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The Effect of Ultraviolet Aging Duration on the Rheological Properties of Sasobit/SBS/Nano-TiO_2-Modified Asphalt Binder

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Abstract: In recent years, nanoparticles have been introduced into warm-mix-modified asphalt to improve asphalt performance after sustaining ultraviolet (UV) aging, yet the evaluation of aging performance is often a descriptive characterization of rheological properties. This study extends rheological characterization with viscoelastic mechanical modeling to evaluate resistance to UV aging using Sasobit and SBS compound-modified binder blended with nano-titanium dioxide (TiO_2). The extended method comprises characterizations using several rheological properties and a viscoelastic mechanical model, named the 2S2P1D model, on modified asphalt after 3 days, 6 days and 9 days of ultraviolet (UV) aging. The rheological properties of the UV-aged binders were tested at high and medium temperatures in terms of viscosity, complex modulus, phase angle and fatigue factor. Rheological test results showed that nanoparticles generally had no apparent effect on the complex modulus of aged binders regardless of UV aging times. However, the aged binder with nanoparticles showed better fatigue resistance than aged binders without nanoparticles after 3 days of UV aging. As an extension, the black space diagram and 2S2P1D model were used to investigate the viscoelastic properties of these aged binders. The k and h values, as important model parameters, were almost the same and less than one for all UV-aged binders. All investigated aged asphalt binders showed characteristics of a viscoelastic solid in terms of the master curves of the complex modulus and phase angle, and the master curves of the phase angle for all UV-aged binders did not meet the time–temperature equivalence. Moreover, these observations from the 2S2P1D model revealed that aging durations did not affect the viscoelastic mechanical characteristics of warm mix asphalt in this study. The method adopted in this study may promote a comprehensive evaluation of asphalt properties after UV aging, especially considering the viscoelastic mechanical performance.

Keywords: ultraviolet aging (UV); aging times; viscoelastic properties; nano-titanium dioxide (TiO_2)

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1. Introduction

As a result of its benefits in terms of high and intermediate-temperature properties under increased traffic loads, Styrene–butadiene–styrene copolymer (SBS)-modified asphalt binder has attracted extensive attention in recent decades [1]). However, SBS-modified asphalt binder shows higher viscosity than conventional binders, requiring higher mixing and compaction temperatures [2–4]. Higher temperatures in mixing and compaction are generally associated with more energy consumption during the production process and can introduce more severe aging in asphalt binders. Warm mix asphalt (WMA) techniques have been used to reduce mixing and compaction temperatures during the paving process. WMA additives can decrease the viscosity of asphalt binder without compromising the other physical and mechanical properties [3–6]. One of the most widely used WMA additives is Sasobit, a type of long-chain aliphatic polyethylene hydrocarbon produced from coal gasification using the Fischer–Tropsch process [4,7,8]. According to a study from Xiao et al. [9], WMA binder made with Sasobit showed significantly better high-temperature properties and inferior low-temperature properties compared to that made with other WMA additives such as Cecabase, Evotherm and Rediset. In addition, a study by Kök et al. [4] reported that compound-modified asphalt binders with Sasobit and SBS has better fatigue resistance than either the Sasobit or SBS-modified binders.

Despite the potential benefits of Sasobit/SBS compound asphalt binder, the properties of bitumen in the field would irreversibly deteriorate under the influence of heat, sunlight, oxygen and the combination of these factors. The oily components, chemical composition and molecular structuring of asphalt would change due to aging. Aging methods can be divided into two main methods: thermal aging and ultraviolet aging. Many standards for conducting thermal aging have been established and can be followed, including the rolling thin-film oven test (RTFOT), thin-film oven test (TFOT) and Pressure Aging Vessel (PAV) [10–13]. Zhang et al. [14] found that ultraviolet (UV) aging shows a more obvious influence on the degradation of SBS copolymers in comparison with PAV aging. With increasing temperatures due to global warming and corresponding decreases in the UV protection layer, the effect of UV radiation on the aging of asphalt has gained more attention from researchers [15]. Based on the amount of UV radiation, a variety of methods have been proposed to avoid the UV-aging of asphalt [16].

Nano materials have been applied in the paving industry in recent years. Among them, Nano-titanium dioxide (Nano-TiO_2) is a type of photocatalytic material capable of facilitating the reactions of nitrogen oxide and sulfur oxide decomposition due to the effects of ultraviolet radiation. TiO_2 has been used as a coating material for asphalt pavement to eliminate the aging of asphalt materials [17–19]. Yang et al. [20] pointed out that the resistance to ultraviolet (UV) aging of asphalt binder can be improved significantly by adding nano-TiO_2 at a reasonable dosage. The rheological properties of Sasobit/SBS compound binder modified with nano-TiO_2 after UV aging and PAV aging procedures were investigated by Yang et al. [20]. However, there are few efforts to investigate the combined effects of nano-TiO_2, Sasobit and SBS on the ultraviolet aging resistance of asphalt binder.

The aging of asphalt binder is induced by chemical or physicochemical changes in the emissions of ultraviolet radiation during its service life. Currently, assessments of the aging degree of asphalt binder are primarily conducted by measuring certain chemical, physical and rheological indices before and after aging [21]. However, the capability of these indices to characterize modified asphalt binders has been questioned. The time–temperature superposition principle (TTSP), related to the equivalency between time and temperature for viscoelastic materials, is usually used to build the complex modulus and phase angle master curves. The master curves can be used to describe changes in viscoelastic properties in a wide range of temperatures and frequencies. Therefore, it is widely used to evaluate the aging performance of asphalt. The 2S2P1D model, a simple combination of physical elements (two springs, two parabolic elements and one dashpot), was used to assess the rheological properties of asphalt binders by Yusoff et al. [22] before and after thermal aging. However, few studies have applied the 2S2P1D model to construct master curves of warm SBS throughout UV irradiation.

The primary goal of this study was to evaluate the influence of UV aging procedures on the properties of titanium dioxide nanoparticle (nano-TiO_2)-modified warm-SBS asphalt binder. To achieve this, the rheological properties of compound-modified binders were investigated in a medium and high-temperature range. Moreover, the 2S2P1D model was built in terms of the black diagram and master curve, providing further understanding of the influence of UV aging duration on nano-TiO_2 modified warm-SBS asphalt binder.

2. Materials and Methods

2.1. Raw Materials

An asphalt binder with a penetration grade of 60/80 was used as the base asphalt binder in this study. The properties of the base binder are listed as follows: penetration, 71.7 dmm at 25 °C; ductility, 1305 cm at 15 °C; and softening point, 48.0 °C. Four types of asphalt modifiers, Sasobit, styrene–butadiene–styrene (SBS), polyethylene glycol (PEG-

10000) and anatase-based titanium dioxide nanoparticles (nano-TiO_2 with a purity of 99.9%), were applied. The average size of nano-TiO_2 is 18 ± 5 nanometers. More information on the raw materials is detailed in the previous study [20].

2.2. Laboratory Testing Program

2.2.1. Mixing and Conventional Test

A high-shear mixer was used to prepare the modified asphalt binders. The WMA binder was prepared by mixing 3% Sasobit (by the weight of the base asphalt) with the fluid asphalt binder for 20 min at 150 °C and with a mixing speed of 4000 rpm. Then, 4% SBS (by the weight of the base binder) was mixed with the WMA binder at the same temperature and mixing speed for another 20 min. The 4% SBS was selected based on the recommendations of the manufacturer. Finally, nano-TiO_2 with various dosages (i.e., 0%, 1% and 3% by the weight of the base binder) was added to prepare the compound-modified asphalt binders. Based on the recommendations of Yang et al. [23], PEG-10000 with a dosage of 0.2% (by the weight of the base binder) was added to the compound-modified binder as a physical dispersant. The flowchart of the experimental procedure for this article is listed in Figure 1.

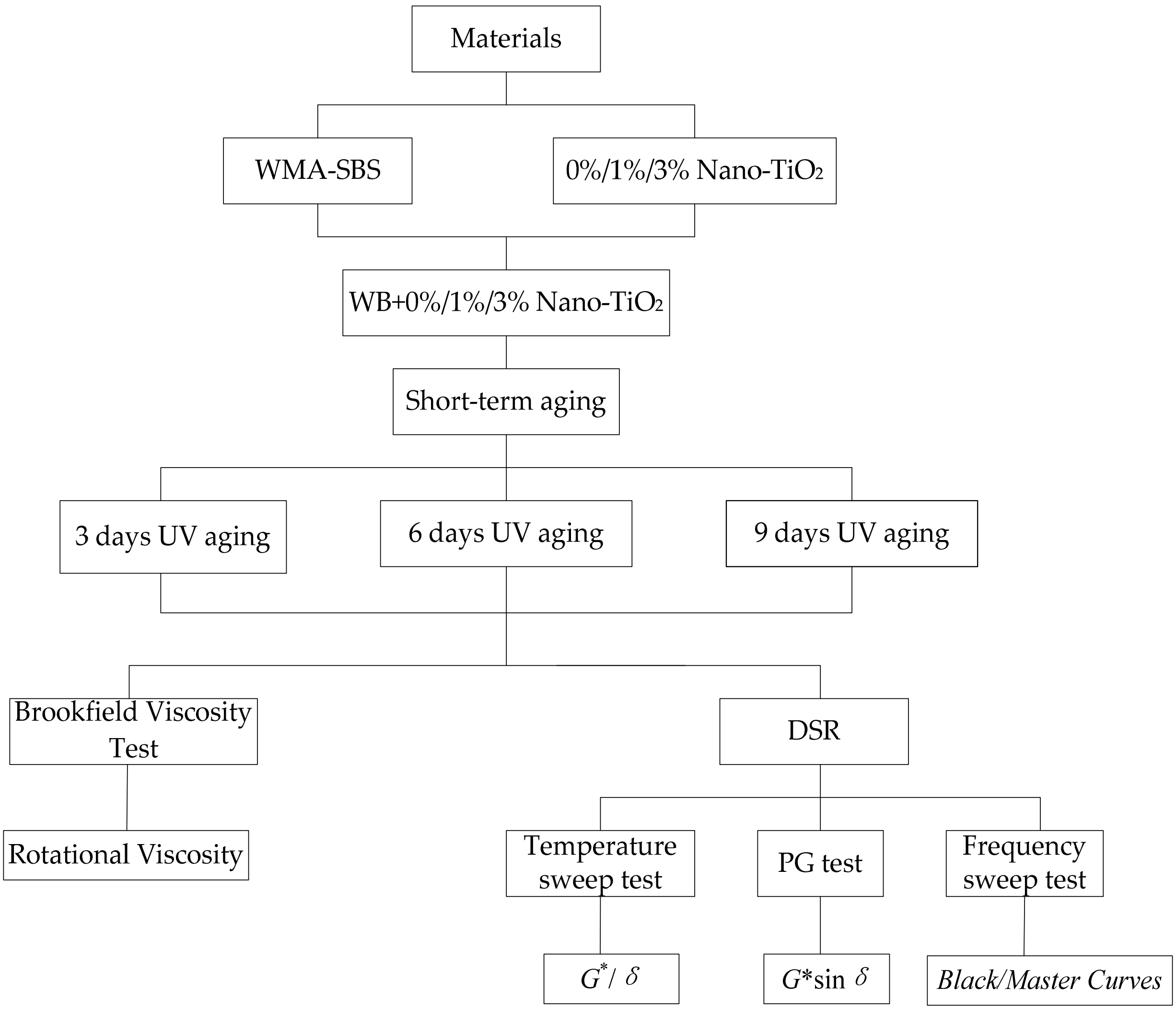

Figure 1. Flowchart of experimental procedure.

2.2.2. Aging Procedures

The warm-SBS (named WB) and nano-TiO_2 modified WB binders were aged through the rolling thin-film oven test (RTFO) procedure according to ASTM D2872. The pans, with 50 g of RTFO-aged binder, were placed into a UV aging box for 3 days, 6 days and 9 days. The UV radiation intensity of the box was set to 8 w/m^2, and the temperature was 60 °C. Details of the UV aging procedure were introduced in previous research [24].

2.2.3. Brookfield Viscosity Test

The rotational viscosity of the UV-aged binders was tested using a Brookfield rotational viscometer to measure the flow characteristics of the UV-aged binders at a temperature of 135 °C according to ASTM D4402.

2.2.4. DSR Test

The high and intermediate-temperature properties of the developed compound-modified asphalt binders were measured using a dynamic shear rheometer (DSR) by following AASHTO T 315. The geometry, with a 2 mm gap and an 8 mm diameter, was applied. The intermediate performance grading (PG) test for asphalt binders was conducted at various temperatures (i.e., 19, 22 and 25 °C) to assess the fatigue cracking resistance of the proposed compound-modified asphalt binders. The fatigue factor ($G^*\sin\delta$) was obtained at each temperature, and the intermediate failure temperature was determined based on ASTM 7643. A temperature sweep test was also conducted using the DSR. The temperature varied from 30 to 90 °C. The strain control mode (1%) was applied, and the loading frequency was 10 rad/s. In addition, four various temperatures (i.e., 10 °C, 22 °C, 34 °C, and 46 °C) with different loading frequencies ranging from 100 to 0.1 rad/s were chosen in the frequency sweep test to evaluate the aged samples.

The 2S2P1D model was applied to analyze and model the frequency sweep testing data. The 2S2P1D model has five components: two springs, two parabolic elements and one dashpot. Equation (1) shows the 2S2P1D model for the complex modulus (G^*). The shift factor ($\alpha(T)$) was calculated based on the Williams–Landel–Ferry theory (Equation (2)).

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

$$\log \alpha_T = -\frac{C_1(T - T_0)}{C_2 + T - T_0} \tag{2}$$

$$\tau = \tau_0 \times \alpha_T(T) \tag{3}$$

where the variables are as follows:
ω = angular frequency, rad/s;
i = complex number ($i^2 = -1$);
k, h = dimensionless constants, with $0 < k < h < 1$;
G_0 = static modulus, when $\omega \to 0$, Pa;
G_g = glassy modulus, when $\omega \to \infty$, Pa;
τ = characteristic time as a function of temperature; τ_0 is the characteristic time at the reference temperature (T_0);
β = dimensionless constants as functions of η and τ;
$\alpha(T)$ = shift factor as a function of temperature;
C_1 = dimensionless constants;
C_2 = constants/°C.

3. Results

3.1. Brookfield Viscosity Test

Figure 2 shows the Brookfield viscosity test results for all the aged binders. As shown in Figure 2, the viscosity of the aged binders changed with increased UV aging time.

In addition, the nanoparticle-modified binder showed higher viscosity than the binder without nanoparticles under different durations. However, the viscosity of the binder with nanoparticles had no obvious changes compared to the blank sample. Based on these analyses, the viscosity of all aged binders had a growth trend with the increases in UV aging times. However, the viscosity value of the asphalt with nanoparticles increased less significantly compared to the blank sample.

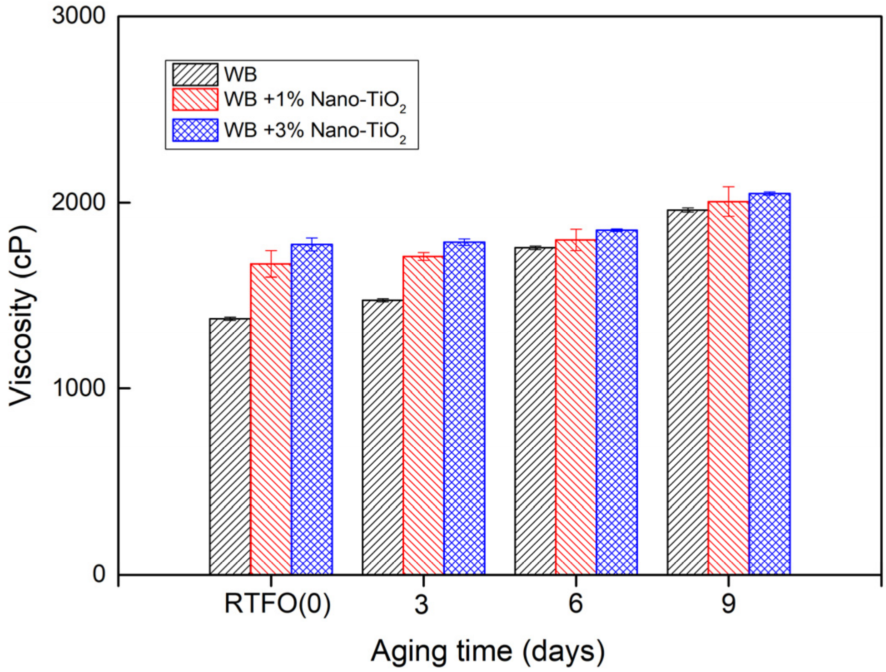

Figure 2. Viscosity of UV-aged binders at 135 °C.

3.2. Temperature Sweep

In order to determine the temperature dependence of the complex modulus (G^*) and the phase angle (δ) of the WB and nano-TiO_2 binders subjected to various times of UV aging, a temperature sweep test was conducted in the study, with a temperature range of 30 to 90 °C at an interval of 2 °C/min. Figure 3 shows the temperature sweep test results for different durations of UV-aged binders. As shown in Figure 3, a constantly increased complex modulus was observed in the isochronal plots over the entire range of temperatures for all aged binders. However, the phase angle in the isochronal plots had an opposite trend, increasing as temperature increased. This result is consistent with other researchers' findings [5,25]. In addition, the complex modulus G^* did not exhibit any significant change as the percentage of nanoparticles increased over the temperature domain, regardless of the aging time. In other words, control-aged binders generally have similar G^* values with a binder containing nano-TiO_2 regardless of aging time.

The phase angle (δ) is generally considered to be more sensitive to the chemical structure; therefore, the modification of bitumen is greater compared to the complex modulus [26]. The figures clearly illustrate the temperature dependence of the modified binders and the effectiveness of the aging time. With increased temperatures, the phase angles for all aged bitumen present a plateau region between 45 °C and 65 °C. It has been reported that the plateau phenomenon can be attributed to the formation of a polymer network in SBS-modified asphalt binders [1,26,27]. This indicates that the spatial structure of SBS was not destroyed with the increase in UV aging time for all aged binders with or without nano-TiO_2.

In the case of Figure 3a, the addition of nanoparticles resulted in a decrease in the phase angle of all aged bitumen at high temperatures. However, in Figure 3b,c, there is a similar increase in the phase angle towards more viscous behavior for all aged binders with the addition of nanoparticles at high temperatures. This significant increase in modification is a result of the UV aging resistance of WB with nanoparticles.

3.3. Fatigue Factor

Figure 4 shows the fatigue factor ($G^*\sin\delta$) results of compound-modified asphalt binders subjected to UV aging. As shown in Figure 4a, nano-TiO_2 had effects on the fatigue resistance of WB. Under a given UV aging state (3 days of UV aging), the fatigue factor of the WB increased with the increase in nano-TiO_2 dosages, and it is implied that the addition of nano-TiO_2 can degrade the fatigue properties of asphalt binder. However, as the UV aging period increased from 3 days to 6 days, the fatigue factor of the WB decreased with the addition of nano-TiO_2. Therefore, it can be inferred that adding nano-TiO_2 can improve the fatigue cracking resistance of Sasobit and SBS-modified asphalt binders to some extent.

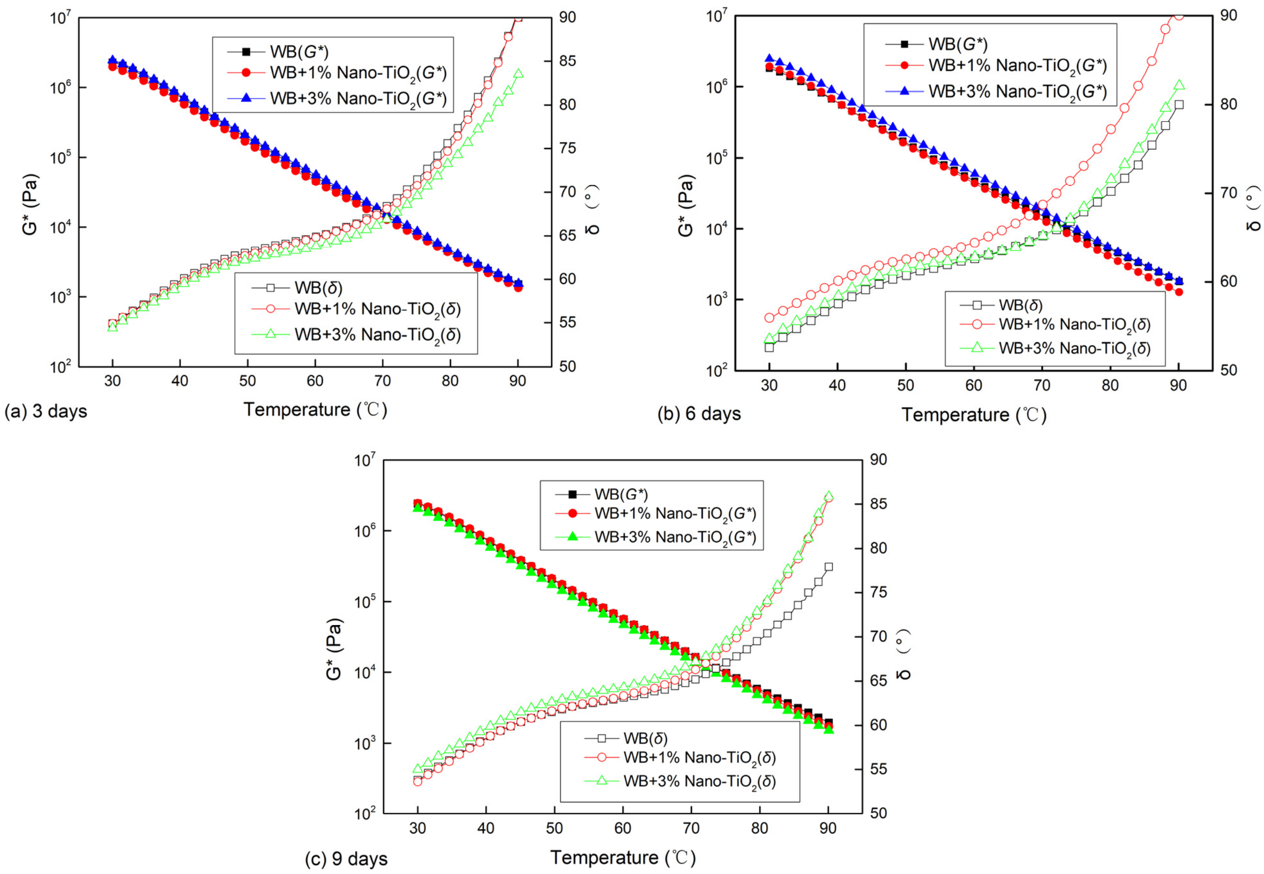

Figure 3. Complex moduli and phase angles of UV-aged binders increased with the tested temperature: (**a**) UV aging for 3 days; (**b**) UV aging for 6 days [20]; (**c**) UV aging for 9 days.

3.4. Failure Temperature

Figure 5 illustrates the failure temperature test results of all long-term aged binder samples. The failure temperature can more accurately identify the temperature grade of asphalt. It can be seen from Figure 5 that the failure temperature value of the UV-aged control binder increases with the aging times, indicating that aged binders were susceptible to fatigue damage in the process of UV aging. However, the failure temperature of the binder with nanoparticles showed a slight change as the aging time increased. Moreover, as can be seen from the figure, for binders subjected to 3 days of the UV aging process, the failure temperature increased with the increase in nano-TiO_2 dosage, which implies that the asphalt binders with nano-TiO_2 were more prone to fatigue cracking than those without nano-TiO_2. However, it should be noted that the binders with nano-TiO_2 subjected to more than 6 days of UV aging exhibited lower fail temperatures than binders without the nanoparticles, indicating that nanoparticle-modified asphalt is less susceptible to fatigue damage.

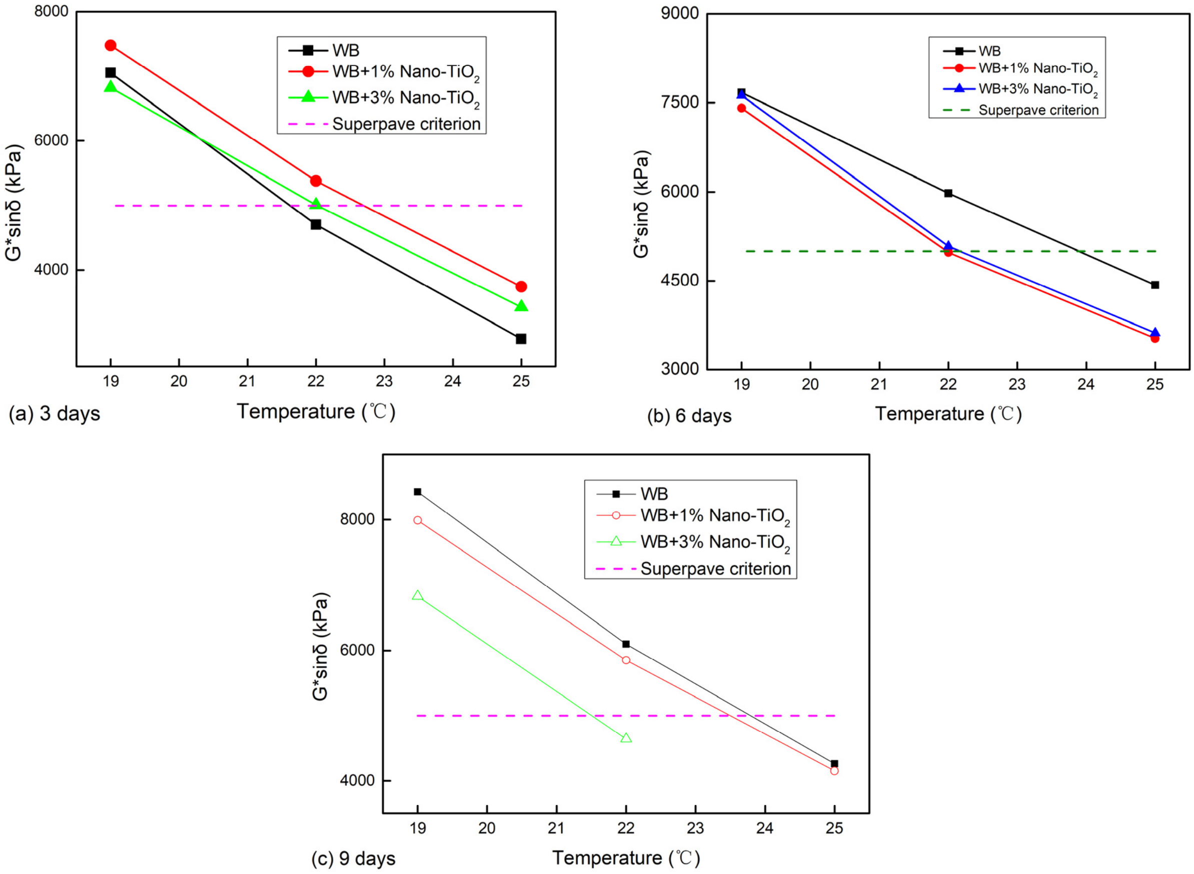

Figure 4. $G^*\sin\delta$ values of UV-aged binders: (**a**) 3 days; (**b**) 6 days [21]; (**c**) 9 days.

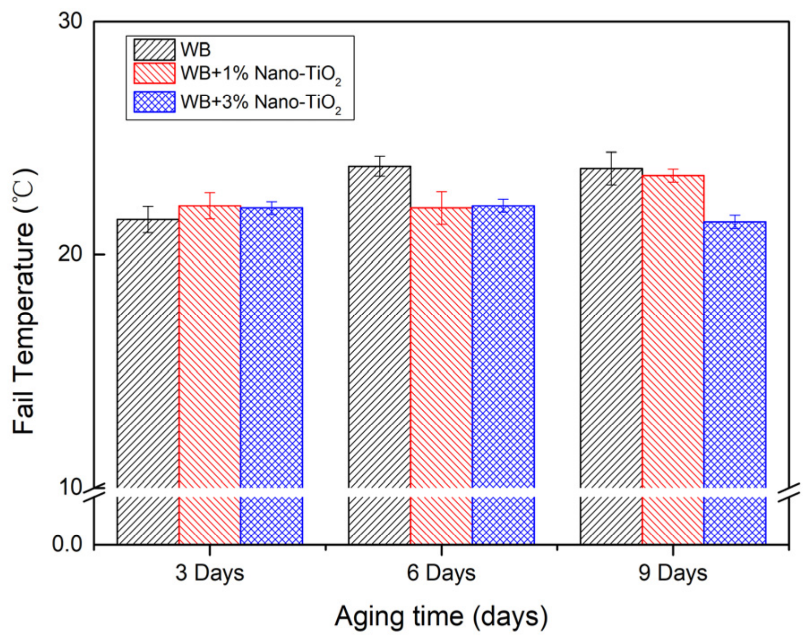

Figure 5. Fail temperatures of UV-aged binders.

3.5. Black Diagram

Figure 6 shows black diagrams of the Nano-TiO_2-modified WB binder at various aging times. The time–temperature superposition (TTS) theory was examined using the black diagrams. Like Cole–Cole, Han and Wicket plots, black diagrams do not function with loading frequency and temperature, i.e., the sufficiency/accuracy of the TTS to the studied asphalt binders can be easily detected using the black diagrams.

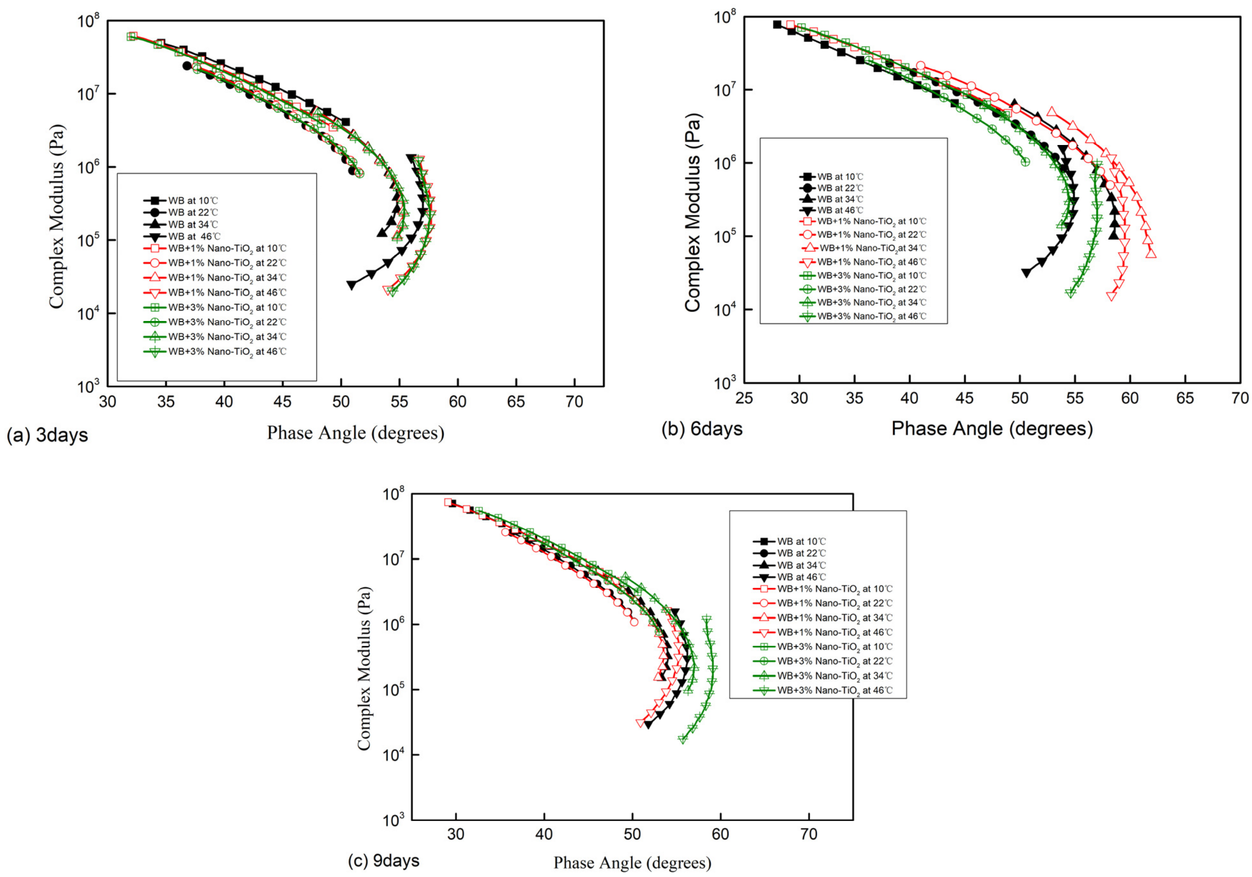

Figure 6. Black diagrams of WB binders with various UV-aging times: (**a**) 3 days; (**b**) 6 days; (**c**) 9 days.

Figure 6 focuses on the performance of WB-modified asphalt with Nano-TiO_2 compared to the WB asphalt bitumen. In Figure 6, the time–temperature superposition (TTS) holds for all aged binders at temperatures ranging from 10 to 34 °C. The black diagram plots lose their single-curve characteristics above 34 °C, suggesting the breakdown of TTS. However, the viscoelastic information reflected by the black curve is still not comprehensive because the viscoelastic material has partial time–temperature equivalence properties.

3.6. Master Curves

The master curves' complex moduli and phase angles of the 2S2P1D model for all UV-aged binders are given in Figure 7. It can be seen from Figure 7a,b that the 2S2P1D model can represent the complex modulus, but the phase angle test value is discontinuous. This shows that the asphalt binders after UV aging only meet part of the time–temperature equivalence principle, i.e., the viscoelastic causality between the viscoelastic parameters is not satisfied. However, compared with the black space diagram, the master curve can more clearly reflect the discontinuity of the viscoelastic parameters (such as the complex modulus and phase angle), i.e., the parameters of time–temperature failure. It can also be seen from Figure 7 that the complex modulus is a constant at low frequencies, and the main curve of the phase angle shows characteristics of a Wicket diagram, indicating that the asphalt still shows characteristics of a viscoelastic solid after different UV aging times. As seen in Figure 7, the aging times and the contents of nanoparticles had no obvious influence on the master curve of the complex modulus and phase angle of asphalt, indicating that the master curve is not very appropriate for evaluating the UV-aging process.

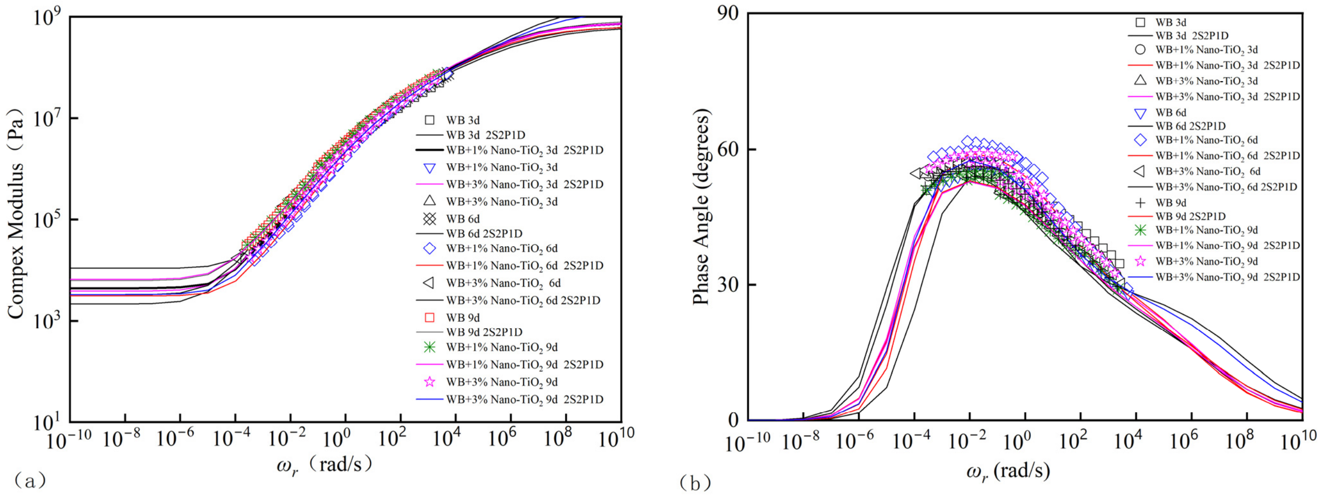

Figure 7. Master curves of viscoelastic parameters for UV-aged WB binder modified with various contents of Nano-TiO_2 at a reference temperature of 22 °C (**a**) complex modulus; (**b**) phase angle.

The parameters of the 2S2P1D model for all UV-aged binders are shown in Table 1. The G_0 values of all UV-aged binders are constant, indicating that nanoparticle-modified WB binders have characteristics of a viscoelastic solid. The G_g values obtained by Di Benedetto et al. [28] vary from 0.9×10^9 to 2×10^9 Pa, and a value of less than 1×10^9 Pa was used in this study. The k and h values for binders with various UV-aging times and contents of nanoparticles are almost the same and less than one, even though an evidently higher value of h is observed. The α values for all UV-aged binders are higher than those obtained by Yusoff et al. [22] in this study. The τ_0 values are related to the horizontal position for master curves.

Table 1. Model coefficients for various UV-aged binders.

Sample	UV/d	G_0/Pa	G_g/Pa	k	h	α	τ_0	β	C_1	C_2	R_c^2	R_a^2
WB	3	2150	6.4×10^8	0.28	0.64	7.73	3.0×10^{-4}	3.3×10^4	39	343.8	0.920	0.502
WB + 1% NANO-TiO_2	3	4378	7.6×10^8	0.33	0.67	8.27	3.3×10^{-4}	3.6×10^4	140	1326.5	0.994	0.883
WB + 3% NANO-TiO_2	3	3830	7.5×10^8	0.31	0.65	8.07	2.7×10^{-4}	3.8×10^4	114	1028.4	0.990	0.838
WB	6	10,740	6.6×10^8	0.29	0.66	7.62	3.4×10^{-4}	4.2×10^4	11	90.1	0.995	0.858
WB + 1% Nano-TiO_2	6	3088	6.4×10^8	0.31	0.69	7.24	3.2×10^{-4}	4.2×10^4	11	93.7	0.997	0.937
WB + 3% NANO-TiO_2	6	3281	9.8×10^8	0.32	0.67	12.42	4.5×10^{-4}	1.9×10^4	238	1930.5	0.988	0.763
WB	9	6146	8.7×10^8	0.29	0.63	7.25	2.0×10^{-4}	5.0×10^4	267	2440.2	0.996	0.874
WB + 1% Nano-TiO_2	9	4378	7.6×10^8	0.33	0.67	8.27	3.3×10^{-4}	3.6×10^4	140	1326.5	0.997	0.903
WB + 3% Nano-TiO_2	9	2150	6.4×10^8	0.28	0.64	7.73	3.0×10^{-4}	3.3×10^4	39	343.8	0.991	0.886

Moreover, Yusoff et al. [22] pointed out that the parameter β is linked to the Newtonian viscosity of the model and has a large influence in this behavior domain. However, with the increase in aging time, parameter β of the 2S2P1D model has no obvious change. The goodness-of-fit of complex modulus is greater than 0.9 and greater than that of the phase angle. This shows that the master curves of the complex modulus gain better fitting in comparison with the phase angle curves for all UV-aged binders.

4. Conclusions

Based on the test results of various durations of UV aging for Nano-TiO_2-modified SBS/Sasobit compound binders, the following conclusions can be drawn:

1. With the increase in UV-aging times, the viscosity had a growth trend for all aged binders. However, the viscosity value of asphalt with nanoparticles increased less significantly compared to the blank sample.
2. The nanoparticles showed insignificant effects on the complex modulus of the asphalt binder at different temperatures. However, nanoparticles can enhance the property of resistance of UV aging in terms of phase angle.
3. The binders with nanoparticles after UV aging showed better fatigue-cracking resistance than the aged binders without nanoparticles due to lower fatigue factors.
4. The results show that the 2S2P1D model can accurately characterize the complex modulus of all UV-aged asphalt binders. However, the measured phase angle value is in poor agreement with the main curve, indicating that the asphalt binders after UV aging only met part of the time–temperature equivalence principle. In addition, the k and h values, as important model parameters, were almost the same and less than one for all UV-aged binders.
5. According to the black space curves and the master curves of the 2S2P1D model, all aged asphalt binders showed viscoelastic solid mechanical characteristics. Compared with the black space curve, the main curve of the 2S2P1D model could better identify whether the time–temperature equivalence principle was satisfied.
6. The black space diagram and the 2S2P1D model were used to investigate the viscoelastic properties of UV-aged binders. In further studies, validation of the model may require longer UV-aging durations for indoor simulations and longer outdoor exposure for all aged binders.

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