

# Rolling Thin Film Oven Test and Pressure Aging Vessel Conditioning Parameters

## Effect on Viscoelastic Behavior and Binder Performance Grade

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This paper presents a sensitivity analysis of two parameters applied for the rolling thin film oven test (RTFOT) and pressure aging vessel (PAV) aging on the viscoelastic behavior and performance grade of asphalt binder PG 58-22. For the RTFOT, the temperature was varied from the default temperature of 163°C to 143°C and to 183°C. For the PAV, the binder film thickness was varied from the default 3.2 mm to 1.0 and 5.0 mm. Dynamic shear rheometer (DSR) tests were run with a temperature sweep from 46°C to 82°C at a frequency of 1.59 Hz on the virgin binder, as well as on the RTFOT and RTFOT+PAV-aged samples. Bending beam rheometer (BBR) tests were carried out at -12°C, -18°C and -24°C on the RTFOT+PAV-aged samples. The effect of the mentioned conditioning parameters on  $|G^*|$  and  $\delta$ —as well as on the upper and lower performance grade—was investigated. Results show that the effect of RTFOT temperature on the  $|G^*|$  and  $\delta$  is more distinct for lower DSR temperatures. In general, the effect of a change in the RTFOT temperature on the viscoelastic parameters can be considered as small. At 46°C DSR, a change of 1°C in the RTFOT temperature shifts  $|G^*|$  by 2.2% and  $\delta$  by 0.08°. The PAV binder film thickness has an exponential effect on  $|G^*|$  and  $\delta$ . A reduced RTFOT temperature leads to reduced long-term aging after PAV. Reduced short-term aging owing to reduced production temperatures results in significantly reduced long-term aging. The effect of RTFOT temperature and PAV binder film thickness on upper and lower performance grade was found to be insignificant.

Asphalt binder as a product from a crude oil refinery is an organic material and is thus susceptible to changes in chemical composition, microstructure, and mechanical behavior over its life span (1–5). These changes are generally referred to as aging. Aging of an asphalt binder in road construction is divided into short-term aging during transportation, mix production, and paving. Short-term aging is characterized by high temperatures, a high specific surface during mix production, and thus, evaporation of the remaining volatile components and fast oxidation (6, 7). Long-term aging comprises changes in the binder during the in-service life of a pavement layer

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in the field. It is triggered mainly by slow oxidation and the effects of ultraviolet radiation (8). Ultraviolet radiation is especially relevant for polymer modified binders (9, 10).

Various lab methods exist that simulate aging to assess the stability of a binder to short-term and long-term aging. Of these, the rolling thin film oven test (RTFOT) (11–13) and the pressure aging vessel (PAV) (14, 15) are validated, standardized, and commonly used methods to simulate short-term and long-term aging, respectively.

Two issues are worthy of examining more closely for both aging methods:

- In recent years, warm-mix asphalt with reduced production temperatures has achieved a significant share in asphalt mix production. One question of interest is how the change in production temperatures affects the short-term and long-term aging of the asphalt binder. Is a potential benefit owing to reduced short-term aging also beneficial for long-term aging?

- In regard to PAV aging, it is known that the binder film thickness in standard PAV (3.2 mm) is significantly higher than the binder film on aggregates in asphalt mixes (16, 17). Although PAV never aimed to simulate a realistic binder film thickness, understanding how the binder film thickness in PAV affects the long-term aging of asphalt binders is still seen as an interesting topic.

### OBJECTIVES AND APPROACH

From the issues stated above, this paper aims for the following objectives:

- Investigate the effect of the RTFOT conditioning temperature on short-term binder aging by varying the RTFOT temperature and testing aged samples by means of the dynamic shear rheometer (DSR).

- Investigate the effect of PAV binder film thickness on long-term binder aging by varying the film thickness and testing aged samples by means of the DSR and bending beam rheometer (BBR).

- Analyze the effect of changes in the RTFOT temperature and PAV binder film thickness on the performance grading of the asphalt binder.

- Investigate the effect of RTFOT temperature on long-term binder aging to determine whether lower production temperatures have a potentially beneficial effect on long-term aging.

TABLE 1 Binder Characteristics

Parameter	Value
Penetration at 25°C (1/10 mm)	91
Softening point ring and ball (°C)	46.0
PG (-)	58-22

## MATERIALS

One unmodified asphalt binder (PG 58-22, 70/100 pen) was used for the purposes of this study. The main characteristics of the binder are shown in Table 1.

## TEST PROGRAM

### Rolling Thin Film Oven Test

Simulation of short-term aging of the asphalt binder was carried out by the RTFOT according to ASTM D2872-12e1. To study the effect of temperature on short-term aging, three temperatures were applied: 143°C, 163°C, and 183°C.

### Pressure Aging Vessel

Simulation of long-term aging of the asphalt binder was realized by PAV according to ASTM D6521-13 at a temperature of 100°C. Five binder film thicknesses were used in the study to analyze the effect on long-term aging.

Table 2 shows the variations in the RTFOT temperature and PAV film thickness considered in the study. The film thicknesses of 1.0, 2.0, 3.3, 4.0, and 5.0 mm correspond to 15, 30, 50, 61, and 76 g of binder per PAV pan, respectively.

### Dynamic Shear Rheometer

The rheological properties—complex modulus  $|G^*|$  and phase lag  $\delta$ —of the virgin and aged binder samples at high temperatures were obtained by DSR tests according to ASTM D7175-08 at 1.59 Hz and a temperature sweep from 46°C to 82°C with 6°K steps. The

samples were tested with 25 oscillations at each temperature. Most of the test was carried out with triple repetition.

### Bending Beam Rheometer

BBR tests were run on all PAV-aged samples according to ASTM D6648-08 to measure the stiffness and relaxation capability at low temperatures. Samples were tested at -12°C, -18°C, and -24°C.

## RESULTS AND DISCUSSION

### Complex Modulus and Phase Lag at High Temperatures: DSR Tests

The complex modulus  $|G^*|$  and the phase lag  $\delta$  from DSR tests are presented and discussed in this section to analyze the effect of conditioning parameters in RTFOT and PAV on the high temperature behavior of the binder.

All DSR tests for the virgin binder and the RTFOT-aged samples were run with triple repetition. The PAV-aged binders were tested with triple repetition for samples RTFOT aged at 143°C and RTFOT aged at 183°C. The PAV-aged binders from samples RTFOT aged at 163°C were tested only once since it was found that the scattering of results is similar regardless of the conditioning temperature in RTFOT.

#### Impact of RTFOT Conditioning Temperature

Figure 1 shows the effect of the RTFOT temperature on the complex modulus in the left diagram and on the phase lag in the right diagram. Two DSR temperatures were chosen for the presentation in the diagrams: 46°C and 82°C, respectively. The diagrams contain the mean values of three repetitions and error bars indicating the standard deviation.

In regard to the complex modulus, the diagram shows the relative change in  $|G^*|$  compared with the virgin binder. A value of 0 would indicate that no change in  $|G^*|$  was derived. At all test temperatures, there is a linear trend between RTFOT temperature and change in  $|G^*|$ . At lower temperatures (46°C), the effect of a change in the aging temperature was more severe than for higher temperatures (82°C). A change of 1°C RTFOT temperature leads to a change of 2.2% in  $|G^*|$  at 46°C and 1.4% at 82°C.

For the phase lag (right diagram), similar trends can be found. The diagram shows the absolute change in phase lag between virgin and RTFOT aged binder. A value of 0 would indicate that no change in the phase lag occurred. Again, the effect of the RTFOT temperature is stronger for lower temperatures. At 46°C, a change of 1°C in the RTFOT temperature leads to a change of 0.08° in the phase lag, at 82°C this change is only 0.01°.

Taking into account the mean value and standard deviation of the RTFOT aged sample at 163°C (standard aging conditions), a *t*-test was carried out to investigate which change in the RTFOT temperature will lead to significant changes in the behavior of the respective aged binder: at 46°C, significant changes in  $|G^*|$  can be expected when the RTFOT temperature changes by more than 33°C and in  $\delta$  when the RTFOT temperature changes by more than 12°C. At 82°C, significant changes in  $|G^*|$  may occur when the RTFOT temperature changes by more than 10°C and in  $\delta$  when the RTFOT temperature changes by more than 11°C.

TABLE 2 Variation of RTFOT Temperature and PAV Binder Film Thickness

Binder Film Thickness (mm)	RTFOT Temperature (°C)		
	143	163	183
1.0	x	x	x
2.0		x	
3.3	x	x	x
4.0		x	
5.0	x	x	x

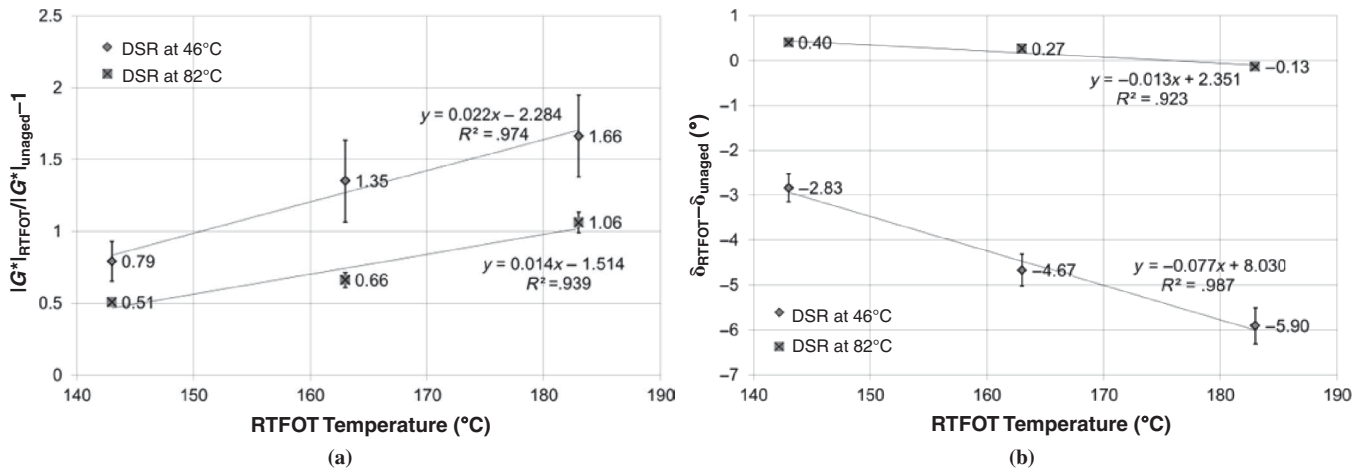


FIGURE 1 Impact of RTFOT temperature on (a) complex modulus and (b) phase lag.

*Impact of PAV Binder Film Thickness*

Figure 2 presents the effect of the binder film thickness in PAV pans on the high-temperature behavior of RTFOT+PAV aged samples. PAVs were carried out on samples from RTFOT aging at different RTFOT temperatures. The left diagram shows relative changes in  $|G^*|$  compared with  $|G^*|$  of the respective RTFOT aged sample. The right diagram shows the absolute change in  $\delta$  compared with the respective RTFOT aged sample. A value of 0 would indicate that no change in  $|G^*|$  occurs between RTFOT and PAV aging. Since the largest changes were found to occur at the lowest DSR temperature, only results from the DSR tests at 46°C are shown here. Similar trends can be seen for higher test temperatures but with the binder film thickness having a smaller effect.

Two essential observations can be made from the data depicted in Figure 2:

- No significant difference can be traced back to the RTFOT temperature when data from the PAV-aged samples are compared with the respective samples from RTFOT aging. The change in binder behavior from short-term to long-term aging does not seem to be affected by the RTFOT temperature.

- The binder film thickness has a strong effect on the long-term aging in PAV. As shown in the left diagram, there is an exponential trend. This trend seems to be rational since an increase in the binder film thickness will bring the long-term aged  $|G^*|$  asymptotically toward the short-term aged  $|G^*|$ . A reasonable explanation for this trend toward the short-term aged stage is that during a standard PAV with a duration of 20 h, only a thin layer of the sample is actually aged. The binder below this layer stays on the RTFOT aged level. Increasing the binder film thickness does not increase the layer of aged binder after PAV; it only increases the mass of binder below this layer that is still at an RTFOT aged level after PAV aging. Thus, increasing binder film thickness leads to a decrease in aging of the complete sample. In the theoretical case of indefinite binder film thickness, the binder behavior after RTFOT+PAV aging would be equal to the behavior after RTFOT. Analog statements can be made about the phase lag.

While Figure 2 compares changes between RTFOT and PAV-aged binder, Figure 3 compares changes between virgin and PAV-aged binders. With the data in Figure 3, the effect of RTFOT temperature and PAV binder film thickness on the complete short-term and long-term aging process can be assessed.

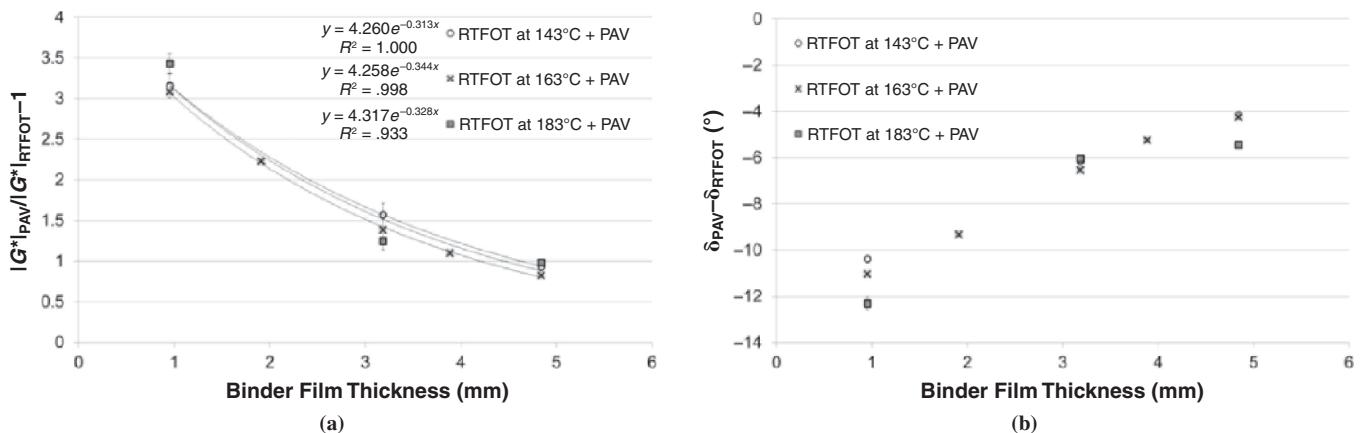


FIGURE 2 Impact of PAV film thickness on (a) complex modulus and (b) phase lag, RTFOT-aged binder versus PAV-aged binder.

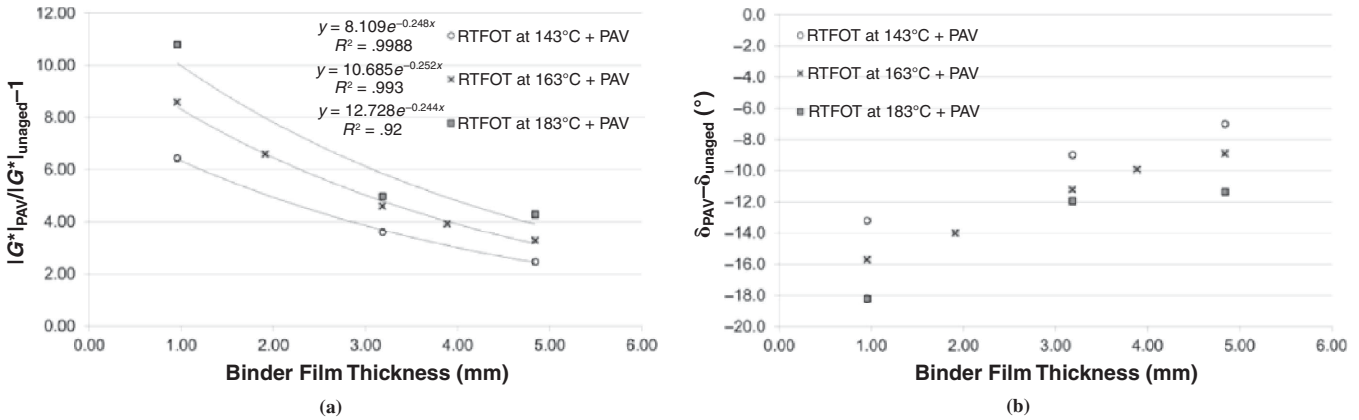


FIGURE 3 Impact of PAV film thickness on (a) complex modulus and (b) phase lag at 46°C, PAV-aged binder versus virgin binder.

Similar to in Figure 2, the change in  $|G^*|$  follows an exponential trend, proving the strong effect of the binder film thickness in PAV. Different from Figure 2, here the RTFOT temperature has a clear effect on the change in  $|G^*|$ . A higher temperature used for short-term aging leads to a stronger increase in stiffness after PAV compared with the virgin binder. While the effect of the binder film thickness is not affected by the RTFOT temperature (a similar exponent of regression function for all samples), the offset of the regression function changes with the RTFOT temperature. To provide an example at standard PAV conditions (3.3-mm PAV binder film thickness corresponding to 50 g of binder), a change from 163°C to 183°C RTFOT temperature results in a 22% higher  $|G^*|$ . However, a reduction from 163°C to 143°C RTFOT temperature leads to a 23% lower  $|G^*|$ . Again, similar trends can be observed for the phase lag.

With these results, it can be stated that the temperature during short-term aging has a significant effect on the behavior of the long-term aged binder.

**Performance Grading**

Results from the DSR and BBR testing were also analyzed relative to changes in the Superpave® performance grading. The effect of the RTFOT temperature on the upper performance grade and the effect of RTFOT temperature and PAV binder film thickness on the lower performance grade are shown in Figures 4 and 5, respectively. For

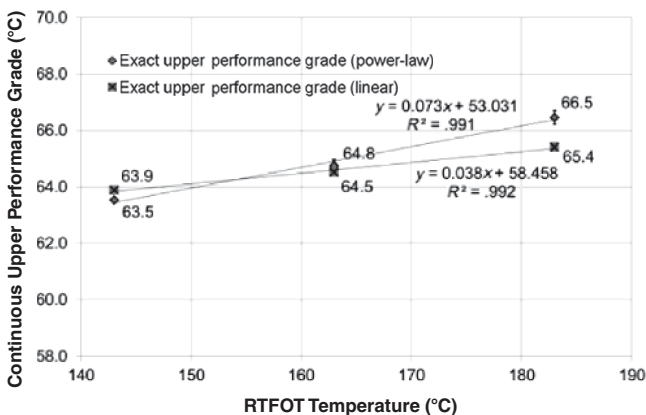


FIGURE 4 Impact of RTFOT temperature on upper performance grade.

both performance grades, the continuous values were calculated from the results of the DSR and BBR tests.

From the data in Figure 4, the tested binder would represent an upper performance grade of 64°C. However, DSR testing of the unaged binder sample resulted in an upper performance grade of 58°C. Thus, the binder is labeled PG 58-22.

For the upper performance grade (Figure 4), two ways of obtaining the continuous performance grade were chosen. In the linear case, the  $|G^*|/\sin(\delta)$  values were interpolated between each test temperature in a linear way. This linear interpolation was used to calculate the exact temperature at which 2,200 kPa was reached [continuous upper performance grade (linear) in Figure 4].

The  $|G^*|/\sin(\delta)$  versus temperature curve shows a power-law trend with a high coefficient of correlation ( $R^2 > .99$  for all samples) of the following form:

$$\frac{|G^*|}{\sin(\delta)}(T) = a \cdot T^b \tag{1}$$

where  $T$  is the test temperature. The respective curves were fitted with a power-law regression, and the exact temperature at which 2,200 kPa was reached was obtained from the regression [continuous upper performance grade (power-law) in Figure 4].

The method using the linear interpolation shows the RTFOT temperature having a smaller effect on the upper performance grade with a slope of 0.038 compared with the method using the power-law regression with a slope of 0.073. At the standard RTFOT temperature of 163°C, the two methods result in similar continuous upper performance grades. With the linear interpolation, to change the upper performance grade by 1°C, the RTFOT temperature would need to change by 27°C. For the power-law regression, a change of 14°C in the RTFOT temperature is necessary to change the upper performance grade by 1°C. Thus, it can be stated that the upper performance grade is relatively insensitive to inaccuracies in the RTFOT temperature.

For the continuous lower performance grade derived from the RTFOT+PAV aged samples that were tested in the BBR, the standard benchmark values of stiffness  $S \leq 300$  MPa and  $m$ -value  $\geq 0.3$  were used. Since the evolution of  $S$  and  $m$ -value versus the BBR test temperature is linear, the continuous lower PGs shown in Figure 5 were derived by linear interpolation of  $S$  and  $m$ -values between the BBR test temperatures.

The left diagram in Figure 5 presents the effect of RTFOT temperature and PAV binder film thickness on the lower performance

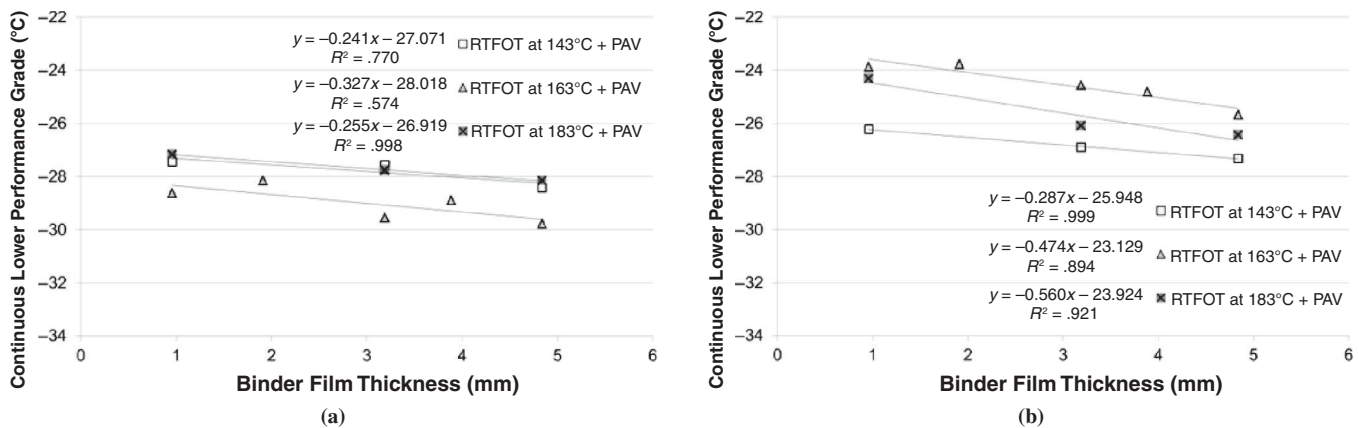


FIGURE 5 Impact of RTFOT temperature on lower performance grade with regard to (a) BBR stiffness and (b)  $m$ -value.

grade with regard to the stiffness  $S$ . The effect of the binder film thickness is consistent for all RTFOT temperatures. A change of 1 mm in the binder film thickness shifts the lower performance grade by 0.24°C to 0.33°C, whereas a change of 10 g of binder in a PAV pan (corresponding to a change of 0.6 mm in the binder film) leads to a change in the lower performance grade of 0.15°C to 0.21°C. From these data, the effect of the binder film thickness on the lower performance grade seems to be rather insignificant.

No consistent statement can be made for the effect of the RTFOT temperature. Results from RTFOTs at 143°C and 183°C are on the same level and produce slightly worse lower performance grades than RTFOTs at 163°C.

The right diagram in Figure 5 shows the effect of aging conditioning parameters on the lower performance grade with regard to the  $m$ -value. The effect of the binder film thickness is more distinct than for stiffness. A change of 1 mm in film thickness changes the lower performance grade by 0.28°C to 0.56°C. When a change in the binder mass in a PAV pan is taken into account, a change of 10 g of binder shifts the lower performance grade by 0.18°C to 0.36°C. The higher the RTFOT temperature is, the stronger is the effect of the binder film thickness. Although the effect of the binder film thickness on the lower performance grade is more pronounced for the  $m$ -value than for the stiffness, it can still be seen as rather insignificant.

Looking at the effect of RTFOT temperatures, again, no clear statement can be made. RTFOTs at 143°C lead to the lowest performance grades, which seems to be a rational result. But the ranking of RTFOTs at 163°C and 183°C is not consistent since the higher RTFOT temperature leads to a better low temperature performance grade.

## SUMMARY AND CONCLUSIONS

This paper presents a study of the effect of two essential parameters in RTFOT and PAV on the viscoelastic behavior and performance grade of an asphalt binder, PG 58-22 (70/100 pen). For the RTFOT, the temperature was varied from the default temperature of 163°C to 143°C and to 183°C. For the PAV, the binder film thickness was varied from the default 3.2 mm to 1.0 and 5.0 mm. DSR tests with a temperature sweep from 46°C to 82°C were carried out on the virgin binder, as well as on the different RTFOT and RTFOT+PAV-aged samples. The frequency was fixed at 1.59 Hz. BBR tests were carried out at -12°C, -18°C and -24°C on the RTFOT+PAV-aged samples. The effect of the mentioned conditioning parameters on

$|G^*|$  and  $\delta$ —as well as on the upper and lower performance grades—was investigated. The following conclusions can be drawn from the results:

- In general, the effect of a change in the RTFOT temperature on the complex modulus and phase angle is higher for lower test temperatures. A change of 1°C in the RTFOT temperature leads to an increase of 2.2%  $|G^*|$  or 0.08° phase lag at 46°C. At 82°C, this change is only 1.4%  $|G^*|$  and 0.01° phase lag, respectively.
- On the basis of a  $t$ -test, it can be stated that significant changes in  $|G^*|$  will occur when the RTFOT temperature changes by more than 33°C at a DSR temperature of 46°C and by more than 10°C at 82°C. For the phase lag, the necessary changes in RTFOT temperatures are 12°C at a DSR temperature of 46°C and 11°C at 82°C. Thus, the effect of the RTFOT temperature on the viscoelastic behavior appears to be small.
- The PAV binder film thickness has an exponential effect on  $|G^*|$  and the phase lag.
- An effect of the RTFOT temperature can also be detected after PAV long-term aging. A change in the RTFOT temperature from 163°C to 183°C brings a 22% increase in  $|G^*|$ , and a reduction from 163°C to 143°C brings a 23% decrease in  $|G^*|$ . Similar trends can be observed for the phase lag. According to these data, it can be concluded that a change in the production temperature of asphalt mixes does not have an effect on short-term aging only. Reduced short-term aging because of reduced production temperatures results in significantly reduced long-term aging.
- The effect of RTFOT temperature on upper performance grade is rather insignificant. The upper performance grade changes by 1°C when the RTFOT temperature is changed by 27°C. Analog statements can be made about the effect of PAV binder film thickness on the lower performance grade. A change of 10 g of binder per PAV pan shifts the lower performance grade by up to 0.36°C in the worst case. Thus, RTFOT and PAV seem to be rather robust to changes in the two investigated parameters.

## REFERENCES

1. Hofko, B., F. Handle, L. Eberhardsteiner, M. Hospodka, R. Blab, J. Füssl, and H. Grothe. Alternative Approach Toward Aging of Bitumen and Asphalt Mixes. Presented at 94th Annual Meeting of the Transportation Research Board, Washington, D.C., 2015.
2. Hofko, B., L. Eberhardsteiner, J. Füssl, H. Grothe, F. Handle, M. Hospodka, D. Grossegger, S.N. Nahar, A.J.M. Schmets, and A. Scarpas.



- Impact of Maltene and Asphaltene Fraction on Mechanical Behavior and Microstructure of Bitumen. *Materials and Structures*, 2015, pp. 1–13.
3. Mouillet, V., J. Lamontagne, F. Durrieu, J.P. Planche, and L. Lapalu. Infrared Microscopy Investigation of Oxidation and Phase Evolution in Bitumen Modified with Polymers. *Fuel*, Vol. 87, No. 7, 2008, pp. 1270–1280.
  4. Stulirova, J., and K. Pospisil. Observation of Bitumen Microstructure Changes Using Scanning Electron Microscopy. *Road Materials and Pavement Design*, Vol. 9, No. 4, 2008, pp. 745–754.
  5. Rebelo, L. M., J. S. de Sousa, A. S. Abreu, M. P. M. A. Baroni, A. E. V. Alencar, S. A. Soares, J. Mendes, and J. B. Soares. Aging of Asphaltic Binders Investigated with Atomic Force Microscopy. *Fuel*, Vol. 117, 2014, pp. 15–25.
  6. Galal, K. A., and T. D. White. SHRP PG Classification and Evaluation of In-Service Asphalts After Eight Years. In *Progress of Superpave (Superior Performing Asphalt Pavement): Evaluation and Implementation* (R. N. Jester, ed.), ASTM, Philadelphia, Pa., 1997, pp. 135–150.
  7. Im, S., and F. J. Zhou. Laboratory Short-Term Aging Protocol for Plant-Mixed and Laboratory Compacted Samples. *Construction and Building Materials*, Vol. 89, 2015, pp. 1–12.
  8. Chipps, J. F., R. R. Davison, and C. J. Glover. A Model for Oxidative Aging of Rubber-Modified Asphalts and Implications to Performance Analysis. *Energy and Fuels*, Vol. 15, No. 3, 2001, pp. 637–647.
  9. Wu, S. P., L. Pang, and G. J. Zhu. The Effect of Ageing on Rheological Properties and Chemical Conversions of Asphalts. *Advances in Fracture and Damage Mechanics VII*, 2008, pp. 481–484.
  10. Feng, Z. G., S. Xu, Y. B. Sun, and J. Y. Yu. Performance Evaluation of SBS Modified Asphalt with Different Anti-Aging Additives. *Journal of Testing and Evaluation*, Vol. 40, No. 5, 2012, pp. 728–733.
  11. Migliori, F., and J. F. Corte. Comparative Study of RTFOT and PAV Aging Simulation Laboratory Tests. *Asphalt Mixture Components*, Vol. 1638, 1998, pp. 56–63.
  12. Bahia, H. U., H. C. Zhai, and A. Rangel. Revisions of the Superpave Binder Specifications to Include Modified Binders. *Materials and Construction: Exploring the Connection*, 1999, pp. 657–663.
  13. Naskar, M., K. S. Reddy, T. K. Chaki, M. K. Divya, and A. P. Deshpande. Effect of Ageing On Different Modified Bituminous Binders: Comparison Between RTFOT and Radiation Ageing. *Materials and Structures*, Vol. 46, No. 7, 2013, pp. 1227–1241.
  14. Bahia, H. U., and D. A. Anderson. The Pressure Aging Vessel (PAV): A Test to Simulate Rheological Changes due to Field Aging. *Physical Properties of Asphalt Cement Binders*, Vol. 1241, 1995, pp. 67–88.
  15. Puello, J., N. Afanasjeva, and M. Alvarez. Thermal Properties and Chemical Composition of Bituminous Materials Exposed to Accelerated Ageing. *Road Materials and Pavement Design*, Vol. 14, No. 2, 2013, pp. 278–288.
  16. Khedaywi, T. S., and E. Tons. Aggregate Rugosity and Size Effect on Bituminous Mixes. In *Transportation Research Record: Journal of the Transportation Research Board*, No. 1619, TRB, National Research Council, Washington, D.C., 1998, pp. 26–36.
  17. Jung, Y., H. Lee, M. J. Lee, S. Choi, and Y. H. Cho. Concept of Film Thickness Applied to a New Approach for Polymer Concrete Mix Design for Airport Pavement Repair. In *Transportation Research Record: Journal of the Transportation Research Board*, No. 2440, Transportation Research Board of the National Academies, Washington, D.C., 2014, pp. 103–109.

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