Modelling short-term aging of asphalt binders using the rolling thin film oven test

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Abstract: Simulation of short-term aging of asphalt binders is a widely used procedure in asphalt binder characterization for predicting the binder response to plant mixing and paving under controlled laboratory conditions. There are two laboratory test methods for evaluating the short-term aging of asphalt binders: (*i*) a method using rotating pans filled with a thin asphalt film termed thin film oven test (TFOT) and (*ii*) a method using rolling cylindrical asphalt containers termed rolling thin film oven test (RTFOT). In this paper, an attempt is made to develop generalized models for short-term aging effects using the RTFOT aging time as a benchmark. Six binder types representing two PG grades and three source suppliers are conditioned to varying levels of RTFOT aging and tested using the dynamic shear rheometer (DSR). Aging effects are modelled using independent temperature shift models for the shear modulus and phase angle. The paper discusses the sources of errors in producing generalized models and some potential applications of aging models. The research revealed that it is possible to develop and implement such models for unmodified binders.

Key words: asphalt, aging, RTFOT, DSR, binder rheology, shear modulus, phase angle.

Résumé : La simulation du vieillissement à court terme des liants asphaltés est une procédure largement utilisée pour la caractérisation des liants asphaltés afin de prédire la réponse du liant au mélange en usine et au pavage sous des conditions de laboratoire contrôlées. Il existe deux méthodes d'essais en laboratoire afin d'évaluer le vieillissement à court terme des liants asphaltés. Une méthode employant des plateaux rotatifs remplis d'un mince film d'asphalte appelé essai d'un film mince au four (« thin film oven test, TFOT ») et une méthode employant des contenants d'asphalte cylindriques roulant appelé essai d'un film mince roulant au four (« rolling thin film oven test, RTFOT »). Dans cet article, une tentative est faite afin de développer des modèles généralisés pour les effets du vieillissement à court terme en utilisant le temps de vieillissement RTFOT comme référence. Six types de liant représentant deux niveaux de performance (« Performance Graded, PG ») et trois fournisseurs ont été conditionnés à différents niveaux de vieillissement RTFOT et testés en utilisant le rhéomètre dynamique de cisaillement (« Dynamic Shear Rheometer, DSR »). Les effets du vieillissement sont modélisés en utilisant des modèles indépendants de changements de température pour le module de cisaillement et l'angle de phase. Cet article discute des sources d'erreurs lorsque des modèles généralisés sont produits, et de quelques applications potentielles des modèles de vieillissement. La recherche a révélé qu'il est possible de développer et de mettre en œuvre de tels modèles pour des liants non-modifiés.

Mots clés : asphalte, vieillissement, RTFOT, DSR, rhéologie des liants, module de cisaillement, angle de phase.

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Introduction

Superpave binder specifications evaluate the performance characteristics of a binder using blind methods that subject each binder material to the same test procedures regardless of project specifics or intended use. The simplified and standardized test methods assign a high and low temperature to each binder within which the binder is expected to perform adequately. In this paper, one of the Superpave parameters, namely the time of the rolling thin film oven test, is closely examined to determine the influence of this parameter on binder selection and the feasibility of modelling binder conditions using short-term aging.

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Written discussion of this article is welcomed and will be received by the Editor until 30 June 2002.

Background to aging methods of asphalt binders

There are two widely used methods for subjecting asphalt binders to age hardening to simulate hot mix plant and placement conditions. These methods are the thin film oven test (TFOT) (ASTM D1754, AASHTO T179) and rolling thin film oven test (RTFOT) (ASTM D2872, AASHTO T240). The TFOT method originated in the 1940s and was adopted by AASHTO in 1959 and by ASTM in 1969. The RTFOT was proposed by the California Department of Transportation in 1959, received ASTM approval in 1970, and accepted by AASHTO in 1973. Both the TFOT and RTFOT are intended to simulate age hardening, which occurs in the pug mill of a hot mix batch plant operated at a maximum temperature of 150°C. Age hardening in a drum facility appears to be less severe because of the presence of

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Table 1. Comparison of TFOT and RTFOT test methods.

Parameter	TFOT (ASTM D1754)	RTFOT (ASTM D2872)
Sample placement	Steel pans	Glass bottle
Dimensions	140 mm O.D., 9.5 mm deep	64 mm O.D., 140 mm deep
Sample size	50 g per pan	35 g per bottle
Rotation	5.5 rev/min	na
Rolling action	na	15 rev/min
Forced air	na	Heated air jet
Test duration	300 min	85 min
Test temperature	163°C	163°C
Limitations	(*i*) Skinning, a thin crust forms on sample surface (*ii*) test duration is too long for quality control/quality assurance	(*i*) Binder may leak from bottle particularly if polymer modified (*ii*) potential for segregation in modified binders

Fig. 1. Rolling thin film oven test: (*a*) oven showing sample placement and (*b*) glass sample containers before and after the test.

(a) (b)

water vapour in the drum, which reduces oxidation (Roberts et al. 1996). The key features of TFOT and RTFOT are shown in Table 1. Although there are many similarities between the two testing methods, several researchers concluded that the two tests are not interchangeable and that the RTFOT appears to be more severe on the majority of binders than TFOT (Zupanick 1994).

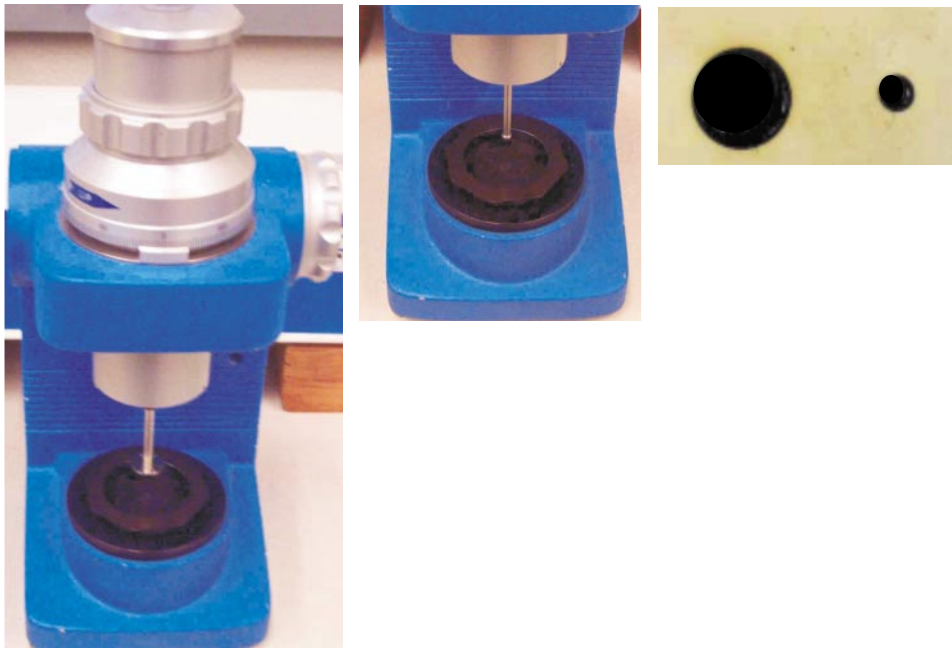
The rolling thin film oven test (Fig. 1) measures the effect of heat and air on a moving film of semi-solid asphaltic binder (ASTM 1995). In this test, the 35 g asphalt sample is placed in a glass bottle, which has a narrow top opening. The glass containers are placed in a carriage such that the axis of revolution is horizontal and the container opening is facing a jet of air. The oven is kept at 163°C and the carriage is rotated in the oven at a rate of 15 rev/min for 85 min. This aging process continuously exposes a film of asphalt binder to heat and oxidation similar to conditions experienced in the production of hot mix asphalt concrete.

The aging time of 85 min was arbitrarily selected to classify all binders to the same extent of aging produced in the TFOT. The aging is expected to represent a majority of conditions where the asphalt is considerably aged from the first exposure to the plant burner and contact with hot aggregates, throughout hauling and paving and until the final compaction takes place. The actual time that an asphalt binder is subjected to heat and oxidation varies from few minutes in the case of hot-in-place recycling to several hours in the case of plant mixing and long hauling distances or paving delays. Temperature management is essential in field operations to maintain the heat in the mix and hence the lower viscosity to achieve the required compaction density. Research leading to the development of the RTFOT test indicated that aging of 85 min produces aging effects comparable to average field conditions, although it is common that asphalt materials are exposed to heat and oxygen to cause hardening patterns significantly different from those produced at 85 min.

The Superpave binder specifications were produced by the Strategic Highway Research Program (SHRP) (FHWA 1995) and adopted RTFOT for short-term aging. The test is followed by conditioning in a pressure aging vessel (PAV) to simulate several years of long-term aging. Binders tested following PAV conditioning have shown no significant difference in DSR and viscosity response between TFOT and RTFOT short-term aging methods, which indicates that the effects of long-term aging far exceeds the limited oxidation caused by short-term aging methods (Phromsorn and Kennedy 1996).

Fig. 2. Dynamic shear rheometer test: (*a*) apparatus with 25 mm loading plate; (*b*) 8 mm loading plate; and (*c*) test samples for high temperature (25 mm diameter) and intermediate temperature (8 mm diameter).

(a) (b) (c)



Problems were encountered when modified asphalts were used in SHRP RTFOT protocols (Sirin et al. 1998). First, asphalts modified with crumb rubber and styrene butadiene rubber (SBR) tend to spill out from the container bottles during the RTFOT process. When the TFOT process was used, it was found that a thin skin would tend to form on the surface of some modified asphalt samples, which reduced the homogeneity and retarded the aging of the samples. Alternative aging methods using microwave ovens and a modified rotavapor apparatus have been studied and documented (Sirin et al. 2000; Bishara and McReynolds 1995, 1996). However, these methods remain at the research stage and none are recommended for general use at this time. RTFOT protocols for modified binders are being developed under NCHRP Project 9-10 (Bahia et al. 1998).

Rheological properties of asphalt binders

The viscoelastic nature of asphalt binders and the multitude of states that these materials undergo in the construction and service phases warrant the use of temperature controlled and loading rate controlled testing. This is achieved at least in part by the use of dynamic shear rheometer (DSR) shown in Fig. 2. The test determines the complex shear modulus and phase angle under specified temperature and loading frequency. In Superpave binder specifications, the test is conducted at a rate of 10 rad/s, at both high and intermediate temperature ranges. In the high temperature range of 46–70°C, a 1 mm thick and 25 mm diameter test sample is placed between two parallel plates. In the intermediate temperature range of 7–40°C, a 2 mm thick and 8 mm diameter sample is placed between the two plates. The change in sample size is necessitated by the increase in asphalt viscosity at low temperatures and the limited load capacity of test equipment. The complex binder response can be decomposed into a storage modulus $G' = G^* \cos(\delta)$ and a loss modulus $G'' = G^* \sin(\delta)$, as shown in Fig. 3. The storage modulus represents the elastic component and the loss modulus represents the viscous component of the asphalt binder stiffness. Findings of SHRP research suggested that the fatigue life of a pavement is proportional to the loss modulus G'' or the complex shear modulus G^* (Leahy et al. 1994). The rutting resistance is proportional to $G^*/\sin(\delta)$, G^*, or G'. SHRP research concluded that incorporating the phase angle in the terms $G^* \sin(\delta)$ in fatigue and $G^*/\sin(\delta)$ in rutting is not necessary for neat binders but may have a greater influence on modified binders (Anderson et al. 1994).

Research objectives and methodology

This investigation adopted RTFOT as a benchmark for aging extent to develop and evaluate models for characterization of binders subjected to selected levels of short-term aging. The experimental program is limited to RTFOT aging between 0 min (unaged) and 180 min. The research utilized six types of the commonly used binders in Manitoba to be tested using the DSR temperature sweep in the range of 7–70°C at a loading frequency of 10 rad/s.

Materials

In the penetration grading system, three of the binders qualified as 150–200 pen and the other three were 200–300 pen. As shown in Table 2, the binders were characterized as PG58-28 and PG52-34 according to PG designations. Samples of binders were split and each binder was subjected to one of four levels of RTFOT using aging times of 0 min (unaged), 40 min, 85 min, and 180 min. The binders were tested in the DSR to determine the complex shear modulus

Fig. 3. Interpretation of complex shear modulus G^*, storage modulus G', loss modulus G'', and phase angle δ: (*a*) stress–strain response and (*b*) vectorial relationship between G', G'', and G^*.

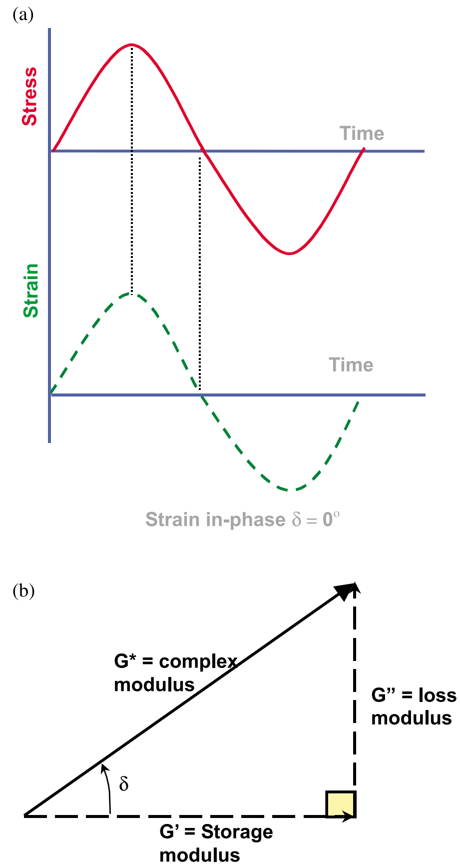


Table 2. Characterization of tested binders using penetration and PG grading systems.

Binder designation	Penetration grade ASTM D946	PG grade ASTM D6373
A1	150–200 pen	PG 58-28
A2	150–200 pen	PG 58-28
A3	150–200 pen	PG 58-28
B1	200–300 pen	PG 52-34
B2	200–300 pen	PG 52-34
B3	200–300 pen	PG 52-34

G^* and phase angle δ for temperatures between 7°C and 70°C. Aging releases volatile components of the binders resulting in mass loss and a stiffening effect. Over the life span of a binder, and as a result of aging and heat exposure during construction, the shear modulus increases by several decades while the phase angle decreases. Both effects lead to a more elastic response to loading.

DSR test results

The $G^*/\sin(\delta)$ for all binders after 85 min of short-term conditioning are shown in Fig. 4. DSR testing in this research program is conducted using a single loading frequency of 10 rad/s. It is common to construct rheological responses of polymer materials at a spectrum of several decades of frequencies; however, this program has only focused on the effects of short-term aging and Superpave test specifications require that testing be conducted at 10 rad/s. The graphs show that while the binders exceed the limit of 2.2 kPa at 58°C required for proper compaction and early strength, they have dissimilar performance characteristics. There is also a visible kink in the obtained response in the temperature range of 30–46°C due to the change in sample size and approaching the upper limit of output measurements for 25 mm samples and the lower limit for measurements for the 8 mm sample. It is evident that this behaviour is a limitation of test equipment and is not due to a physical change of the binder response to loading.

Construction of master curves and models

The experimental DSR test results for binder B3 are shown in Fig. 5 which illustrates sets of G^* and δ at four levels of aging of 0, 40, 85, and 180 min. If a linear viscoelastic binder response is assumed, a master curve can characterize the binder response from a range of test temperatures, aging times, or test frequencies using the principle of time–temperature interchangeability. In this context, the response of an aged binder at a specific temperature is equivalent to the response of a less aged binder under a lower temperature. A master curve is produced from a family of responses by assigning a temperature shift to each response so that it partially overlaps the response at the reference aging time (Fig. 6). The master curve and associated tempera-

Fig. 4. $G^*/\sin(\delta)$ after 85 min of RTFOT at 10 rad/s: (*a*) 150–200 pen (PG 58-28) binders and (*b*) 200–300 pen (PG 52-34) binders.

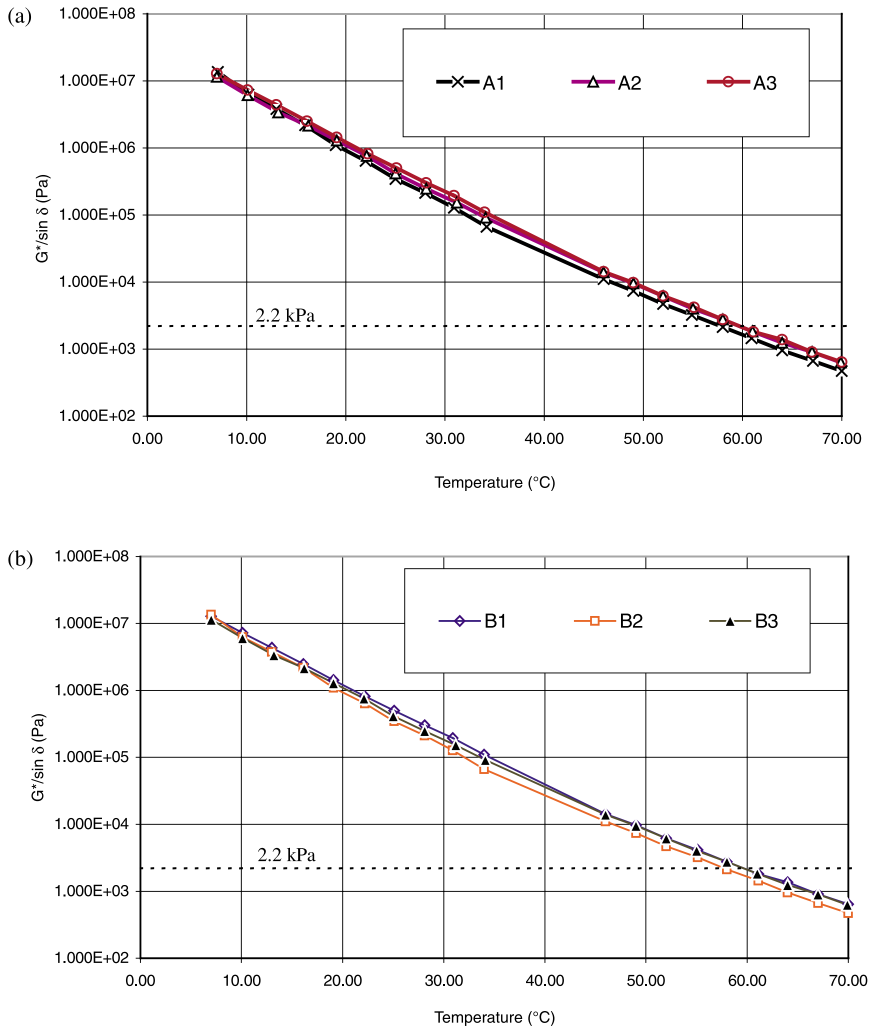


Fig. 5. Rheological response of binder B3 from experimental DSR data at 10 rad/s.

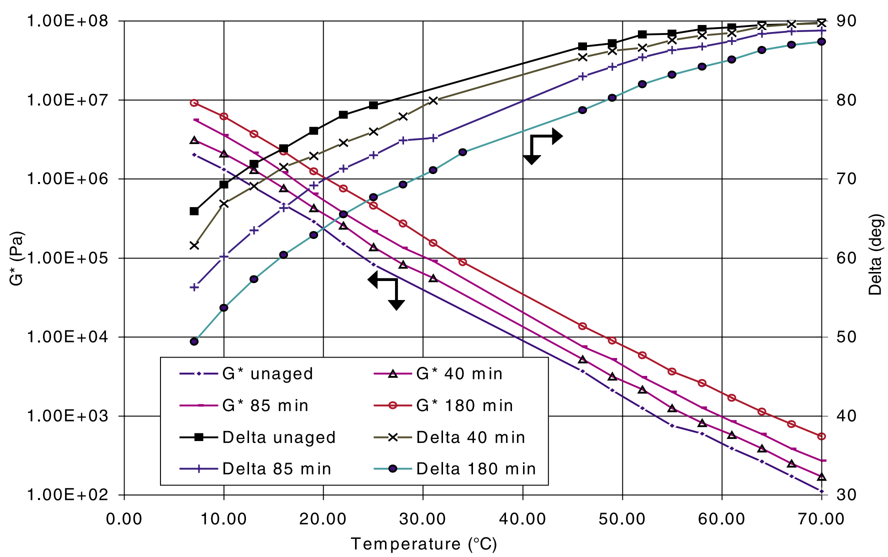


Fig. 6. Construction of a master curve from a family of responses.

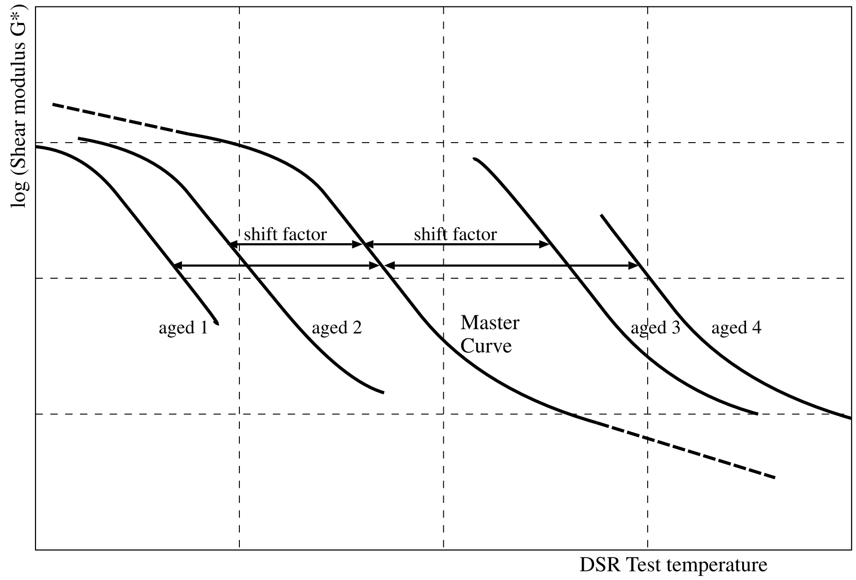
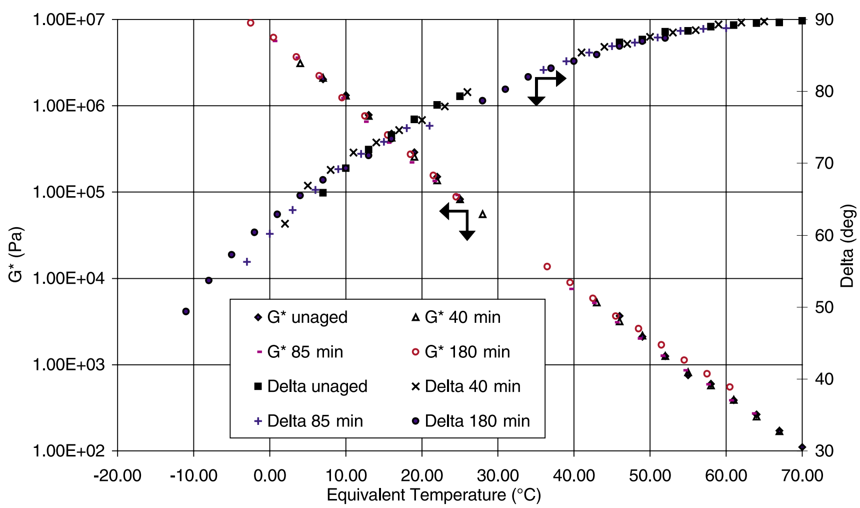


Fig. 7. Master curve of rheological response of binder B3 at 10 rad/s.



ture shift models are expressed by continuous mathematical functions. The original response can be reconstructed from these functions.

Master curves for G^* and δ are produced using visual shifting of the responses of aged binders to the unaged response. Visual shifting is a simple graphical technique that involves selecting shift factors manually by trial and error without the use of optimization techniques. Despite the superiority of computational optimization techniques over visual shifting and their inherent ability to minimize errors, they were not used in this research. It is essential at this stage to demonstrate the validity of the process.

The master curves for binder B3 produced using visual shifting are shown in Fig. 7 and the associated shift factors are shown in Fig. 8. The aging shift factors are modelled as parabolic functions and referenced to unaged binders (0 min). The shift functions are forced through the origin (0, 0) to eliminate shifting of the unaged binder response. The temperature shift coefficients are obtained using least squares regression. An exponential model is selected for the master curve of shear modulus while the phase angle is fitted to a second-degree polynomial. The model forms are as follows:

$$[1] \quad \log_{10}[G^*(t, T)] = a_1 + a_2[T - (a_3t^2 + a_4t)]$$

$$[2] \quad \delta(t, T) = b_1[T - (b_4t^2 + b_5t)]^2 + b_2[T - (b_4t^2 + b_5t)] + b_3$$

where t is the aging time in RTFOT; T is the DSR test temperature; a_1 and a_2 are the G^* model coefficients; a_3 and a_4 are the G^* temperature shift coefficients; b_1, b_2, and b_3 are the model coefficients for phase angle; and b_4 and b_5 are the temperature shift coefficients for phase angle.

The coefficients a_1, a_2, b_1, b_2, b_3 are obtained using least squares regression of eqs. [1] and [2] and using the temperature shift coefficients a_3, a_4 and b_4, b_5. The coefficients of the complex shear modulus models, a_1, ..., a_4, are shown in

Fig. 8. Temperature-aging shifting model of binder B3.

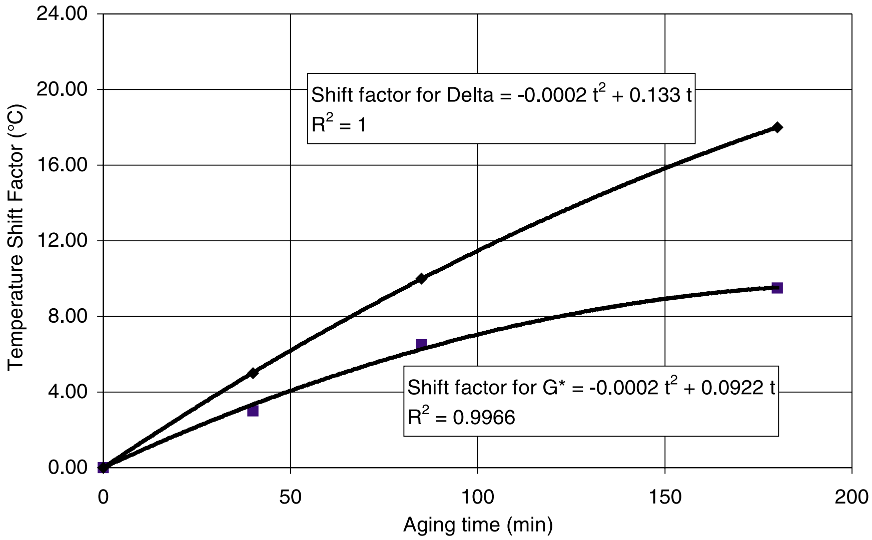


Table 3. Regression model coefficients for shear modulus.

Binder model	Model coefficients			
	a_1	a_2	a_3	a_4
A1	6.95941	–0.06683	–0.00012	0.06842
A2	6.93835	–0.06654	–0.00015	0.08288
A3	7.02731	–0.06944	–0.00013	0.08096
B1	6.84421	–0.06909	–0.00013	0.08096
B2	6.47068	–0.06516	–0.00024	0.11027
B3	6.72566	–0.06864	–0.00022	0.09223

Table 4. Regression model coefficients for phase angle.

Binder model	Model coefficients				
	b_1	b_2	b_3	b_4	b_5
A1	–0.00454	0.77300	57.54632	–0.00021	0.12583
A2	–0.00509	0.82120	56.45225	–0.00027	0.15474
A3	–0.00690	0.94248	56.80479	–0.00026	0.16878
B1	–0.00551	0.94763	50.36991	–0.00025	0.14579
B2	–0.00496	0.74738	61.77470	–0.00017	0.14705
B3	–0.00657	0.85507	61.33055	–0.00018	0.13301

Table 3 and the coefficients of phase angle models, b_1, ..., b_5, are shown in Table 4. The reconstructed response of binder B3 obtained from the models is shown in Fig. 9.

Comparison of model estimates and experimental measurements

The prediction error is defined as

$$[3] \qquad \text{percent error}(t, T) = [\text{model } G^*(t, T) - \text{experimental } G^*(t, T)] / \text{experimental } G^*(t, T) \times 100$$

The percent of root-mean-squared error %RMSE is calculated for each binder type using

$$[4] \qquad \%\text{RMSE} = \sqrt{\frac{\sum_{i=1}^{m}\sum_{j=1}^{n}[\text{percent error}(t_i, T_j)]^2}{mn}}$$

where t_i is the aging time, T_j is the test temperature, m is the number of aging times, and n is the number of test temperatures.

The model statistics are shown in Table 5. The errors are higher close to the middle range of temperatures (30–46°C) as well as at extreme low and high temperatures as shown in Fig. 10 due to the variability introduced by changing the sample size from 8 to 25 mm. To that effect, the extrapolated model values may in fact produce a better representation of binder properties than experimental measurements. In other words, by removing the undesirable bias in the experiment, the model values are closer to the true binder behaviour than experimental measurements. The root-mean-squared percent error in G^* is much higher than that in δ. The upper and lower bounds of the errors represent the absolute highest and lowest percent difference between estimated and measured values and are shown in Table 6. As the range of G^* spans many decades of magnitudes, these errors are not at all surprising. The change in binder shear stiffness is very sensitive to temperature with an average decline of 20% for each 1.0°C rise in DSR test temperature. Conversely, the phase angle increases by 1% under the same conditions. These values can also be easily verified from the models. The measurement and control of test temperature is usually accurate to 0.1°C. The error in estimating the phase angle δ appears to be acceptable.

The predictions can be improved if independent models are used for the high temperature and the intermediate temperature; however, this will result in an increased complexity of the models and associated shift factors. The error in δ is insignificant except at the lowest test temperature of 7°C, which reflects the inability of the model to capture this extreme range.

The results presented in this paper relied on the visual shifting and fitting of the generated models. It is possible to

Fig. 9. Reconstructed rheological response of binder B3 from model.

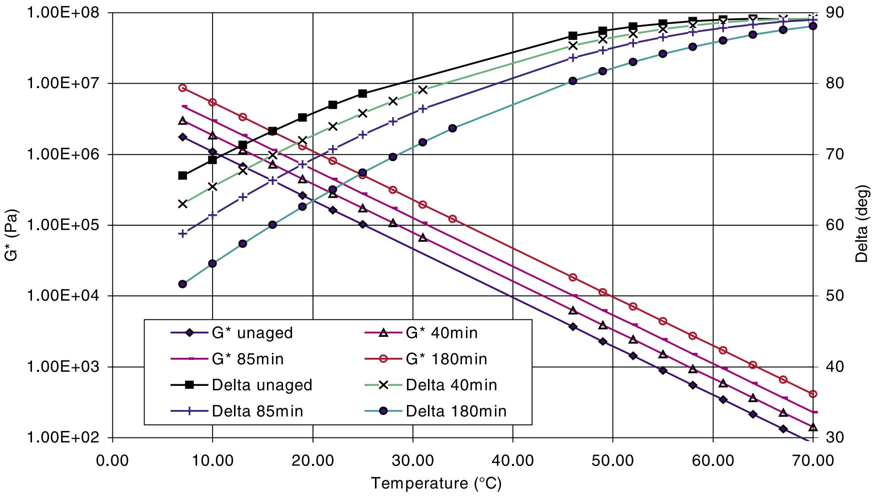


Table 5. Associated statistics for short-term aging models.

	Binder model					
Model statistics	A1	A2	A3	B1	B2	B3
Shear modulus *G**						
R^2	0.967	0.966	0.956	0.911	0.968	0.997
%RMSE	20.101	20.723	18.481	22.297	21.098	16.013
Phase angle δ						
R^2	0.993	0.991	0.994	0.997	0.997	0.994
%RMSE	1.474	1.778	1.568	1.356	0.896	1.218

Note: R^2, coefficient of determination; %RMSE, root-mean-squared percent errors.

minimize prediction errors by implementing a mathematical optimization technique. Such a procedure was not implemented in this analysis, since there are more significant sources of error in the experimental setup, repeatability and test equipment that must be addressed prior to implementing optimized aging models.

Model applications

Under some foreseen conditions, construction delays and long hauls can be accounted for at the design stage. A change in binder grade or source may be warranted if the short-term aging effects can result in a significant change of the binder properties. This analysis may help resolve some compaction issues such as mix tenderness or difficulties in attaining required density.

Discussion

RTFOT evolved to replace TFOT as a short-term aging procedure. The test is part of the Superpave protocol for short-term aging. Under this protocol, binders are tested after RTFOT to have a $G^*/\sin(\delta)$ exceeding 2.2 kPa in DSR at a grade-dependent test temperature, then aged in PAV, and retested in DSR to have a $G^* \sin(\delta)$ lower than 5 MPa at a grade-dependent test temperature.

A methodology is developed to analytically determine the effects of short-term aging and test temperature on binder response or aging sensitivity. At this stage, the models seem to be reasonably able to simulate binder response at any aging or temperature condition. The shear modulus of asphalt generally changes by several decades within the tested range of 7–70°C. In order to improve the test precision and because of the limitations of DSR equipment, it is required to use a smaller sample size at intermediate temperatures than the sample size used at high temperatures. There is a loss of accuracy associated with the change in sample size particularly at the intermediate temperatures of +30 to +46°C. The models determine a best fit across the entire temperature range and help reduce the errors associated with changing the sample size. The fitted models also alleviate deviations of individual test results providing a smoother transition over the full test range. The models allow for the extrapolation of RTFOT aging time and test temperature to estimate binder properties outside the test range or outside the capability of DSR test equipment. Although extensive testing was performed in order to develop the master curves, it is anticipated that a much smaller number of tests will be required once the model forms are established. Master curves for unmodified binders can be developed from tests at two aging levels and four test temperatures.

The models utilize the aging time in the RTFOT test as a benchmark for aging. It is feasible to correlate the RTFOT aging time to hot mix plant and construction aging. The models can then determine the extent of aging and the impact on mix performance under a variety of scenarios, such

Fig. 10. Percent prediction error (model to experiment) for binder A1: (*a*) error in shear modulus and (*b*) error in phase angle.

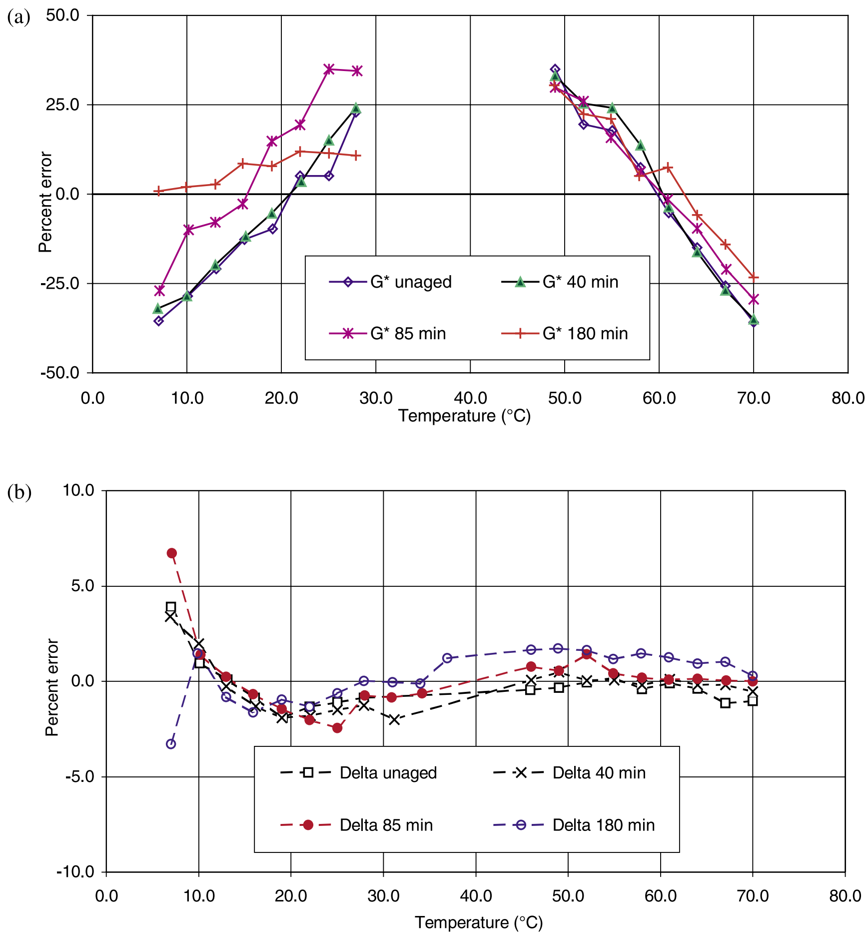


Table 6. Upper and lower percent error bounds of binder models.

Model error bounds	Error (%) A1	A2	A3	B1	B2	B3
Shear modulus *G**						
Lower bound	–35.80	–50.06	–38.23	–28.01	–27.62	–25.00
Upper bound	34.98	27.57	30.44	69.60	59.00	31.43
Phase angle δ						
Lower bound	–3.28	–2.53	–2.23	–2.96	–1.05	–2.18
Upper bound	6.72	9.22	5.61	3.25	3.51	4.59

as potential of delayed paving, long hauling distance, or prolonged exposure to plant burners. Binder models or charts can be developed by oil suppliers and verified or spot-checked by user agencies. Although the current PG grading specifications utilize a pass–fail system and a set of standardized conditions, development of aging models can allow for specific conditions where delays, short mixing time, or short hauling distances are anticipated. A more informed selection of the binder would be made using this method.

Conclusions

The RTFOT test is selected to establish a benchmark for short-term binder aging and produce binder aging models. Using a temperature sweep test, sufficient data were generated to model the effects of short-term aging on six binders using the DSR. The paper shows that models can be developed for binder aging using independent model forms and independent temperature shift factors for each of *G** and δ. The models consisted of a temperature–time shift parabolic function and an exponential master curve for complex shear modulus and a second-degree polynomial for phase angle. The models appear to capture the binder characteristics adequately and can have several practical uses. The temperature shift factors for *G** are consistently smaller in magnitude than those for δ for all the binders tested in this program, indicating that *G** is less sensitive to short-term aging than δ. The analytical modelling approach can be used to resolve issues related to mix tenderness, mix compactibility, and similar events sensitive to short-term aging. To facilitate the use of RTFOT as a benchmark for aging, there is a need to quantify the effects of mixing and construction in the RTFOT time scale.

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