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Developing unified rheological curves for polymer-modified asphalts – Part I. Theoretical analysis

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ABSTRACT

Rheological data for polymer-modified asphalts are obtained by measurements on the dynamic shear rheometer (DSR) and expressed in terms of |G∗|, G″ and |G∗| / Sin δ versus frequency curves. In each case, the material's volumetric-flow rate MVR (in cc / 10 minutes) through a predefined die under conditions of constant temperature and stress can be obtained for the polymer-modified asphalts using a simple flow measurement device (FMD). A theoretical relationship between the fundamental rheological properties got from the DSR and the MVR obtained from the FMD shows that unified curves of viscoelastic properties of polymer-modified asphalts can be obtained within a determined temperature range.

RÉSUMÉ

Les données rhéologiques pour les bitumes modifiés par des polymères sont obtenues par mesure sur Rhéomètre à Cisaillement Dynamique (DSR) et exprimées en terme de courbes |G∗|, G″ et |G∗| / Sin δ en fonction de la fréquence. Pour chaque cas, le débit volumétrique du matériau MVR (en cc / 10 minutes), à travers un capillaire prédéfini, dans des conditions de température et de contrainte constantes, peut être obtenu pour les bitumes modifiés par des polymères en utilisant un système simple de mesure du débit (FMD). Une relation théorique entre les propriétés rhéologiques fondamentales obtenues d'une part à l'aide du DSR et d'autre part à partir du MVR montre que les courbes unifiées des propriétés viscoélastiques des bitumes modifiés par des polymères peuvent être obtenues dans un domaine de température déterminé.

1. INTRODUCTION

Asphalts in road paving applications are often modified [1, 2] through the use of small amounts (4-8 wt %) of polymers and there are more than 20 listed reasons [2, 3] for such modification. Whatever be the reason for the addition of polymers to asphalts, their presence changes the rheological characteristics of the original base asphalt. The rheology of polymer-modified asphalts has received considerable attention over the years [4-34].

Based on the findings of SHRP [35, 36] (a five-year $150 million dollar United States research effort established and funded under the 1987 Surface Transportation and Uniform Relocation Assistance Act), it was concluded that fundamental viscoelastic behavior of asphalts under different levels of stresses and temperatures needs to be understood for performance-related specifications to address major pavement distresses.

The 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. Rapid rheological measurements, on the other hand, normally give information on the consistency of the asphalts but do not provide all the fundamental rheological knowledge about the material.

A good compromise would be if one is able to identify a very simple, yet reasonably accurate rheological parameter that could be determined rapidly on an equally simple, low-cost instrument and then relate it to fundamental rheological data. This has been done for unmodified asphalts and it has been shown [37] that unified curves of fundamental rheological data can be obtained through a simple flow rate parameter. In the present work, the method of unification is discussed and applied to two special cases of polymer-modified asphalts.

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2. UNIFICATION CONCEPTS

2.1 Simple rheological parameter

The chosen parameter 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) shown in Fig. 1. This equipment is borrowed from the polymer industry [38] where it is routinely used to measure the melt flow index of the polymers.

2.2 Definition of 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 [38], namely, the melt flow index MFI, 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.

2.3 Theoretical basis for unification

The expressions for shear stress τ and shear rate $\dot{\gamma}$ in the FMD (on the assumption that the fluid is Newtonian as a first approximation) can be written in the following well-known conventional forms [38]:

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

$$\dot{\gamma} = \frac{4Q}{\pi R_N^3} \quad (2)$$

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

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

Since the geometry of the equipment is fixed as given above, equations (1) and (2) give:

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

and

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

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 asphalt sample at a given temperature, the shear stress and shear rate values corresponding to those test conditions can be obtained from equations (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. However, equations (4) and (5) essentially imply that the shear stress and shear rate values will always change in such a way so as to maintain the proportionality constants shown above. Thus, instead of the conventional plots of shear stress versus shear rate on a log-log scale, if a plot of τ/L versus $\dot{\gamma}$/MVR on log-log scale is made, then the result should be a unified curve [38].

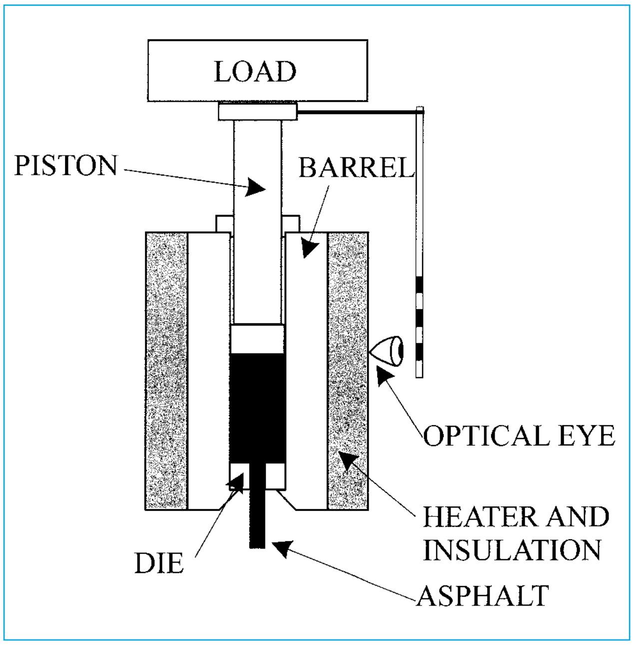

Fig. 1 – Schematic diagram showing the main parts of the Flow Measurement Device (FMD) that is used for determining the Material's Volumetric-Flow Rate (MVR).

SHRP asphalt binder research findings [35, 36] have indicated that dynamic shear rheometer data are the preferred fundamental rheological properties of 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 frequencies. There are a number of diverse methods [39-44] for correlating dynamic and steady shear data. According to the Cox-Mertz [39] method,

$$\eta(\dot{\gamma}) = |\eta^*|(\omega) \text{ at } \dot{\gamma} = \omega \quad (6)$$

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 ω and shear rate $\dot{\gamma}$.

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

Using the fact that $\eta = \tau/\dot{\gamma}$ and $|\eta^*| = |G^*| / \omega$ by definition, the following expression is written.

$$\tau/\dot{\gamma} = |G^*| / \omega \text{ at } \dot{\gamma} = \omega \quad (7)$$

Thus, equations (4) and (5) can be rewritten using the Cox-Mertz rule as follows.

$$|G^*| / L = \text{constant} = 9.13 \times 10^4 \quad (8)$$

and

$$\omega/MVR = \text{constant} = 1.83 \quad (9)$$

By following the arguments similar to those that were put forth earlier, it can be concluded that a plot of $|G^*| / L$ versus ω/MVR on log-log scale should result in a unified curve.

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'', it is necessary to use one of the theoretical models available in the literature [40-44]. In the present case, the Spriggs [42] model has been chosen for correlating the dynamic and steady state rheological characteristics. Based on the Spriggs [42] model, the loss modulus G'' which is the dynamic function is expressed as follows:

$$G'' = \frac{\eta_0}{\lambda Z(\bar{a})} \sum_{p=1}^{\infty} \frac{(\omega\lambda)^2}{p^{2\bar{a}} + (\omega\lambda)^2} p^{\bar{a}} \quad (10)$$

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

$$\tau = \frac{\eta_0}{c\lambda Z(\bar{a})} \sum_{p=1}^{\infty} \frac{(c\dot{\gamma}\lambda)^2}{p^{2\bar{a}} + (c\dot{\gamma}\lambda)^2} p^{\bar{a}} \quad (11)$$

where η_0, λ, $\bar{a}$ and $Z(\bar{a})$ are model parameters and c is an arbitrary adjustable constant expressed in terms of an independent parameter $\bar{e}$ as:

$$c^2 = \frac{1}{3}\left(2 - 2\bar{e} - \bar{e}^2\right) \quad (12)$$

Comparing equations (10) and (11) yields the following:

$$\tau = c^{-1} G'' \text{ at } \omega = c\dot{\gamma} \quad (13)$$

Thus, it is obvious from equation (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 Spriggs [42] needs to be followed, namely, of superimposing the plot of $\eta(\dot{\gamma})/\eta_0$ versus $c\dot{\gamma}$ on the plot of $\eta'(\omega)/\eta_0$ versus ω. For example, a value of $c = \sqrt{2/3}$ has been found by Saini and Shenoy [45] 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 equation (13) in equations (4) and (5), the following can be written:

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

and

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

Equations (14) and (15) again imply that a plot of G''/L versus ω/MVR on log-log scale should result in a unified curve, if arguments similar to those that were put forth earlier when discussing equations (4) and (5) are followed. Knowing the interrelationships between various dynamic functions, unified curves can be expected for G', $\tan\delta$, and even $|G^*|/\sin\delta$ given the following.

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

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

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

Based on the theoretical developments discussed above, it is clear that the unified curves would have a normalizing parameter for the y-axis (*i.e.*, load L), 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 much easier to regenerate fundamental rheological information and also to derive useful conclusions from the unified curve. An improved representation of the unification is therefore attempted.

From the unified curve, the following equality can be written:

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

This is equivalent to stating that:

$$\tau / L = \Phi\{\dot{\gamma} / MVR\} \quad (20)$$

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

$$\tau = \eta_0 \dot{\gamma}(L/MVR) \quad (21)$$

where η_0 is the proportionality constant. η_0 (L/MVR) is equivalent to the Newtonian viscosity. Equation (21) would imply that a plot of τ versus $\dot{\gamma}(L/MVR)$ would be unique and, therefore, in turn, a plot of $|G^*|$ versus $\omega(L/MVR)$ 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 equations (19) and (20) are not linear. The non-linearity in this relationship can be taken into account by using the Ostwald-de Waele power-law model [46-48] within small ranges of shear rates and frequencies, and equation (20) can be written as follows:

$$\tau / L = K (\dot{\gamma} / MVR)\eta_0 \quad (22)$$

where K is the proportionality constant {K (L / MVR^n) is termed as the consistency index} and n is the power-law constant that is normally termed as the pseudoplasticity index or the shear susceptibility index. Based on equation (22), the following relationships can then be written:

$$\tau = \Phi \{\dot{\gamma} (L^{1/n} / MVR)\} \quad (23)$$

and

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

Equation (24) implies that a plot of $|G^*|$ versus modified frequency $\omega (L^{1/n} / MVR)$ should then give a unique curve. It follows that G″ versus $\omega (L^{1/n} / MVR)$ and $|G^*| / \sin\delta$ versus $\omega (L^{1/n} / MVR)$ 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 devise a method to determine the n value in each case.

2.4 Determination of the n value

Since the τ versus $\dot{\gamma}$ curve and the $|G^*|$ versus ω 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:

$$\tau = K (\dot{\gamma})^n \quad (25)$$

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 equations (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 (MVR1 and MVR2) 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). For example, if the MVR value has been determined at, say, 2.16 kg, then one MVR value at, say, 3.06 kg and another at, say, 1.00 kg are determined. In order to get the value of n, equation (25) is rewritten using equations (4) and (5) as:

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

and

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

Solving equations (26) and (27), gives:

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

3. CONCLUDING REMARKS

A systematic theoretical analysis shows that, in principle, the data obtained from the DSR can be unified using the value of the MVR on the sample. This needs to be verified and this has been accomplished through experimental data in the Part II of this paper [49]. The unified curves have far-reaching implications. Once the unified curves have been developed, no further data from the DSR needs to be generated. Simply by generating MVR data and making use of the unified curves can give all viscoelastic properties on the sample. The technique can thus be used for quality control / quality assurance on a routine basis. In Part II of the paper [49], the experimental verification is followed by details on implications of the unified curves and on how to predict viscoelastic data from the unified curves.

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Developing unified rheological curves for polymer-modified asphalts – Part II. Experimental verification

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ABSTRACT

The theoretical analysis in Part I of the paper has shown that unified curves can be obtained, in principle, if the rheological data obtained by measurements on the dynamic shear rheometer (DSR) are normalized through the use of the material's volumetric-flow rate (MVR) generated from a simple flow measurement device (FMD). In Part II of the paper, experimental verification of the unification process is done through systematic data analysis on selected polymer-modified asphalts. The unified curves have far-reaching implications and these have been brought out explicitly. Since MVR is so simple to determine quite accurately on a relatively inexpensive, easy-to-use flow measurement device (FMD), this parameter can be generated on paving sites or at refineries. The MVR can be used as a quality control / quality assurance parameter to ensure batch-to-batch invariance and also as an excellent indicator of the fundamental rheological parameters through the use of the unified curves.

RÉSUMÉ

L'analyse théorique dans la partie I de l'article a montré que des courbes unifiées peuvent être obtenues, en principe, si les données rhéologiques issues d'un Rhéomètre à Cisaillement Dynamique (DSR) sont normalisées à partir de l'utilisation du débit volumétrique du matériau (MVR) obtenu grâce à un simple système de mesure du débit (FMD). Dans la partie II de l'article, une vérification expérimentale du processus d'unification est réalisée à travers une analyse systématique des données sur des bitumes modifiés par des polymères sélectionnés. Les courbes unifiées sont bien mises en évidence et ceci est clairement explicité. Dans la mesure où le MVR permet une détermination simple, assez précise à partir d'un système de mesure du débit peu onéreux et facile d'utilisation, ce paramètre peut être obtenu sur chantiers ou en raffineries. Le MVR peut être utilisé comme un paramètre de contrôle et d'assurance qualité afin d'assurer la constance de production, et aussi comme un excellent indicateur des paramètres rhéologiques à travers l'utilisation des courbes unifiées.

1. INTRODUCTION

As discussed in Part I of the paper [1], fundamental viscoelastic behavior of asphalts under different levels of stresses and temperatures needs to be understood for performance-related specifications to address major pavement distresses.

Equipments that provide the fundamental rheological information cannot easily be taken to the field or on-site, normally require highly trained operators and are also relatively much more expensive. Rapid rheological measurements would be preferred, but are known to normally give only limited rheological information about the material.

The theoretical analysis in Part I of the paper [1] shows that a very simple, yet reasonably accurate rheological parameter that could be determined rapidly on an equally simple, low-cost instrument can be related to fundamental rheological data. This theoretical possibility needs experimental verification and this has been done in this Part II of the paper. It has also been shown that the unified curves have far-reaching implications and that the concept can be used for routine quality control / quality assurance tests.

2. EXPERIMENTAL VERIFICATION

The theoretical development in the Part I of the paper [1] 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 need to be made on different polymer-modified asphalts that have widely different rheological characteristics. These 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.

2.1 Equipment used

(1) The Rheometrics Dynamic Shear Rheometer (DSR) was used for generating the dynamic data at four different temperatures between 52°C and 76°C with a set of parallel plates of 25 mm diameter following the procedure given in the AASHTO provisional specifications [2]. 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 any 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 for all three flags sequentially. 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.

2.2 Materials used

The two polymer-modified asphalts chosen for the present study were those that were used in the Accelerated Loading Facility (ALF) experiment [3] at the

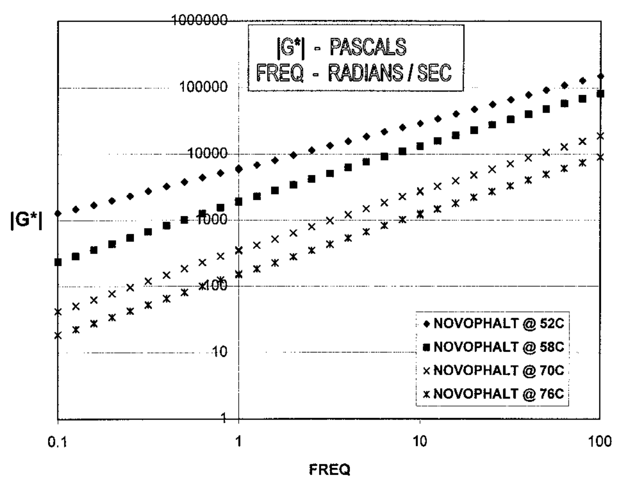

Fig. 1 – Variation of the dynamic complex modulus |G*| with frequency ω at four different temperatures of 52°C, 58°C, 70°C and 76°C for Novophalt original unaged sample.

Turner-Fairbanks Highway Research Center of the Federal Highway Administration. These were (a) Novophalt and (b) Styrelf.

Novophalt (PG76-22) is manufactured by Advanced Asphalt Technologies (AAT), Sterling, VA. The base asphalt used for the ALF Novophalt is AC-10. The asphalt is mixed with a certain amount (probably about 6.5%) of low density polyethylene (unknown grade as the information is proprietary). The material is run through a high shear mixer at about 145°C and then fed back into the mixer and run again to get a better dispersion. The final mixed material is not stabilized and tends to separate if correct handling procedures are not followed.

Styrelf (PG82-22) is presently manufactured by Koch Materials, Wichita, KS. The base asphalt used for Styrelf is AC-20. This asphalt is first blown to AC-40 grade and then styrene-butadiene (SB) is added to it. Sulphur is added for the reactions to occur in order to achieve chemical links with asphaltenes and other reactive species in the asphalt.

3. UNIFICATION PROCESS

The first attempt to test the unification technique was with polymer-modified asphalt Novophalt. Fig. 1 shows the |G*| versus ω curves for four different temperatures, namely, 52°C, 58°C, 70°C and 76°C which were generated from the DSR on Novophalt original unaged sample. The load versus MVR values obtained from the FMD for the Novophalt original unaged sample at four identical temperatures are given in Table 1. Using the corresponding values of load, MVR and n at each temperature, the four curves in Fig. 1 were replotted in terms of |G*| versus ω ($L^{1/n}$ / MVR), to give a unified curve as shown in Fig. 2. Thus, the 33 data points from each curve generated from the DSR were modified using a single data point from the FMD. A total of 132 data points have been unified into a single curve in Fig. 2. The same procedure when applied to G″ and |G*| / Sin δ data for Novophalt also gave unified curves (not shown here).

It must be noted that Novophalt is an unstablized system and hence it is naturally difficult to get reproducible rheological data. Whenever DSR data is obtained on two different Novophalt samples, there is a considerable variation in the rheological characteristic even if all other conditions (such as temperature of measurement, the plate diameters, frequency range of measurements, etc.) are maintained the same. One would

Table 1 – Load versus MVR data for original unaged sample

Asphalt ID	Temperature, °C	Load, kg	MVR, cc/10min	n
Novophalt	52	12.50 14.90 10.00	18.00 28.64 15.71	0.664
	58	12.50 14.90 10.00	47.10 64.46 38.07	0.757
	70	5.00 10.00 5.00	82.42 197.87 48.82	0.846
	76	2.16 3.05 1.00	57.29 104.33 31.98	0.946
Styrelf	58	14.90 17.40 10.00	14.47 19.80 10.15	0.829
	64	14.90 22.40 10.00	40.43 65.63 24.69	0.825
	70	7.06 10.00 5.00	39.06 57.57 26.74	0.904
	76	5.00 7.06 2.16	62.89 87.68 24.38	0.925

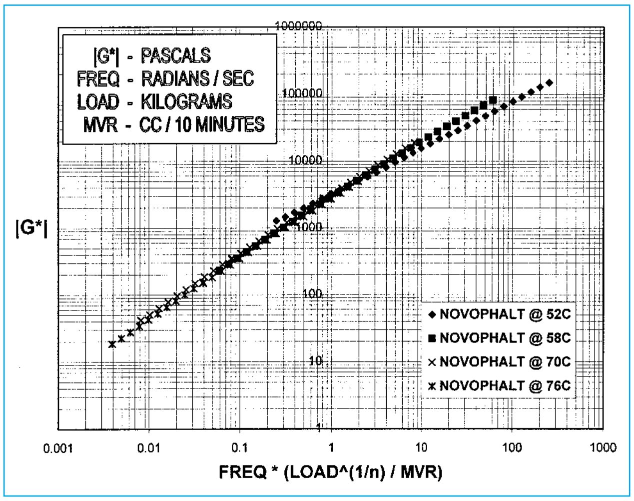

Fig. 2 – Unified curve of the dynamic complex modulus |G*| with modified frequency ω($L^{1/n}$ /MVR) covering temperature range of 52°C - 76°C for Novophalt original unaged sample.

have expected this to cause a problem in the unification; however, it does not. The unification is based on getting DSR data and FMD data on rheologically identical samples. This is achievable if care is taken to pour the sample for the DSR and the FMD measurement from the same container at around the same time and the same temperature. When such care is taken, the sample measured on the DSR and the FMD are rheologically identical and they will then unify elegantly on the curve. In case a replicate is taken and the two DSR data do not match, it will be found that the MVR data on the replicate will also be offset accordingly in such way that the data will unify. This is because the term ($L^{1/n}$ / MVR) will take care of the corresponding changes.

Based on the theoretical development, it is evident that the unification should hold independent of the type of polymer-modified asphalt being considered, though of course each different type of polymer-modified asphalt may have its own unified curve. In order to confirm this, the polymer-modified asphalt Styrelf is considered and the G″ curves are used.

Fig. 3 shows the G″ versus ω curves for four different temperatures, namely, 58°C, 64°C, 70°C and 76°C which were generated from the DSR on Styrelf original unaged sample. The load versus MVR values obtained from the FMD for the Styrelf original unaged sample at four identical temperatures are given in Table 1. Using the corresponding values of load, MVR and n at each temperature, the four curves in Fig. 3 were replotted in terms of G″ versus ω ($L^{1/n}$ / MVR), to give a unified curve as shown in Fig. 4. Again, the 33 data points from each curve generated from the DSR were modified using a single data point from the FMD. A total of 132 data points have been unified into a single curve in Fig. 4. The same procedure when applied to |G*| and |G*| / Sin δ data for Styrelf also gave unified curves (not shown here).

The plot of ln($L^{1/n}$ / MVR) versus 1/T for the two polymer-modified asphalts are shown in Fig. 5, from which the variation of the normalizing parameter with temperature can be adjudged.

It can be seen that the unified curves show a band within which all the data points coalesce. The percentage errors considering all cases were seen to be in the range of 7-20%. An estimate of the error bounds for the individual DSR measurements were found to be 8-22% while for the FMD measurements were found to be 1-4%. Thus, the error bound range of 7-20 % in the unified curves is probably only a reflection of the errors in the original DSR data.

Figs. 2 and 4 confirm that the unification technique works for at least two polymer-modified asphalts. The polymer-modified asphalts chosen in this study are different in many respects. Firstly, they are not made from the same base asphalt. Secondly, whereas one is modified using a thermoplastic homopolymer - low density polyethylene, the other is modified using a block copolymer - styrene butadiene styrene. The rheological behaviors of the modifiers in their pure melt form are radically different [4] and hence their presence in the

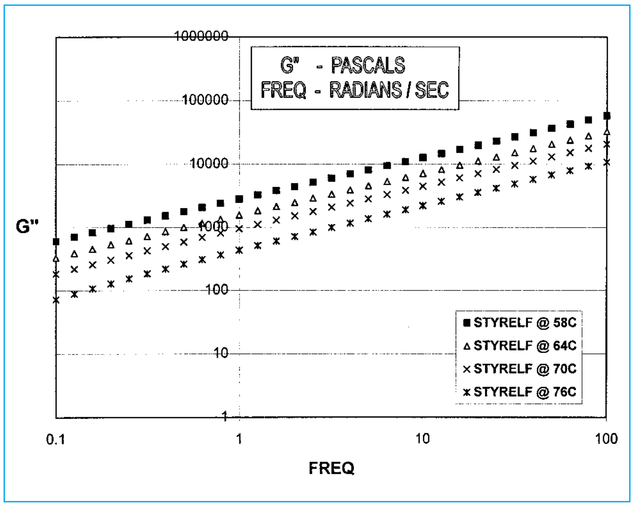

Fig. 3 – Variation of the dynamic loss modulus G″ with frequency ω at four different temperatures of 58°C, 64°C, 70°C and 76°C for Styrelf original unaged sample.

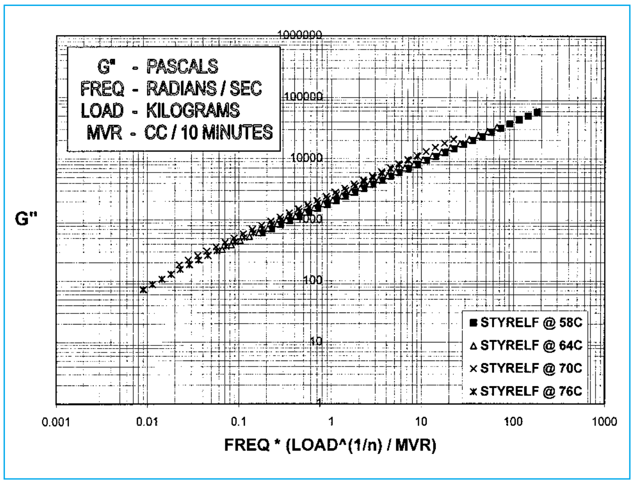

Fig. 4 – Unified curve of the dynamic loss modulus G″ with modified frequency ω($L^{1/n}$ /MVR) covering temperature range of 58°C - 76°C for Styrelf original unaged sample.

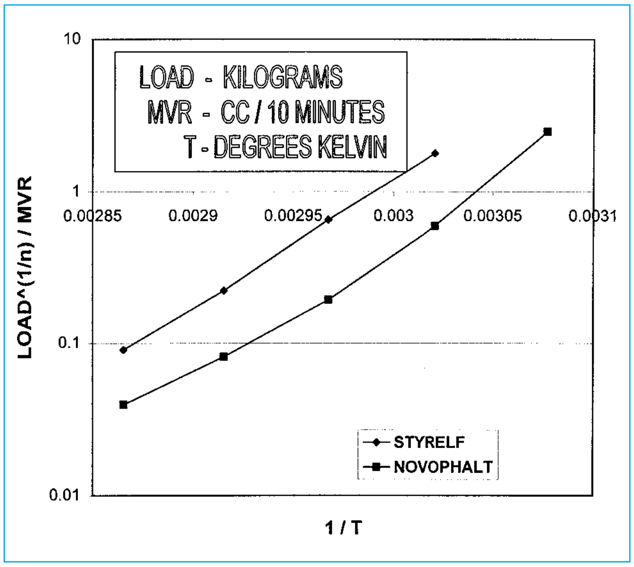

Fig. 5 – Variation of ($L^{1/n}$ / MVR) with (1 / T) covering the temperature range of 52°C - 76°C for Styrelf and Novophalt.

asphalt depicts quite different rheological characteristics. Despite these diversities, it has been shown that the unification approach works well in both cases. It may therefore, not be unrealistic to expect that the unification technique would in general be applicable to all polymer-modified asphalts. Of course, this needs to be confirmed, and can be done over a period of time in future.

The main accent of the present paper was essentially to suggest the methodology to achieve proper unification based on good theoretical foundations (Part I) and verify it through experimental data (Part II). This has been done through the analysis of rheological data on the two chosen polymer-modified asphalts. The unified curves of fundamental rheological data have many advantages and hence the beneficial implications of this unification are discussed next.

4. IMPLICATIONS OF THE UNIFIED CURVES

1) In the Superpave© binder performance grading system, there is a specification for minimum limit requirement of $|G^*|/\mathrm{Sin}\delta$ (≥ 1 kPa) for unaged asphalts at a frequency of 10 radians/s, which is assumed to simulate traffic loading when vehicles are moving at 50 to 60 mph. From the unified curves of $|G^*|/\mathrm{Sin}\delta$ (not shown here), a value of $\omega\,(L^{1/n}/MVR)$ was determined for the specification limit as:

$$\omega\,(L^{1/n}/MVR) = 0.245 \qquad (1)$$

(Minimum requirement for Novophalt and Styrelf)

Since a frequency of 10 radians/s is presently accepted as the value to simulate actual traffic conditions, the requirement can be rewritten as follows:

$$(L^{1/n}/MVR) = 0.0245 \qquad (2)$$

(Minimum requirement for Novophalt and Styrelf)

The implicit advantage of the unification technique is that if at all the Superpave © binder specification is changed from the present 1 kPa to some different value, then no new data need to be generated. One could simply read out the new requirements corresponding to equation (1) from the unified curves. New research continually brings in new ideas and refinements of the specifications are not to be ruled out. New analysis or traffic conditions in the future could warrant the imposition of a higher frequency than 10 radians/s in the specifications. With the unified curves available, there would be no need to again generate fresh data. Equation (2) simply takes on a new value by using the new frequency in equation (1).

2) If a database which includes all important parameters of asphalts is to be developed, then the unified curves will greatly reduce the amount of data that are needed to be stored in the information base. In fact, for each polymer-modified asphalt, the complete rheological data will be capsuled in just one single curve corresponding to each fundamental material function within the temperature range of interest.

3) The unified curves can be fitted with appropriate rheological equations so that predictions can be made in future, not by reading values from the plots but by simple mathematical calculations. These rheological equations will give unique relationships between $|G^*|$ versus $\omega\,(L^{1/n}/MVR)$, G'' versus $\omega\,(L^{1/n}/MVR)$, $|G^*|\,/\,\mathrm{Sin}\,\delta$ versus $\omega\,(L^{1/n}/MVR)$ and so on for each polymer-modified asphalt. These equations would be directly useful to those who are modeling the performance of the asphalts and attempting to relate the performance characteristics with the chemistry and physical structure of the asphalts.

5. PREDICTING VISCOELASTIC DATA FROM UNIFIED CURVES

The idea behind developing the 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. The procedure is illustrated for Novophalt data. From the unified curve, the average values of the viscoelastic parameter of interest are obtained at different values of modified frequency $\omega\,(L^{1/n}/MVR)$. Using these values, theoretical unified curve is drawn as shown in Fig. 6 for $|G^*|\,/\,\mathrm{Sin}\,\delta$. From this theoretical unified curve, it is now possible to estimate $|G^*|\,/\,\mathrm{Sin}\,\delta$ values at different frequencies for Novophalt at any temperature between 52°C - 76°C when the MVR value at a convenient load L and the parameter n are known.

In order to check the predictability, Novophalt data at 64°C is chosen. It should be noted that data at this temperature was not used when the unified curve was formed in the first place. At this temperature of 64°C, the MVR is determined to be 80.17 cm^3/10min at load L=10 kg and the value of n is determined from two other MVR measurements as equal to 0.84. Thus a value of (L^(1/n) / MVR) is calculated as 0.193. Using this value,

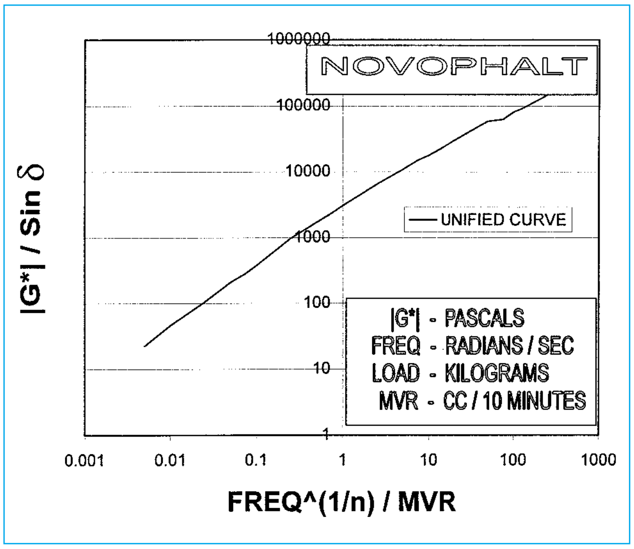

Fig. 6 – Theoretical unified curve of the SHRP parameter $|G^*|\,/\,\mathrm{Sin}\,\delta$ with modified frequency $\omega(L^{1/n}/MVR)$ at temperatures of 52°C - 76°C for Novophalt.

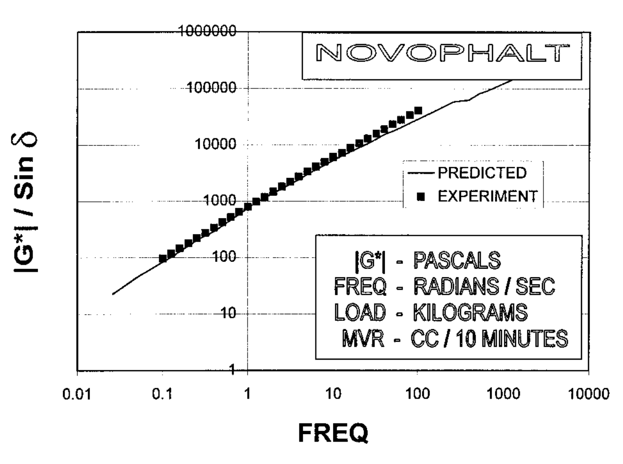

Fig. 7 – Comparison of the predicted curve and experimental data for the SHRP parameter |G*| / Sin δ with frequency ω at temperature of 64°C for Novophalt.

Table 2 – Details of the time required for generating MVR data

Activity	Time, Minute(s)
Sample weighing	1
Heating sample in oven @ 163°C	3
Pouring sample in FMD barrel	1
Temperature stabilization	10
Testing time for one MVR reading Flag of 6.35 mm	
For MVR 1.5	3.0
4.5	0.9
10	0.4
20	0.2
50	0.08
Time for remnant material to be discharged	3
Cleaning time	5

the variation of |G*| / Sin δ is calculated and shown by the solid line in Fig. 7. Actual DSR data are plotted on the same figure and the match between predicted curve and experimental data is seen to be good.

6. CONCLUDING REMARKS

The unification of fundamental rheological data for polymer-modified asphalts provides a rather powerful tool to reduce subsequent experimentation and to ease the generation of rheological information in the future. It also provides the possibility of introducing new specification parameters that have the advantage of being easy to determine and at the same time being more flexible to changes, in case such a need for a specification change is felt in the future.

The FMD that is used for 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.

In order to generate MVR data accurately, a few important points should be borne in mind:

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 may take about five minutes but there is no compromise on getting parts spotlessly clean.

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 as can be seen from Table 2. However, 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 $1.3 < MVR < 50$ lies between 0.08 to 3.3 minutes, which is quite reasonable. It was found that for MVR = 0.2, the testing time was about 20 minutes and for 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 as far as possible, 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 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 reproducible. In fact, in terms of repeatability of data, the FMD performs better than DSR. There are a number of other benefits in using the FMD. The evaluations of the benefits of the FMD are to be based on a number of factors such as initial equipment cost, opera-

Table 3 – Comparison between DSR and FMD		
Considered Feature	DSR	FMD
Initial Equipment Cost	$ 35000	✓ $5500*
Operational Cost	Power supply Air pressure Circulating water bath	✓ 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	✓ 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	✓ 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 Replicate data has to be generated on a fresh sample for viscoelastic systems and needs an entire new run	✓ Not at all difficult to guess the load conditions in order to achieve MVR values between 1 and 50 to get most accurate data ✓ Three MVR values are generated one after another for each sample loading.
Sample Preparation Time	✓ Heating the asphalt to 163°C and pouring in the silicone mold and cooling (about 5 minutes)	✓ Heating the asphalt to 163°C and pouring into the barrel (about 5 minutes)
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	✓ Equilibrates very quickly at all temperatures and the entire data generation takes only about 15 minutes (See Table 2 for details)
Specimen Size	✓ 1 gm (For one set of data)	10 gms (For three sets of data)
Variability of Output (% STD / AVG)	7 to 22 (Depending on plate size, gap and temperature)	✓1 to 4 (For the standard die)
Data reduction method	Requires a computer and Windows'95 to run the software for calculation of various rheological functions	✓ Requires no computer as all calculations are done by the built-in software which does not require any other support program either as it does not calculate any other rheological functions except MVR
Information Obtained	✓ Extensive information on the basic rheological properties of asphalts can be got in terms of $\|G^*\|$, G'', G', etc.	✓ 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.	✓ Relatively light weight, Requires no air pressure, Needs no water-bath and thus is portable.

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

tional cost, maintenance cost, operator training requirements, experimental ease, testing time, specimen size, variability of output, data reduction method, information obtained, and mobility or portability. A comparison between DSR and FMD is shown in Table 3. It can be seen that the FMD beats out DSR on most counts except specimen size and the information obtained. 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 as can be seen from Table 2, which gives the details of the time involved for getting three MVR readings on one asphalt. This makes the measurement particularly attractive. The FMD can be used for routine quality control / quality assurance testing in order to ensure that there is no batch-to-batch variation in the quality of the polymer-modified asphalt during the manufacturing process.

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