RHEOLOGICAL ANALYSIS OF CRUMB RUBBER MODIFIED BINDER

G A J MTURI and J O'CONNELL*

CSIR Built Environment, Transport Infrastructure Engineering, Pretoria 0001, South Africa
Tel: +27 12 841 2234; E-mail: gmturi@csir.co.za
*Tel: +27 12 841 2295; E-mail: joconnell@csir.co.za

ABSTRACT

In South Africa, the use of crumb rubber modified (CRM) bitumen has increased over the years as increased traffic loads have resulted in higher performance requirements. Empirical binder characterisation of CRM bitumen remains widespread internationally, even though empirical properties cannot predict asphalt mix performance. Advanced rheological characterisation of CRM bitumen using a dynamic shear rheometer (DSR) is a superior alternative once the morphological challenge of the binary binder system has been overcome. This paper documents a method for the rheological analysis of CRM bitumen with ageing and attempts to relate such characterisation to the performance of CRM bitumen in an asphalt mix.

1 INTRODUCTION

In South Africa, the main distresses contributing to asphalt pavement failures are fatigue cracking, permanent deformation and thermal cracking. Such distresses are affected by the rheological properties of the binder in the asphalt pavement. Bituminous binder behaves as a visco-elastic material when subjected to loading. The linear visco-elastic behaviour of the binder is influenced by loading time and temperature; and changes with ageing. Fatigue and thermal cracking is associated with lower temperatures and aged binder of high viscosity, while permanent deformation is associated with higher temperatures where its rheology approaches Newtonian behaviour.

An ideal binder should, therefore, display adequate elastic behaviour at higher pavement temperatures to resist permanent deformation with a reduced rate of ageing and lower viscosity at lower temperatures to prevent fatigue and thermal cracking. In order to attain such ideal rheological behaviour, thermoplastic polymers have been used extensively internationally, to improve the properties of unmodified bitumen.

Nationally, rubber crumbs are a commonly used modifier due to their proven field performance in CRM bitumen (Potgieter *et al.*, 1998]). Until recently, empirical characterisation remained the only means of predicting performance of these binders, short of constructing pavement test sections. An improved characterization is provided in rheological characterisation using a DSR. However, the binary morphology of CRM bitumen makes it a challenge to test using current methods and equipment. Furthermore, the heterogeneous state of the recovered binder makes it impossible to monitor the rheological properties of the binder in an asphalt mix with time. An appropriate DSR method is presented in this paper for the rheological testing and characterisation of CRM bitumen with ageing and related to asphalt mix performance.

1.1 Empirical characterisation of CRM bitumen

In South Africa, CRM bitumen is manufactured through blending penetration grade bitumen (72 – 82%), rubber crumbs (18 – 24%) and extender oil (0 – 4%) (TG1, 2007) at elevated temperatures of between 190 - 210ºC. The blending is done by a high speed stirring device for 1 to 4 hours until the bitumen is considered modified.

The typical base bitumen used in South Africa is 80/100pen grade bitumen according to SANS 307 (2005) requirements. Table 1 shows the results of the 80/100pen grade bitumen used in this investigation. The extender oil is produced as per COLTO (Committee of Land Transport Officials) specification requirements (COLTO, 1998). Rubber crumbs are obtained through the ambient process of shredding vulcanized tyres. The crumb rubber particles used essentially passed the 1.18mm sieve and the majority retained on the 0.6mm sieve. The resultant CRM bitumen was blended by Much Asphalt (Pty) Ltd. The test results in Table 2 indicate that the CRM bitumen blend conformed to TG1 specification requirements for all tests except resilience, which was slightly above specification.

Table 1: Bitumen Grade Requirements for an 80/100pen grade binder

PROPERTY	Requirements	Results	Test Method
Penetration (1/10 mm)	80-100	81	ASTM D5
Softening Point (°C)	42-51	45.2	ASTM D36
Viscosity @ 60°C (Pa.s)	75-150	97	ASTM D4402
Viscosity @ 135°C (Pa.s)	0.15-0.40	0.30	ASTM D4402
After Rolling Thin Film Oven Treatment (RTFOT)			
Mass Change (%)	0.3 Max	0.05	ASTM D2872
Viscosity @ 60°C (% of original)	300 Max	229	ASTM D4402
Softening Point (°C)	44 Min	50.4	ASTM D36
Increase in Softening Point (°C)	7 Max	5.2	ASTM D36
Retained Penetration (% of original)	50 Min	64	ASTM D5

Table 2: CRM bitumen properties

PROPERTY		Unit	Results	Test Method	Class: A-R1
Softening Point		°C	62.8	MB-17	55-65
Dynamic Viscosity @ 190°C		dPa.s	35	MB-13	20-50
Compression Recovery	5 mins	%	86.6	MB-11	>80
	1 hour		88.6		>70
	4 days		N/A		N/A
Resilience @ 25°C		%	42	MB-10	13-40
Flow		mm	14	MB-12	10-50

1.2 Digestion viscosity curve of CRM bitumen

CRM bitumen properties change with temperature, digestion time and energy consumed during the digestion process.

The various stages of CRM bitumen blends can be defined in terms of viscosity as depicted in Figure 1. Stage 1 is characterised by an increase in viscosity initially upon blending. In this phase, the rubber particle dimensions increase as the oil and/or lighter components of the bitumen diffuse into the three dimensional rubber networks of poly-isoprene and poly-butadiene linked by sulphur-sulphur bridges. The diffusion process varies according to the amount of cross-links in the rubber, the molecular compatibility between the rubber and the diffusing particles as well as the molecular weight of the latter (Treolar, 1975 and Airey *et al.*, 2002). Thereafter, an additional viscosity increase occurs from a further incorporation of the diffusing matter into the rubber particles as the sulphur-sulphur bonds thermally dissociate.

The thermal dissociation process continues until a maximum viscosity point is reached referred to as Stage 2. The viscosity then decreases with digestion time in Stage 3 as the network disintegrates due to the loss of the sulphur linkages. Once the decrease in viscosity reaches a point of constant viscosity, the CRM bitumen blend is referred to as terminal. This has been depicted as Stage 4 in the digestion viscosity curve.

Figure 2 shows the CRM bitumen blend exhibiting a gradual viscosity increase. This indicates that the blend was in Stage 1 of the digestion circle at the time of testing. Figure 3 shows both Stage 3 and 4 viscosity behaviour of the blend with over digestion.

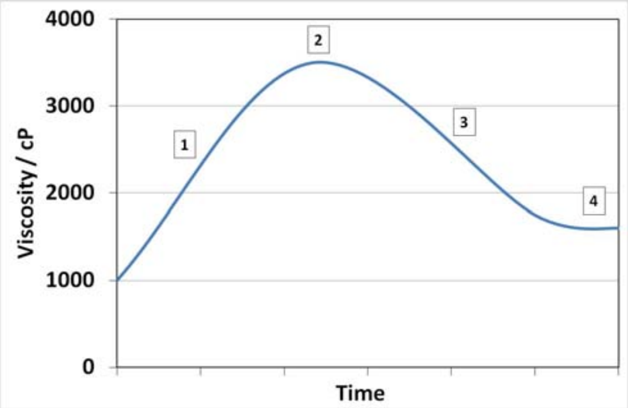

Figure 1: Typical digestion viscosity curve of CRM bitumen.

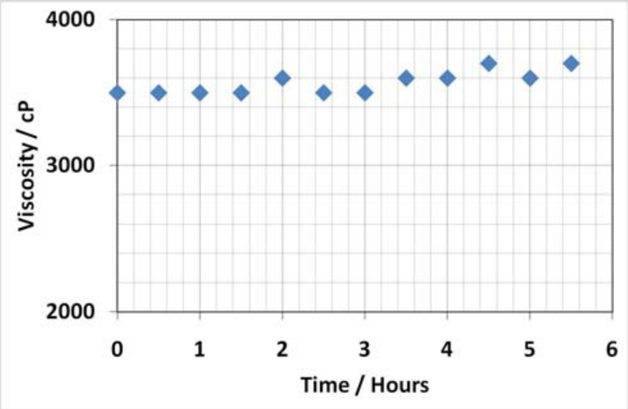

Figure 2: Viscosity vs. Time of the CRM bitumen blend at 190°C.

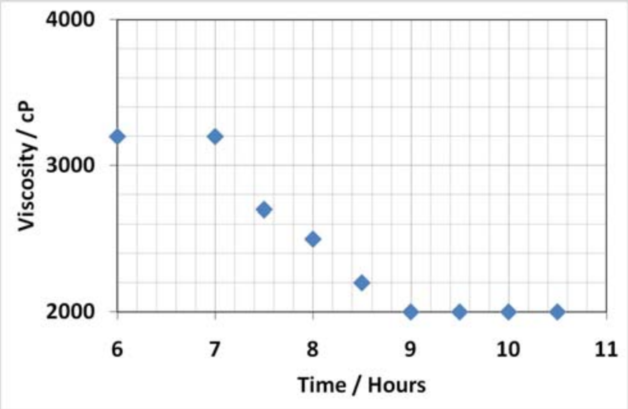

Figure 3: Viscosity vs. Time of the CRM bitumen blend at 210°C (after initial digestion as shown in Figure 2).

2 RHEOLOGICAL TESTING OF CRM BITUMEN

The development of the Performance Graded (PG) specification system for binders in the United States by the Strategic Highway Research Program (SHRP) program, focused on selecting the proper binder grade for the climate in which the binder is to be utilized. The PG system uses parameters measured with a DSR to predict rutting and fatigue resistance at various temperatures. This system was a major improvement over empirical testing and the intent was to develop binder specifications that could be applied universally to all binders.

The determination of binder rheological properties using a DSR is specified in AASHTO (American Association of State Highway and Transportation Officials) Test Method T 315 (2005). According to the test method, the limits of the test temperature and frequency ranges are a function of the binder stiffness (which is affected by binder grade, type of modification, etc.) and the capacity of the DSR. The following guidelines (SHRP-A-370, 1994) are used in approximation for selecting plate diameters and sample thickness (gap):

- 8-mm parallel plates with a 2-mm gap are recommended when the absolute value of G* ranges from 0.1 to 30 MPa.
- 25-mm parallel plates with a 1-mm gap are recommended when G* ranges from 1.0 to 100 kPa.
- 50-mm parallel plates (less common) are recommended when G* < 1 kPa.

2.1 Limitations of the PG specification system

The majority of the SHRP research was conducted using straight penetration grade binders. The resultant test methods and binder specifications were not verified for modified binders. Consequently, the PG specification system does not fully characterize polymer modified binders (such as CRM bitumen) with proven field performance.

Furthermore, AASHTO T315 (2005) is also incapable of correctly characterizing CRM bitumen rheologically due to the requirements of its testing system. The plate gap thickness would require adjustment to avoid interference from the crumb rubber particle size (Airey *et al.*, 2002, McGennis, 1995 and Shen *et al.*, 2005).

2.2 DSR Analysis of CRM bitumen

An unmodified 60/70pen grade bitumen of similar stiffness to CRM bitumen was used as a control sample. Frequency sweeps of the two binders were measured at 55°C for various gap settings. All measurements were done within the linear visco-elastic (LVE) range using a 25-mm diameter parallel plate configuration. Table 3 contains results of the measured complex moduli (G*) and phase angle (δ) properties at various frequencies and gap settings for the two binders.

The frequency sweep results for the 60/70pen grade bitumen in Table 3 shows good reproducibility in terms of G* and δ at all gap thicknesses between 1-2mm. This implies there was adequate adhesion of the binder sample to the two DSR plates as well as homogenous deformation of the sample throughout the gap distance.

On the other hand, the results for the CRM bitumen blend shows poor reproducibility of both G* and δ values at the 1-mm gap setting and towards lower frequencies. As the gap thickness was increased the reproducibility improved until a gap range is reached where the frequency sweeps became reproducible, as observed for the results at 2-mm gap.

The results show that a minimum DSR gap is required for any CRM bitumen, probably dependent on the maximum particle size of the rubber crumbs in the blend. The maximum rubber particle size would depend on the initial size of the crumb rubber prior to blending, the diffusion process as well as the stage of the blend in the digestion viscosity curve at the time of testing. Fortunately, the type, amount and size of rubber crumbs in South African blends are fairly consistent. It is therefore recommended that repeated DSR testing of local CRM bitumen blends be done at a 2-mm gap thickness. The calculated standard deviation should establish whether a gap adjustment is necessary for future tested blends.

Table 3: Standard deviation at different frequencies and gap thickness of binder samples measured at 55°C

Binder Type	**Gap / mm**	**Frequency**									
		0.0126 Hz		**0.126 Hz**		**1.26 Hz**		**12.6 Hz**		**50.2 Hz**	
		δ /°	**G* /Pa**	**δ /°**	**G* /Pa**	**δ /°**	**G* /Pa**	**δ /°**	**G* /Pa**	**δ /°**	**G* /Pa**
60/70pen	1.0	89.6	40.0	88.5	397	85.8	3 710	82.2	31 900	80.3	110 000
60/70pen	1.0	89.6	41.0	88.4	402	85.6	3 750	82.0	32 100	80.0	111 000
60/70pen	1.0	89.6	41.0	88.5	403	85.7	3 750	81.8	32 200	78.6	111 000
60/70pen	1.0	89.5	40.4	88.4	396	85.6	3 690	82.0	31 700	79.4	109 000
60/70pen	1.0	89.6	39.0	88.5	387	85.8	3 620	82.0	31 200	79.0	109 000
60/70pen	1.0	89.6	39.0	88.6	382	85.9	3 570	82.4	30 800	80.7	107 000
Average	**1.0**	**89.6**	**40.1**	**88.5**	**395**	**85.7**	**3682**	**82.1**	**31650**	**79.7**	**109500**
σ (60/70pen)	**1.0**	**0.04**	**0.95**	**0.08**	**8**	**0.12**	**72.8**	**0.21**	**546.8**	**0.80**	**1516.6**
Coeff. of Variation/%	**1.0**	**0.0**	**2.3**	**0.1**	**2**	**0.1**	**2**	**0.3**	**2**	**1.0**	**1**
60/70pen	1.0	89.6	40.0	88.5	397	85.8	3 710	82.2	31 900	80.3	110 000
60/70pen	1.1	89.6	40.3	88.5	398	85.8	3 730	82.3	32 200	80.5	112 000
60/70pen	1.2	89.6	39.7	88.4	393	85.7	3 660	81.9	31 500	78.8	110 000
60/70pen	1.3	89.6	39.2	88.3	384	85.7	3 580	81.9	30 800	78.8	107 000
60/70pen	1.4	89.6	40.4	88.3	397	85.6	3 700	81.7	31 700	78.0	110 000
60/70pen	1.5	89.6	39.1	88.6	385	85.9	3 620	82.1	31 300	79.0	109 000
60/70pen	1.6	89.4	40.9	88.2	400	85.6	3 730	81.9	31 900	79.1	110 000
60/70pen	1.7	89.4	40.3	88.2	393	85.6	3 650	81.8	31 300	78.7	109 000
60/70pen	1.8	89.5	40.9	88.2	397	85.6	3 700	81.9	31 800	79.2	110 000
60/70pen	1.9	89.4	40.4	88.2	397	85.6	3 690	81.9	31 700	79.1	110 000
60/70pen	2.0	89.3	40.2	88.2	392	85.6	3 650	82.2	31 400	80.3	109 000
Average	**1-2**	**89.5**	**40.1**	**88.3**	**394**	**85.7**	**3675**	**82.0**	**31591**	**79.3**	**109636**
σ (60/70pen)	**1-2**	**0.11**	**0.59**	**0.15**	**5.2**	**0.11**	**47.2**	**0.19**	**383.3**	**0.78**	**1206.0**
Coeff. of Variation/%	**1-2**	**0.1**	**1.5**	**0.2**	**1**	**0.1**	**1**	**0.2**	**1**	**1.0**	**1**
CRM Bitumen	1.0	28.2	1 430	43.8	3 500	51.1	12 500	52.6	47 200	54.6	107 000
CRM Bitumen	1.0	24.4	1 600	41.7	3 640	50.7	12 600	52.9	47 600	55.5	108 000
CRM Bitumen	1.0	29.5	1 190	46.3	3 120	52.4	11 800	53.4	45 500	54.9	105 000
CRM Bitumen	1.0	25.4	1 710	41.6	3 870	50.1	13 300	52.1	49 300	53.7	112 000
CRM Bitumen	1.0	28.1	1 580	43.4	3 850	50.0	13 400	51.8	48 800	54.9	108 000
CRM Bitumen	1.0	23.5	1 820	40.4	3 980	49.5	13 400	52.4	49 600	55.0	112 000
CRM Bitumen	1.0	28.8	1 310	44.8	3 280	52.0	12 000	53.6	46 500	55.5	107 000
CRM Bitumen	1.0	29.1	1 320	45.4	3 370	51.7	12 500	52.9	47 200	54.8	108 000
CRM Bitumen	1.0	27.8	1 400	44.4	3 440	51.9	12 600	53.1	48 600	54.2	112 000
Average	**1.0**	**27.2**	**1484**	**43.5**	**3561**	**51.0**	**12678**	**52.8**	**47811**	**54.8**	**108778**
σ (CRM Bitumen)	**1.0**	**2.19**	**206.2**	**1.95**	**293.5**	**1.02**	**584.8**	**0.59**	**1363.3**	**0.58**	**2587.4**
Coeff. of Variation/%	**1.0**	**8.1**	**14**	**4.5**	**8**	**2.0**	**5**	**1.1**	**3**	**1.1**	**2**
CRM Bitumen	2.0	45.9	676	56.7	2 670	55.6	11 700	54.4	46 700	56.1	108 000
CRM Bitumen	2.0	46.0	655	57.6	2 650	55.9	11 800	54.3	47 100	55.8	109 000
CRM Bitumen	2.0	45.5	688	56.8	2 680	55.9	11 800	54.6	47 200	55.9	109 000
CRM Bitumen	2.0	45.0	675	57.4	2 660	56.1	11 900	54.5	47 600	55.4	111 000
CRM Bitumen	2.0	45.5	688	56.8	2 680	55.9	11 800	54.6	47 200	55.9	109 000
Average	**2.0**	**45.6**	**676**	**57.1**	**2668**	**55.9**	**11800**	**54.5**	**47160**	**55.8**	**109200**
σ (CRM Bitumen)	**2.0**	**0.40**	**13.5**	**0.41**	**13.0**	**0.18**	**70.7**	**0.13**	**320.9**	**0.26**	**1095.4**
Coeff. of Variation/%	**2.0**	**0.9**	**2**	**0.7**	**0**	**0.3**	**0**	**0.2**	**1**	**0.5**	**1**

3 RHEOLOGICAL PROFILE OF CRM BITUMEN WITH AGEING

On doing a frequency sweep at various temperatures, a black diagram of the bitumen can be plotted. This characterises the bitumen in terms of rheology, at various temperatures and frequencies for various conditions of ageing. Figure 4 shows black diagrams of a 40/50pen grade bitumen unaged, after Rolling Thin Film Oven Treatment (RTFOT) ageing (to simulate ageing that occurs during manufacture and laying of the mix); and after Pressure Ageing Vessel (PAV) ageing (to simulate long term ageing). When a 40/50pen grade ages there is an increase in stiffness and a reduction in phase angle. The reduction in phase angle is more prominent at lower temperatures than it is at higher temperatures. This is because straight run bitumen experiences oxidative hardening with ageing.

The effect of ageing on modified binders differs to unmodified bitumen. Figures 5 and 6 show the complicated nature of binary systems; it shows black diagrams for an SBS-modified binder and a CRM bitumen with ageing, respectively. Unaged modified binders exhibit increased complex moduli with decreased phase angle at higher temperatures compared to unmodified bitumen. Upon ageing, these modified binders partially lose their proportional elastic contribution at higher temperatures but such elastic contribution remains much higher than that for unmodified binders. At lower temperatures, ageing of modified binders results in decreased phase angle. For SBS-modified binder there is an accompanying increase in G* associated with oxidative ageing, but for CRM bitumen oxidative ageing is juxtaposed against S-S bond scission, leading to unpredictable changes in G*.

The rheological monitoring of binder ageing in asphalt mixes is very complicated for CRM bitumen. The binder cannot be recovered as a single entity and the recovered elements cannot be re-combined to produce the same binder as it existed before in the asphalt. In the recovery process, the binder is dissolved in benzene then separated from the aggregates before it is recovered back again through distillation. Crumb rubber particles do not dissolve in benzene hence they are separated out with the aggregate. The solvent recovery process destroys the CRM bitumen network and the chemical equilibrium of the blend; this makes it impossible to re-blend the separated components to reproduce the binder as it occurred previously in the asphalt. As a result only the base binder, the extender oil together with the benzene soluble polymer fractions from the crumb rubber can be recovered and tested. Figure 7 shows black diagrams of the recovered base binder mixture from the CRM bitumen blend. The ageing observed is a combination of oxidative hardening of the base bitumen together with the increased incorporation of de-linked polymers into the recovered bitumen.

In essence, the ageing observed for the recovered base binder cannot be related to that of the CRM bitumen blend, but remains the only practical way of monitoring ageing of these binders. Further research is required.

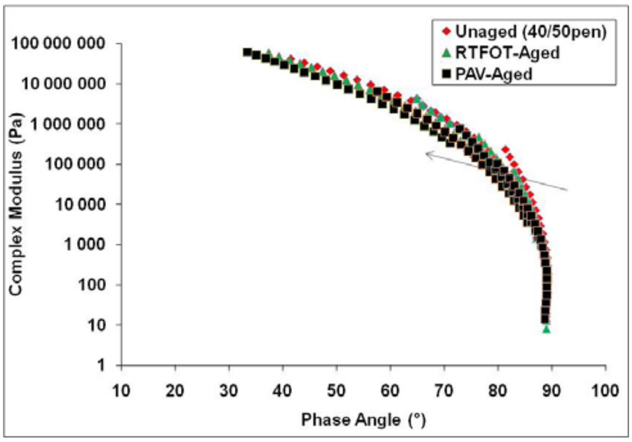

Figure 4: Black diagram of the 40/50pen grade bitumen with ageing (Mturi *et al.*, 2010).

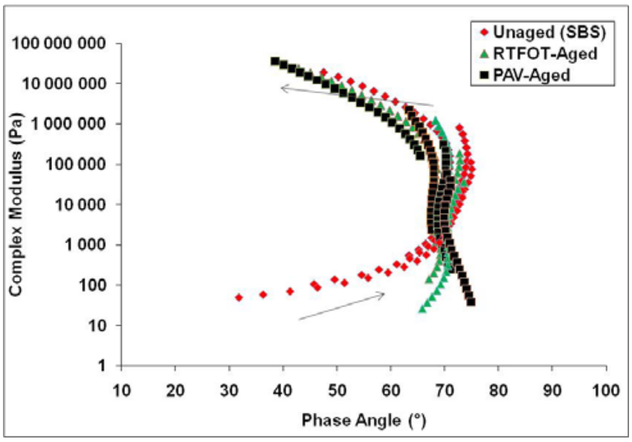

Figure 5: Black diagram of an SBS-modified binder with ageing (Mturi *et al.*, 2010).

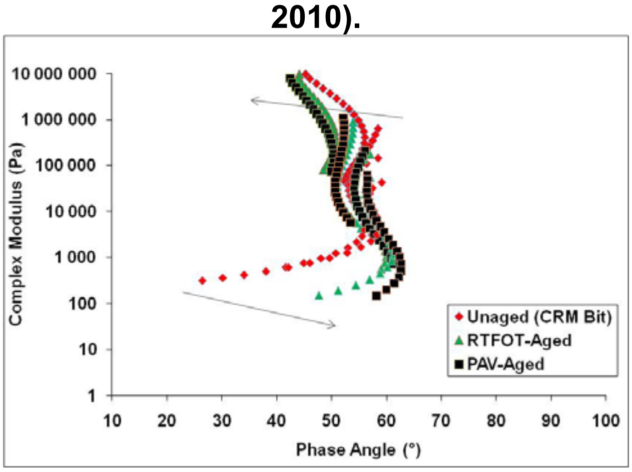

Figure 6: Black diagram of CRM bitumen binder with ageing.

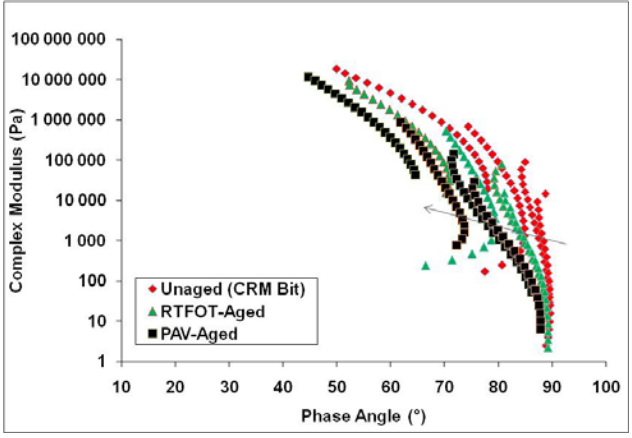

Figure 7: Black diagram of recovered base binder from CRM bitumen at various stages of ageing.

4 USING CRM BITUMEN RHEOLOGY TO PREDICT ASPHALT MIX PERFORMANCE

For mechanistic-empirical design purposes, the dynamic modulus of a mix is an important input parameter for damage models pertinent to the design model. It is not always practical to determine the dynamic modulus of a mix, and so use is made of predictive equations which relate the viscosity of a bituminous binder after RTFOT to the dynamic modulus value.

The NCHRP guide, "Guide for Mechanistic-Empirical Design" as published by the National Cooperative Highway Research Program (NCHRP, 2004) recommends the use of the Witczak predictive equation as given in Equation 1.

$$\begin{aligned}\log\left|E^*\right| &= 3.750063 + 0.029232P_{200} - 0.001767(P_{200})^2 - 0.002841P_4 - 0.058097V_a \\ &- 0.802208\frac{V_{beff}}{(V_{beff}+V_a)} + \frac{\left[3.871977 - 0.0021P_4 + 0.003958P_{38} - 0.000017(P_{38})^2 + 0.00547P_{34}\right]}{1+e^{(-0.603313-0.313351\log f-0.393532\log\eta)}}\end{aligned} \tag{1}$$

where:

E* = dynamic modulus, psi.
η = bitumen viscosity, 10^6 Poise.
f = loading frequency, Hz.
V_a = air void content, %.

V_{beff} = effective bitumen content, % by volume.
P_{34} = cumulative % retained on the ¾ in (19.0mm) sieve.
P_{38} = cumulative % retained on the 3/8 in (9.5 mm) sieve.
P_4 = cumulative % retained on the No. 4 (4.75mm) sieve.
P_{200} = % passing the No. 200 (75 micron) sieve.

Viscosity can be determined from the complex shear modulus generated by the DSR by using the conversion equation 2:

$$\eta = \frac{G^*}{10}\left(\frac{1}{\sin\delta}\right)^{4.8628} \tag{2}$$

where;
G^* = complex modulus of the binder, Pa.
δ = phase angle.
η = viscosity, Pa.s.

The Hirsch predictive equation 3 (Christensen *et al.*, 2003) is also widely used.

$$|E^*|_{mix} = P_c\left[4{,}200{,}000\left(1-\frac{VMA}{100}\right)+3\,|G^*|_{binder}\left(\frac{VFA\ xVMA}{10{,}000}\right)\right]+\frac{1-P_c}{\left[\frac{\left(1-\frac{VMA}{100}\right)}{4{,}200{,}000}+\frac{VMA}{3VFA\,|G^*|_{binder}}\right]} \tag{3}$$

$$P_c = \frac{\left(20+\frac{VFA\,x\,3\,|G^*|_{binder}}{VMA}\right)^{0.58}}{650+\left(\frac{VFA\,x\,3\,|G^*|_{binder}}{VMA}\right)^{0.58}}$$

where:
$|E^*|$ = dynamic modulus, psi.
$|G^*|_{binder}$ = shear complex modulus of binder, psi.
VMA = voids in mineral aggregates, %.
VFA = voids filled with binder, %.
P_c = aggregate contact factor.

Both these equations were evaluated using a BRASO asphalt mix (CRM bitumen with a semi-open aggregate grading) manufactured in the CSIR Built Environment pavement materials laboratory. The binder rheology was determined using a DSR and is summarized in Figure 8.

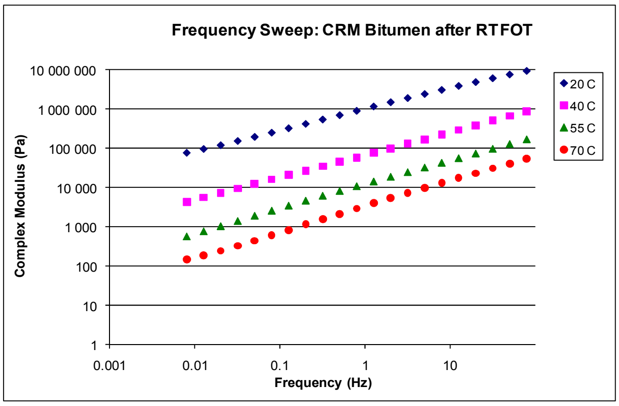

Figure 8: Complex Shear Modulus after RTFOT.

The dynamic modulus (E*) test was conducted on samples compacted to design and field voids using a CSIR protocol for asphalt mixtures in South Africa (Anochie-Boateng, 2009) and a commercially available Universal Testing Machine (UTM-25) testing device.

Predicted dynamic modulus values are compared with measured values in Figures 9 and 10 (E* unit of measurement is MPa).

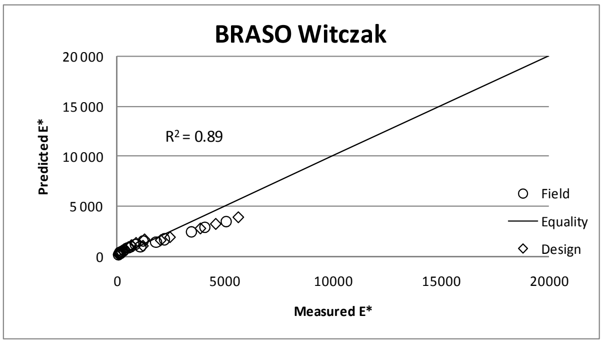

Figure 9: Predicting Dynamic Modulus using the Witczak Equation.

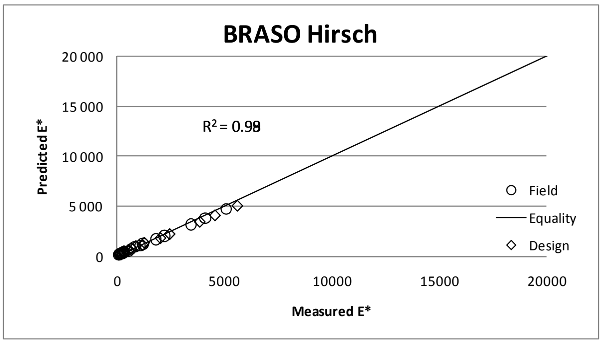

Figure 10: Predicting Dynamic Modulus using the Hirsch Equation.

The results indicate that the Hirsch equation provided a better prediction of the measured dynamic modulus than the Witczak equation did for this particular mix**.**

Generally, alternative rheological indicators such as apparent viscosity (as determined by the Brookfield viscometer) may also be used in the Witczak prediction equation. However, Brookfield viscosity cannot be determined accurately for a two-phase CRM bitumen. This emphasizes the importance of the rheological characterisation of CRM bitumen with the aid of a DSR, considering that no alternative rheological tests would provide usable viscosity values.

5 CONCLUSION

A method for determining the rheology of a CRM bitumen using a DSR has been demonstrated. The results were used successfully for the prediction of resilience response of a BRASO asphalt mix.

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