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Rheological and Fatigue Properties of Epoxy Asphalt Binder

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Abstract: Epoxy asphalt binder is a different thermosetting material from asphalt or common polymer modified asphalt binders. It is difficult to evaluate epoxy asphalt binder performance using traditional asphalt characterization methods. This makes it hard to understand for engineers or researchers. In this study, theology and fatigue properties of epoxy asphalt binder were investigated. The dynamic shear rheometer (DSR) test using column specimens was conducted to examine the rheological and fatigue properties of epoxy asphalt binders. The time temperature superposition principle (TTS) was employed to calculate the rheological properties at different temperatures and frequencies. Mathematical equations and mechanical models were employed to describe the rheological properties of epoxy asphalt binder. Fatigue performance of epoxy asphalt binder was researched using stress-controls, and testing temperature and loading were also considered in this study. The results indicated that the epoxy asphalt binder demonstrates a better ability to maintain an elastic / viscous capability. In addition, the time temperature superposition (TTS) principle can be applied to evaluate epoxy asphalt binder, and the linear viscoelastic behavior of epoxy asphalt binder can be satisfactorily described using Huet-Sayegh modle. The results of fatigue life can be predicted using the phenomenological fatigue model. The dynamic shear rheometer (DSR) test using column specimens is a reliable test method for evaluating thermosetting epoxy asphalt binder's performance.

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***Key words**: Column sample; Epoxy asphalt binder; Fatigue; Rheological property.*

Introduction

Epoxy asphalt concrete (EAC) is a thermosetting polymer modified asphalt concrete that contains epoxy asphalt binder and aggregates. The epoxy asphalt binder is a two-phase chemical system in which the continuous phase is a thermosetting epoxy and the discontinuous phase is a mix of specialized asphalt and epoxy cross-linker [1-3]. The thermosetting epoxy binder provides EAC with high temperature stability, anti-cracking, water stability, and fuel resistance [4, 5]. The use of EAC as a surfacing material on steel bridge deck has grown since 2000 when it was first used to pave the surface of the No.2 Nanjing Yangtze River Bridge in China [6]. During the past decade, more than 20 steel bridge decks have been paved using EAC.

Rheology is a very powerful tool for characterizing and quantifying material properties. It has been well established that the rheological properties of asphalt binder affect the pavement performance [7, 8]. The behavior is characterized in the Superpave specification by the complex shear modulus measured using dynamic shear rheometer (DSR).

Epoxy asphalt contains a great deal of asphalt, and it exhibits the rheological behavior at different temperature and frequency conditions [9-11]. Studying its rheological properties is essential to analyze pavement structure response, performance, and evolution of distress caused by different loads. Although a number of papers have been published on determining the creep stiffness, dynamic modulus, and flexural stiffness of EAC [12-15], few studies have focused on the model capable of describing the rheological properties of EAC over the range of temperatures and frequencies. In addition, because epoxy asphalt binder is a thermosetting material, it is difficult to prepare test samples like the thermoplastic materials. Thermosetting materials need some time or temperature to curing that can show the performance of the application. Thus, most research about epoxy asphalt binder is difficult to achieve for common engineer. It is important to find a simple and effective test method to test the performance of epoxy asphalt binder.

DSR is widely used to characterize the viscous and elastic behavior of asphalt binders at medium to high temperatures. This characterization is also used in the Superpave PG asphalt binder specification. The DSR test uses a thin asphalt binder sample sandwiched between two circular plates [16-18]. In general, asphalt or common polymer modified asphalt is thermoplastic material. Under A given temperature, it can make a thin sample to test by the upper plate (or rotor). But epoxy asphalt binder is a thermosetting material. The uncured epoxy asphalt binder can make a thin test sample. But it must be cured before the test. And the curing epoxy asphalt binder requires a lot of time, sometimes even two days. It is not practical to test the performance of epoxy asphalt binder using DSR. However, the cured epoxy asphalt binder cannot be further shaped by heating [19-21]. In addition, some recent studies already mentioned adhesion failure between circular plate and asphalt binder during DSR testing.

Mortar column specimens are used in various DMA and DSR tests, giving insight into mortar response and mortar fatigue. And the production of mortar sample a special mould was designed by in TU Delfts mechanical workshop DEMO [22-25]. It is convenient to

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evaluate the epoxy asphalt rheological behavior to avoid the problem of adhesion failure between circular plates and binder. DSR time sweep test with this new type of specimen was put forward as a new way to evaluate the rheological and fatigue performance of epoxy asphalt binders.

In this study, the rheological properties and fatigue performance of epoxy asphalt binder were tested using mortar column specimen, and rheological model and fatigue equation were evaluated. The time temperature superposition principle (TTS) was employed to calculate the rheological properties at different temperatures and frequencies. Master curves, frequency sweep, Cole-Cole plots, and black diagram were generated. The Marasteanu Anderson (MA) Model and Williams-Landel-Ferry (WLF) equation were employed to describe the rheological properties of epoxy asphalt binder. And the linear viscoelastic behavior of epoxy asphalt binder was evaluated by Huet-Sayegh model. Fatigue performance of epoxy asphalt binder was researched using stress-controls, and testing temperature and loading were considered in this study.

Experimental Program

Materials

The 60/80 pen grade asphalt used was obtained from Shell Asphalt Co Ltd. in Xi'an province, China, with penetration of 73 (0.1 mm at 25^{o}C 100g and 5s), softening point of 46.8^{o}C , ductility of 148cm (at 15^{o}C) and viscosity of 0.35Pa·s (at 135^{o}C). The epoxy resin used was diglycidyl ether of bisphenol A, and its epoxy value was 0.52 mol/100g. It was obtained from Shanghai Xinhua Resin Co Ltd., Shanghai, China. Aromatic amine curing agent was provided by Jiaxing Fine Chemical Co. Ltd., Zhejiang province, China. All materials were commercially available and used as received. The solubilizer was prepared in a laboratory, and it had surfactant properties and contained a hydrophobic and hydrophilic group.

The epoxy resin modified asphalt was a two-part (Part A and Part B) product blend before it was used. Part A was diglycidyl ether of bisphenol A. Part B was prepared as follows: asphalt binder was heated to 150℃±5℃ in an oil-bath heating container until it flowed freely. Appropriate amounts of solubilizer and modified amine agent were then added into the blend and mixed for 30 minutes with a lab mixer at fast speed (usually 500 rpm) to create a small vortex in nitrogen atmosphere. The mixes were cooled down to room temperature. The epoxy resin modified asphalt was obtained when the desired amount of Part A (epoxy resin) was added into the Part B and mixed until blended. The blend was fully solidified under the certain condition (60℃, 7-day).

The properties of epoxy resin modified asphalt Part A and Part B are shown in Table 1, and the properties of the fully solidified epoxy resin modified asphalt are shown in Table 2. The 35 wt.% epoxy resin was added into asphalt to prepare epoxy resin modified asphalt in this study.

Test Sample

Epoxy resin modified asphalt is a thermal setting material and it will get harder after being fully solidified, which means that the epoxy resin modified asphalt cannot be shaped like asphalt [26-28].

Table 1. Properties of Epoxy Resin Modified Asphalt Part A and Part B.

Property	Part A	Part B	Test Method
Viscosity at 25℃ (Pa·s)	12	-	ASTM D445
Viscosity at 100℃ (Pa·s)	-	0.17	ASTM D2983
Epoxide Equivalent Weight	192	-	ASTM D1652
Flash Point (℃)	235	215	ASTM D92
Moisture Content (%)	0.01	0.01	ASTM D6304
Specific Gravity at 25℃	1.158	1.02	ASTM D1475
Color, Gardner	2	-	ASTM D1544

Table 2. Properties of Fully Solidified Epoxy Resin Modified Asphalt.

Property	Measured Value	Test Method
Tensile Strength at 25℃ (MPa)	2.9	ASTM D412
Tensile Elongation at 25℃ (%)	315	ASTM D412
Heat Deflection Temperature (℃)	-22	ASTM D648

Therefore, shear tests on columns were used to evaluate the rheological properties of epoxy resin modified asphalt. The process of moulding can be illustrated as follows: two components of Part A and Part B were first mixed homogenously, then the specimen was moulded in a knock-down moulding clamp, and the asphalt binder was fully cured. The mould was disassembled and the cylindrical mortar columns were ready to be tested using the DSR test equipment. The total height of the specimen was 20 mm, in which a column with 10 mm height and 6 mm diameter was the effective section for testing. At each end, a steel ring with an inner-diameter of 7 mm, outer-diameter of 8 mm, and a height of 4 mm was placed for the purpose of fastening. Considering that the elastic-visco characteristics of mortar would result in loose contact due to the relaxation effect, the clamps designed did not touch the mortar directly, but on two steel rings at both ends. The two components of epoxy asphalt binder were carefully poured into a designed mould. After the epoxy asphalt binder was fully cured, the cylindrical mortar columns were ready to be tested using the DSR. The specimen is shown in Fig. 1 [29].

Results and Discussions

Black Diagram

For a dynamic experiment, a graph of the magnitude (or norm) of the complex shear modulus (|G*|) versus the phase angle (δ) is called a black diagram. The frequency and the temperature are therefore eliminated from the plot, thus allowing all the dynamic data to be presented in one plot without performing TTS manipulations of raw data. A smooth curve in a black diagram is a useful indicator of time-temperature equivalence, while a disjointed curve indicates the breakdown of the TTS and the presence of a high wax content bitumen, a high asphaltene structured bitumen, or a highly polymer-modified bitumen. Fig. 2 represents a black diagram for epoxy asphalt binder. As shown, the curve is continuous. In this typical case, the time temperature equivalence principle can be applied.

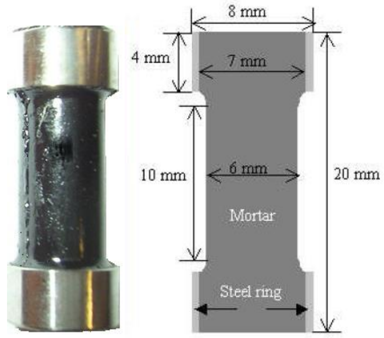

Fig. 1. Column Specimens for the DSR Test.

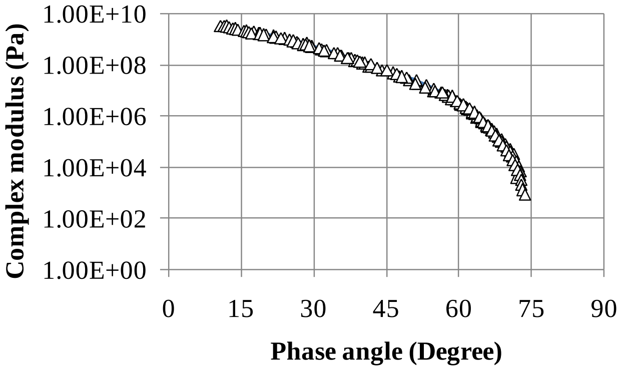

Fig. 2. Black Diagram for the Epoxy Asphalt Binder.

In pavement engineering practice, a Cole-Cole diagram is defined as a graph of the loss modulus (G'') as a function of storage modulus (G'). The Cole-Cole diagram provides a means of presenting the viscoelastic properties of the bitumen without incorporating frequencies and temperature as one of the axes. Figs. 3 and 4 show the Cole-Cole diagram for the epoxy asphalt binder. The results indicated that the storage modulus is much higher than the loss modulus for an epoxy asphalt binder in test temperature range. In the Cole-Cole diagram of epoxy asphalt binder, curves for different temperatures of modulus will overlap according to TTS.

Master Curve

Frequency sweep tests were conducted with the DSR at various temperatures (from 0°C to 60°C) and frequencies (from 0.1 rad/s to 300 rad/s). Master curves of shear complex modulus and angle were obtained using the time temperature superposition (TTS) principle. This principle allows shifting the response data at different temperatures with respect to time or frequency to selected reference temperature. The curve obtained using this principle shows the frequency or loading time dependency of the complex shear modulus and phase angle over a wide loading time or frequency range.

The amount of shifting required at each temperature to the reference temperature was determined using the Williams-Landel-Ferry (WLF) equation [30-32]:

$$\log(\alpha_T) = \frac{-C_1(T - T_0)}{C_2 + (T - T_0)} \quad (1)$$

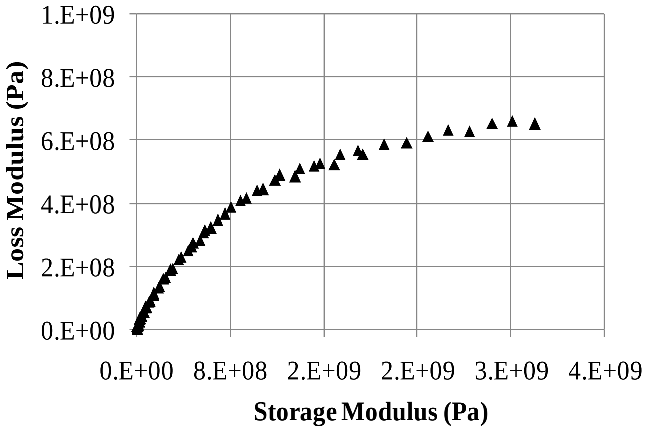

Fig. 3. Cole-Cole Diagram for the Epoxy Asphalt Binder.

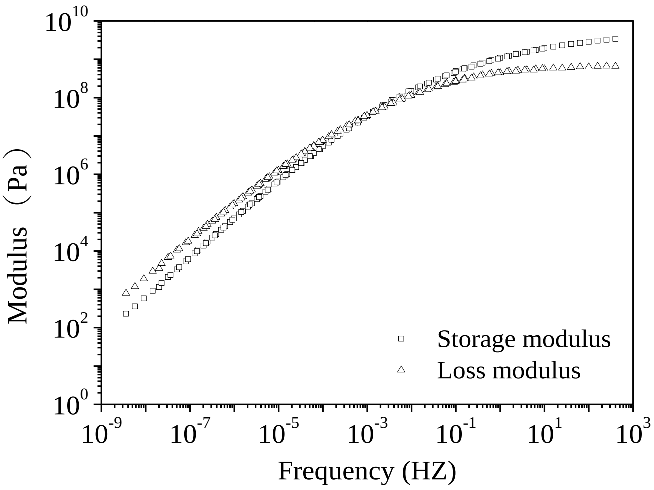

Fig. 4. The Modulus of Epoxy Asphalt Binder.

where: α_T is the shift factor at a temperature of T; T_0 is the reference temperature and C_1 and C_2 are constants.

The master curve data, complex modulus and phase angle, are described with Marasteanu Anderson (MA) Model given in Eq. (2) [33, 34].

$$\left|G^*(\omega)\right| = G_g\left[1 + \left(\frac{\omega_c}{\omega}\right)^v\right]^{-\frac{w}{v}} \quad (2)$$

The parameter v is described by the following equation.

$$v = \frac{\log 2}{R} \quad (3)$$

The parameter w addresses the issue of how fast or slow the phase angle converges to the ninety and zero degree asymptotes as the frequency goes to zero or infinity, respectively. It can best be interpreted from the expression for the phase angle as following equation.

$$\delta(\omega) = \frac{90w}{1 + \left(\frac{\omega}{\omega_c}\right)^v} \quad (4)$$

Fig. 5 presents the master curve data obtained at a reference temperature of 0℃. The correlation coefficient (R^2) between the master curve model with MA model and the actual master curve was calculated. An R^2 value of 0.993 was obtained for the complex modulus master curve obtained from the DSR. The results indicated that the epoxy asphalt binder shows high complex modulus and low phase angle in the test temperature range. In other words, the epoxy asphalt binder demonstrates a higher ability to maintain an elastic/viscous capability.

The MA model parameters for describing the master curve are given in Table 3. WLF factors for obtaining the shift factors for different temperatures are also included in Table 3.

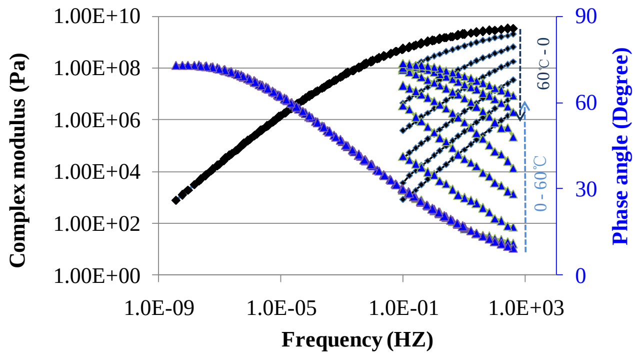

Fig. 5. Master Curve at T_0=0℃ for the Mortar Data.

Table 3. MA Model Parameters and WFL Factors at T_0 = 10℃.

Parameters	ω_c	v	w	G_g	R^2
MA Model	1.8	0.2	1.3	3.74E+11	0.9927
WLF	C_1		C_2		R^2
Factors	21.59		112.12		0.9934

Linear Viscoelastic Behavior of Epoxy Asphalt Binder

Most rheological models for the description of the viscoelastic behavior of asphalt binder have been developed. The Huet-Sayegh model was proposed for characterizing the viscoelastic property of materials [35]. This model consists of the combination of two springs (G_0 and G_∞ - G_0) and two parabolic creep elements (k and h). The physical representation of the Huet-Sayegh model is shown in Fig. 6.

The Huet-Sayegh model can be described mathematically using the following formula:

$$G^*(i\omega\tau) = G_0 + \frac{G_\infty - G_0}{1 + \alpha(i\omega\tau)^{-k} + (i\omega\tau)^{-h}} \tag{5}$$

where G^* is the complex modulus, G_0 is the elastic modulus when $\omega \rightarrow 0$, G_∞ is the limit of the complex modulus $\omega \rightarrow \infty$, and α is a dimensionless constant. The k and h are the two constants ($0 < k$, $h < 1$).

The parameter τ is referred to as the characteristics time and it is a temperature dependent variable calculated using the following equation:

$$\ln\tau = a + bT + cT^2 \tag{6}$$

where a, b and c are regression parameters representing the material characteristics.

The Huet-Sayegh model required six parameters (δ, k, h, G_∞, G_0 and τ) to determine the linear viscoelastic behavior of the considered material over a wide frequency range at a given temperature. The model parameters were determined by obtaining the best fit for the measured complex modulus values plotted in the Cole-Cole and black diagrams.

The obtained values of the Huet-Sayegh model parameters and the regression coefficients are given in Table 4. The best fit of the curves to the experimental data confirms the applicability and appropriateness of the model to describe the linear viscoelastic behavior of epoxy asphalt binder in the small strain domain for any range of frequencies and temperatures.

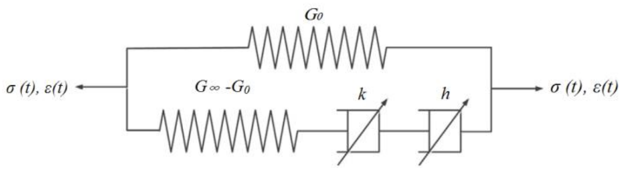

Fig. 6. Huet-Sayegh Model.

Table 4. Parameters of the Huet-Sayegh Model at T_0 = 0℃.

G_0 (Pa)	G_∞ (Pa)	δ (1/s)	k	h	τ	R^2
36	4.05E+11	2.425	0.231	0.472	2.83E-04	0.9724

Fatigue of Epoxy Asphalt Binder

The asphalt binder/mastic phase of asphalt concrete is the most critical for resisting fatigue damage, and thus it must be evaluated for fatigue performance. But evaluation of fatigue damage in the binder alone was challenging, as it typically requires multiple repeated load cycles over a testing period that can last for hours.

As is shown in Fig. 7, typical fatigue curves show three distinct phases on a variation of the complex modulus plotted versus the number of cycles. An initial decrease assumed to be self heating of the material under shear, then, a smooth, and finally a drop related to failure propagation. The decrease of complex modulus with load repetitions can be used as an indicator of fatigue. As is shown in Fig. 7, the fatigue failure point N_f was defined as the point where the complex modulus began to enter the failure propagation drop section. The fatigue failure occurs where all of the properties change very rapidly with additional load repetitions.

The test sample as above was employed to study the fatigue performance of epoxy asphalt binder. Test temperature varied from -10℃ to 20℃. All the oblique shear tests were done at a frequency of 10 Hz with a pure sine-wave.

The internal stress level of an asphalt pavement is always extremely variable and depends on many factors such as its structural capacity, thickness of the layers, temperature conditions and bearing capacity of the subgrade. Thus, it is fundamental to analyze the effect of stress level on the fatigue property of asphalt binders which are an important part of the pavement. The fatigue tests have been conducted with a range of temperature, frequency and applied stress. The adhesion between epoxy asphalt binder and steel ring was found to be strong enough and adhesion failure was

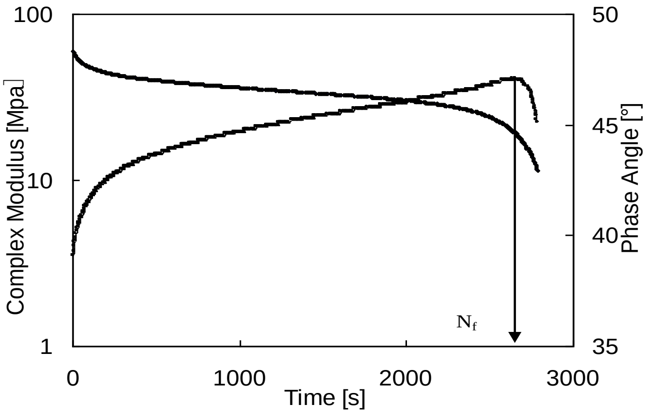

Fig. 7. Time Versus Fatigue Life Using Modulus and Phase Angle.

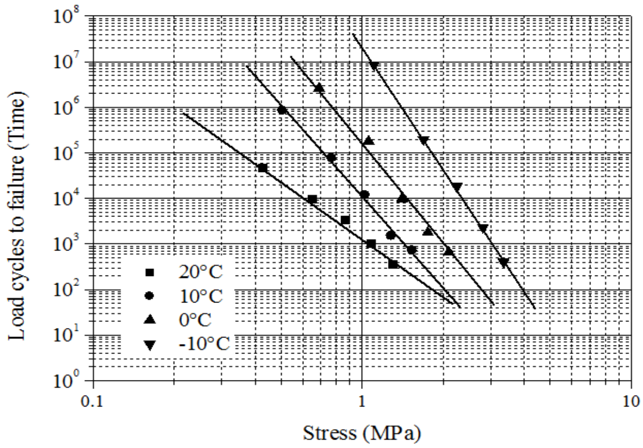

Fig. 8. Fatigue Life of Epoxy Asphalt Binder.

not observed during all tests. Thus, DSR time sweep test with column specimen can avoid adhesion failure between circular plate (or rotor) and epoxy asphalt binder when the fatigue property was evaluated. Based on the results of the fatigue tests using cylindrical column specimen, the stress level has a significant effect on the fatigue life of the epoxy asphalt binder.

Fig. 8 shows that the fatigue life of the epoxy asphalt binder at different stresses and temperatures. When compared with the fatigue life of the epoxy asphalt binder at a high stress level, the loading repetitions to failure (N_f) of epoxy asphalt binder are increased remarkably when the stress level is dropped. For example, the N_f at 20℃ for an epoxy asphalt binder with different stress levels are 367 (1.291 MPa stress), 3468 (0.864 MPa stress), 9889 (0.649 MPa stress) and 48398 (0.426 MPa stress), respectively. The fatigue life substantially dropped with the increase in stress level at the same temperature. In addition, the fatigue life increased with the drop of temperature. The effect of temperature on the fatigue life of cylindrical column specimen has a great dependence on the level of stress. The fatigue life of the epoxy asphalt binder dropped with the increase of the temperature from -10℃ to 20℃ at the test range. The reason may be that the concentrated stress produced by fatigue loading can be dispersed and the development of micro-cracks can be delayed at low stress. Otherwise, at low stress levels and/or temperature, elastic strain acts on the dominant action in the fatigue process. The elastic strain recovered after unloading. In contrast, the plastic deformation becomes a dominant action with the temperature or stress level increasing. Previous research has shown that epoxy resin can increase the elasticity of asphalt and the fatigue property of epoxy asphalt binder is enhanced significantly at low stress levels.

There are several phenomenological fatigue models developed to predict fatigue performance of asphalt or asphalt concrete. Fatigue models are divided into two main types, the strain-controlled and the stress-controlled mode based models [36, 37]. In the strain-controlled based model, the strain level or deflection remains constant during the test. The strain-controlled relation can be expressed mathematically in the following equation:

$$N_f = K(\varepsilon_0)^{-n} \tag{7}$$

where N_f is the fatigue life; ε_0 is the applied stress; K, n are material constants.

In case of stress-controlled mode, the nominal stress level or load is maintained constant during the fracture life. Epoxy asphalt binder has high tensile strength. Large stress is needed if the strain-controlled is employed to investigate the fatigue life of the epoxy asphalt binder. Excessive load that may exceed the range of the instrument will destroy the instrument or affect test accuracy. Thus the stress-controlled fatigue test was performed in this research. The stress-controlled relation can be expressed mathematically in the following equation:

$$N_f = K(\sigma_0)^{-n} \tag{8}$$

where N_f is the fatigue life; σ_0 is the applied stress; K, n are material constants.

Table 5 gives the regression constants of epoxy asphalt binder at different temperature. It can be observed that the values of K and n decrease when the temperature increases, which results in an decrease in cycle numbers to failure for epoxy asphalt binder, especially at higher stress levels. Moreover, it can be seen that the fatigue life increases with applied stress decreasing. Furthermore, the fatigue properties of epoxy asphalt binder were predicted by fatigue equation.

Conclusions

In this study, rheological and fatigue properties of epoxy asphalt binder were investigated. The dynamic shear rheometer (DSR) test using column specimens was conducted to examine the rheological and fatigue properties of epoxy asphalt binders. Based on the laboratory test results and analysis of the rheological and fatigue properties of the epoxy asphalt binders, the following conclusions were drawn:

1. The dynamic shear rheometer (DSR) test using column specimens is a reliable test method for evaluating thermosetting epoxy asphalt binder's performance.
2. The epoxy asphalt binder demonstrates a higher ability to maintain an elastic/viscous capability. And the time temperature superposition (TTS) principle can be applied to evaluate epoxy asphalt binder.
3. The master curve obtained from the response data is well

Table 5. Fatigue Equation of Epoxy Asphalt Binder.

Temperature (°C)	Fatigue Equation	Regression Coefficient		R^2
		K	*n*	
-10	$N_f = 8.0\mathrm{E}+06(\sigma_0)^{-7.89}$	8.0E+06	7.89	0.9789
0	$N_f = 2.2\mathrm{E}+06(\sigma_0)^{-6.38}$	2.2E+06	6.38	0.9854
15	$N_f = 9.4E+05(\sigma_0)^{-5.61}$	9.4E+05	5.61	0.9623
20	$N_f = 1.6E+05(\sigma_0)^{-4.82}$	1.6E+05	4.82	0.9712

described by the Marasteanu Anderson (MA) Model and Williams-Landel-Ferry (WLF) equation.

4. The linear viscoelastic behavior of epoxy asphalt binder can be satisfactorily described using the Huet-Sayegh model. The linearity limits of epoxy asphalt binder decrease as temperature increases and frequency decreases.
5. The results of fatigue life can be predicted using the phenomenological fatigue model. This approach seems to have the potential to be used in the prediction of fatigue life of the epoxy asphalt binder or epoxy asphalt concrete based on fatigue test result.

Acknowledgements

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