Rheological Properties of Chemically Modified Asphalts

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Asphalt chemically modified with furfural and furyl acrolein was performance graded (PG), and its performance was predicted according to the criteria developed by the Strategic Highway Research Program (SHRP). These asphalts were aged in a thin film oven, a rolling thin film oven, and a pressure aging vessel to investigate the aging behaviors of the chemically modified asphalts. The rheological properties were determined from 10°C through 70°C by the dynamic shear rheometer and from −25°C through −5°C by the bending beam rheometer. By using the SHRP criteria chemical modification was shown to increase the temperature range over which the asphalt can be used by primarily increasing the high-temperature stiffness of the asphalt. Differences in properties and PG grading were observed when the asphalts were aged in a rolling thin film oven compared with that when they were aged in the thin film oven, especially for the modified asphalts. The resistance to rutting was predicted to increase with chemical modification. The resistance to fatigue was predicted to change insignificantly at the range of temperatures considered in the PG specifications. The low-temperature properties were not significantly affected because of modification.

Conventionally refined asphalts have been modified by various means to extend the temperature range at which they can be used. These modifications have been made through the addition of rubber-related products and polymers and through chemical modification. Researchers at FHWA have been investigating the modification of asphalts by chemicals such as maleic anhydride, chromium trioxide, and furfural (*1*). Among these modifiers furfural-modified binders showed better adhesion to aggregate and less temperature susceptibility (of viscosity), especially at low temperatures. Consequently, further research was performed on furfural modification to study the effects of certain reaction variables on the physical properties of the products of the chemical reaction between asphalt and furfural (*2,3*). From that study the conditions for the method of preparation of an AC-20-modified asphalt from an AC-5 asphalt and furfural were obtained. Subsequent research at FHWA (*2–4;* this study) used this method to prepare AC-20 or higher asphalt cement (AC)-grade modified asphalts with furfural and AC-5 or AC-10 asphalts. Since Strategic Highway Research Program (SHRP) binder testing was not fully developed when those studies (*2–4*) were performed, there has been little effort to optimize the furfural reaction according to the performance criteria used in the performance-graded (PG) grading.

The nature of the reaction of furfural with asphalt was studied by using partial furfural analogs such as furyl acrolein, furan, allyl alcohol, and furanone (*3*). The results indicated that the furan ring was the principal component in producing modified asphalts with increasing stiffness at high temperatures, and furan with or without a conjugated aldehyde group was important in improving the low-temperature properties. Memon and Chollar (*4*) showed that the furfural reacts with the phenolic groups in the asphalt by a condensation reaction in the presence of an acid catalyst. Initially, the rheological properties of the chemically modified asphalts were evaluated primarily by traditional methods such as penetration at 10°C and 25°C and viscosity at 60°C and 135°C. In addition, a dynamic shear rheometer equipped with a cup and stepped plate geometry was also used to characterize the rheology of unaged reaction products (*1,2*).

Anderson and Kennedy (*5*) presented new criteria for evaluating the distress modes that were developed by SHRP for the new binder specifications. These performance criteria were based on the fundamental properties of properly aged asphalt binders measured at the appropriate distress temperatures. At the beginning of the study SHRP had recommended that either the thin film oven test (TFOT) or the rolling thin film oven test (RTFOT) be used for specification purposes. The AASHTO provisional specifications later adopted the RTFOT aging procedure for the performance grading of asphalt binders (*6*).

The objectives of the present study were threefold: to evaluate the benefits of chemical modification on the predicted performance based on the SHRP criteria, to understand the effect of chemical modification on the rheological properties of asphalts, and to study the aging characteristics of chemically modified asphalts.

BACKGROUND

The PG grading system evaluates the resistance of an asphalt to the primary distress modes in the pavements: rutting, fatigue cracking, and low-temperature cracking. This information is then used to define a climate range based on the temperature at which the asphalt can be used for a designed period of time without causing the pavement to fail. Certain rheological parameters were chosen to evaluate the resistance of the asphalt to these distress modes (*5*). In this section the basis for the selection of these rheological parameters, as available in the literature, are summarized.

Rutting

Rutting of flexible pavements is effectively the total permanent deformation accumulated in the pavements due to the repeated application of loads (*5,7*). Since the pavements rut because of plastic deformation, one approach to quantifying the rutting behavior would be to look at the energy dissipated (as plastic deformation, among other mechanisms) during each loading cycle under stress-controlled conditions. This work dissipated in a loading cycle is given by (*7*)

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$$W_c = \pi \sigma_0 \epsilon_0 \sin(\delta) \quad (1)$$

where

σ_0 = maximum stress amplitude,

ϵ_0 = maximum strain amplitude, and

δ = loss angle.

For a stress-controlled loading ϵ_0 can be substituted with σ_0/G^*, where σ_0 is the applied stress and G^* is the complex shear modulus, to obtain

$$W_c = \pi \sigma_0^2 \left[\frac{1}{G^*/\sin(\delta)} \right] \quad (2)$$

Equation 2 relates the work dissipated per loading cycle, a measure of rutting, to $G^*/\sin(\delta)$, the parameter used in the specification. In another variation Bouldin (*8*) related the loss compliance J'' to the total accumulated deformation as follows:

$$\frac{1}{J''} = \frac{\sigma_0}{y_{acc}} = \frac{G^*}{\sin(\delta)} \quad (3)$$

where y_{acc} is the accumulated strain. In either case the greater the $G^*/\sin(\delta)$, the lower the tendency of the pavement to rut. Since a pavement is most susceptible to rutting soon after it is laid down, the specifications call for a minimum $G^*/\sin(\delta)$ for short-term-aged asphalt at the maximum temperature for which the pavement is designed.

Fatigue

Fatigue cracking of pavements occurs under repeated application of loads of magnitudes far less than the fracture strength. The current criterion for fatigue cracking specification is based on a measure of the total dissipated energy, which includes the energy expended in propagating cracks in the pavement (*9*). Under a strain-controlled loading, typical for a thin pavement layer, the dissipated work per cycle W_c is given by (*5*)

$$W_c = \pi \sigma_0 \epsilon_0 \sin(\delta) \quad (4)$$

where ϵ_0 is the strain amplitude applied, or

$$W_c = \pi \epsilon_0^2 [G^* \sin(\delta)] \quad (5)$$

since σ_0 is equal to $G^*\epsilon_0$ (*5*). Thus, the tendency of the asphalt pavement to crack because of fatigue is related to the loss shear modulus G'', which is equivalent to $G^* \sin(\delta)$. At higher values of G'' there is an increased tendency for the pavement to crack because of fatigue. The SHRP specification limits the G'' for pressure aging vessel (PAV)-aged asphalts to 5 MPa at a temperature 4°C above the midpoint between the highest and the lowest design temperatures of the pavement.

Low-Temperature Cracking

The low-temperature cracking tendency in pavements is evaluated by two independent tests: the direct tension test and the bending beam rheometer test. The strain at failure at a 1-mm/min loading rate (as determined by the direct tension procedure) is a direct measure of the failure properties of asphalt. However, for asphalt the strain to failure is related to the stiffness (*5*), leading to the concept of a limiting stiffness (when pavement will crack) and a limiting stiffness temperature (when the pavement will attain the limiting stiffness). As the stiffness of asphalt increases at low temperatures the tendency of asphalt pavements to crack increases. The stiffness of asphalt at a 60-sec loading time was measured on a bending beam rheometer.

EXPERIMENTAL

An Alaskan North Slope asphalt with the SHRP designation AAV was used as the base asphalt. This asphalt was reacted with furfural and furyl acrolein in the presence of a catalyst to yield an AC-20 asphalt with furfural modification and an AC-30 asphalt with furyl acrolein modification (*2*). These products were coded FUM for the furfural-modified asphalt and FAM for the furyl acrolein-modified asphalt. The various asphalts tested and the characterization techniques used are summarized in Table 1.

The asphalts were aged by standard TFOT and RTFOT procedures and in a PAV according to the AASHTO provisional standard procedure (*10*). The complex modulus and the loss tangent for all of the asphalts were measured with a dynamic shear rheometer (DSR) (Model RDA-II; Rheometrics, Inc. Piscataway, New Jersey) at temperatures of 10°C, 20°C, 30°C, and 40°C with an 8-mm parallel plate geometry and at 40°C, 50°C, 60°C, and 70°C with a 25-mm parallel plate geometry by the procedure in the AASHTO pro-

TABLE 1 Asphalts, Aging Treatments, and Tests Conducted

	AAV	FUM	FAM
Unaged	DSR	DSR	DSR
TFO aged	DSR	DSR	DSR
RTFO aged	DSR	DSR	DSR
TFO-PAV aged	DSR, BBR	DSR, BBR	DSR, BBR
RTFO-PAV aged	DSR, BBR	DSR, BBR	DSR, BBR

All dynamic shear rheometer (DSR) measurements were made at 10, 20, 30 and 40°C with 8 mm parallel plate and at 40, 50, 60 and 70°C with 25 mm parallel plate geometry.
All bending beam rheometer (BBR) measurements were made at -5, -15 and -25°C

TABLE 2 PG of Chemically Modified Asphalts

	Specification limits	*TFOT BASED* AAV	FUM	FAM	*RTFOT BASED* AAV	FUM	FAM
Tank							
G*/sin(δ)	Min: 1000 Pa	56.5	67.0	67.0	56.5	67	67
TFOT/RTFOT Residue							
G*/sin(δ)	Min: 2200 Pa	55.0	67.0	64.0	58	62.5	61
PAV Residue							
G*.sin(δ)	Max: 5000 kPa	17.5	17.5	16.0	16	19	19
S(60)	Max: 300 MPa	-19.0	-20.0	-20.0	-18	-18	-18
m(60)	Min: 0.3	-18.0	-18.0	-19.0	-19	-18	-17
PG GRADE		**52-28**	**64-28**	**64-28**	**52-28**	**58-28**	**58-22**
Continuous grade		55-28	67-28	64-29	57-28	63-28	61-27
Useful temperature range		83	95	93	85	90	88

Useful temperature range is the temperature range in which the asphalts can be used and is the difference between the upper and lower limit temperatures. For example, for PG 55-28 (in the continuous grade) the useful temperature range is 55-(-28)=83

visional specifications (*11*). For each measurement three repetitions (three separate samples from a single reaction) were made and averaged. All DSR measurements reported in this paper were measured at 10 rad/sec unless stated otherwise. The flexural creep stiffnesses of the PAV-aged asphalts were measured at temperatures of −5°C, −15°C, and −25°C with a bending beam rheometer (Cannon Instruments, State College, Pennsylvania). A constant load of 100 g was applied, and the deflection was recorded as a function of time. The creep stiffnesses and the slope of the logarithm of creep stiffness–logarithm of time curve (*m*-value) at 60 sec were measured according to the procedures described in the AASHTO provisional specification (*12*).

RESULTS AND DISCUSSION OF RESULTS

Table 2 gives the SHRP PG grading (*6*) of these asphalts as determined by both the TFOT and RTFOT procedures for short-term aging. The differences between PG for these asphalts were determined by the TFO and RTFO aging techniques. For AAV the PG was 52–28 when both TFOT and RTFOT procedures were used. In the case of furfural- and furyl acrolein-modified asphalts, the TFO route graded PG 64−, whereas the RTFO route graded PG 58− because of factors discussed later. The low-temperature limit for all asphalts was unchanged after RTFO aging except for the furyl acrolein-modified asphalt. This change was primarily due to the lower *m*-value for this asphalt.

Effect on Rutting Characteristics

Figure 1 shows the isochronal curve for *G**/sin(δ) at 10 rad/sec illustrating the effect of modifications for the unaged and TFO- and RTFO-aged asphalts. Each datum point in the curve is an average of three independent measurements, with the error bars denoting ± 1 standard deviation. The modification caused a substantial increase in *G**/sin(δ) over the entire temperature range for the unaged asphalts. The difference in *G**/sin(δ) between the furfural- and furyl acrolein-modified asphalts was not significant. In the case of TFOT residues, the increase in *G**/sin(δ) due to modification was significant, but it was not as dramatic as that for the unaged asphalts. Also, the increase in *G**/sin(δ) was greater at higher temperatures. For RTFOT residues the increase in *G**/sin(δ) due to chemical modification was reduced even further. Also, the furfural-modified

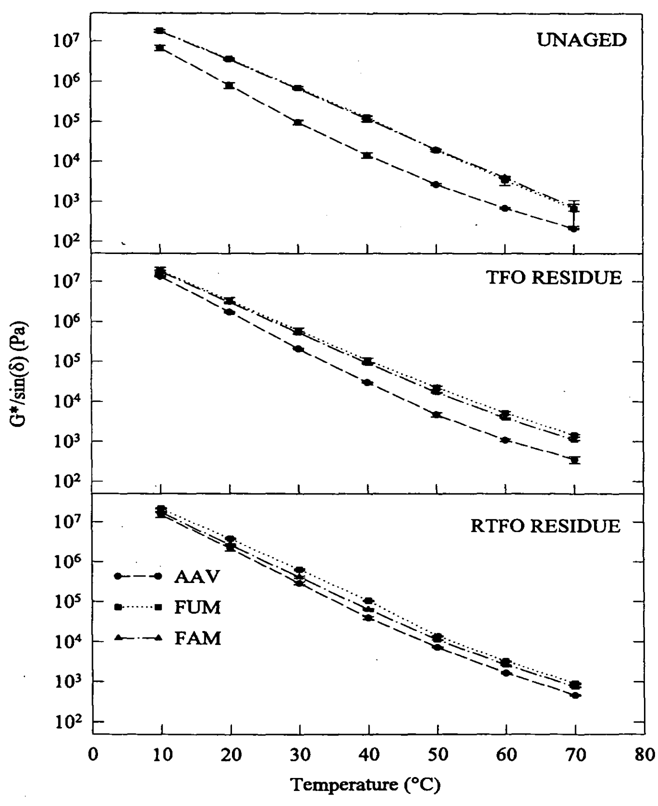

FIGURE 1 Effect of modification on *G/sin(δ) for unaged and short-term-aged asphalts.**

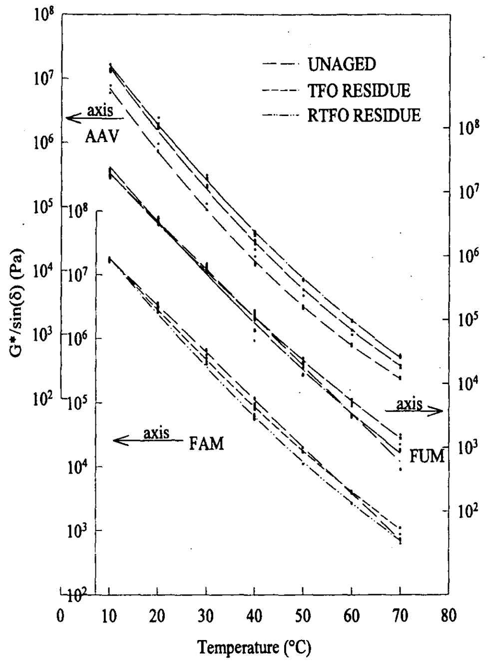

FIGURE 2 Effect of short-term aging on $G^*/\sin(\delta)$ of unmodified and modified asphalts.

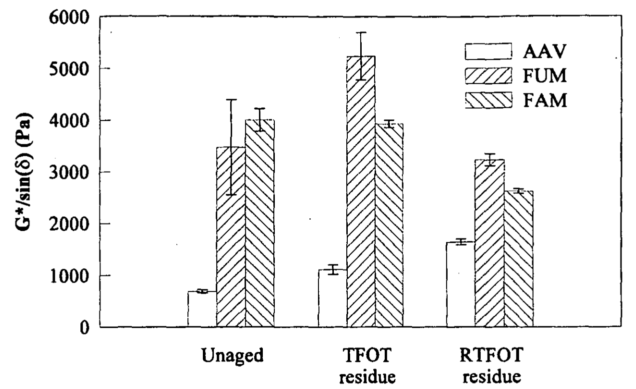

FIGURE 3 Effect of short-term aging on $G^*/\sin(\delta)$ at 60°C for unmodified and modified asphalts.

product had a significantly higher $G^*/\sin(\delta)$ value than the furyl acrolein-modified product.

From the same data the effect of aging the asphalts by the TFO and RTFO methods can also be studied. Such a study will yield information on the rheological nature of the modified asphalts. In Figure 2 the isochronal curves from Figure 1 are grouped by asphalts to enable an analysis of the effects of aging. In Figure 2 the three measurements that made up the average $G^*/\sin(\delta)$ at a given temperature (Figure 1) were plotted as separate datum points and were fitted with a second-order polynomial (shown in Figure 2). Such a fit will also take the measurement error into consideration to better estimate the significance of the observed changes. Each of the curves in Figure 2 had a regression coefficient greater than 0.997, indicating a good fit. For the unmodified asphalt aging shifts the curve toward a higher $G^*/\sin(\delta)$ value without changing its shape significantly. In the case of furyl acrolein-modified asphalts the shape of the isochronal curve for the unaged asphalt is markedly different from that of the aged asphalt. The curvature of the curve for the unaged asphalt was much less than that of the curves for aged asphalts. Consequently, at temperatures of about 30°C and 40°C, the TFO- and RTFO-aged samples had $G^*/\sin(\delta)$ values significantly lower than those for the unaged asphalt. Such a difference in the shapes of the curves suggests that the unaged furyl acrolein-modified asphalt has properties rheologically different from those of the unmodified asphalts. However, the aged furfural- and furyl acrolein-modified asphalts behave similarly to the unmodified asphalt. Furfural-modified asphalt also shows a similar behavior, but it is not as definitive as that of the furyl acrolein-modified asphalt.

Since the asphalts are in the PG 52- through PG 64- grade range, it is useful to compare the $G^*/\sin(\delta)$ values at 60°C for these asphalts, as illustrated in Figure 3. For the unmodified asphalt, $G^*/\sin(\delta)$ increases from unaged to TFO-aged to RTFO-aged, in that order. This observation is consistent with the conclusions of Zupanick (*13*), who has shown the RTFOT to have a more severe aging effect than TFOT on asphalts. However, in the case of the furfural-modified asphalt, the $G^*/\sin(\delta)$ values for the RTFO residues were comparable to those for the unaged samples, whereas the TFO residues had $G^*/\sin(\delta)$ values higher than those for the unaged samples. In the case of furyl acrolein-modified samples, $G^*/\sin(\delta)$ values for TFO-aged samples remained essentially unchanged compared with the value for unaged samples, whereas they decreased for RTFO-aged samples aging.

One can interpret these observations as being two mechanisms that are operating during aging and that are trying to affect $G^*/\sin(\delta)$ in an opposite manner. One mechanism is the normal aging characteristics, which tend to increase $G^*/\sin(\delta)$ as the severity of aging increases. The other mechanism decreases $G^*/\sin(\delta)$ as the severity of aging increases. The combined actions of these two mechanisms could lead to the behavior observed in Figure 3. Similar observations can be made by examining the data in Table 2 for the temperatures at which the asphalt meets the specification. These temperatures for AAV are 56.5°C, 55.0°C, and 58.0°C for unaged, TFOT-aged, and RTFOT-aged asphalts, respectively. For the furfural-modified asphalt the corresponding temperatures are 67.0°C, 67.0°C, and 62.5°C, and for the furyl acrolein-modified asphalts they are 67.0°C, 64.0°C, and 961.0°C.

It is unknown what mechanism could be operating to decrease the $G^*/\sin(\delta)$ values for asphalts during short-term aging. Perhaps one of two things could be happening. The modification reaction products may be unstable when they are heated and partially break down to smaller molecules that volatilize upon heating. On the other hand the reaction could be reversible at high temperatures, causing the chemical modification to partially reverse into asphalt and furfural, and the latter then volatilizes, causing a decrease in $G^*/\sin(\delta)$. The loss of $G^*/\sin(\delta)$ for the furyl acrolein-modified asphalt is more than that for the furfural-modified asphalts.

Effect on Fatigue

Figure 4 shows the isochronal curves of $G^*/\sin(\delta)$ (or G'') at 10 rad/sec, which were used to study the effects of modifications under various aging conditions. G'' increased on modification except at the lower temperatures. At temperatures below 25°C,

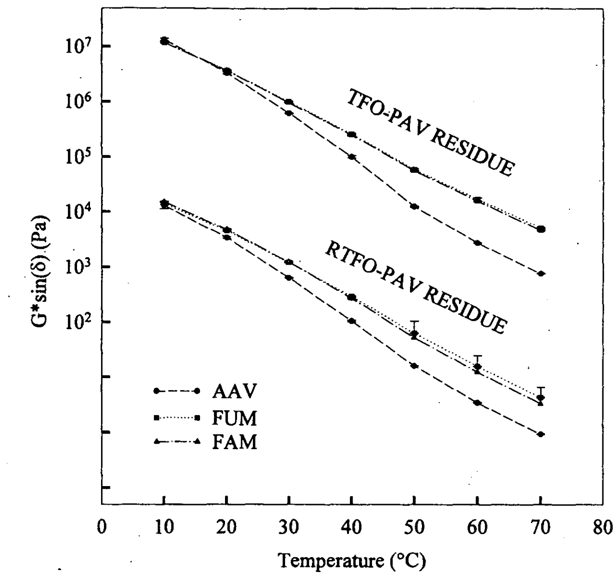

FIGURE 4 Effect of modification on $G^*/\sin(\delta)$ for TFO-PAV- and RTFO-PAV-aged samples.

which is the temperature range at which fatigue cracking would be a problem, G'' values for the modified asphalts are only slightly higher than, if not equal to, those for the unmodified asphalts. This indicates that for these temperatures modification had little effect on fatigue resistance.

Figure 5 shows $G^*/\sin(\delta)$ (or G'') at 10°C and 30°C for these asphalts comparing the TFO-PAV and RTFO-PAV aging procedures. Several observations can be made from Figure 5, as follows:

1. No significant differences were found between samples of AAV aged by TFO-PAV and RTFO-PAV at both 10°C and 30°C, whereas significant differences were apparent in chemically modified asphalts.
2. At 30°C modification increased G'' by about 60 percent for the TFOT-PAV-aged residues and 100 percent for the RTFOT-PAV-aged residues. At 10°C modification decreased G'' for the TFO-PAV-aged residues, whereas it showed an insignificant increase for the RTFO-PAV-aged residues.
3. For the modified asphalts at both 10°C and 30°C the RTFO-PAV-aged residues had higher G'' values than the TFO-PAV-aged residues. This is in contrast to the short-term-aged (modified) asphalts, for which the $G^*/\sin(\delta)$ values for the TFO-aged residues were higher than those for the RTFO-aged residues. It was verified that this observation was also true when G^* alone was considered.

These data point to an interesting case when the SHRP ranking of asphalts for fatigue is different at various temperatures. Table 2 shows the temperatures at which G'' is 5 MPa for asphalts subjected to both TFO-PAV and RTFO-PAV aging. This temperature increased for RTFO-PAV-aged residues compared with that for unaged asphalt. For TFO-PAV-aged residues it remained the same for furfural-modified asphalts and decreased for furyl acrolein-modified asphalts. This indicates that the fatigue resistance may be predicted to remain the same or increase (based on TFO-PAV aging) for chemically modified asphalts. When the aging is more severe during mixing with aggregates, as simulated by RTFO aging, the chemically modified asphalts show higher G'' values, probably predicting slightly decreased fatigue resistance. Such an interpretation can also be made from the data in Figure 5 at 10°C. At 30°C, however, modification increases G''on TFO-PAV aging, and it increases further for RTFO-PAV aging. Thus, at 30°C the ranking of these asphalts is different from that at 10°C and the SHRP ranking.

Effect on Low-Temperature Cracking

Figure 6 shows the creep stiffness of asphalts at a 60-sec loading time, $S(60)$, at various temperatures. Because repetitions were not made, a measurement error of 6 percent was used (based on prior determinations) to help interpret the differences between the curves. From the plot in Figure 6 it can be seen that except for RTFO-PAV-aged AAV at −25°C, all points overlap, indicating that the modification has little effect on the low-temperature creep stiffness. This behavior confirms the data in Table 2, which simply show that for RTFOT-PAV-aged asphalts the temperature at which $S(60)$ is equal to 300 MPa remains the same after chemical modification. For TFO-PAV-aged residues, however, chemical modification decreases the temperature at which $S(60)$ is 300 MPa. On the basis

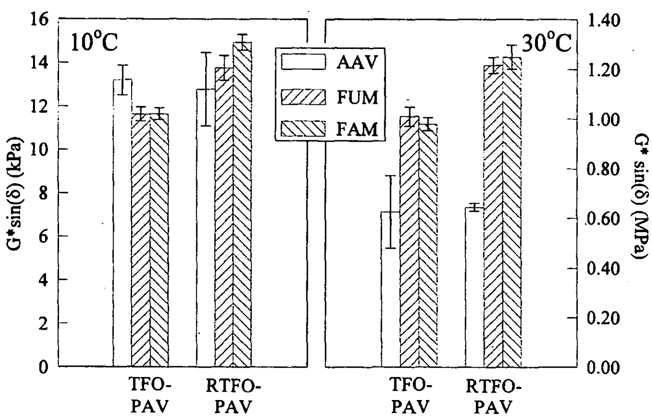

FIGURE 5 Effect of aging on $G^*/\sin(\delta)$ at 30°C and 10°C at 10 rad/sec.

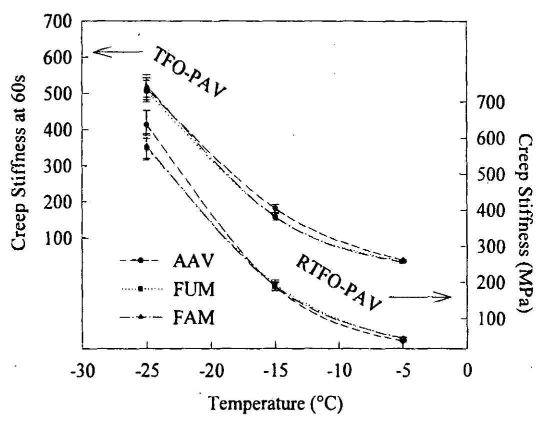

FIGURE 6 Effect of modification on the flexural creep stiffness of unmodified and modified asphalts.

of $S(60)$ alone it is concluded that chemical modification has no significant effect or a slightly beneficial effect on the tendency for low-temperature cracking. What is not obvious from Figure 6 is that the creep stiffness of RTFO-PAV-aged residues of the modified asphalts is higher than that of the TFO-PAV-aged residues, whereas there is no such difference in that of the unmodified asphalts.

Another factor considered in the specification is the m-value. This is related to the rate at which thermal stresses are relieved. The faster the rate of release of the thermal stresses the more durable the pavement. An asphalt with a high m-value would release stresses faster than an asphalt with a lower m-value. Figure 7 shows the m-value as a function of temperature. For TFO-PAV-aged residues at temperatures greater than approximately −19°C the modification decreases the m-value compared with that of the unmodified asphalt, decreasing the rate of release of built-up stresses. At temperatures below approximately −19°C modification has a beneficial effect. For RTFO-PAV-aged residues at temperatures higher than approximately −24°C, chemical modification reduces the m-value. If the PG specification limits the use of the asphalt binders to above an m-value of 0.300, the m-values of the unmodified asphalts are always higher than those of the chemically modified asphalts. In other words, the modification decreases the m-value and therefore has a slight deleterious effect on the thermal stress release rate.

The PG may be too coarse a scale when the effects of modifications (such as polymer and rubber additions or chemical modifications) on the performance of binders are to be studied. In such cases it is useful to consider a continuous grading scale in which the upper and lower temperatures (rounded to the nearest integer) are specified. To determine the continuous grade the actual temperature at which the specification criterion is met is determined for each parameter, as illustrated in Table 2. The upper temperature limit is the lower of the two temperatures that satisfy the high-temperature criteria [$G^*/\sin(\delta)$ for tank- and RTFOT-aged asphalts], and the low-temperature limit is 10°C lower than the higher of the two temperatures that satisfy the low-temperature criteria [$S(60)$ and $m(60)$]. The intermediate temperature is not limiting as long as the temperature for which the intermediate temperature criterion [$G^*\sin(\delta)$ for PAV-aged residue] is met is less than or equal to fatigue specification temperature (4°C higher than the midpoint between the high- and low-temperature limits). This is true for most asphalts and is certainly true for the modified asphalts considered in the present experiments.

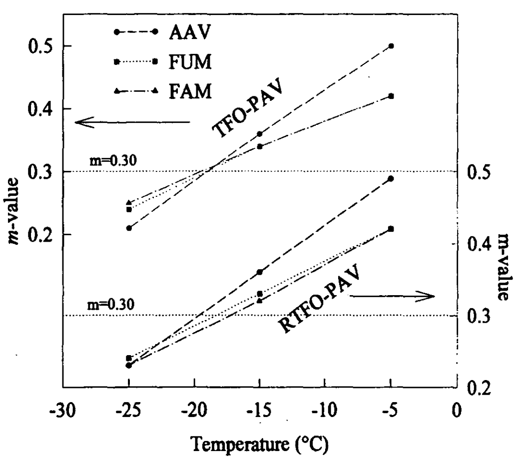

FIGURE 7 Effect of modification on m-value.

The continuous grading is also presented in Table 2. It can be noted that the furfural modification increased the high-temperature limit by 12°C (for TFOT-based aging) or 5°C (for RTFOT-based aging), whereas the low-temperature limit remained the same. The furyl acrolein modification increased the high-temperature limit by 9°C (TFOT-based aging) or 4°C (RTFOT-based aging), whereas the low-temperature limit essentially remained the same. Such an analysis gives more insight into the degree of the effect that modification has on the rheological properties than simply stating that the modification caused an increase of two high-temperature grades. An increase in two such grades could be due to an increase of 7°C or 12°C at the high-temperature end.

CONCLUSIONS

The following conclusions are based on the rheological properties measured for the asphalts chemically modified by furfural and furyl acrolein.

1. Chemical modification increased the $G^*/\sin(\delta)$ values, which is predicted to increase the resistance of the pavements to rutting.
2. The increase in $G^*/\sin(\delta)$ became less pronounced upon short-term aging, but the increase was still significant.
3. On short-term aging it was apparent that another mechanism (other than aging) that tended to decrease the stiffness was active in chemically modified asphalts. However, this mechanism was not identified.
4. $G^*\sin(\delta)$ increased slightly because of modification, indicating that the fatigue resistance was slightly reduced. Even with the slight increase $G^*\sin(\delta)$ was not the limiting parameter in the specification, suggesting that this decrease in fatigue resistance would not lead to pavements that fail because of fatigue cracking.
5. The creep stiffness did not change significantly on modification, but the m-value decreased slightly, indicating that the pavement in which these modified asphalts are used may not be able to relieve thermal stresses at the same rate as that in which the unmodified asphalt is used.
6. The PG of asphalts based on RTFOT as the short-term aging test changed from 52–28 to 58–28 for the furfural modification and 58–22 for the furyl acrolein modification. When TFOT instead of RTFOT was used for short-term aging, the PG changed from 52–28 to 64–28 for both furfural- and furyl acrolein-modified asphalts.
7. Continuous grading provided a more practical way of assessing the degree of effect that modification had on the rheological properties of asphalt.

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