Inc., pp. 59-101, 1997.
[42] Estimating Damage Tolerance of Asphalt Binders Using the Linear Amplitude Sweep, AASHTO TP 101-12-UL, Estimating Damage Tolerance of Asphalt Binders Using the Linear Amplitude Sweep), available on the MARC website: http://uwmarc.wisc.edu/linear-amplitude-sweep, 2011
[43] Interpretation of Dynamic Mechanical Test Data for Paving Grade Asphalt, Christensen, D.W. and Anderson, D.A. , Journal of the Association of Asphalt Paving Technologists, 61, 67-116, 1992
[44] NF EN 14771: Determination of the flexural creep stiffness: Bending beam rheometer (BBR)