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Development of a Matrix Analysis Methodology for Characterization of Short-Term Aging in Asphalt Binders Modified by Synthetic Wax

Ali Jamshidi ^{1,*} , Greg White ¹, Wim Van den bergh ² , Seyed Reza Omranian ² and Meor Othman Hamzah ³

¹ School of Science, Technology, and Engineering, University of the Sunshine Coast, Sunshine Coast 4556, Australia; gwhite2@usc.edu.au

² Energy and Materials in Infrastructure and Buildings, Faculty of Applied Engineering, University of Antwerp, Groenenborgerlaan 171, 2020 Antwerp, Belgium; wim.vandenbergh@uantwerpen.be (W.V.d.b.); SeyedReza.Omranian@uantwerpen.be (S.R.O.)

³ Sustainable Asphalt Research Group (SARG), School of Civil and Environment Engineering, Universiti Sains Malaysia, Nibong Tebal 14300, Pulau Pinang, Malaysia; cemeor@usm.my

* Correspondence: ajamshidi@usc.edu.au

Abstract: In this study, an innovative methodology is proposed to characterize the short-term aging of asphalt binders using the matrix analysis method. The rotational viscosity and complex shear modulus of asphalt binders were chosen as target rheological properties for the analysis of aging. A set of square matrices was developed based on test temperatures and the synthetic additive wax content. Transformational short-term aging matrices were obtained that characterize the trend of the aging process as a function of binder type, temperature sweep, and additive percentage. The results of the matrix analysis show that the trend of short-term aging depends on the binder performance grade and the rheological characteristic chosen for the analysis of aging. In addition, transformational aging matrices can provide detailed information about the range of the aging rate and the trend in aging for each binder type. Furthermore, the components of the transformational matrices clearly show the sensitivity of the binders to aging. In conclusion, the matrix analysis of aging can be used to compare the effects of short-term aging of different asphalt binders.

Keywords: warm mix additive; viscosity; viscoelastic properties; transformational aging matrix; complex shear modulus

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1. Introduction

Short-term aging of asphalt binders is a physico-chemical reaction that occurs during the mixing of aggregate particles and hot asphalt binders in mixing facilities, transportation, and construction. Therefore, the physical and chemical properties of asphalt binders change after the oxidation and volatilization that take place within the mixing process and during subsequent placement on-site. There are various procedures to simulate the complex mechanisms for the short-term aging of asphalt binders. In this regard, artificial laboratory aging procedures and protocols have been developed. For example, the thin-film oven test (TFOT), rolling thin-film oven test (RTFOT), rotating flask test, shell microfilm test, rolling microfilm test, tilt oven durability test, thin-film accelerated aging test, and modified RTFOT are experimental methodologies developed based on extended heat procedures [1,2]. Of these, TFOT and RTFOT are commonly used procedures for the simulation of short-term aging of asphalt binders. The aging depends on various factors, including binder type, source, mix construction temperature, mixing time, test procedure, aggregate type, gradation, anti-stripping additives, filler type, rejuvenator type, and asphalt binder modifiers [3–15]. There are some prediction models to evaluate the effects of short-term aging on the rheological properties of binders and the performance of mixtures. For example, Omranian et al. [16]

proposed a model-based artificial neural network to choose binder types based on the rheological properties of short-term aged binders. In addition, Haghshenas et al. [17] proposed a RTFO-based methodology for better simulation of short-term aging in unmodified and modified binders. In this regard, a statistical model using response surface methodology was developed that indicates that the time, temperature, and weight of the sample are significant independent variables on the rheological and chemical characteristics of aged binders.

To characterize the aging rate, the aging index (AI) is used, which is defined as the ratio between a rheological property of an aged binder or mix and the same property of the binder or mix sample when unaged [11,18,19]. However, the AI does not give any information about the synergistic effects of the asphalt binder additive content and test temperature. Furthermore, the AI does not demonstrate the process of aging occurring on the unaged binder as an input (Figure 1). Moreover, the interaction between the variables makes analysis of short-term aging complicated. Therefore, it is necessary to find a simpler method to study aging, especially when there are several variables, such as asphalt binder type and additive type, content, and variations in test temperatures. In this regard, the unaged binder can be assumed as a given raw material (Phase 1 or upstream), while the short-term aged binder is proposed as the output material (Phase 3 or downstream), as conceptualized in Figure 1. The artificial short-term aging conditioning is also supposed as processing (Phase 2 or middle part).

Figure 1. Schematic illustration of asphalt binder short-term aging process.

Phase 2 is an aging process carried out using laboratory facilities to simulate aging during mixture production and paving. Then, the output, or Phase 3, is the short-term aged asphalt binder. The AI values only show the changes in the rheological properties because of short-term aging, while it is not possible to characterize such changes and show how these changes happen in the chosen rheological properties (short-term aging process). Meanwhile, AI values are measured at each test temperature separately, while it would be realistic for the short-term aging process to be characterized at a temperature range chosen to characterize aging, because the aged binder may be exposed to various temperatures. This study fills these gaps:

- Proposing a new integrated methodology that simultaneously characterizes the effects of the test temperature and asphalt binder modifier.
- Characterizing the interaction between the effect of the binder type and the asphalt modifier content during the short-term aging process.

2. Materials and Methods

2.1. Asphalt Binder

Three types of asphalt binders were used. Table 1 shows the rheological properties of asphalt binders. PG 76-28 was a polymer-modified asphalt.

Table 1. Rheological characteristics of asphalt binders.

Aging State	Binder Type	Property	Values	Standard	
Unaged	PG 64-22	Viscosity at 135 °C (mPa.s)	340	ASTM D4402 [20]	
		$G^*/\sin \delta$ at 64 °C (kPa)	1.14	ASTM D 7175 [21]	
Short-term		Viscosity at 135 °C (mPa.s)	580	ASTM D4402 [20]	
		$G^*/\sin \delta$ at 64 °C (kPa)	2.68	ASTM D 7175 [21]	
Long-term		m-value at −18°C	0.35	ASTM D 6648 [22]	
		Stiffness at −18 °C (MPa)	277	ASTM D 6648 [22]	
		$G^*\sin \delta$ at 25 °C (MPa)	2.70	ASTM D 7175 [21]	
Unaged		PG 70-22	Viscosity at 135 °C (mPa.s)	575	ASTM D4402 [20]
			$G^*/\sin \delta$ at 70 °C (kPa)	1.04	ASTM D 7175 [21]
Short-term			Viscosity at 135 °C (mPa.s)	812.50	ASTM D4402 [20]
			$G^*/\sin \delta$ at 70 °C (kPa)	2.02	ASTM D 7175 [21]
Long-term			m-value at −18 °C	0.33	ASTM D 6648 [22]
	Stiffness at −18 °C (MPa)		292	ASTM D 6648 [22]	
	$G^*\sin \delta$ at 25 °C (MPa)		4.50	ASTM D 7175 [21]	
Unaged	PG 76-28		Viscosity at 135 °C (mPa.s)	3125	ASTM D4402 [20]
			$G^*/\sin \delta$ at 76 °C (kPa)	1.51	ASTM D 7175 [21]
Short-term			Viscosity at 135 °C (mPa.s)	3150	ASTM D4402 [20]
			$G^*/\sin \delta$ at 76 °C (kPa)	3.07	ASTM D 7175 [21]
Long-term			m-value at −18 °C	0.33	ASTM D 6648 [22]
		Stiffness at −18 °C (MPa)	281	ASTM D 6648 [22]	
		$G^*\sin \delta$ at 25 °C (MPa)	1.65	ASTM D 7175 [21]	

2.2. Asphalt Additive

Synthetic wax, namely Sasobit, was used as the asphalt modifier for each binder. Sasobit is used to produce warm mix asphalt (WMA) in the asphalt industry. The WMA technology allows the reduction of production and compaction temperatures of asphalt mixes, thereby decreasing the energy requirements and environmental burdens of pavement construction [23,24]. The percentage of Sasobit was from 1% to 4%, at 1% increments by mass of the asphalt binder.

2.3. Sample Preparation

The Sasobit and the hot binder samples were blended at 160 °C and stirred for 30 min to achieve a homogenous blend. Then, the binder sample was subjected to short-term aging by the rolling thin-film oven (RTFO) in accordance with the procedures outlined in ASTM D2872 [25].

2.4. Rotational Viscometer Test

A Brookfield rotational viscometer (RV) was used to measure the viscosity of the unaged and short-term aged asphalt binder samples. The temperature sweeps were performed from 120 °C to 160 °C, at 10 °C increments. Three readings were recorded for each measurement and the mean value was calculated as the final result.

2.5. Dynamic Shear Rheometer (DSR)

Viscoelastic properties of asphaltic materials can be characterized through the complex shear modulus (G^*) derived using the DSR. G^* values of the unaged and aged binder samples were measured at 10 Hz. The temperature sweep was performed from 46 °C to 82 °C, at increments of 6 °C. Three readings were recorded for each measurement and the mean value was calculated as the final result.

2.6. Proposed Methodology

In aging, Parameter A transforms to Parameter B, via a function of ψ . Mathematically speaking, ψ is a transition for two different moduli from Space A (unaged) to Space B (short-term aged), as shown in Equation (1).

$$\{A\} \cdot \{\psi\} = \{B\} \quad (1)$$

Therefore, ψ can be a constant value, linear operator, or multivariable function, depending on the number and trend of parameters included in the analysis of aging. In this study, the characterization of the short-term aging process is a two-dimensional problem, including the test temperature and Sasobit content. In other words, ψ indicates the short-term aging process as a transformation with two independent variables. Parameter A can be a rheological characteristic of the asphalt binder or engineering property of a mixture. The rotational viscosity and G^* were chosen as Parameter A—Equations (2) and (3):

$$\{\mu\}_u \cdot \{\psi\}_\mu = \{\mu\}_s \quad (2)$$

$$\{G^*\}_u \cdot \{\psi\}_{G^*} = \{G^*\}_s \quad (3)$$

where $\{\mu\}_u$ is the unaged viscosity, $\{\mu\}_s$ is the short-term aged viscosity, $\{\psi\}_\mu$ is the transformation operator of short-term aging (Step 2 in Figure 1) in terms of viscosity, $\{G^*\}_u$ is unaged G^* , $\{G^*\}_s$ is short-term aged G^* , and $\{\psi\}_{G^*}$ is the transformation based on the G^* of the asphalt binders.

To characterize the short-term aging process in terms of independent variables, a square matrix ($n \times n$) for Moduli A and B is proposed, as shown in Figure 2.

$$\begin{array}{c}
 \begin{array}{c} \updownarrow \\ n \end{array}
 \begin{array}{c} \leftarrow n \rightarrow \\ \begin{array}{ccc} a_{iu} & \dots & a_{nu} \\ \dots & \dots & \dots \\ \dots & \dots & \dots \\ \dots & \dots & \dots \\ Z_{mu} & \dots & Z_{ju} \end{array} \end{array}
 \cdot \{\psi\} =
 \begin{array}{ccc} a_{js} & \dots & a_{ns} \\ \dots & \dots & \dots \\ \dots & \dots & \dots \\ \dots & \dots & \dots \\ Z_{ms} & \dots & Z_{js} \end{array}
 \end{array}$$

Unaged matrix
Short-term aging matrix

where a_{iu} , a_{nu} , Z_{mu} , and Z_{ju} are components of the matrix of unaged asphalt binder, and a_{is} , a_{ns} , Z_{ms} , and Z_{js} are components of the short-term aged binder.

Figure 2. Matrix of unaged and short-term aged asphalt binders.

The column and row are the independent variables chosen to study the short-term aging process. The components of each asphalt binder type can be different compared with the other asphalt because of various viscosities and G^* . In the matrix analysis, it is assumed that there is a unique output for each input. For example, a_{iu} , the unaged property of the asphalt binder or mixture, corresponds to a_{js} (short-term aged property of the binder). The main advantage of the proposed matrix analysis of short-term aging is that

the transformational aging matrix shows the trend of each component in terms of the independent variables, such as Sasobit content and test temperature. Therefore, the short-term aging process can be characterized in terms of any rheological characteristics chosen as the indicator of short-term aging, such as rotational viscosity and G^* . Moreover, there is no limitation to the number of inputs and outputs. However, it is recommended to build a square matrix to find the transformational matrix based on a linear operator. The big matrices require mathematical software to find the operator.

Figure 3 shows input and output matrices of asphalt binders in square matrices (5×5). In other words, the five columns indicate the test temperature, and the five rows show the Sasobit percentages.

Figure 3. Matrix of viscosity and G^* .

For example, components of the right-side matrix show unaged viscosities and G^* of PG 64-22, PG 70-22, and PG 76-22 binders, while the left-side matrices show the viscosity and G^* of binders subjected to short-term aging conditions. As another instance, the G^*

of unaged PG 64-22 containing 2% Sasobit, tested at 64 °C, is 1.96 kPa, highlighted in Figure 3d, that resulted in 3.7 kPa because of the short-term aging process modeled through operator Ψ , also highlighted in Figure 3d.

2.7. Analysis of Aging Operator

To find the operator via analysis of the matrices, MATLAB software was used. The operator can be an equation, a constant value, or a matrix, depending on the boundary conditions of the problem and mathematical relationships between the components of the input and output matrices.

3. Results

Figure 4 shows the transformational matrix of the unaged into the short-term aging state. In other words, the short-term aging process using RTFO is characterized in a set of 5×5 matrices for various asphalt binder types shown in Figure 3.

$$\{\psi\} = \begin{vmatrix} 1.44 & 1.44 & 1.41 & 1.37 & 1.22 \\ 1.39 & 1.25 & 1.25 & 1.08 & 1.07 \\ 1.33 & 1.33 & 1.38 & 1.18 & 1.26 \\ 1.30 & 1.22 & 1.19 & 1.22 & 1.34 \\ 1.43 & 1.32 & 1.25 & 1.20 & 1.20 \end{vmatrix}$$

(a) Transforming short-term aging matrix of viscosity of PG 64-22

$$\{\psi\} = \begin{vmatrix} 1.53 & 1.52 & 1.67 & 1.66 & 1.42 \\ 1.42 & 1.42 & 1.60 & 1.49 & 1.28 \\ 1.31 & 1.34 & 1.54 & 1.40 & 1.27 \\ 1.26 & 1.29 & 1.51 & 1.32 & 1.18 \\ 1.22 & 1.26 & 1.43 & 1.38 & 1.31 \end{vmatrix}$$

(b) Transforming short-term aging matrix of viscosity of PG 70-22

$$\{\psi\} = \begin{vmatrix} 1.17 & 1.21 & 1.23 & 1.28 & 1.37 \\ 1.18 & 1.15 & 1.15 & 1.21 & 1.65 \\ 1.19 & 1.01 & 1.12 & 1.02 & 1.09 \\ 1.16 & 1.03 & 1.16 & 1.06 & 1.13 \\ 1.09 & 1.08 & 1.12 & 1.06 & 1.09 \end{vmatrix}$$

(c) Transforming short-term aging matrix of viscosity of PG 76-28

$$\{\psi\} = \begin{vmatrix} 2.07 & 2.11 & 2.15 & 1.95 & 1.82 \\ 1.35 & 1.60 & 1.83 & 1.82 & 1.75 \\ 2.17 & 2.02 & 1.88 & 1.60 & 1.52 \\ 1.45 & 1.35 & 1.43 & 1.64 & 1.83 \\ 1.58 & 1.52 & 1.42 & 1.30 & 1.22 \end{vmatrix}$$

(d) Transforming short-term aging matrix of G^* of PG 64-22

$$\{\psi\} = \begin{vmatrix} 1.66 & 1.57 & 1.91 & 1.86 & 1.94 \\ 1.77 & 1.21 & 1.96 & 1.83 & 1.90 \\ 1.82 & 1.81 & 1.71 & 1.81 & 1.90 \\ 1.68 & 1.63 & 1.74 & 1.46 & 1.59 \\ 1.58 & 1.50 & 1.39 & 1.34 & 1.41 \end{vmatrix}$$

(e) Transforming short-term aging matrix of G^* of PG 70-22

$$\{\psi\} = \begin{vmatrix} 1.59 & 1.57 & 1.23 & 1.22 & 1 \\ 1.90 & 1.64 & 1.47 & 1.46 & 1.05 \\ 2.72 & 1.64 & 1.41 & 1.68 & 1.33 \\ 1.20 & 1.71 & 1.39 & 1.60 & 1.36 \\ 1.04 & 1.86 & 1.30 & 1.24 & 1.33 \end{vmatrix}$$

(f) Transforming short-term aging matrix of G^* of PG 76-28

Figure 4. Transforming short-term aging matrix of viscosity and G^* .

For example, the short-term aging process resulted in a viscosity of PG 64-22 containing 4% Sasobit, tested at 120 °C, with an increase of 1.22, as shown in the transforming matrix in Figure 4a. It can be seen that the maximum increase is for the control PG 64-22 (without Sasobit) and the sample containing 1%, which is 1.44, as also shown in Figure 4a. Regarding the characterization of the short-term aging process in PG 70-22, as in Figure 4b, the components of the aging matrix based on viscosity are higher than the components of the matrix of PG 64-22, indicating the higher aging. For example, the short-term aging increased the viscosity of the PG 70-22 modified binder with 3% Sasobit at 130 °C, by 1.49, while the corresponding values for PG 64-22 and PG 76-28 were 1.08 and 1.21, respectively. In other words, the aging matrix of PG 70-22 shows more sensitivity to short-term aging in terms of viscosity, compared with the other binders.

Furthermore, from the transformational aging matrices in Figure 4a,b, the components of the Sasobit-modified samples are often lower than those of the control binders. As an instance, the components that correspond to the control binder tested at 130 °C is 1.39, while the corresponding values for the Sasobit-modified PG 64-22 vary from 1.07 to 1.25. Therefore, incorporation of Sasobit can decrease stiffness due to short-term aging of PG 64-22, which is consistent with the trend observed by Lee et al. [26] and Kim et al. [27]. The same general trend can be observed for the PG 76-28 asphalt binder. However, the component related to PG 76-28 containing 4% Sasobit and tested at 130 °C is 1.65, which is 40.6% higher than the control binder, as shown in Figure 4c. In addition, the components of the aging matrix of PG 76-28 are generally lower than those of PG 64-22 and PG 70-22, indicating less hardening due to oxidation and volatilization. This means that the polymer-modified binders are less prone to aging, which is reasonable. For instance, the component of PG 76-28 modified by 3% Sasobit, tested at 150 °C, is 1.06 in comparison with 1.22 and 1.32 for the PG 64-22 and PG 76-28, respectively. The lower short-term aging results in less stiffness after mixing. Therefore, the mixes can be more workable. Moreover, long-term aging decreases, which results in less cracking. However, the components of the transformational aging matrix, in terms of viscosity, depend on Sasobit content, binder type, and test temperature. As a result, it is recommended that the effect of short-term aging, using viscosity as the aging indicator, is evaluated at various temperatures, because analysis of aging at just one or two temperatures can be misleading. Therefore, use of a transformational matrix that indicates the values of aging at various temperatures, as well as the Sasobit content, can provide detailed information about the trends in short-term aging, based on the viscosity of the binders.

Figure 5 shows the range of components of the transformational aging matrix of each asphalt binder type. From the Figure, the ranges of PG 64-22, PG 70-22, and PG 76-28 are 1.07 to 1.44, 1.26 to 1.66, and 1.03 to 1.37, respectively. The bigger range of PG 70-22 indicates more sensitivity to aging than the other binder types. However, the ranges of PG 64-22 and PG 76-28 values are similar.

The ranges mentioned in Figure 5 are based on the viscosity of asphalt binders at the temperature sweep according to the RV test. Obviously, the range can change for different asphalt binder types, contents, chemical structures, and temperature ranges. Therefore, the boundary conditions of the tests play a pivotal role in the ranges of the components of the transforming aging matrices of asphalt binders.

Figure 4d–f shows the transforming aging matrix of asphalt binders based on G^* . The components of the matrices are often higher than the components of the matrix based on rotational viscosity shown in Figure 4a–c. The reason behind this difference could be the higher stiffness of the binders at the lower temperature ranges. That is, Sasobit forms networks of crystals in the binder structure at temperatures lower than 100 °C, which increases the binder stiffness. As a result, the synergistic effects of asphalt hardening and crystal networks caused by Sasobit increase the stiffness of the binder.

Figure 5. Ranges of components of the aging matrix developed based on RV results.

In addition, Figure 4d shows that the components of the aging matrix of PG 64-22 are lower than those of the other binder types, indicating less aging or lower hardening. The reason can be attributed to the lower stiffness of the asphalt binder. However, this trend was different from the trend observed for the aging matrix of PG 64-22, based on the viscosity.

Comparing PG 76-28, in Figure 4f, with the other binders clearly shows that the components of PG 76-28 are generally lower than the corresponding components, excluding 2.72, which is the component related to zero-Sasobit and a test temperature of 64 °C. Furthermore, some components of the Sasobit-modified binders are less than the control binders, which means that Sasobit decreases the stiffening effects of aging. For example, the components of PG 70-22 binders containing 1% and tested from 46 °C to 64 °C are lower than the components of the control binder, while the trend change is from 64 °C to 82 °C. In other words, the transformational aging matrix shows that 64 °C can be considered the inflection point, because the trend of the components of the PG 70-22 containing 1% Sasobit changes. Such points can be found for the other asphalt binder types and matrices developed using viscosity. It is clear that the inflection points can be different depending on the property selected as the indicator of aging. This means that the type of rheological property selected to develop the aging matrix is an important factor.

Figure 6 shows the ranges of components of the aging matrices. The component range of PG 64-22 varies from 1.22 to 2.17, while the range variations are 1.2 to 1.94 and 1 to 2.7 for PG 70-22 and PG 76-28, respectively. Comparing Figures 5 and 6, the ranges of components are different, indicating the role of the aging indicator in the analysis of the short-aging process. However, the range of PG 64-22 remains almost constant for both the rheological properties. In other words, the asphalt binder performance grade is also an important factor. Therefore, short-term aging is a complex process because of the interaction between various factors and oxidative aging.

Figure 6. Ranges of components of the transforming aging matrix developed based on G^* results.

Figure 7 shows the relationships between unaged and aged rheological characteristics for the various binder types. There are linear relationships between the unaged and aged viscosity and G^* of binders for all the Sasobit contents. In other words, the linear relationships are independent of the binder type, temperature range, and rheological characteristics selected to study the short-term aging process. In addition, the relationships between the unaged and aged viscosity, or G^* , are mathematically continuous. Therefore, the slopes of the linear equations show the gradient of variation of short-term-aged properties to unaged ones. In other words, the slopes are the average value of AI in terms of the viscosity and G^* , within the temperature range considered. In addition, the slopes of the equations of PG 76-28 are lower than the corresponding slopes for PG 64-22 and PG 70-22. The lower values of the slope indicate a lower rate of aging for PG 76-22, which is consistent with the results of matrix analysis, shown in Figure 4c,f. In addition, the average of the slopes for the viscosity and G^* vary from 1.00 to 1.85 and 1.02 to 1.54, respectively. These ranges are within the ranges of the components of the matrices in Figures 5 and 6, indicating the same values via two different methodologies.

In other words, the results of AI and the transformational aging matrix are similar. The components of the transforming aging matrix and the slopes of the asphalt binder samples may change, while the short-term aging test parameter, as a boundary condition, also changes. The boundary condition is the duration of short-term aging, which is 90 min, based on ASTM D 2872, or the temperature of aging and air flow rate changes. Any change in the speed of the rotating bottle carriage in the RTFOT device can also result in a different aging matrix. Therefore, any change in the boundary conditions can be included in the new output and input matrix to find a new operator (Ψ).

In addition, such a matrix methodology is suggested to analyze the effects of aging using the results of a simple performance tester—that is, the rows of the matrix are the frequency of loading, and the columns are the temperature sweep. It is clear that the operator differs for various criteria adopted as the indicator of aging.

(a) Relationship between unaged and short-term aged viscosity of PG 64-22.

(b) Relationship between unaged and short-term aged viscosity of PG 70-22.

Figure 7. Cont.

(c) Relationship between unaged and short-term aged viscosity of PG 76-28.

(d) Relationship between unaged and short-term aged G^* of PG 64-22.

Figure 7. Cont.

(e) Relationship between unaged and short-term aged G^* of PG 70-22. (f) Relationship between unaged and short-term aged G^* of PG 76-28.**Figure 7.** Relationship between unaged and short-term aged rheological properties of asphalt binders.

4. Conclusions

Short-term aging is a conditioning process. The effects of short-term aging are often evaluated based on changes in the rheological properties of binders (as output) using the AI. However, the proposed matrix analysis characterizes the effects of the short-term aging procedure as an integrated system, developed based on the various test temperatures and additive contents. Indeed, the effects of short-term aging were characterized through a

matrix operator. The transformational aging matrices showed that the short-term aging processes have different effects on the trends of components, depending on the binder type, Sasobit content, and test temperature. In addition, the rheological characteristics chosen to indicate the level of aging played a key role. The results of the matrix analysis show that polymer-modified binders are less sensitive to aging than unmodified asphalt binders. The components of each transformational aging matrix are close to the average of the AI values, based on the same rheological characteristics, calculated at various test temperatures. As a result, the matrix analysis is proposed as a methodology to characterize the short-term aging of asphalt binders and to better understand the factors that affect binder aging rates.

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