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VALIDATING THE GENERALITY AND PREDICTIVE ABILITY OF UNIFIED RHEOLOGICAL CURVES FOR UNMODIFIED PAVING ASPHALTS

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Abstract

Fundamental rheological properties of unmodified paving asphalts have been shown to form unified curves when the material's volumetric-flow rate MVR (in cc / 10 minutes) is used as a normalizing factor. The generality of the unification technique is shown by analyzing rheological data for a wide variety of unmodified core asphalts under different temperature conditions. Based on the broad spectrum of covered data, it is concluded that the unified curves are applicable to all unmodified paving asphalts. The predictive ability of the unified curves is then checked and found to give reasonably reliable information. Thus, using a simple parameter like MVR that is determined quite accurately on a relatively inexpensive, easy-to-use flow measurement device (FMD), all fundamental rheological parameters of unmodified paving asphalts can be calculated from the unified curves without actual data generation of viscoelastic properties through the dynamic shear rheometer.

Keywords : asphalt rheology, unified curves, viscoelastic parameters, flow measurement device, material's volumetric-flow rate

Introduction

Asphalts on paved roads witness a wide range of static and dynamic stresses at varying temperatures and under different environmental conditions. Hence it is essential to develop a good insight into the rheological properties of the asphalts covering a wide range of shear rates over an equally broad range of temperatures and under simulated environmental conditions.

Paving asphalts exhibit varying degrees of viscoelasticity under different conditions of temperature and loading.

Understanding the rheology of paving asphalts is important from different viewpoints. It is useful as a control tool to distinguish between various asphalts from different crude sources and which are refined using different processes.

Understanding asphalt rheology aids in determining the appropriate temperature for mixing aggregates with asphalt and also to compact the composite material in place so that the final pavement can be prepared well and with ease. It is also important to find out how the rheological properties of asphalts relate to the distresses in the pavements after the lay down process and after years of service. It is, therefore, not surprising that the subject of asphalt rheology has been the focus of a great deal of research as can be seen from even a partial list of references¹⁻¹³ on various aspects of this topic.

The findings of Strategic Highway Research Program (SHRP) have shown⁹ that fundamental viscoelastic behavior of asphalts under different levels of stresses and temperatures need to be understood for performance-related specifications to address major pavement distresses. Equipments that provide the fundamental rheological information have a constraint in that they cannot easily be taken to the field or on-site, normally require highly trained operators and are also relatively much more expensive. On the other hand, rapid methods of rheological measurements normally give information on the consistency of the asphalts but do not provide all the fundamental rheological knowledge about the material.

An attempt to relate fundamental rheological data with a very simple, yet reasonably accurate and rapidly determinable rheological parameter was done through the development of unified curves for polymer-modified asphalts¹⁴ as well as unmodified asphalts¹⁵. Six asphalts were chosen from among the SHRP Materials Reference Library asphalts to serve as representatives during initial verification of the developed theory for unification¹⁵.

These were AAK-1, AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1. Their select properties are shown in Table 1. Though these asphalts cover a wide range of asphaltene content, a broad span of molecular weight, and a good spread of viscosity values, it is essential to validate the generality of the unified curves through a different set of asphalts under different temperature conditions.

The purpose of the present paper is to reinforce the unified curves through data analyses of a new set of asphalts. It is confirmed through the extra data analyses that the unified curves are universal for all unmodified asphalts at least within the studied temperature range of 46°C - 70°C. Since the validation has been done only in the temperature

range of 46°C - 70°C, the use of this work will be limited to high temperature applications. Assessment of the predictive ability of the unified curves is also done. The results show that the simple material's volumetric-flow rate (MVR) is able to give good predictions of the viscoelastic parameters by mere calculations from the unified curves for all unmodified asphalts. It is thus possible to now get all fundamental rheological information on the unmodified paving asphalts in the temperature ranges of 46°C - 70°C by determining the MVR using a simple flow-measuring device (FMD) rather than the dynamic shear rheometer (DSR).

There have been earlier efforts^{4,6} to generate viscosity data using a capillary rheometer. Initially⁴, experiments were conducted at a constant rate of strain but later⁶ a constant stress mode was employed for more rapid testing. The approach did not become popular because of the large shear stresses and strains that were produced during measurement. These could lead to spurious results under certain conditions of high stresses due to the elasticity of the material and in other cases, where experimentally correct data could be produced, the results could be outside the range of practical use because of the unreasonably high strain values. Moreover, their attempt^{4,6} was to generate the entire shear stress versus shear rate data through the capillary rheometer.

In the present case, though a capillary die is used for flow rate measurement, the data is limited to one single value of MVR that is carefully controlled to lie within restricted bounds, thereby keeping the shear rates within acceptable limits. Moreover, the MVR data is collected only under a limited range of dead load conditions, thereby putting an upper bound on the stress level. If at all there are any deficiencies in the measuring technique, these are automatically annulled because the MVR is only used as a normalizing parameter.

Background

Most of the details of the unification concepts are covered elsewhere^{15, 16}. However, a brief account is provided here in order to facilitate the understanding of the present work. In fact, all the salient features of the concepts are covered here so that the present work becomes self-sufficient and it becomes easier to digest the subject matter.

Simple Rheological Parameter

The simple parameter that is chosen to give a good measure of the rheological characteristics of the asphalt is the material's volumetric-flow rate (MVR) that is determined through a closely defined flow measurement device (FMD), whose main parts are shown in Figure 1. This equipment is borrowed from the polymer industry where it is routinely used to measure the melt flow index of polymers. The cylinder of the flow measurement device is made of hardened steel and is fitted with heaters, insulated, and controlled for operation at the required temperature. The thermocouple is buried inside the instrument's barrel. The thermocouple and the associated temperature control electronics are calibrated against NIST traceable temperature probes by the equipment manufacturer. The heating device is capable of maintaining the temperature at 10 mm above the die to within " 0.2°C of the desired

temperature during the test. The temperature of the barrel, from 10 mm to 75 mm above the top of the die, is maintained within " 1% of the set temperature (°C). All this is followed in strict compliance with the ASTM D1238 stipulations. The piston is made of steel and the diameter of its head is 0.075 " 0.015 mm less than that of the internal diameter of the cylinder which is 9.5 mm. Extrusion of the material is done through a hardened steel die with an internal diameter of 2.095 " 0.005 mm.

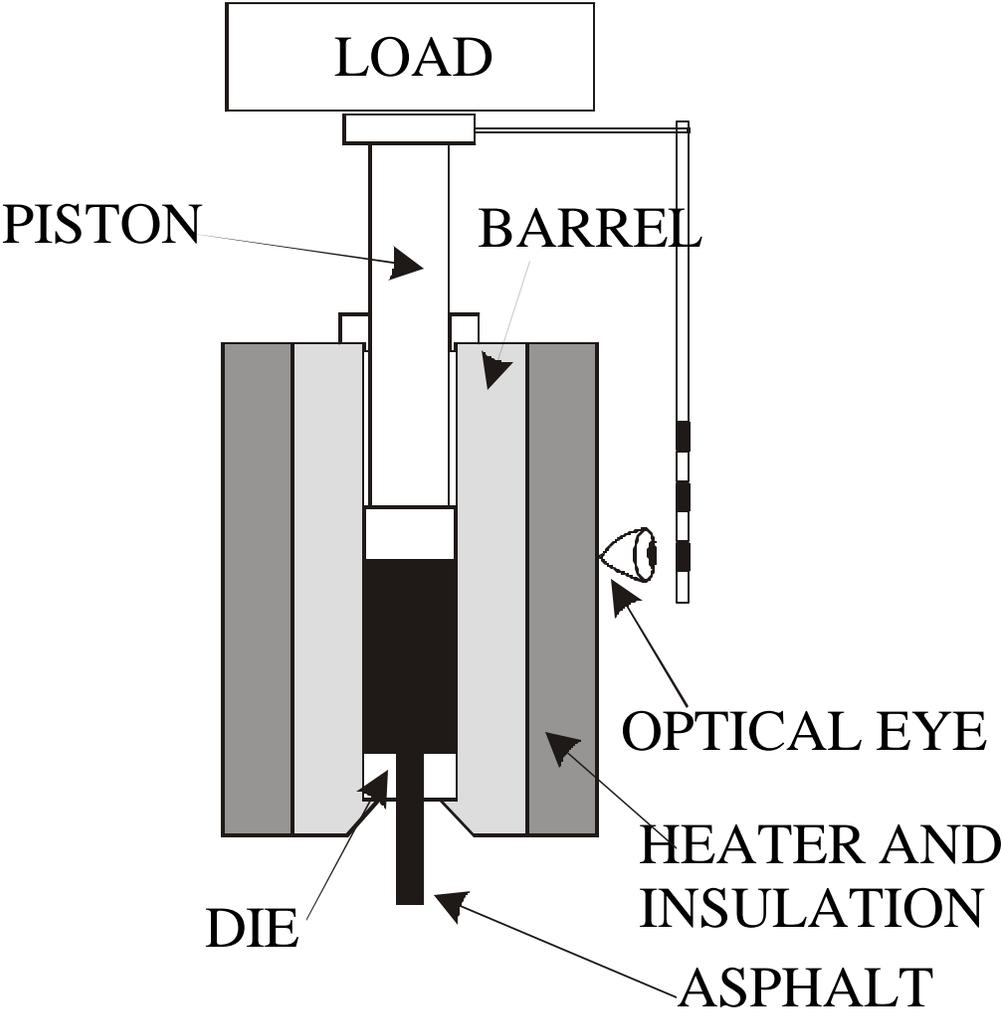


Figure 1 : Schematic diagram showing the main parts of the Flow Measurement Device (FMD) that is used for determining the Material's Volumetric-Flow Rate (MVR).

MVR

The MVR is defined as the volume of the material (in milliliters or cubic centimeters) that is extruded in 10 minutes through the die of specific diameter and length as described above by applying pressure through dead weight under prescribed temperature conditions. This definition is rather an arbitrary one. It has been chosen to be consistent with the well-known rheological parameter used in polymer melt rheology, namely, the melt flow index MFI¹⁶, except that MFI is the weight extruded in 10 minutes while MVR is the volume extruded in 10 minutes. The volume-flow rate is more convenient to measure than the mass flow rate and does not require the knowledge of the density of the material in the calculations. Possible sources of errors in MVR data could be due to a) kinetic energy effect, b) hydrostatic head of the fluid material above the die exit, c) time-dependency of the flow, d) entrance and end corrections, and e) effective wall slip. Correction terms to account for these errors are not included in the unification and the reasons for that are given elsewhere¹⁵.

Unification Theory

The equipment used for measurement of MVR as defined above falls in the category of a circular orifice rheometer¹⁶. Hence the expressions for shear stress J and shear rate u in this equipment can be written in the following well-known conventional forms:

$$J = \frac{R_N F}{2B R_P^2 l_N} \quad (1)$$

$$u = \frac{4 Q}{B R_N^3} \quad (2)$$

where nozzle radius $R_N = 0.105$ cm, piston radius $R_P = 0.4737$ cm, nozzle length $l_N = 0.8$ cm, force $F = \text{load } L \text{ (kg)} \times 9.807 \times 10^5$ dynes and the flow rate Q (cc/s) is related to MVR (cc/10min) as follows by definition.

$$Q = \frac{\text{MVR}}{600} \quad (3)$$

Since the geometry of the measuring equipment is fixed as given above, Equations (1) and (2) can be reduced to give the following:

$$J/L = 9.13 \times 10^4 = \text{constant} \quad (4)$$

and

$$u/MVR = 1.83 = \text{constant} \quad (5)$$

Note that the constant has only geometric values and no material properties.

When the MVR value is generated under a specific load condition for a particular grade of asphalt at a given temperature, the shear stress and shear rate values corresponding to those test conditions can be obtained from Eqs. (4) and (5). At that very temperature, by changing the applied load, a different value of MVR can be generated which corresponds to a new set of shear stress and shear rate values. In this way, it is possible to generate the shear stress versus shear rate curve for the asphalt at that temperature. This curve, which may be generated through the flow measurement device (FMD), should correspond to the shear stress versus shear rate curve generated from any rheometer because the response of the material to stress should be independent of the type of measuring equipment. Generating the full flow curve from the FMD is meaningless, when more sophisticated equipments are available. However, any curve generated from a sophisticated equipment would have a point on it which corresponds to the shear stress and shear rate values from the FMD as given by equations (4) and (5). Thus, if the shear stress J versus shear rate u data from any rheometer were replotted as J/L versus u/MVR , then for all samples, the curves should pass through the co-ordinates of $J/L = 9.13 \times 10^4$ and $u/MVR = 1.83$. This in effect, allows a vertical shift of the curves through division by L and a horizontal shift through division by MVR , thereby resulting in a superposition of the curves.

SHRP asphalt binder research findings have indicated that dynamic shear rheometer data are the preferred fundamental rheological properties of neat asphalts to relate to pavement performance for rutting at high temperatures. Hence, if a unification of rheological data is to be sought, then it is essential to develop a method to coalesce dynamic data in terms of the complex modulus $|G^*|$, loss modulus G'' , and parameter $|G^*|/\sin \delta$.

Earlier investigations of rheological properties (at least for polymer melts) have shown that the data under dynamic conditions can be related to that obtained under steady shear within certain ranges of shear rates and frequencies¹⁷⁻²². The empirical method suggested by Cox and Mertz¹⁸ for relating steady shear viscosity with the absolute value

of complex viscosity O^* gives

$$O(u) = |O^*(T)| \quad \text{at } u = T \quad (6)$$

The relationship simply indicates that for prediction purposes, the magnitude of the complex viscosity is equal to that of shear viscosity at equal values of radial frequency T and shear rate u .

The relationship has been found to largely hold for flexible-chain thermoplastic melts, particularly in the lower and intermediate ranges of u and T . It can be assumed that this relationship would hold well for paving asphalts because they too are thermoplastic materials.

Using the fact that $O = J/u$ and $|O^*| = |G^*| / T$ by definition, the following expression is written.

$$J/u = |G^*| / T \quad \text{at } u = T \quad (7)$$

Thus, the following equations corresponding to Eqs. (4) and (5) can be written using the Cox-Mertz rule.

$$|G^*| / L = \text{constant} \quad (8)$$

and

$$T/MVR = \text{constant} \quad (9)$$

Thus, if the $|G^*|$ versus T data from the DSR are replotted as $|G^*| / L$ versus T/MVR , then for all samples, the curves will pass through the point with co-ordinates given by $|G^*| / L = 9.13 \times 10^4$ and $T/MVR = 1.83$. This in effect, allows a vertical shift of the curves through a division by L and a horizontal shift through a division by MVR , thereby resulting in a superposition of the curves.

Now in order to establish the relationship which is likely to give a unified curve for the other dynamic functions such as the loss modulus G'' , the Spriggs model²⁰ has been chosen for correlating the dynamic and steady state rheological characteristics. Based on the Spriggs model²⁰, the loss modulus G'' which is the dynamic function is expressed as follows:

$$G' = \frac{O_0}{3} \frac{4}{8Z(a)^{p-1} p^2 \tilde{\omega} + (T/8)^2} \quad (10)$$

whereas the shear stress J which is the steady-state function is given as :

$$J = \frac{O_0}{3} \frac{4}{c 8Z(a)^{p-1} p^2 \tilde{\omega} + (c u/8)^2} \quad (11)$$

where O_0 , 8 , a and $Z(a)$ are model parameters and c is an arbitrary adjustable constant expressed in terms of an independent parameter ' as

$$c^2 = (2 - 2' - ' ^2) / 3 \quad (12)$$

Comparing Eqs. (10) and (11) yields the following :

$$J = c^{-1} G' \text{ at } T = c u \quad (13)$$

Thus, it is obvious from Eq. (13) that the dynamic loss modulus would be equivalent to the steady-state shear stress when shifted by an amount c . In order to determine the shift factor c , the procedure suggested by Spriggs²⁰ needs to be followed, namely, of superimposing the plot of $O(u)/O_0$ versus $c u$ on the plot of $O(T)/O_0$ versus T . For example, a value of $c = 1/2/3$ has been found by Saini and Shenoy²³ to correlate the dynamic and steady-state viscoelastic data for a particular grade of linear-low-density polyethylene over a wide range of frequencies and shear rates. Using Eq. (13) in Eqs. (4) and (5) the following equations can be written

$$G' / L = \text{constant} \quad (14)$$

and

$$T / MVR = \text{constant} \quad (15)$$

Eqs. (14) and (15) again imply that a plot of G' / L versus T/MVR on log-log scale should result in a unified curve, if arguments similar to those that were put forth earlier when discussing Eqs. (4) and (5) are followed. Knowing the interrelationships between various dynamic material functions, unified curves can be expected for $G' \tan \delta$, and even $|G^*|/\sin \delta$ given the following.

$$G' = (|G^*|^2 - G''^2)^{0.5} \quad (16)$$

$$\tan \delta = G'' / G' \quad (17)$$

$$|G^*| / \sin \delta = (|G^*|)^2 / G'' \quad (18)$$

The unified curves as defined above have a normalizing parameter for the y-axis (i.e., load), and another normalizing parameter for the x-axis (i.e., MVR). It would certainly be more beneficial to have the normalizing parameter all clubbed together on just one axis like the x-axis. This would make it easier to regenerate fundamental rheological information and also to derive useful conclusions from the unified curve. From Eqs. (8) and (9), the following equality can be written:

$$|G^*| / L = M \{T / MVR\} \quad (19)$$

This is equivalent to stating that

$$J / L = M \{u / MVR\} \quad (20)$$

where M represents a function. If the material is Newtonian in character then the relationship would be linear and one can write

$$J = O_0 u(L / MVR) \quad (21)$$

where O_0 is the proportionality constant. The Newtonian viscosity of the sample is equal to $O_0(L / MVR)$, and can be calculated using the L and MVR values from the FMD at the particular temperature of interest. Eq. (21)

would imply that a plot of J versus $u(L/MVR)$ would be unique and, therefore, in turn, a plot of $|G^*|$ versus $T(L/MVR)$ would also unify provided the material is Newtonian. Transferring the L term from the left-hand-side to the right-hand-side cannot be done in this simple manner if the material is non-Newtonian. Asphalts are known to possess non-Newtonian characteristics and hence the relationships in Eqs. (19) and (20) are not linear. The non-linearity can be taken into account by using the Oswald-de Waele power-law model²⁴⁻²⁶ within small ranges of shear rates and frequencies. Within these ranges, Eq. (20) can be written as follows:

$$J/L = K_0 (u/MVR)^n \quad (22)$$

where n is the power-law constant that is normally termed as the pseudoplasticity index or the shear susceptibility index and K_0 is the proportionality constant. The consistency index of the material is equal to $K_0 (L/MVR^n)$ and can be calculated using the L and MVR values from the FMD at the particular temperature of interest. Based on Eq. (22), the following relationships can then be written

$$J = M \{u (L^{1/n} / MVR)\} \quad (23)$$

and

$$|G^*| = M \{T (L^{1/n} / MVR)\} \quad (24)$$

Eq. (24) then implies that a plot of $|G^*|$ versus modified frequency $T (L^{1/n} / MVR)$ should then give a unique curve. It also follows that G' versus $T (L^{1/n} / MVR)$ and $|G^*| / \sin^*$ versus $T (L^{1/n} / MVR)$ would each give a unique curve taking into account the non-Newtonian behavior of the material system. Since the load L and the MVR value are available from the flow measurement, it is only now necessary to determine the n value in each case.

Since the J versus u curve and the $|G^*|$ versus T curve have continuously changing slopes for a non-Newtonian material, the value of n is constant only in short ranges of shear rates and frequencies. In that limited range, the power-law model can be fitted to the curve as follows:

$$J = K (u)^n \quad (25)$$

Note that this is not the unified curve that is being considered but the curve that could be generated by simply changing the load conditions in the FMD as discussed earlier during the development of Eqs. (4) and (5). In the present context, the value of n has to be chosen in the range of shear stress and shear rate which corresponds closely to those applicable to the MVR value that is used for the normalizing process. Hence, in case the MVR value has been determined at a load L for a particular asphalt sample at a specific temperature, then two more MVR values are to be determined at two other load conditions to estimate the value of n . The two load conditions are chosen in such a way that one is higher than L (i.e., say $L1$) while the other is lower than L (i.e., $L2$). In order to get the value of n , Eq. (25) is rewritten using Eqs. (4) and (5) as follows:

$$L1 = K (MVR1)^n \quad (26)$$

and

$$L2 = K (MVR2)^n \quad (27)$$

Solving Eqs. (26) and (27), the following equation can be written for the estimation of the value of n .

$$n = \log (L1/L2) / \log (MVR1/MVR2) \quad (28)$$

Experiments

The theoretical development in the previous section has shown the possibility of unifying fundamental rheological data through the use of the material's volumetric-flow rate (MVR). In order to verify this, systematic experiments are needed on a number of different asphalts that have widely different rheological characteristics. These asphalts have to be characterized on two different rheometers, one which gives the fundamental rheological properties in terms of the dynamic material functions and the other which gives the material's volumetric flow rate.

Equipment Used

(1) The Rheometrics Dynamic Shear Rheometer (DSR) was used for generating the dynamic data for the first set of asphalts at three different temperatures of 46°C, 58°C and 70°C and for the second set of asphalts at two different temperatures of 52°C and 64°C. All data were generated with a set of parallel plates of 25 mm diameter following the procedure given in the AASHTO provisional specifications²⁷. The samples for the test were prefabricated using a silicone rubber mold. To maintain a specific constant temperature, the samples were completely immersed in temperature controlled water that was circulated throughout the test by a pump-equipped water bath. The rheometer and the temperature-controlled unit were operated through a personal computer and the data acquisition / analysis was done using specialized software running under Windows 95.

The data were generated using a frequency sweep covering a range from 0.1 radians/s to 100 radians/s with 33 data points. It was essential to establish that the generated data is within the linear viscoelastic range of response. The values of the viscoelastic functions are independent of the applied stress amplitudes within the linear range of response, but the moduli begin to show a decrease with increasing stress when the response gets into the nonlinear range. In order to identify the border between the two regimes of response, a few experiments were initially conducted using different stress levels to watch the strain levels when the response changes from linear to nonlinear. The target strains were thus established and used in the frequency sweeps.

(2) The Kayeness Melt Indexer Model D4002 was used as the Flow Measurement Device (FMD) in order to measure the material's volumetric flow rate (MVR). The material's flow characteristic is assessed from the volumetric displacement with time based on the piston's downward movement. The piston's downward travel time is determined from a counter initiated by an optical sensor. The optical eye senses opaque flags on a transparent tape hung off the top of the piston rod. Flags of different lengths are available such as 1/8", 1/4", 1/2" and 1". Multiple flags are also available. In the present case, the transparent tape chosen was the one which had three 1/4" flags spaced at about 1/8" from each other. Such a multiple flag was advantageous to use because three readings for MVR could be obtained in one run of the sample. These may not be exact replicates as they are not taken on different samples. However, they are three measurements on the same sample and help to identify bad data.

The FMD has a built-in computer that can be programmed to set up the experimental conditions. The temperature of MVR measurement and the load conditions are input into the system. While the temperature of the FMD begins to rise towards the set temperature, the asphalt for testing is heated in the oven to a temperature of 163°C so that it is in a pourable condition. Approximately 10 gms of asphalt are gradually poured in a thin continuous stream into the barrel of the FMD and the piston is put in place. The asphalt is then allowed to equilibrate with the set temperature.

This takes from 10 to 15 minutes depending upon the set temperatures, the temperature of the poured asphalt and the quantity of the asphalt that finally sits in the barrel. When the set temperature is reached, the buzzer sounds a signal and shows that the FMD is ready for MVR measurement. At this stage, the predecided weight is placed on the piston and the flag with three black strips is placed on the extending piston arm.

Asphalt begins to flow out of the die as soon as the load is placed. At that stage, the RUN signal is given to the FMD from the main panel of the equipment. Even though the run signal is given, the equipment does not start taking MVR readings until the first scribed mark on the piston is reached, which coincides with the point at which the optical eye sees the first flag. It takes about 8 to 10 minutes for the scribed mark to reach the point when the measurement starts. This time is, of course, variable and can be shortened by pouring less asphalt into the barrel. In the present case, this time is maintained at a value between 8 to 10 minutes because the poured asphalt material was always around 10 gms. Once the optical eye sees the first flag, the MVR is automatically determined sequentially for all three flags. It takes a few seconds for each flag to pass the optical eye. This time is also variable, because it is dependent upon the viscosity of the asphalt. Lower viscosity asphalts flow in shorter times. The flow time also decreases with increasing loads. In the present case, the MVR data was taken under such load conditions as to maintain the MVR values to be between 1 and 50 in most cases.

The three MVR values corresponding to the three flags are automatically recorded by the FMD and then sent to a printer for final printout. The remnant material in the barrel after the MVR readings are recorded is allowed to drain out through the die. This takes about 2 to 5 minutes after which the load, the flag strip, and the piston are removed. The capillary die is removed from the equipment, dipped in a solvent, and cleaned thoroughly using cotton swabs and toothpicks. The piston and barrel are also cleaned with cotton swabs tied to specially designed plungers. The entire cleaning process takes about 5 minutes.

Materials Used

Five asphalts were chosen from among the SHRP Materials Reference Library asphalts to serve as representatives during initial verification of the developed theory for unification¹⁵. These were AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1. For verifying the generality of the unified curves, five more asphalts are chosen from among the SHRP Materials Reference Library. These were AAA-2, AAB-2, AAC-2, AAD-2 and AAF-2. The select properties of the first and second set of asphalts are shown in Table 1.

The asphalts from the dash1 series and the dash2 series together cover a wide range of asphaltene content, a broad span of molecular weight, and a good spread of viscosity values. The samples were each tested in their original unaged form and then again after aging using the rolling thin film oven test (RTFOT) at 163°C for 85 minutes and in the pressure aging vessel (PAV) at 100°C for 20 hours in accordance with the AASHTO provisional standard procedure²⁸.

TABLE 1 : Selected Details about the Asphalts Used

Asphalt ID	Source	Viscosity@140F, poise	Asphaltenes, %	W.Av.Mol.Wt., daltons
<i>Set 1</i>				
AAA-1	Lloydminster	864	16.2	790
AAB-1	WY Sour	1029	17.3	840
AAD-1	California	1055	20.5	870
AAF-1	W Tx Sour	1872	13.3	840
AAM-1	W Tx Inter	1992	4.0	1300
<i>Set 2</i>				
AAA-2	Lloydminster	363	16.2	---
AAB-2	WY Sour	403	16.7	---
AAC-2	Redwater	304	9.8	870
AAD-2	Coastal	600	21.3	---
AAF-2	W Tx Sour	867	13.0	---

Unified Curves

In order to generate unified curves, fundamental rheological properties are obtained on the DSR and the MVR values are got from the FMD on each of the samples. Since the asphalts were expected to be non-Newtonian in character, the MVR data was obtained at more than one load condition in order to estimate the value of n. The load, MVR and n values are shown in Tables 2-4 for *Set 1* (AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1 asphalts) in the original unaged as well as RTFOT / PAV aged forms, respectively.

TABLE 2 : Load, MVR and n data for Original Unaged Samples

Asphalt ID	Temperature, ⁰ C	Load,kg	MVR,cc/10min	n
<i>Set 1</i>				
AAA-1	46	2.16	15.71	0.981
	58	2.16	77.05	0.932
	70	0.32	43.62	0.971
AAB-1	46	3.06	13.26	0.912
	58	3.06	75.31	1.080
	70	0.32	35.86	0.978
AAD-1	46	5.00	24.56	1.000
	58	2.16	60.04	1.000
	70	1.00	101.53	1.000
AAF-1	46	5.00	7.64	1.060
	58	2.16	31.41	0.968
	70	1.22	75.27	1.030
AAM-1	46	2.38	6.29	0.893
	58	2.16	35.93	0.936
	70	0.32	23.20	0.984
<i>Set 2</i>				
AAA-2	52	1.00	43.80	0.984
	64	1.00	172.90	0.972
AAB-2	52	1.00	30.28	0.959
	64	1.00	135.67	1.030
AAC-2	52	1.00	29.00	0.981
	64	1.00	130.83	0.983
AAD-2	52	1.00	27.76	0.955
	64	1.00	110.17	0.959
AAF-2	52	1.00	12.98	0.925
	64	1.00	60.81	1.113

TABLE 3 : Load , MVR and n data for RTFOT Aged Samples

Asphalt ID	Temperature, ⁰ C	Load,kg	MVR,cc/10min	n
<i>Set 1</i>				
AAA-1	46	7.06	20.47	0.901
	58	1.00	15.23	0.938
	70	1.00	67.69	0.984
AAB-1	46	5.00	9.48	0.861
	58	3.06	36.81	0.924
	70	1.00	52.94	0.986
AAD-1	46	5.00	10.62	0.859
	58	1.00	10.34	1.020
	70	0.32	13.32	0.912
AAF-1	46	10.00	6.67	0.834
	58	5.00	26.58	1.020
	70	2.16	63.51	1.040
AAM-1	46	7.06	9.24	0.742
	58	3.06	25.54	0.871
	70	1.00	35.09	0.975
<i>Set 2</i>				
AAA-2	52	1.00	18.99	0.954
	64	1.00	80.74	0.967
AAB-2	52	1.00	11.85	0.920
	64	1.00	66.72	0.961
AAC-2	52	1.00	12.27	0.957
	64	1.00	65.78	1.011
AAD-2	52	2.16	23.16	0.890
	64	1.00	46.82	0.943
AAF-2	52	5.00	27.44	1.007
	64	1.00	31.36	1.034

TABLE 4 : Load, MVR and n data for PAV Aged Samples

Asphalt ID	Temperature, ⁰ C	Load,kg	MVR,cc/10min	n
<i>Set 1</i>				
AAA-1	46	10.00	6.64	0.691
	58	2.16	7.73	0.887
	70	1.00	16.32	0.864
AAB-1	46	10.00	4.32	0.671
	58	3.06	8.63	0.877
	70	1.00	14.16	0.954
AAD-1	46	10.00	4.73	0.716
	58	5.00	14.24	0.725
	70	2.16	26.34	0.691
AAF-1	46	10.00	1.07	0.752
	58	10.00	13.36	0.793
	70	2.16	16.06	0.899
AAM-1	46	10.00	3.80	0.600
	58	3.06	6.33	0.773
	70	2.16	22.06	0.818
<i>Set 2</i>				
AAA-2	52	5.00	20.15	0.799
	64	2.16	44.91	0.886
AAB-2	52	10.00	28.82	0.636
	64	5.00	68.39	0.666
AAC-2	52	2.16	4.63	0.844
	64	2.16	36.98	0.925
AAD-2	52	10.00	21.01	0.698
	64	2.16	17.63	0.801
AAF-2	52	10.00	9.58	0.850
	64	2.16	13.47	0.944

The collective data on all the considered asphalts from *Set 1* were plotted as $|G^*|$ versus $T(L^{1/n}/MVR)$, G^* versus $T(L^{1/n}/MVR)$ and $|G^*|/\sin^*$ versus $T(L^{1/n}/MVR)$ and found to give unified curves. Only plots of $|G^*|/\sin^*$ versus $T(L^{1/n}/MVR)$ are shown in Figures 2 in order maintain brevity. In fact, all through this paper only the unified curves for the SHRP parameter $|G^*|/\sin^*$ are shown, though in practice, unified curves were established for $|G^*|$ and G^* in all cases and these were used for getting the unified curves of $|G^*|/\sin^*$ based on Eq. 18.

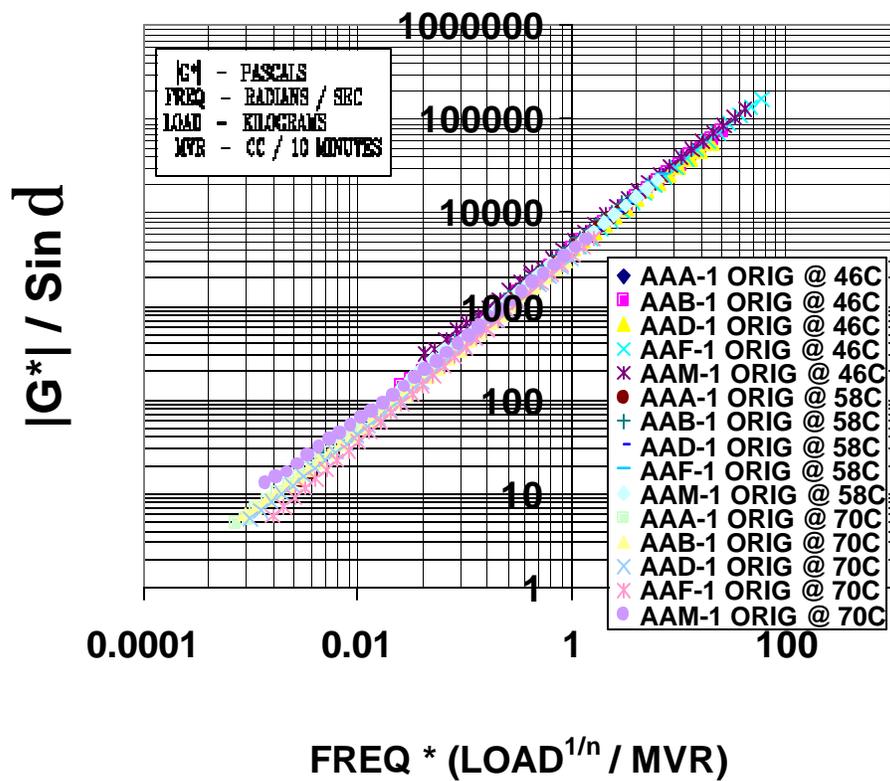


Figure 2(a) : Unified curve of the SHRP parameter $|G^*| / \sin d$ with modified frequency $T(L^{1/n}/MVR)$ covering the temperature range of 46°C - 70°C for five asphalts (AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1) each in original unaged forms.

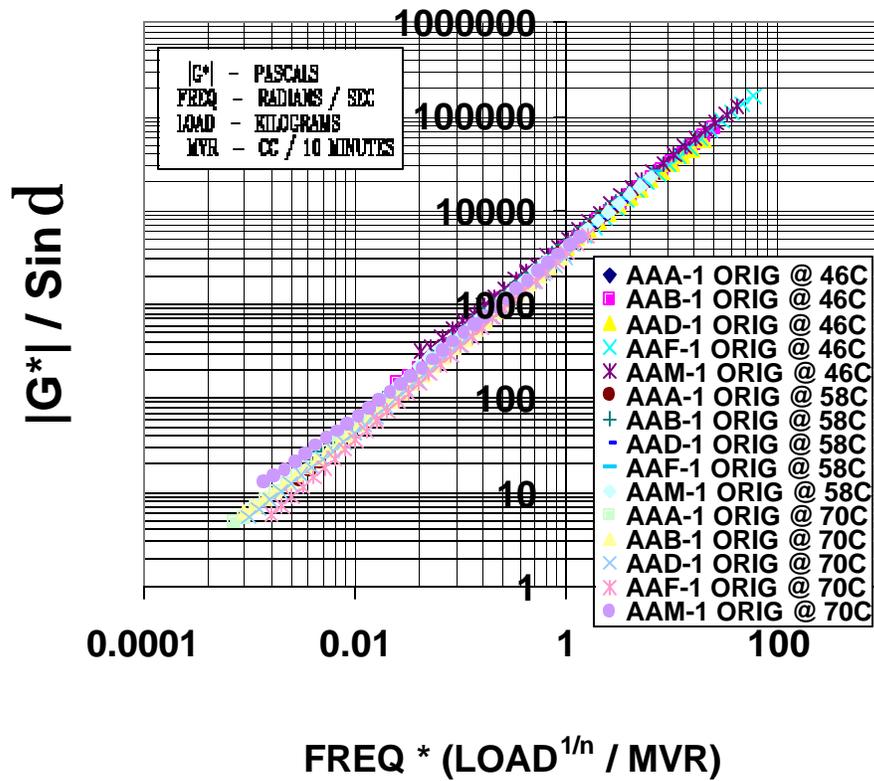


Figure 2(b) : Unified curve of the SHRP parameter $|G^*| / \sin d$ with modified frequency $T(L^{1/n}/MVR)$ covering the temperature range of 46°C - 70°C for five asphalts (AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1) each in RTFOT aged forms.

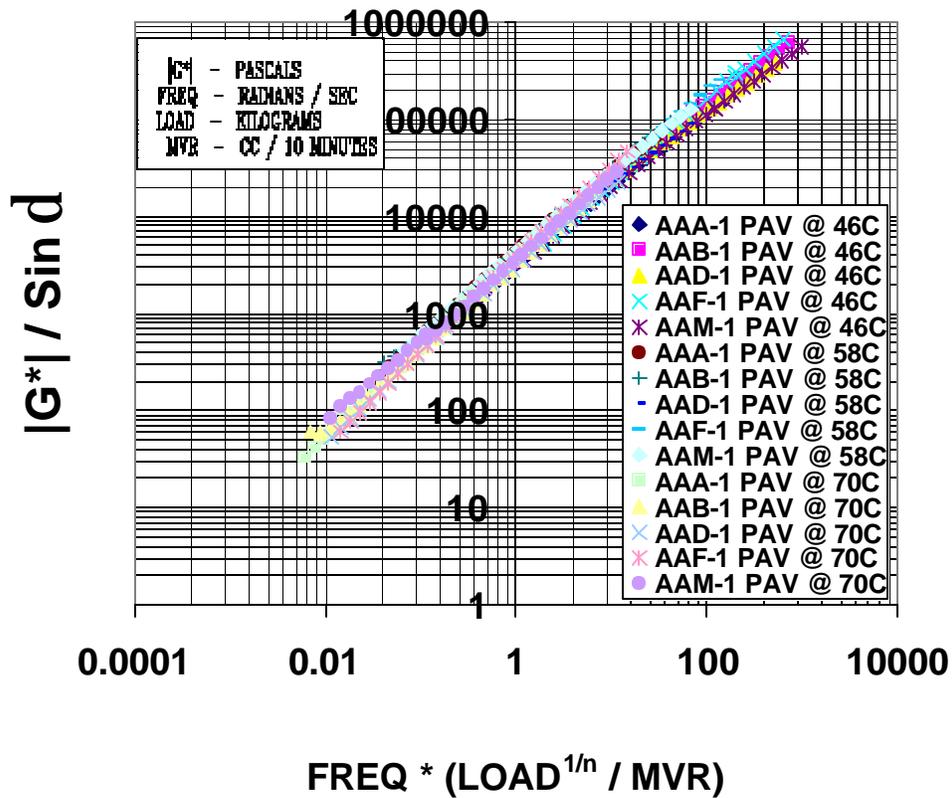


Figure 2(c) : Unified curve of the SHRP parameter $|G^*| / \sin d$ with modified frequency $T(L^{1/n}/MVR)$ covering the temperature range of 46°C - 70°C for five asphalts (AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1) each in PAV aged forms.

It can be seen that the curves in Figures 2 (a)- (c) are unique and independent of the type of asphalt as well as the temperature of measurement. Each unified curve has a total of 495 data points (i.e., 99 data points for each of the five asphalts: AAA-1, AAB-1, AAD-1, AAF-1 and AAM-1). Different unified curves have been shown at different levels of aging, i.e. (a) original unaged, (b) RTFOT aged and (c) PAV aged. This was done on account of two reasons. Firstly, to avoid clogging too much data on one curve and secondly because there was a slight but noticeable difference in PAV aged unified curve which would be otherwise lost if all data were plotted on one curve. It is not clear whether this is an experimental artifact or whether truly the unified curve is different for PAV aged samples.

In Figures 2 (a) - (c), only the dash1 grades of various asphalts were considered. It was important to check whether the rheological data of dash2 grades fall on a unified curve as well and whether there was truly a single curve for the dash1 and dash2 grades. To confirm this, DSR data were generated for asphalts AAA-2, AAB-2, AAC-2, AAD-2 and AAF-2. For each of these asphalts, MVR data were also obtained and the values of n were estimated as well. These are shown in Tables 24 under *Set 2*. Data were generated at two temperatures 52°C and 64°C. It is important to note that these two temperatures (52°C, 64°C) were chosen to be different from the ones (46°C, 58°C, 70°C) that were used for dash1 grades under *Set 1* in Tables 2-4.

The primary check was to confirm that all the dash2 grades of asphalt formed a unified curve.

The collective data on all the considered asphalts from *Set 2* were plotted as $|G^*|$ versus $T(L^{1/n}/MVR)$, G'' versus $T(L^{1/n}/MVR)$ and $|G^*|/\sin \delta$ versus $T(L^{1/n}/MVR)$ and found to give unified curves. The plot of $|G^*|/\sin \delta$ versus $T(L^{1/n}/MVR)$ is shown in Figures 3 (a) - (c).

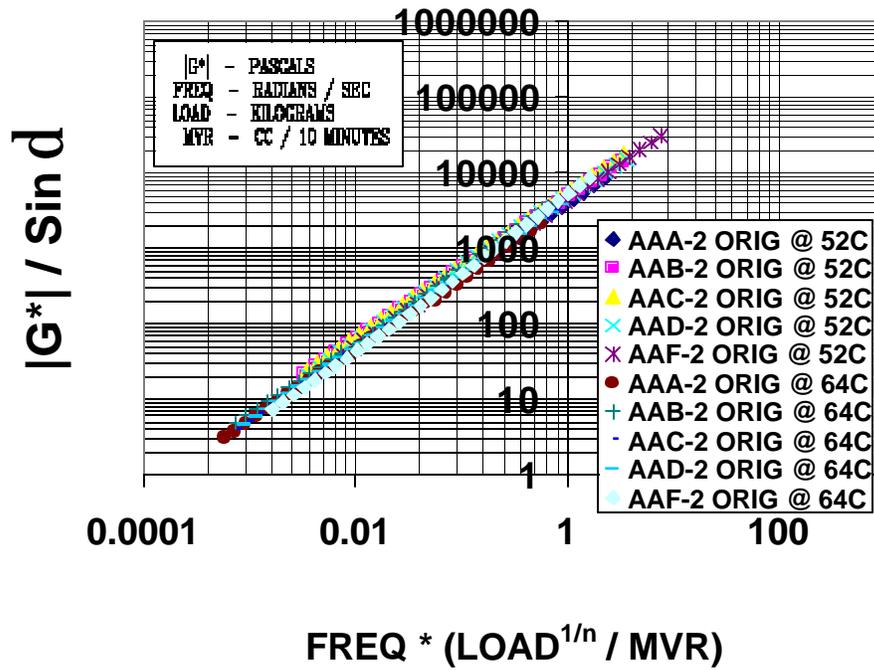


Figure 3(a) : Unified curve of the SHRP parameter $|G^*| / \sin \delta$ with modified frequency $T(L^{1/n} / MVR)$ at temperatures of 52°C and 64°C for five asphalts (AAA-2, AAB-2, AAC-2, AAD-2 and AAF-2) each in original unaged form.

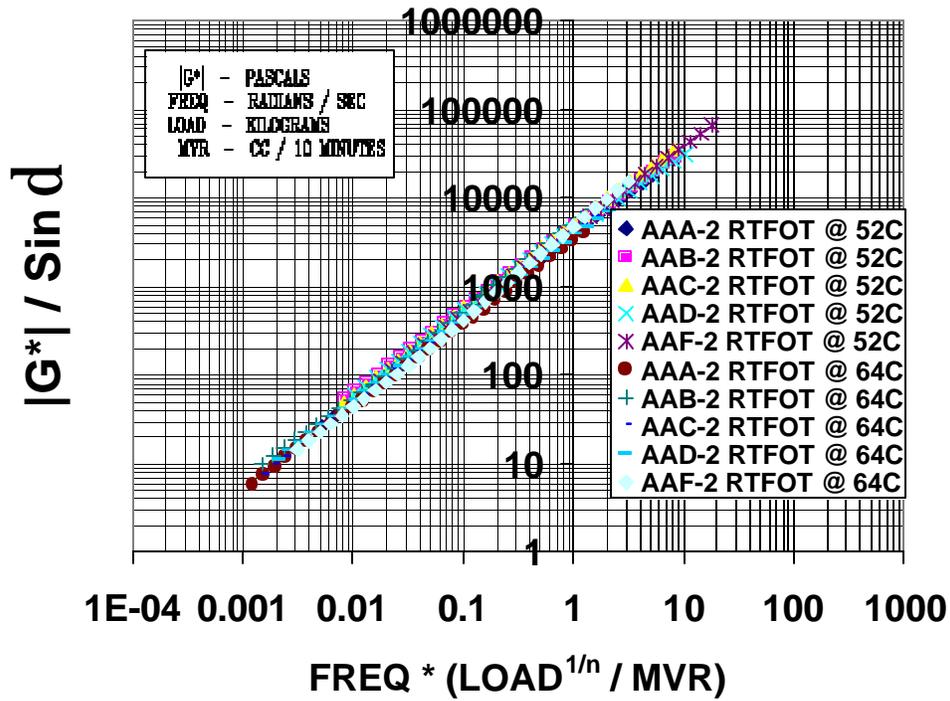


Figure 3(b) : Unified curve of the SHRP parameter $|G^*| / \sin \delta$ with modified frequency $T(L^{1/n} / MVR)$ at temperatures of 52°C and 64°C for five asphalts (AAA-2, AAB-2, AAC-2, AAD-2 and AAF-2) each in RTFOT aged form.

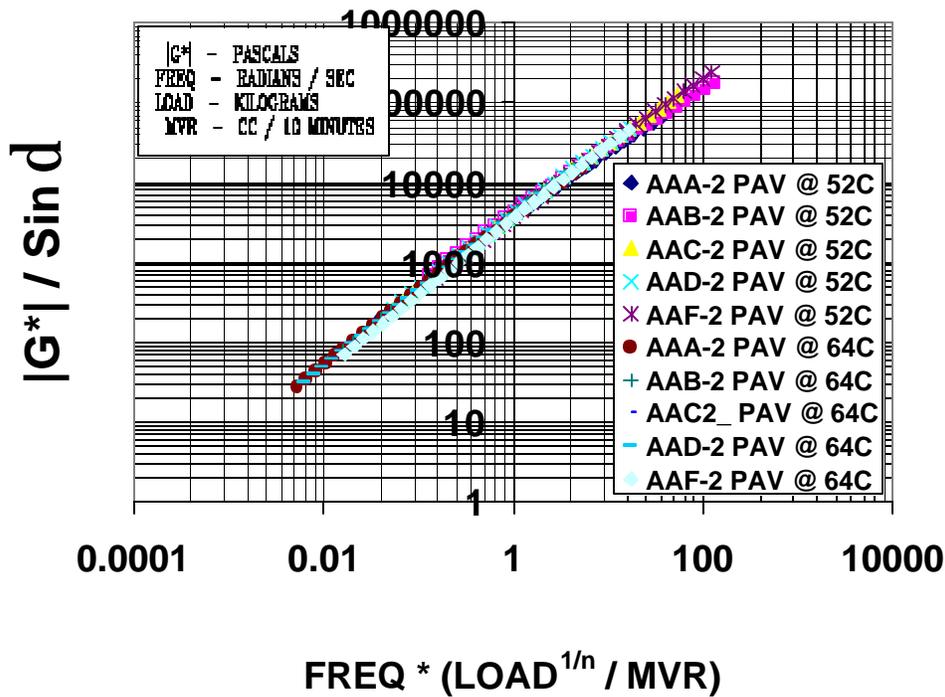


Figure 3(c) : Unified curve of the SHRP parameter $|G^*| / \sin \delta$ with modified frequency $T(L^{1/n} / \text{MVR})$ at temperatures of 52°C and 64°C for five asphalts (AAA-2, AAB-2, AAC-2, AAD-2 and AAF-2) each in PAV aged form.

The other check was to see whether the dash1 and dash2 grades of asphalt from one source formed a unified curve. In order to view this, AAB-1 data at 46°C, 58°C and 70°C were plotted along with AAB-2 data at 52°C and 64°C as shown in Figure 4. When other pairs of dash1 and dash2 were considered, results were similar and hence have not been shown here.

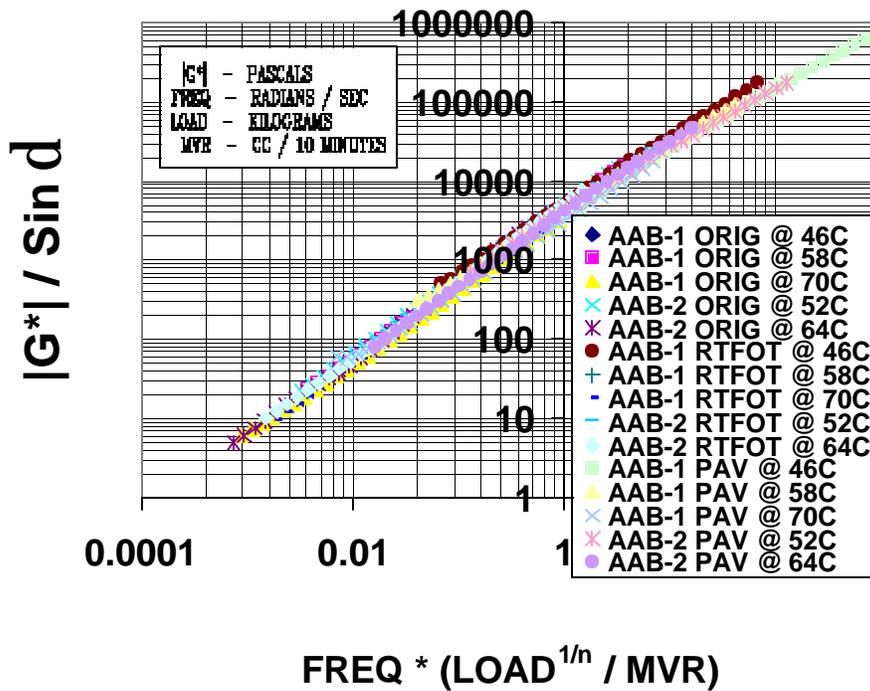


Figure 4 : Unified curve of the SHRP parameter $|G^*| / \sin \delta$ with modified frequency

$T(L^{1/n} / MVR)$ at temperatures of 46°C, 58°C and 70°C for asphalt AAB-1 as well as at temperatures of 52°C and 64°C for asphalt AAB-2, each in original unaged and (RTFOT & PAV) aged forms.

Figures 2-4 confirm that the unification technique works for at least ten different asphalts. A closer look at the selection of the ten asphalts indicates that they span a wide range of viscosity levels, have varied asphaltene content, varied wax content as well as high to low molecular weights. When such a wide spectrum of differences can be unified by the suggested approach, it may not be too unrealistic to expect that all unmodified asphalts would fall on the same curve.

The unified curves show a band within which all the data points coalesce. An estimate¹⁵ of the bandwidth gave an error bound range of 3-16%, most of which was a reflection of the errors in the original DSR data. Reinforcement of the curves with more data and further refinement of the curves to reduce the error bounds can be accomplished to finally establish the decisive shapes of the unified curves. The main accent of the present work was essentially to establish the unification technique and suggest the methodology that needs to be followed in order to achieve proper unification based on good theoretical foundations, and this could be achieved through the analysis of rheological data on the ten chosen asphalts. The unified curves of fundamental rheological data for all unmodified asphalts have many advantages. The beneficial implications of this unification have been discussed elsewhere¹⁵.

Predicting Viscoelastic Data from Unified Curves

The idea behind developing unified curves is to get a method for predicting viscoelastic parameters without actually generating DSR data. In order to check whether this is possible, the following steps need to be followed. From Figures 2-4, the average values of $|G^*| / \sin \delta$ at different values of modified frequency $T(L^{1/n} / MVR)$ are obtained. Using these values, the unified curve is drawn in Figure 5, from which it is now possible to estimate $|G^*| / \sin \delta$ values at different frequencies for any unmodified asphalt at any temperature between $46^{\circ}\text{C} - 70^{\circ}\text{C}$ when the MVR value at a convenient load L and the parameter n are known.

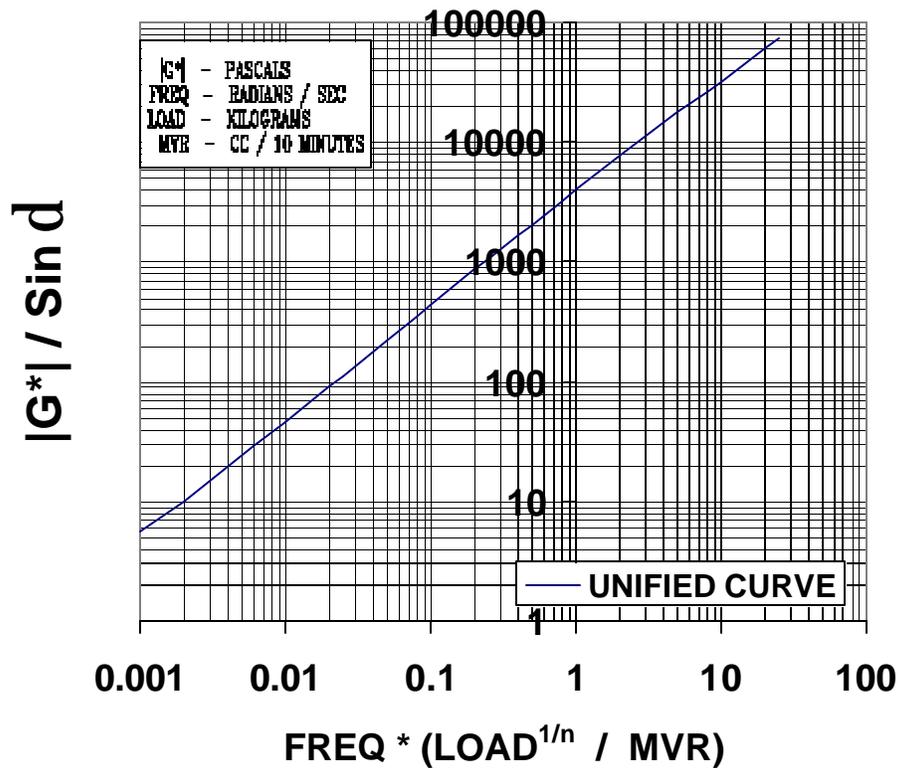


Figure 5 : Unified curve of the SHRP parameter $|G^*| / \sin \delta$ with modified frequency

$T(L^{1/n} / MVR)$ at temperatures of 46°C - 70°C for unmodified original unaged asphalts.

In order to check the predictability, unmodified asphalt AAF-2 is chosen. It should be noted that this asphalt was not used when the unified curve was formed in the first place in Figure 5. Asphalt AAF-2 is a West Texas Sour with a viscosity of 867 poise @ 140°F and has about 13% asphaltenes.

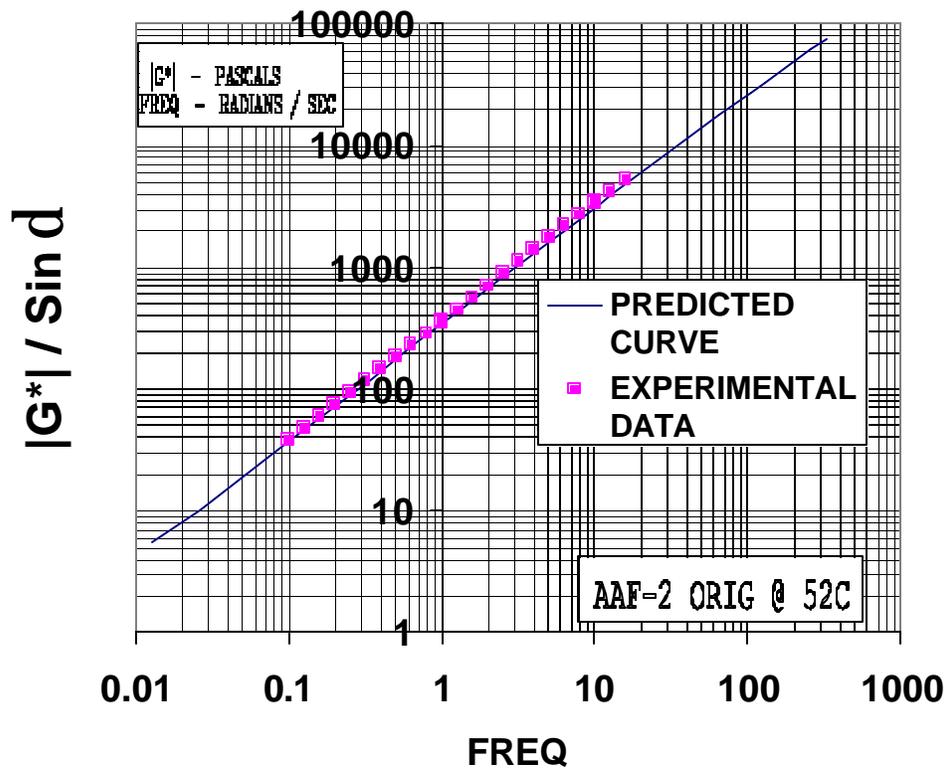


Figure 6 : Comparison of the predicted curve and experimental data for the SHRP parameter $|G^*| / \sin \delta$ with frequency ω at temperature of 52°C for unmodified original unaged asphalt AAF2.

The test temperature is chosen as 52°C . At this temperature, for original unaged AAF-2, the MVR is determined to be $12.97 \text{ cm}^3/10\text{min}$ at load $L=1 \text{ kg}$ and the value of n is determined from two other MVR measurements as equal to 0.925. The value of $(L^{1/n} / \text{MVR})$ is calculated as 0.077. Using this value in Figure 5, the variation of SHRP parameter is calculated and shown by the solid lines in Figure 6. Actual DSR data are plotted on the same figures and the match between predicted curve and experimental data is seen to be good.

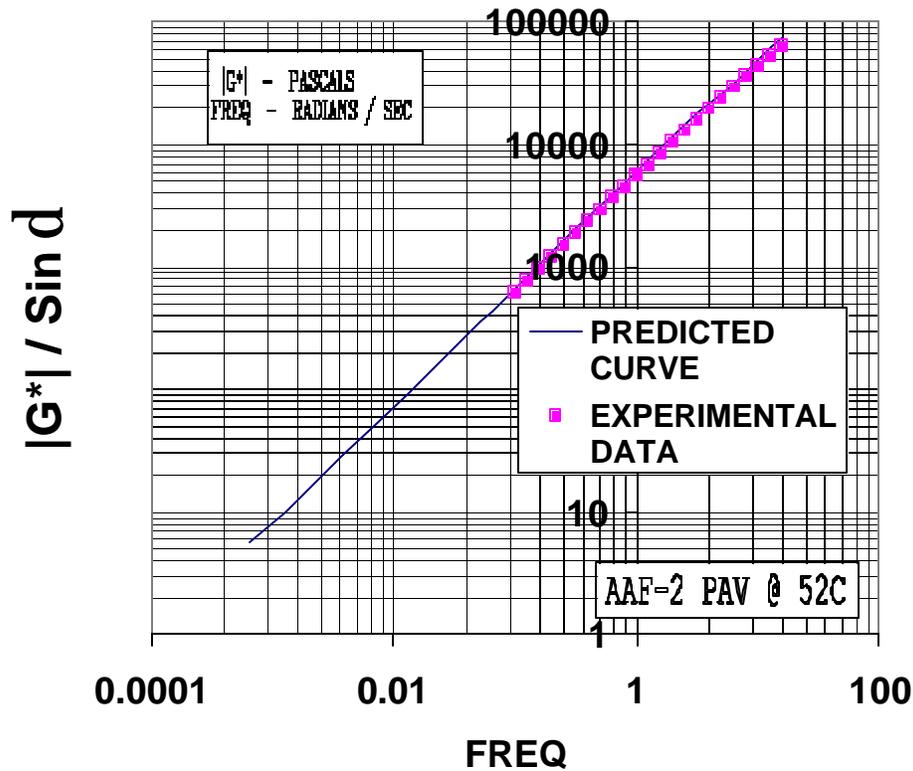


Figure 7 : Comparison of the predicted curve and experimental data for the SHRP parameter

$|G^*| / \sin \delta$ with frequency ω at temperature of 52°C for unmodified PAV aged asphalt AAF2.

A similar exercise is carried out for PAV-aged asphalt AAF-2 at 52°C. The MVR is determined to be 9.58 cm³/10min at load L=10 kg and the value of n is determined as equal to 0.85. The value of $(L^{1/n} / MVR)$ is calculated as 1.566. Following the same procedure, again it can be seen that the match between predicted curve and experimental data is excellent in Figure 7.

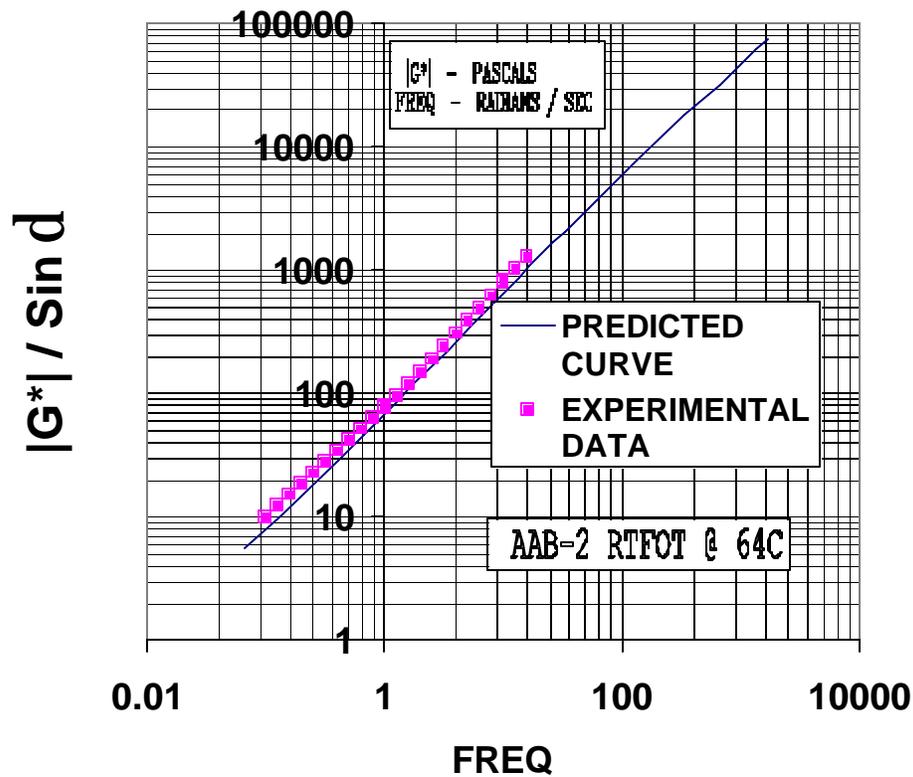


Figure 8 : Comparison of the predicted curve and experimental data for the SHRP parameter

$|G^*| / \sin \delta$ with frequency ω at temperature of 64°C for unmodified RTFOT aged asphalt AAB2.

For confirmation of predictability, the same calculations are done for asphalt AAB2 that was RTFOT-aged. This time the test temperature is chosen as 64°C. For this case the MVR value is determined to be 66.72 cm³/10min at load L=1 kg and the value of n is determined as equal to 0.96. The value of $(L^{1/n} / MVR)$ is calculated as 0.015. Again, the match between the predicted curve and experimental data can be seen to be excellent from Figure 8.

Concluding Remarks

The unification of fundamental rheological data for unmodified asphalts provides a rather powerful tool to reduce subsequent experimentation and to ease the generation of rheological information in the future. The FMD that is used for the generation of MVR data is a relatively simple and inexpensive piece of equipment and can be carried from place to place because of its relative light weight. It neither needs any arrangements for air pressure nor requires a circulating water-bath to maintain a constant temperature environment. Since this equipment was originally built for taking polymer melt data at high temperatures (125°C - 300°C), it has an excellent temperature control system with variations of about 0.1°C, especially in the temperature range applicable to paving asphalts. When generating MVR data from the FMD, a few important points should be borne in mind in order to assure accuracy.

- 1) The barrel, piston and die of the FMD should be meticulously cleaned before every measurement. Cotton swabs dipped in mineral spirits can be used for the barrel and the piston for scrubbing out the residual asphalt. The die can be dropped into a bowl of mineral spirits for about two minutes and then cleaned with a toothpick dipped in mineral spirits. The cleaning process takes no more than five minutes.
- 2) When pouring hot asphalt into the barrel, care should be taken to pour in a thin uniform stream so that no air pockets are formed due to jerky filling. When air gets trapped in asphalt due to faulty pouring, the asphalt will not flow uniformly out of the die. In fact, an audible sound of a burst bubble will be heard when there is a discontinuity in the flow. Any reading taken during the time when such a sound is heard must be discarded, as it is erroneous. Based on the present experimental experience during generation of MVR data for asphalts considered herein, it can be said that the air entrapment may happen no more than 2% of the time. However, it is worth being aware of this in order to distinguish spurious readings from good ones.
- 3) The total time for experiment is quite small. Care should be taken to maintain the MVR value between proper limits by a judicious choice of the load condition. Note that the testing time for $1.3 < \text{MVR} < 50$ lies between 0.08 and 3.3 minutes, which is quite reasonable. It was found that for $\text{MVR} = 0.2$, the testing time was about 20 minutes and for $\text{MVR} = 100$, it was about 0.045 minutes using a Flag of 6.35 mm. In the former case, the flow is too slow while in the latter case the flow is too fast. Hence, it is recommended that the load condition should be chosen in such a way as to get MVR value between 1 and 50 when the Flag of 6.35 mm is used. By opting for a different Flag, these limits can be relaxed to a certain extent (for example, for $\text{MVR} = 22$ the flow time of about 0.2 minutes with Flag of length 6.35 mm can be increased to 0.8 minutes by using a Flag of length 25.4 mm).

If the above points are borne in mind, it will be found that MVR data generated from the FMD is very highly reproducible¹⁵. In fact, in terms of repeatability of data, the FMD was found¹⁵ to perform better than DSR. There are a number of other benefits in using the FMD. The evaluation of the benefits of the FMD is to be based on a number of factors such as initial equipment cost, operational cost, maintenance cost, operator training requirements, experimental ease, testing time, specimen size, variability of output, data reduction method, information obtained, and mobility or portability. The FMD beats out DSR on all counts except specimen size and the information obtained as can be seen from Table 5. While from the DSR, all fundamental rheological parameters can be obtained, the FMD can only give a single value measurement of MVR at a fixed load condition. However, in the foregone text, it has been shown how this single value can be correlated with each of the fundamental rheological parameters from the DSR. This new unification technique thus upgrades the simple flow rate parameter to a level of high utility. The fact that the FMD is a relatively inexpensive equipment, operational costs are low, MVR data generation requires minimal training and the output has low level of variability, and the equipment is portable could merit its use at the paving sites or at the refineries. The actual time for data generation is also very low and this makes the measurement particularly attractive.

TABLE 5 : Comparison between DSR and FMD

Considered Feature	DSR	FMD
Initial Equipment Cost	\$ 35000	U \$5500*
Operational Cost	Power supply Air pressure Circulating water bath	U Power supply only No air pressure No circulating water bath
Maintenance Cost	Complex Electronics Very high repair cost Expensive replacement parts Need for maintenance contract	U Simple Electronics Little that can go wrong At best, die may need replacement (\$60 each) No need for maintenance contract
Operator training	Complete day training is required in order to minimize errors during operation	U Minimal or almost nil training is needed, thereby reducing the chances of error during operation
Experimental Ease	Not very easy to guess the strain levels to be used in a frequency sweep and hence extra data needs to be collected to make sure that obtained information is in the linear viscoelastic region	U Not at all difficult to guess the load conditions in order to achieve MVR values between 1 and 50 to get most accurate data
Sample Preparation Time	U Heating the asphalt to 163°C and pouring in the silicone mold and cooling (about 5 minutes)	U Heating the asphalt to 163°C and pouring into the barrel (about 5 minutes)

* Melt Flow Indexer Model D4002 from Kayeness (Morgantown, PA)

TABLE 5 : Comparison between DSR and FMD (Continued)

Considered Feature	DSR	FMD
Testing Time	Takes a long time to reach equilibrium temperature and the entire data generation takes over an hour without taking into account the time for establishing that the data is in linear viscoelastic range	U Equilibrates very quickly at all temperatures and the entire data generation takes only about 15 minutes (See Table 5 for details)
Specimen Size	U 1 gm (For one set of data)	10 gms (For three sets of data)
Variability of Output (% STD / AVG)	6 to 12	U 0.2 to 2.0
Data reduction method	Requires a computer and Windows '95 to run the software for calculation of various rheological functions	U Requires no computer as all calculations are done by the built-in software which does not require any other support program either
Information Obtained	U Extensive information on the basic rheological properties of asphalts can be got in terms of $ G^* $, G' , G'' etc.	U Only a single value of MVR at a fixed load L condition can be got. But this value can be related to all fundamental rheological properties generated from DSR
Mobility or Portability	Very heavy, Requires air pressure, Needs circulating water-bath and hence is not portable.	U Relatively light weight, Requires no air pressure, Needs no water-bath and thus is portable.

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