THE USE OF THE DYNAMIC SHEAR RHEOMETER (DSR) TO PREDICT THE PENETRATION OF BITUMEN

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Abstract.

Bitumen has historically been graded by two empirical tests; Penetration and viscosity. The penetration grading system was created to establish different bitumen grades for binder selection in varying climates and applications. Penetration is the most commonly used method of measuring the consistency of bitumen and it is also used to monitor bitumen quality during its production process. This study was conducted to develop a method by which bitumen can be analysed in a short space of time so as to allow the refinery to make the necessary changes quickly to maximise bitumen production without having to wait for the time consuming penetration test. More specifically, the study is aimed at identifying a relationship between penetration properties and the dynamic shear rheometer test results using the Strategic Highway Research Program performance grading binder test protocol.

To determine the correlation between the dynamic shear rheometer and penetration results, bitumen samples of 80/100 penetration grade bitumen were analysed on two Paar Physica dynamic shear rheometers using the testing protocol as per performance grading requirements for long term aged bitumen. The tests were all performed at 25 °C to keep the test conditions for both analyses the same.

The penetration values showed a good correlation with the complex modulus (G*) at 25 °C and it would thus be possible to use the dynamic shear rheometer value in a statistical prediction model to predict a penetration value.

KEYWORDS: bitumen, penetration, dynamic shear rheometer, complex modulus.

1. INTRODUCTION

Several programs have been undertaken to find possible correlations between the results of traditional empirical test methods (such as the penetration test) for bitumen and the Strategic Highway Research Program (SHRP) Performance Grading (PG) parameters as determined using a dynamic shear rheometer (DSR). The DSR is used to characterize the viscous and elastic behaviour of bitumen. This is achieved by measuring the viscous and elastic properties of a thin bitumen binder sample sandwiched between an oscillating and a fixed plate.

Saal and Tabout (1958) found that for both unmodified and modified bitumens, the Penetration test correlates well with the stiffness of the bitumen as measured using the DSR, at the same temperature (25°C) and at a frequency of 0,4 Hz, with the equivalent loading time (0.4 seconds). In rheological terms, a good correlation has been identified between log(G*), the complex shear modulus and log(Pen). The correlation can be written as a linear equation:

$$\log(G^*_{T=25\,°C,\ t=0.4}) = 2.923 - 1.9\log(Pen) \qquad \text{[Eq. 1]}$$

This relationship was confirmed by Gershkoff (1995), see Figure 1, and by Francken and Vanelstraete (1996, 1997), see Figure 2, by analysing new and laboratory aged virgin and modified bitumen, as well as extracted binders from cores of pavements after 12 years of field service ageing. This correlation was however used to predict the stiffness of the binder from a conventional Penetration result.

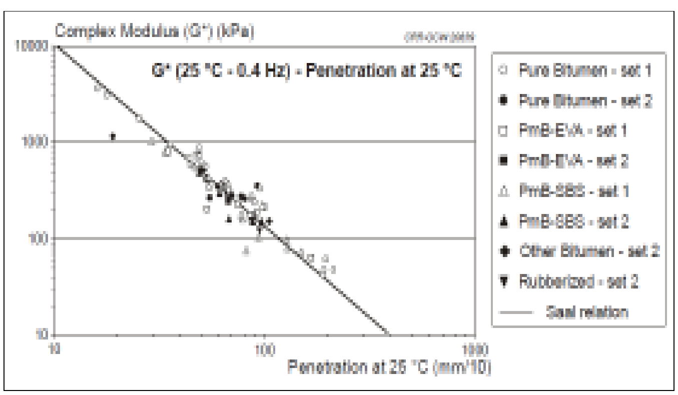

Figure 1. *Binder modulus at 25 °C and 0.4 Hz versus Penetration values for about 100 binders of different origin (Gershkoff, 1995)*

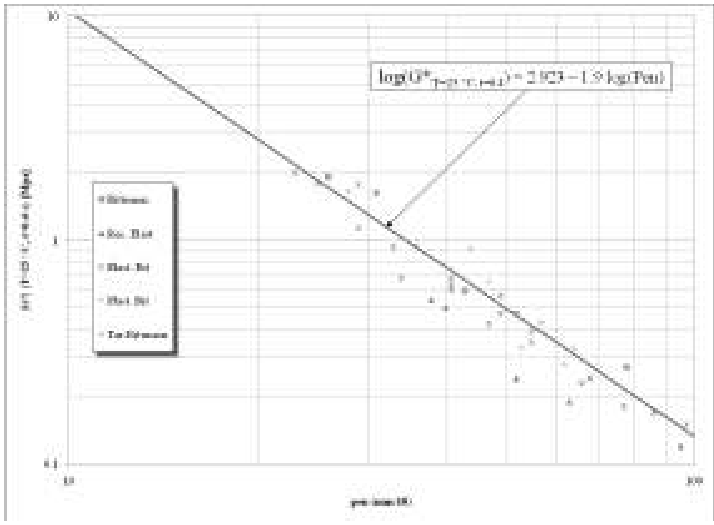

Figure 2. *Correlation between the complex modulus at 25 °C and 0.4 Hz and Penetration (Francken and Vanelstraete, 1996, 1997)*

In a more recent study, where the results of SHRP and conventional binder tests were compared, it was found that the penetration value has a strong correlation with G*×sinδ at 25°C (Lee *et al.*, 2004). The DSR tests in this study were all conducted at 25°C with the rheometer setting complying with the SHRP PG system requirements. Figure 3 shows the plot of Penetration values versus the G*sinδ at 25°C for six asphalt binders before and after being subjected to the rolling thin film oven test (RTFOT) and pressure aging vessel (PAV) processes. G*sinδ (loss modulus) increases as the penetration decreases.

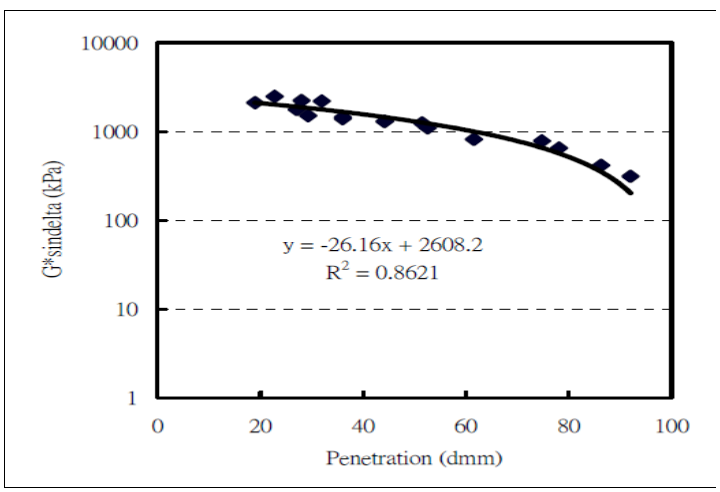

Figure 3. *Relationship between Penetration values and G*.sinδ before and after the RTFOT and PAV at 25 °C (Lee et al, 2004)*

The South African specification for bitumen is based on penetration and viscosity tests. Not surprisingly, most of the South African refineries use the Penetration and viscosity properties of bitumen therefore as quality control parameters during the production of

80/100 penetration grade bitumen. During the changeover from fuel production mode to the bitumen mode, these parameters are closely monitored and, when the specification limits on both are met, the bitumen is run down to the final product tanks. However, the penetration test (using the ASTM D5 protocol) requires a time consuming conditioning step before a sample can be analysed, during which time product that potentially could be run down as bitumen is routed to fuel oil.

Using the DSR to do quality assessment during the changeover from the fuel production campaign to a bitumen production, the complex modulus results can be available in as little as 20 minutes. The objective of this study is to find a correlation between the DSR results from 80/100 Penetration grade bitumen using the conditions prescribed by the SHRP PG system at 25°C and the Penetration test results at 25°C, and to confirm previously reported results.

2. METHODOLOGY

To determine the correlation between the results obtained from a DSR and penetration, more than 100 bitumen samples of 80/100 Penetration grade bitumen were analysed on two different models of Paar Physica DSR instruments (UDS 200 and MCR 301). The DSR tests were done according to ASTM D7175 - Standard Test Method for Determining the Rheological Properties of Asphalt Binder using a Dynamic Shear Rheometer, and more specifically the method as prescribed for analysing the long term aged material after aging in the PAV. The Penetration was determined using ASTM D5 - Standard Test Method for Penetration of Bituminous Materials. All conditioning and settings prescribed in these methods were followed precisely.

These tests were all performed at 25°C to keep the test conditions for the penetration determination and DSR analysis the same. These results were then used to develop a penetration prediction model by doing simple linear regression on the data set.

2.1 Bitumen testing on the DSR

A hot bitumen sample/specimen (0.15-0.20 g) was transferred onto the DSR spindle using a spatula and the spindle was inserted into the rheometer. The two test plates were moved together until the gap between the plates equalled the testing gap (2 mm) plus the gap closure required to create a suitable bulge in the test specimen. The excess bitumen was trimmed by moving a heated trimming tool around the edges of the plates so that the bitumen sample was flush with the outer diameter of the plates. After the trimming was complete, the gap between the test plates was decreased by a predetermined distance to form a slight bulge at the outside face of the test specimen.

The test specimen is maintained at the test temperature of 25.0 ± 0.1°C by enclosing the upper and lower plates in a thermally controlled environment or test chamber. The strain was set at 1 % as per the PG method and the angular frequency was set at 10 radian per second (equivalent to 1.59 Hz). The Complex Shear Modulus (G*) is calculated automatically as part of the operation of the rheometer using proprietary computer software supplied by the instrument manufacturer.

The test period for each sample was 10 minutes to ensure that the samples between the two parallel plates were in thermal equilibrium. Thirty (30) data points were taken over this period and the average of the last 15 data points were used as the G* result for this study. By doing this, it is ensured that the temperature throughout the sample is uniform and it also eliminates any fluctuation in temperature during the test.

A simple linear regression analysis was conducted using MS Excel on the results from the DSR and the penetration test to determine whether there is a correlation between the penetration and G* at 25°C.

2.2 Validation of the model

A number of 80/100 penetration grade bitumen samples from different refineries were collected and the penetration values of these bitumen samples were determined. These samples were also subjected to DSR testing. The measured Complex Shear Modulus (G*) of the bitumen of these samples was used to establish the robustness of the model in terms of the origin of the bitumen, i.e. actual penetration test results were compared with the predicted penetrations for the samples.

3. RESULTS AND DISCUSSION

Figure 4 shows the plot of log(Penetration) versus log(G*) at 25°C for the more than 100 bitumen samples mentioned above. G* (complex modulus) increases as the penetration value decreases, i.e. both properties show an increase in “stiffness” of the bitumen. A good correlation coefficient (R^2=0.99) was observed.

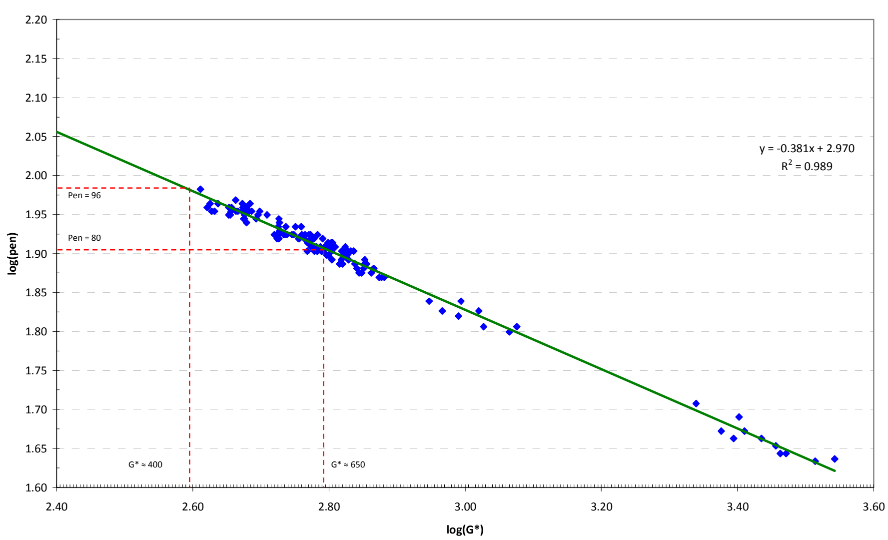

Figure 4. *Relationship between log(Pen) and log(G*) at 25 °C penetration grade bitumen*

Figure 4 provides the regression line for the transformed DSR and penetration data. By superimposing the South African specification range for penetration onto the graph, a range for the corresponding G* can be predicted in which the bitumen will conform to the penetration specification requirement with a 95 % confidence. This means that 95 samples out of 100 analysed with the DSR and the data projected by the regression model will conform to the current Penetration specification requirement.

The results from a simple linear regression of log(Pen) vs. log(G*) are shown in Table 1.

***Table 1**. Regression analysis of Penetration results versus G* at 25°C*

Regression Statistics	
Multiple R	0.9949
R Square	0.9899
Adjusted R Square	0.9898
Standard Error	0.0079
Observations	134

ANOVA

	Df	***SS***	***MS***	***F***	***Significance F***
Regression	1	0.796	0.796	12907.150	1.565E-133
Residual	132	0.008	6.169E-05		
Total	133	0.804			

	Coefficients	***Standard Error***	***t Stat***	***P-value***	***Lower 95%***	***Upper 95%***
β_0	2.971	0.010	312.408	2.864E-191	2.952	2.990
β_1	-0.381	0.003	-113.610	1.565E-133	-0.388	-0.374

The model based on the sample provided in Figure 4 and the regression analysis in Table 1 can now be given by:

$$\text{Log(Pen)} = 2.971 - 0.381\ \text{Log}(G^*) \qquad \text{[Eq. 2]}$$ [1]

With a correlation of 0.9899 and coefficient of determination R^2 = 98.99%, there is clearly a strong linear correlation between log(Pen) and log(G*).

The predicted log(Pen) can be easily converted back to a Penetration value. The predicted Penetration results were compared to the actual Penetration values for the same samples as determined by ASTM D5. The predicted Penetration obtained from the model can be plotted against the actual measured Penetration results (Figure 5) to test the validity of the prediction model. The high R^2 (0.9898) is a further indication that the linear model that was fitted is appropriate.

[1] The coefficients in this equation will change as the data set is expanded.

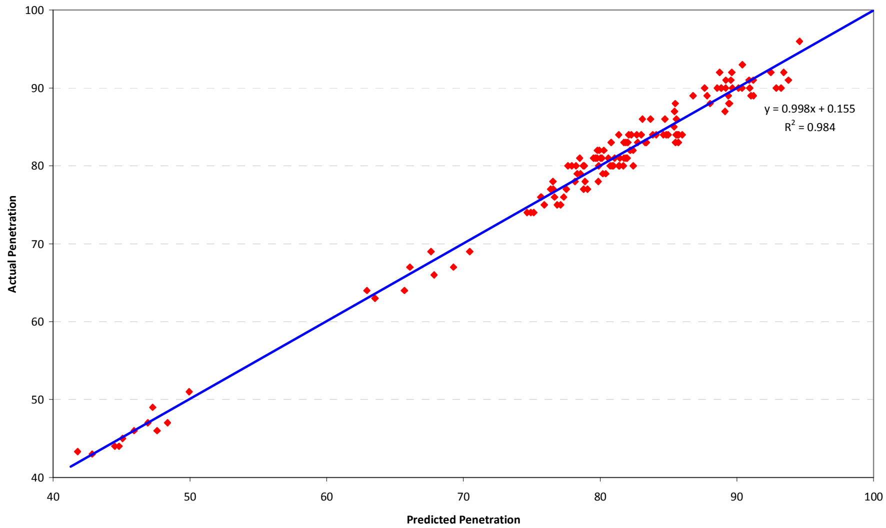

Figure 5. *Actual Penetration versus Predicted Penetration of regression data*

3.1 Validation of model

A new set of samples were collected from different refineries and the G* and Penetration of the samples were determined. The complex modulus, G*, of the validation samples were converted to the log(G*) and substituted into the equation to obtain the log(Pen) (Equation 2). Table 2 presents the results for properties of the bitumen from different origins as determined in this study.

Table 2. *Laboratory results (Penetration and G*) and predicted values of Penetration*

Sample#	**Penetration**	**G* (DSR)**	**log(G*)**	**Predicted log(Pen) (Equation 1)**	**Predicted Pen**
Ref A1	96	431.3	2.635	1.97	92.7
Ref A2	93	444.3	2.648	1.96	91.7
Ref A3	90	457.3	2.660	1.96	90.7
Ref B1	83	596.2	2.775	1.91	82.1
Ref B2	85	599.7	2.778	1.91	81.9
Ref B3	82	597.8	2.777	1.91	82.0
Ref B4	80	597.8	2.777	1.91	82.0
Ref C	86	537.1	2.730	1.93	85.4

Ref A, B and C represents samples of bitumen source from different refineries.

Figure 6 shows a plot of log(Pen) versus log (G*) for the same set of data as in Figure 4, but with the prediction interval superimposed and the results of the samples used for the validation of the model are shown as points on the graph.

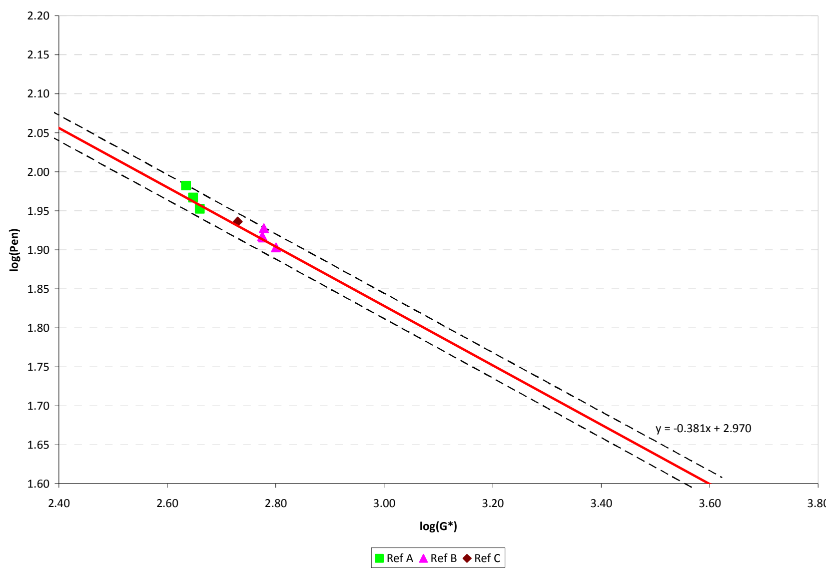

Figure 6. *95th Percentile Limits for the Regression Line*

The 95% prediction limits is the area which will contain the true values with 95% confidence. The data points of the tested samples all fall within the “boundaries” set by the prediction intervals, which is a further indication that there is a strong linear correlation between Penetration and G*.

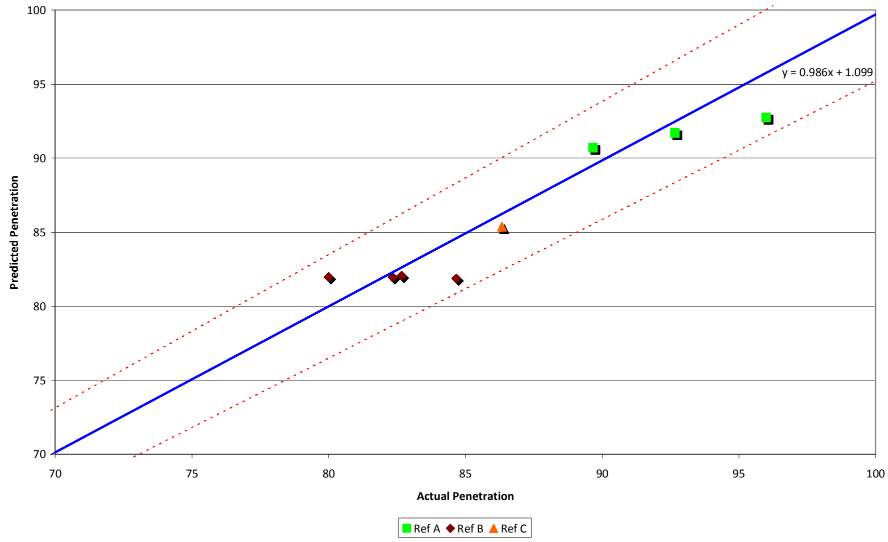

Figure 7. *Actual Penetration versus Predicted Penetration using Model, plotted against Reproducibility Limits for ASTM D5*

Figures 6 and 7 show that the model that has been developed is applicable to most bitumen crude slates that are processed by South African refineries and that the model is also not particularly sensitive to the process by which the bitumen is produced. The predicted values all fall within the reproducibility limits for the ASTM D5 test method for penetration. In using the above-mentioned relationship between G* and penetration, the DSR result can be converted into an equivalent penetration value which could save a lot of analysis time by eliminating the need for prolonged conditioning of test samples.

4. CONCLUSIONS

The measured penetration values show a good correlation with complex shear modulus (G*) at 25°C and it is possible to predict a penetration value using the DSR value in the prediction model (Equation 2). The predicted values all fall within the reproducibility limits for the ASTM D5 test method for penetration. The penetration values, as predicted from results obtained from the two different rheometers, verified the robustness of the prediction equation. This formula could therefore be applied to results from any rheometer. The model that has been developed can be seen as crude oil and refinery process "blind", meaning that it can be used to predict the Penetration value for any 80/100 Penetration grade bitumen produced from any of the four South African crude oil refineries.

The calculation of a Penetration value from the Complex Shear Modulus of bitumen as determined in the DSR at 25°C applied in the linear regression model, provides a result in the equivalent time that it takes to determine the viscosity in a Brookfield Viscometer. This expedites the decision making process. Therefore, the DSR and the penetration model can provide extremely useful results for quality control during crude slate changeovers as well as for final product quality assurance. In choosing the complex modulus limits to incorporate the Penetration specification limit and using the confidence interval, one could, with high degree of confidence, predict whether the Penetration specification will be met.

The model that has been developed for virgin 80/100 Penetration Grade bitumen in this paper, can with further work be extended to include the other Penetration Grades as well as homogeneous modified binders.

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