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Effect of Thermal Oxygen Conditions on the Long-Term Aging Behavior of High-Viscosity Modified Bitumen

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Abstract: High-viscosity modified bitumen is affected by a complex thermal oxygen environment during long-term service. However, the existing standard long-term thermal oxygen aging test cannot fully simulate the effect of different thermal oxygen conditions on the aging of high-viscosity modified bitumen. In this study, on the basis of the standard pressure aging vessel test, high-viscosity modified bitumen was aged under different oxygen conditions through adjusting test parameters. Then, the analysis of the complex moduli, phase angles, and creep and recovery properties was conducted to evaluate the rheological properties of high-viscosity modified bitumen before and after aging. Moreover, gel permeation chromatography was performed to evaluate the molecular size distribution of high-viscosity modifiers during aging. The results indicate that aging improves the modulus of high-viscosity modified bitumen and changes the phase angle of that. Temperature, pressure, and time are the factors affecting the high-temperature sensitivity and viscoelastic properties of high-viscosity modified bitumen. With respect to the creep and recovery property, different high-viscosity modified bitumen exhibits different aging characteristics with the change of thermal oxygen conditions. Gel-permeation-chromatography results directly illustrate that thermal oxygen conditions influence the degradation of high-viscosity modifiers at the initial stage of long-term aging, which is the key factor affecting the rheological properties of high-viscosity modified bitumen.

Keywords: high-viscosity modified bitumen; thermal oxygen conditions; aging; rheological properties; molecular size distribution

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1. Introduction

Currently, a variety of advanced material technologies are applied to improve the service level of bituminous pavement [1–5]. Porous bituminous pavement is one of the effective means for sponge city because of its ecological and environmental benefits, such as water permeability, noise reduction, and low heat absorption. In order to ensure the strength and stability of the skeleton-void structure of porous bituminous mixture, high-viscosity modified bitumen (HVMB) is often used as binders in Asia because of its excellent performance [6–8]. Specifically, HVMB is a kind of modified bitumen with high polymer content, whose ingredients include thermoplastic polymer, resin, plasticizer, and so on [9].

During the long-term service life of HVMB, the degradation of high-viscosity modifiers and the oxidation of bitumen would occur under the influence of environmental factors, including high temperature, water, ultraviolet (UV) light, and oxygen, thus affecting the service level of porous pavement [10–13]. Particularly, compared with dense bituminous mixtures, bitumen is more likely to be aged due to a large number of void structures of porous mixtures [14]. For the purpose of understanding the aging behavior, some scholars used laboratory tests to simulate the aging of HVMB and then conducted various tests to evaluate the properties of HVMB before and after aging [10,11]. Considering the complex environmental effect of HVMB during service, Sun et al. [10] adopted standard pressure aging vessel (PAV) tests and accelerated weather aging tests to simulate the long-term aging of HVMB and found that the aging of HVMB consisted of base-bitumen oxidation and polymer degradation. After the weather aging, the aging degree reached its highest. Furthermore, Zhang et al. [11] explored the simultaneous aging effects of moisture, temperature, and ultraviolet light on HVMB with the help of a self-designed coupling aging system. The research results indicated that the carbonyl index and sulfoxide index were suitable for evaluating the effect of ultraviolet light and temperature, respectively. Similarly, the research conducted by Li et al. [12] demonstrated that the combination of a high temperature, UV-light, and acid-rain-solution environment had a significant impact on the viscoelastic properties of HVMB. Among the many factors affecting bitumen aging, thermal oxygen aging is one of the key factors affecting HVMB properties. Currently, thin-film oven tests (TFOT) and rolling thin-film oven (RTFO) tests were always used to simulate the short-term thermal oxygen aging of bitumen, and PAV tests conducted at 100 °C under 2.1 MPa of air for 20 h were adopted to simulate the long-term thermal oxygen aging of bitumen [15]. The research conducted by Jiang et al. [16] revealed that the effect of thermal long-term aging on HVMA exceeded that of short-term aging. Furthermore, Hu et al. [13] found that after standard PAV aging, aged HVMB showed increased thermal stability, viscosity, and elastic recovery and presented a rapid increase in carbonyl contents, which revealed that the oxidation of base bitumen dominated the properties of HVMB. The above studies only simulated the aging behavior of HVMB in a specific thermal oxygen environment because of the specific parameters of standard PAV tests (100 °C and 2.1 MPa). Actually, due to the difference of climate, HVMB used in different regions is affected differently by the thermal oxygen environment. Moreover, Hu et al. [17] found that the concentration of reactive oxygen species has a significant effect on the aging of HVMB. However, there are no systematic studies that have investigated the effects of temperature and pressure on the long-term aging behavior of HVMB. Therefore, it is necessary to pay attention to the effects of thermal oxygen conditions on the long-term aging behavior of HVMB.

Considering that current research generally ignores the effect of a thermal oxygen environment on the aging of HVMB, the purpose of this study is to investigate the long-term aging behavior of HVMB under various thermal oxygen environments. Specifically, two kinds of HVMB and one kind of base bitumen were aged through changing the parameters of PAV tests, including pressure, temperature, and time. Afterwards, temperature sweep (TS) tests and multiple-stress creep recovery (MSCR) tests were conducted to explore the rheological properties of HMVB before and after aging in multi-temperature domains. Furthermore, gel-permeation-chromatography (GPC) tests were carried out to systemically explain the chemo-rheological evolution characteristics of HVMB with aging. Finally, the effect of various factors on the aging of HVMB was systematically analyzed. The flow chart of the present study is shown in Figure 1.

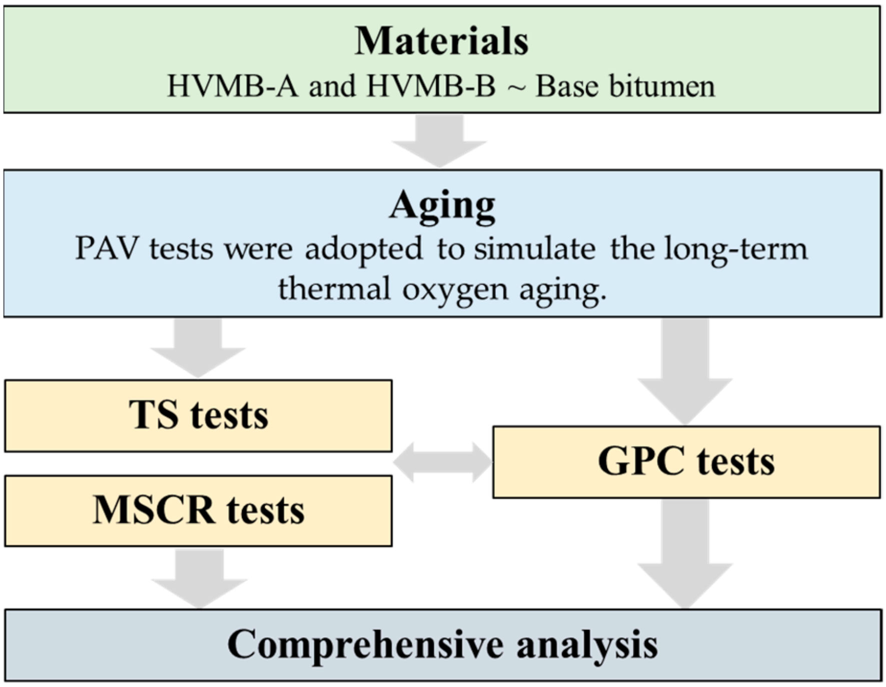


Figure 1. The flow chart of the present study.

2. Materials and Methods

2.1. Materials

Two kinds of commercial HVMB produced by two local Chinese companies were adopted in this study. Moreover, one base bitumen was selected as the experimental control group because HVMB is prepared by base bitumen and high-viscosity modifiers. The basic properties of bitumen are listed in Table 1.

Table 1. Basic properties of bitumen.

Property	HVMB-A	HVMB-B	Base Bitumen	Test Method
Penetration (25 °C, 100 g, 5 s, 0.1 mm)	35	49	67	ASTM D5
Softening point (°C)	92	87	50	ASTM D36
Dynamic viscosity (60 °C, Pa·s)	>400,000	>50,000	-	ASTM D2171

2.2. Aging Methods

Given that a previous study found that bitumen samples aged by PAV for 5 h were comparable to those aged by the standard RTFO test from the standpoint of physicochemical effects [18], HVMB was aged under 2.1 MPa and at 100 °C for 5 h in PAV confinement to simulate the short-term aging. Afterwards, PAV tests were adopted to simulate the long-term thermal oxygen aging of HVMB. For the purpose of simulating the long-term aging of HVMB under different thermal oxygen environments, different temperatures, pressures, and times were selected. The specific test parameter and corresponding sample names are presented in Table 2. For example, A, 100, 2.1, and 20 in HVMB-A-100-2.1-20 represent bitumen type, aging time, aging temperature, and aging pressure, respectively. Moreover, two kinds of HVMB and one kind of base bitumen before aging were named HVMB-A-virgin, HVMB-B-virgin, and Base-virgin, respectively.

Table 2. Aging parameters and corresponding sample names.

Bitumen Type	**Test Parameters of Long-Term Aging**			**Sample ID**
	Temperature/°C	**Pressure/MPa**	**Time/h**	
HVMB-A	100	2.1	20	HVMB-A-100-2.1-20
	100	2.1	40	HVMB-A-100-2.1-40
	100	2.1	60	HVMB-A-100-2.1-60
	100	1.8	20	HVMB-A-100-1.8-20
	100	1.8	40	HVMB-A-100-1.8-40
	100	1.8	60	HVMB-A-100-1.8-60
	80	2.1	20	HVMB-A-80-2.1-20
	80	2.1	40	HVMB-A-80-2.1-40
	80	2.1	60	HVMB-A-80-2.1-60
HVMB-B	100	2.1	20	HVMB-B-100-2.1-20
	100	2.1	40	HVMB-B-100-2.1-40
	100	2.1	60	HVMB-B-100-2.1-60
	100	1.8	20	HVMB-B-100-1.8-20
	100	1.8	40	HVMB-B-100-1.8-40
	100	1.8	60	HVMB-B-100-1.8-60
	80	2.1	20	HVMB-B-80-2.1-20
	80	2.1	40	HVMB-B-80-2.1-40
	80	2.1	60	HVMB-B-80-2.1-60
Base bitumen	100	2.1	20	Base-100-2.1-20
	100	2.1	40	Base-100-2.1-40

2.3. TS Tests

TS tests were performed to collect the complex modulus and phase angle of HVMB and base bitumen before and after aging at 58 °C, 64 °C, 70 °C, 76 °C, and 82 °C. In order to compare the rheological properties of aged bitumen under different thermal oxygen environments, the same test parameters were selected for all samples. Specifically, a sinusoidal oscillating load with a frequency of 10 rad/s ± 0.1 rad/s was applied in a strain-controlled mode with a strain level of 10%.

2.4. MSCR Tests

The MSCR tests were performed to obtain a nonrecoverable creep compliance along with percent recoveries of bitumen at 0.1 and 3.2 kPa with reference to AASHTO T-350 [19,20]. Specifically, this test was conducted through a constant stress creep of 1 s, followed by a zero-stress recovery period of 9 s, with 20 cycles at 0.1 kPa and then 10 cycles at 3.2 kPa. The testing temperatures consisted of 58 °C, 64 °C, 70 °C, and 76 °C.

2.5. GPC Tests

GPC tests were used to measure the molecular size distribution of HVMB before and after aging. Before tests, approximately 35 mg of HVMB was dissolved in 10 mL of tetrahydrofuran to prepare a bituminous solution. Afterwards, the prepared solution was injected into the manual sample injector of GPC. After about 40 min of testing, the raw distribution curve of the molecular size was exported. Considering the effect of bitumen concentration on the collected signal response, the normalization method was used to eliminate the effect of different sample concentrations on the results, which is helpful to qualitatively compare the molecular weight change caused by aging [21]. Based on the research conducted by Daly et al. [22], the GPC chromatogram of polymer-modified bitumen can be divided into three components, including polymers, asphaltenes, and maltenes, as shown in Figure 2.

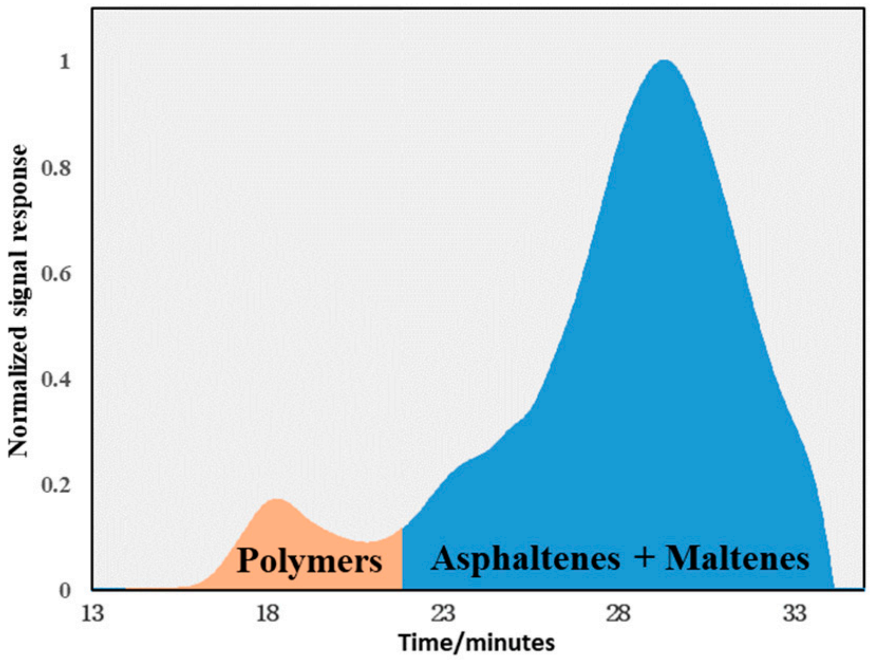


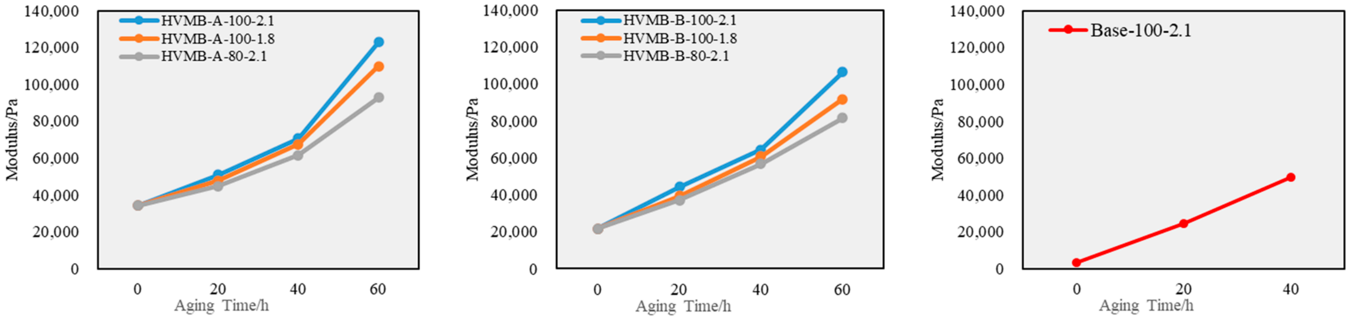
Figure 2. The schematic of the GPC chromatogram division.

3. Results and Discussion

3.1. Complex Moduli and Phase Angles

3.1.1. Complex Moduli

The complex moduli of two kinds of HVMB and one kind of base bitumen aged under different thermal oxygen conditions were obtained through TS tests. Figure 3 shows the measured complex moduli at 58 °C. It can be seen that HVMB has the higher complex modulus compared with base bitumen. With the extension of aging time, the complex moduli of two kinds of HVMB increase gradually. Furthermore, under the same aging time, the higher the temperature and pressure, the higher the aging degree of HVMB. Compared with pressure, temperature has a more significant effect on aging. Similarly, the measured complex modulus at 64, 70, 76, and 82 °C also presents a consistent law. Due to space constraint, the specific results are no longer listed.

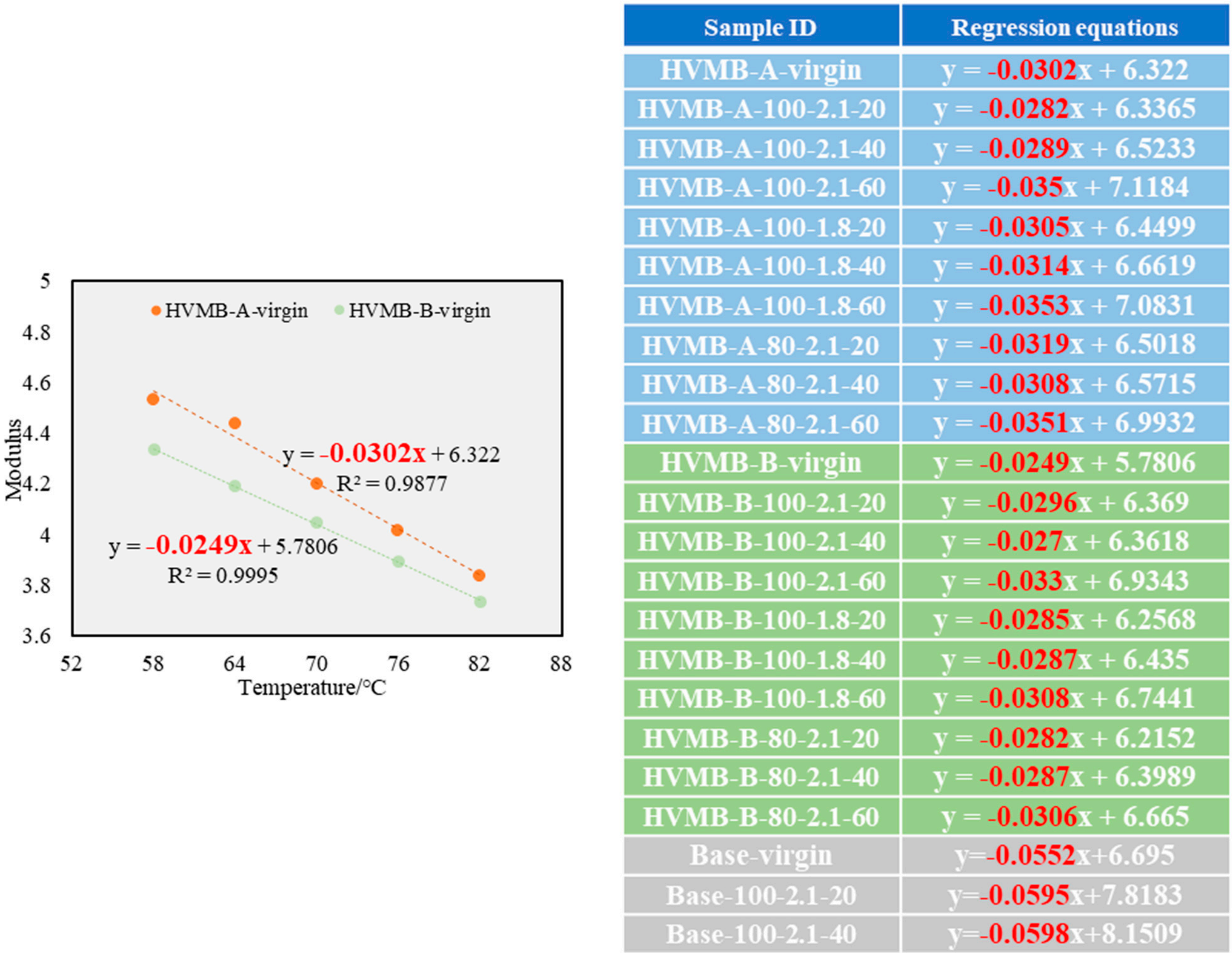


(**a**) (**b**) (**c**)

Figure 3. Measured complex moduli at 58 °C: (**a**) HVMB-A; (**b**) HVMB-B; and (**c**) base bitumen.

Considering that the logarithmic complex modulus exhibits a good linear change with temperature at high temperature [23], the high-temperature sensitivity of HVMB can be evaluated by calculating the sensitivity coefficient. Specifically, the high-temperature sensitivity coefficient is the value of the slope obtained by regression, as shown in Figure 4a. When the absolute value of the slope is larger, the sample is more sensitive to high temperature. Based on this method, the regression equations were obtained, as shown in Figure 4b.

Then, the high-temperature sensitivity coefficient of each sample before and after aging was collected, as illustrated in Figure 5. It can be found that although the complex modulus of HVMB increases gradually with the extension of aging time, the high-temperature sensitivity does not show a consistent monotone change. Specifically, when the aging time of the PAV was set to 20 h, samples aged by different thermal oxygen conditions, including temperature and pressure, present different changes of the temperature sensitivity. Given that the temperature sensitivity of base bitumen changed monotonously with the progress of aging in Figure 5c, this non-monotonic change of high-temperature sensitivity of HVMB can be attributed to the effect of thermal oxygen conditions on the degradation of high-viscosity polymers and the aging of base bitumen.



Sample ID	Regression equations
HVMB-A-virgin	y = -0.0302x + 6.322
HVMB-A-100-2.1-20	y = -0.0282x + 6.3365
HVMB-A-100-2.1-40	y = -0.0289x + 6.5233
HVMB-A-100-2.1-60	y = -0.035x + 7.1184
HVMB-A-100-1.8-20	y = -0.0305x + 6.4499
HVMB-A-100-1.8-40	y = -0.0314x + 6.6619
HVMB-A-100-1.8-60	y = -0.0353x + 7.0831
HVMB-A-80-2.1-20	y = -0.0319x + 6.5018
HVMB-A-80-2.1-40	y = -0.0308x + 6.5715
HVMB-A-80-2.1-60	y = -0.0351x + 6.9932
HVMB-B-virgin	y = -0.0249x + 5.7806
HVMB-B-100-2.1-20	y = -0.0296x + 6.369
HVMB-B-100-2.1-40	y = -0.027x + 6.3618
HVMB-B-100-2.1-60	y = -0.033x + 6.9343
HVMB-B-100-1.8-20	y = -0.0285x + 6.2568
HVMB-B-100-1.8-40	y = -0.0287x + 6.435
HVMB-B-100-1.8-60	y = -0.0308x + 6.7441
HVMB-B-80-2.1-20	y = -0.0282x + 6.2152
HVMB-B-80-2.1-40	y = -0.0287x + 6.3989
HVMB-B-80-2.1-60	y = -0.0306x + 6.665
Base-virgin	y=-0.0552x+6.695
Base-100-2.1-20	y=-0.0595x+7.8183
Base-100-2.1-40	y=-0.0598x+8.1509

(**a**) (**b**)

Figure 4. Regression analysis: (**a**) calculation example of regression and (**b**) regression equations.

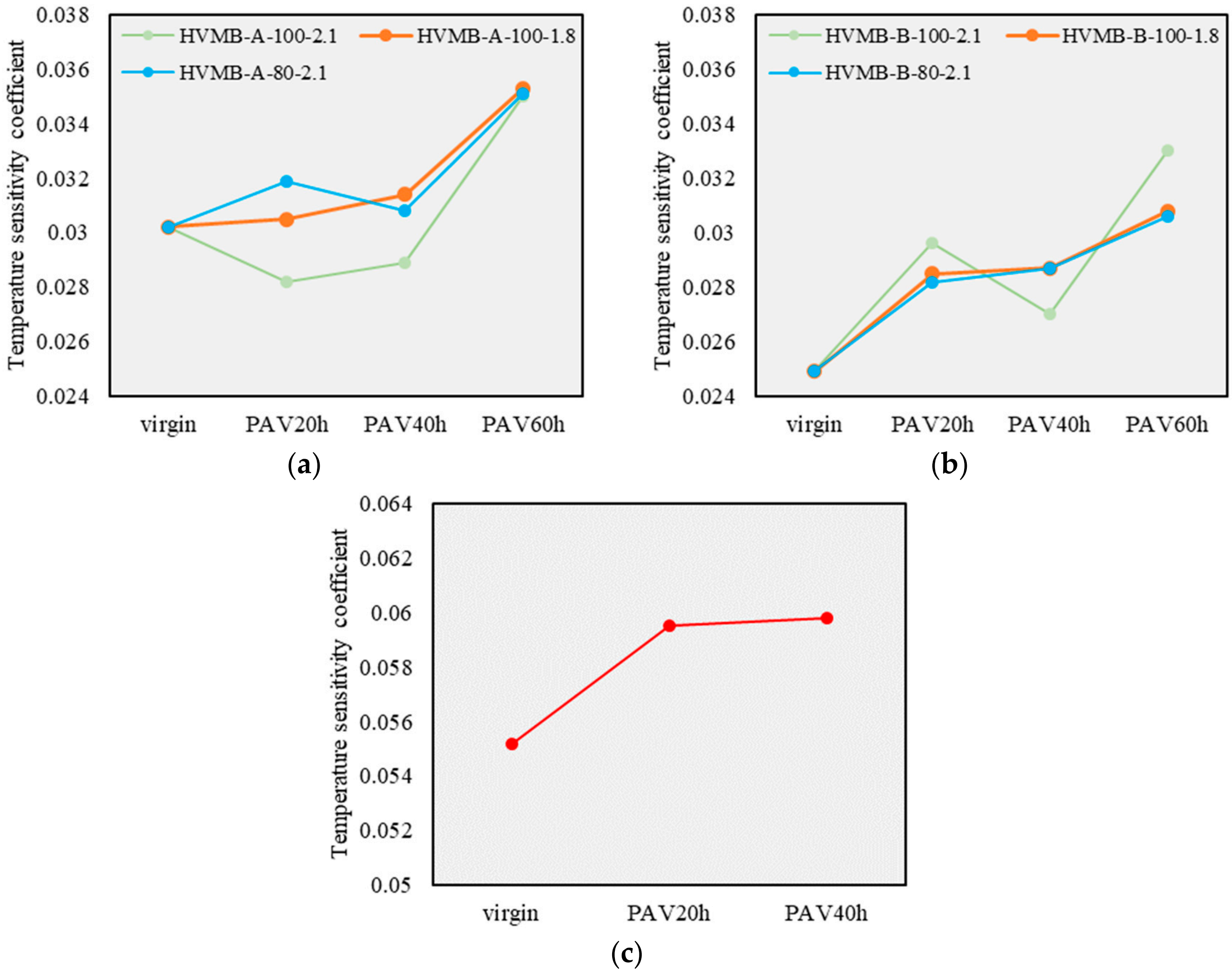
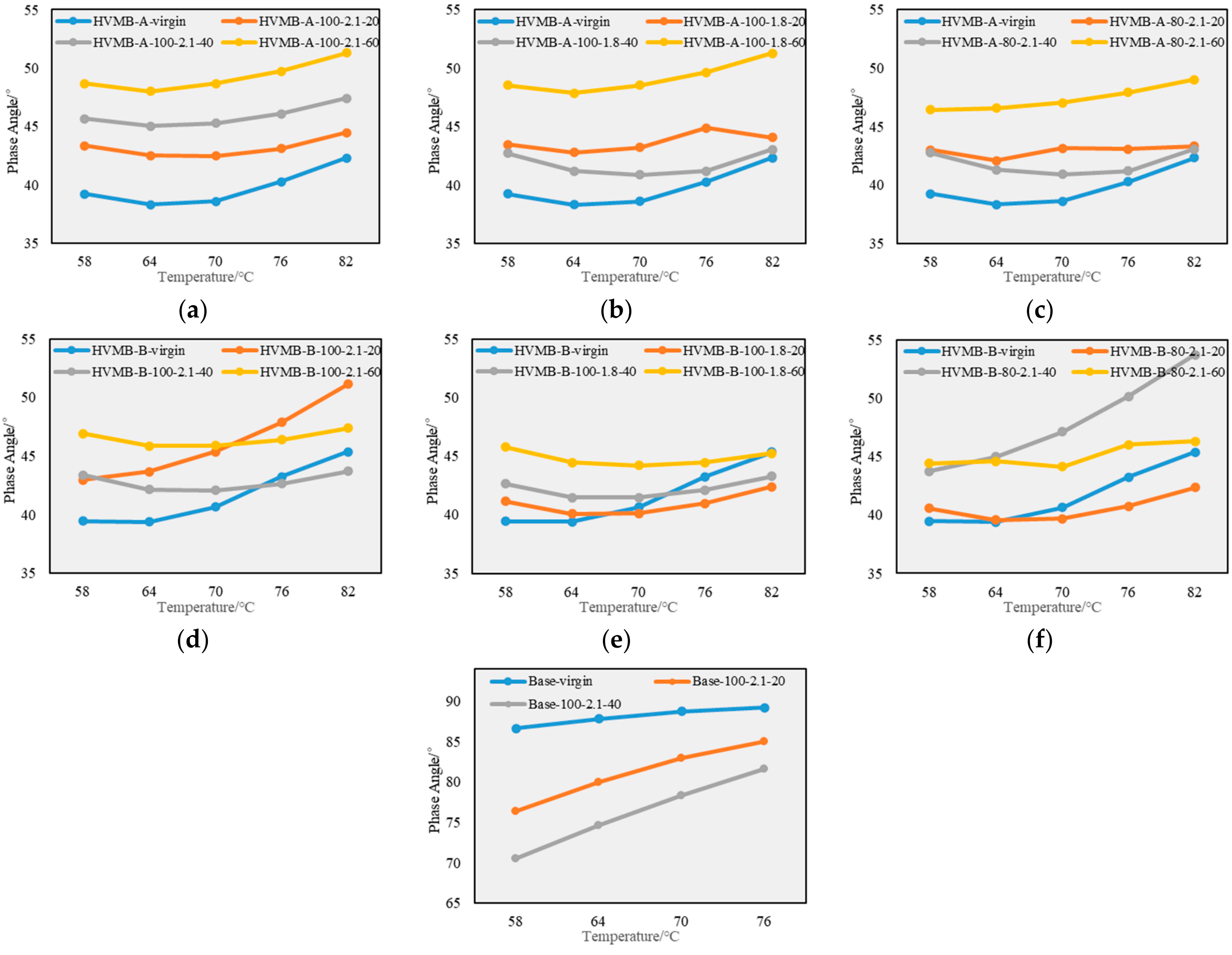


Figure 5. Calculated temperature sensitivity coefficients: (**a**) HVMB-A; (**b**) HVMB-B; and (**c**) base bitumen.

3.1.2. Phase Angles

At the same time as the collection of the complex moduli, the phase angles of the samples were obtained, as shown in Figure 6. It can be found that compared with base bitumen, two kinds of HVMB have smaller phase angles. With the increase in test temperature, the phase angle of HVMB did not increase monotonously, especially for some aged samples, which is different than the change law of phase angles of base bitumen in Figure 6g. This occurred because the apparent viscoelasticity of HVMB is the result of competition between base bitumen and polymers, whose temperature sensitivity is different. Furthermore, through comparing the change of phase angles with the extension aging time of samples aged by different pressures and temperatures, it can be found that various samples of HVMB present different change laws of phase angles when aged by PAV for 20 h and 40 h, while samples show a similar change law of phase angles when aged by PAV for 60 h. This interesting phenomenon indicates that pressure and temperature directly affect the relative rate of polymer degradation and base bitumen aging because the relative rate determines the measured phase angle. Moreover, the similar change laws of phase angles of various samples aged by PAV for 60 h reveal that the effect of pressure and temperature is no longer significant when the aging time is long enough.



(**a**) (**b**) (**c**)

(**d**) (**e**) (**f**)

(**g**)

Figure 6. Phase angles of samples at different temperatures: (**a**) HVMB-A-100-2.1; (**b**) HVMB-A-100-1.8; (**c**) HVMB-A-80-2.1; (**d**) HVMB-B-100-2.1; (**e**) HVMB-B-100-1.8; (**f**) HVMB-B-80-2.1; and (**g**) base bitumen.

3.2. Creep and Recovery Properties

3.2.1. Creep and Recovery Characteristics of Different Cycles

Given that Jnr 3.2 of SBS-modified bitumen under each cycle is obviously discrete according to previous research [19,21], the creep and recovery characteristics of HVMB under different cycles were analyzed through taking HVMB-A-virgin as an example. Specifically, the strain variation of each cycle under 3.2 kPa was calculated according to the related method [19,21], as shown in Figure 7a. It can be intuitively found that the strain change of each cycle under 3.2 kPa of stress is not completely consistent. Furthermore, for the purpose of quantitatively characterizing the difference of strain in each cycle, the coefficient of variation of Jnr 3.2 in different cycles was calculated according to the order from back to front, as shown in Figure 7b. At the initial stage of loading, the strain change of each cycle is quite different. With the increase in the number of cyclic loadings, the difference of each cyclic strain gradually decreases. In detail, the coefficient of variation in the last six cycles is less than 10%. Also, other HVMB before and after aging presents similar characteristics. Therefore, in this study, the R 3.2 and Jnr 3.2 were calculated using the strain data of the last six cycles. However, it should be noted that compared with SBS-modified bitumen in the other research [19], HVMB has more obvious strain instability, which can be attributed to the higher polymer content. Whether the loading cycle of 3.2 kPa in AASHTO T-350 for only ten times is small needs to be further verified in the future.

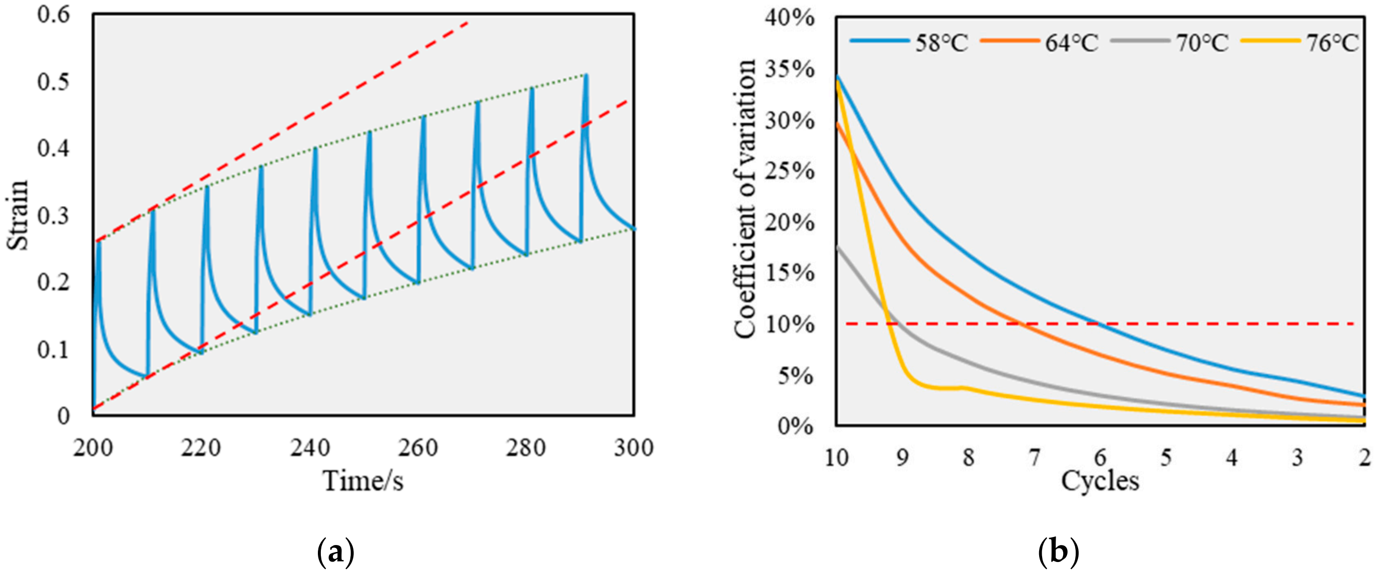
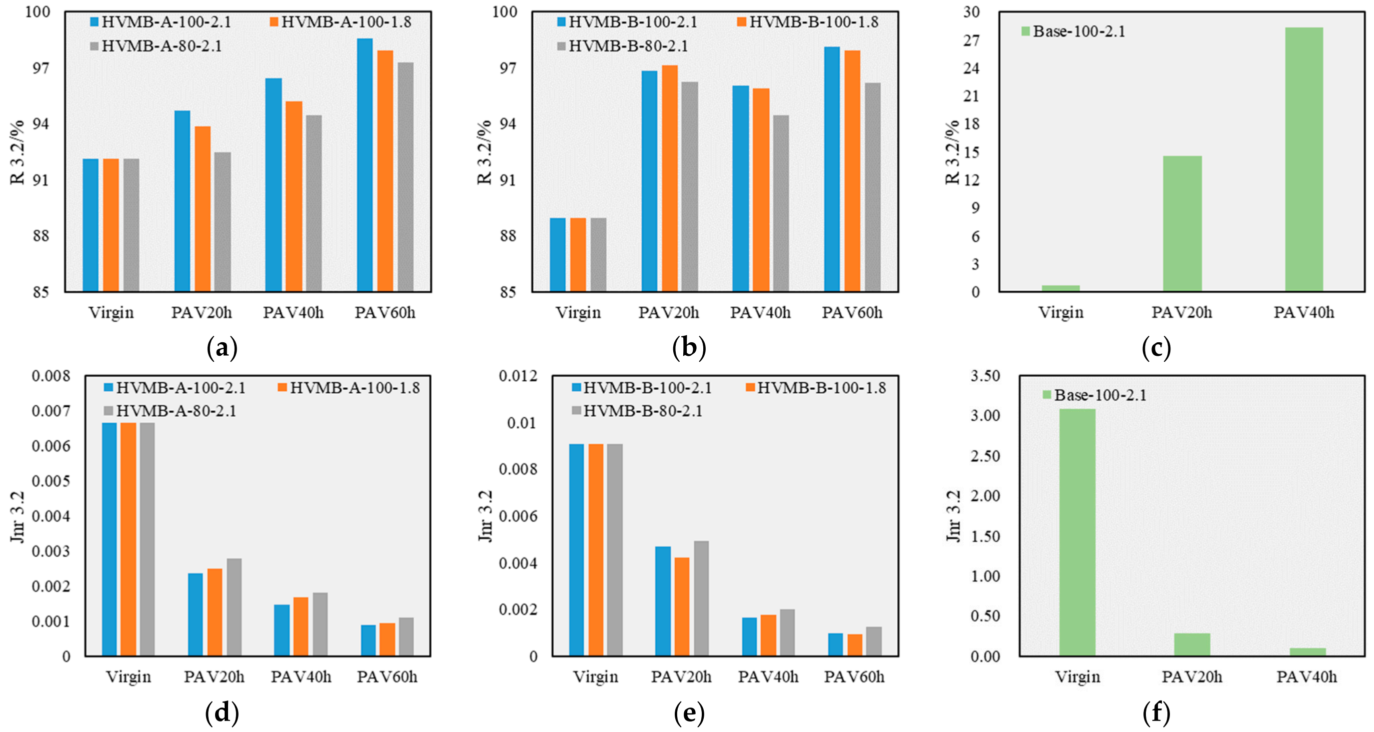


Figure 7. Creep and recovery characteristics of different cycles of HVMB-A-virgin: (**a**) strain variation of each cycle under 3.2 kPa and (**b**) coefficient of variation of Jnr 3.2 under different cycles.

3.2.2. Creep and Recovery Properties before and after Aging

Based on the above findings, the R 3.2 and Jnr 3.2 of two kinds of HVMB and one kind of base bitumen before and after aging were obtained at 58 °C, as shown in Figure 8. Obviously, compared with base bitumen, HVMB has higher R 3.2 and lower Jnr 3.2, which indicates that HVMB has the better ability of elastic recovery and anti-deformation. After long-term aging, the elastic recovery of bitumen increases, while the creep deformation decreases significantly. For HVMB-A, the extension of aging time leads to the increase in R 3.2 and the decrease in Jnr 3.2, which is consistent with the characteristics of base bitumen. Moreover, the increase in temperature and pressure results in the increase in elastic recovery and decrease in creep deformation. Similarly, the Jnr 3.2 of HVMB-B decreases with the extension of aging time, as illustrated in Figure 8e. However, the R 3.2 of HVMB-B does not change monotonously with aging, which is different with the aging law of base bitumen in Figure 8c. This interesting finding can be attributed to the degradation of modifiers and the aging of base bitumen. Actually, the degradation of high-viscosity modifiers reduces the elasticity of modified bitumen, while the aging of base bitumen increases the elasticity of modified bitumen. Thus, because of higher R 3.2, it can be deduced that when HVMB-B is aged by PAV for 20 h, the aging of base bitumen is more significant because of the characteristics of base bitumen aging in Figure 8c. Then, the degradation of modifiers is more obvious. Furthermore, it can be found that when HVMB-B is aged by PAV for 20 h, with the decrease in pressure, creep and recovery properties show an increase in R 3.2 and decrease in Jnr 3.2, which is different from the law of creep and recovery under other aging times. This finding indicates that for HVMB-B, the reduction of pressure inhibits the degradation of high-viscosity modifiers.



(**a**) (**b**) (**c**)

(**d**) (**e**) (**f**)

Figure 8. Results of MSCR tests conducted at 58 °C: (**a**) R 3.2 of HVMB-A; (**b**) R 3.2 of HVMB-B; (**c**) R 3.2 of base bitumen; (**d**) Jnr 3.2 of HVMB-A; (**e**) Jnr 3.2 of HVMB-B; and (**f**) Jnr 3.2 of base bitumen.

3.3. Molecular Size Distribution

GPC tests were selected to evaluate the molecular size distribution of HVMB-A before and after aging. Figure 9 presents the normalized GPC chromatograms. Obviously, polymer peaks continuously move to the right with aging, which verifies that high-viscosity modifiers degrade and then convert into relatively small molecule compounds. In contrast, maltenes transform into asphaltenes with aging, showing enhanced response between 22 and 27 min. Considering that the molecular weight of degraded modifiers partially overlaps with that of asphaltenes [24], only the polymer phase is magnified to further analyze the effect of thermal oxygen conditions on the degradation of high-viscosity modifiers. It can be found from Figure 10a that the degradation degree of HVMB-A aged by PAV for 20 h under different thermal oxygen conditions is significantly different. Thus, temperature and pressure can be considered as the key factors affecting the degradation of high-viscosity modifiers. Specifically, increasing temperature and pressure can accelerate the degradation of modifiers. Compared with pressure, the effect of temperature is more significant. Furthermore, after PAV aging for 40 h, the GPC chromatograms coincide with the changes of aging time and thermal oxygen conditions, as shown in Figure 10b,c, which indicates that high-viscosity modifiers had been fully degraded.

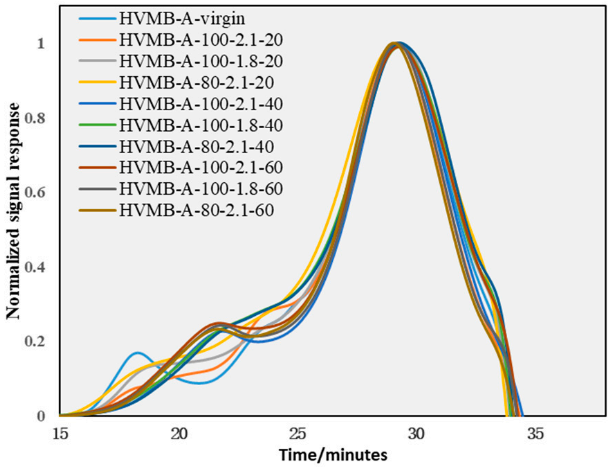
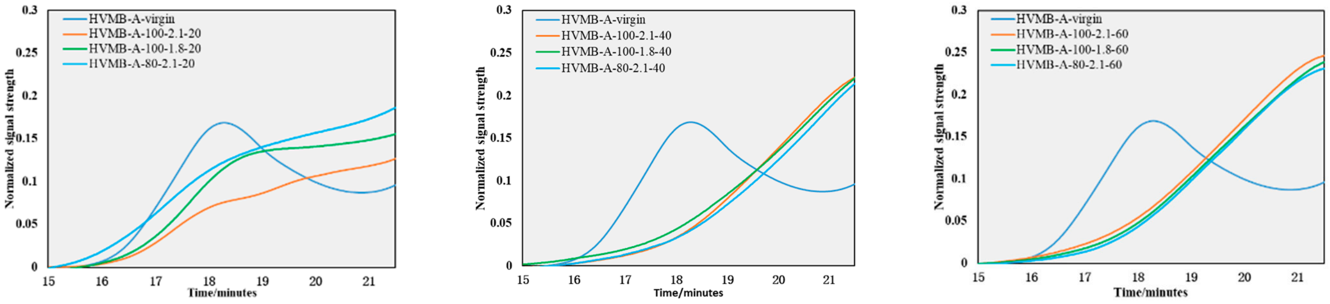


Figure 9. The GPC chromatograms of HVMB-A before and after aging.



(**a**) (**b**) (**c**)

Figure 10. The local chromatograms of aged HVMB-A. (**a**) PAV for 20 h; (**b**) PAV for 40 h; (**c**) PAV for 60 h.

3.4. Comprehensive Analysis

The modulus of HVMB increases gradually with the extension of aging time, indicating that HVMB is aged continuously. Given that the degradation of high-viscosity modifiers and aging of base bitumen have the opposite effect on the change of viscoelasticity, the collected viscoelastic properties are the results of the competition between the degradation of high-viscosity modifiers and aging of base bitumen. This directly illustrates that the temperature sensitivity, phase angles, R 3.2, and Jnr 3.2 of two kinds of HVMB after aging do not show consistent regular change. Furthermore, it can be clearly found that the changes of temperature and pressure do not affect the monotonic growth of the modulus of HVMB but has a significant effect on the viscoelastic law before and after aging, which indicates that thermal oxygen conditions directly affect the relative rates of modifier degradation and bitumen aging.

Considering that the molecular distribution obtained by GPC can characterize the degradation of modifiers, the GPC results are always used to explain the macroscopic test result [19,25]. It can be found from Figures 5a and 10 that the different change laws of temperature sensitivity of HVMB-A aged by PAV for 20 h under different thermal oxygen conditions are related to the degradation degree of modifiers. After aging by PAV for 40 h, the degradation of high-viscosity modifiers gradually stops, while the aging of base bitumen is dominant, which shows that the change of the temperature sensitivity coefficient under different thermal oxygen conditions gradually tends to be the same. Similarly, in Figure 6, when HVMB-A is aged by PAV for 60 h, the phase angles under different thermal oxygen conditions tend to be the same. In contrast, the inconsistency of phase angles under different thermal oxygen conditions before aging by PAV for 60 h can be explained by the different degradation degree of high-viscosity modifiers.

4. Conclusions

This present study investigates the effect of thermal oxygen conditions on the long-term aging behavior of HVMB through DSR and GPC methods. Firstly, the long-term aging of HVMB and base bitumen under different thermal oxygen conditions was simulated by adjusting the parameters of PAV tests. Then, the rheological properties of HVMB and base bitumen before and after aging were tested by TS tests and MSCR tests. Finally, GPC tests were conducted to monitor the molecular size distribution of HVMB-A. According to the collected results, the following conclusions can be drawn:

(1) Decreasing aging temperature and pressure can reduce the aging degree of HVMB. Also, the aging temperature and pressure affect the change of temperature sensitivity and viscoelastic properties, which can be attributed to the effect of the relative rate of modifier degradation and base bitumen aging.

(2) The creep and recovery of HVMB have obvious instability under each cycle. In the future, it is necessary to further explore the MSCR test suitable for HVMB. With the extension of aging time, the creep and recovery properties of two kinds of HVMB are not completely the same, but HVMB-A and HVMB-B aged by PAV for 60 h show the characteristics of larger elastic recovery and less creep compared with virgin HVMB.

(3) The results of the GPC tests show that pressure and temperature significantly affect the degradation rate of high-viscosity modifiers at the initial stage of long-term aging. With the progress of long-term aging, there is no obvious degradation of modifiers when degrading to a certain extent. These results demonstrate that at the initial stage of long-term aging, the rheological properties of HVMB are mainly affected by the degradation of modifiers. Afterwards, the rheological properties of aged HVMB are mainly affected by the aging of base bitumen.

Author Contributions: Conceptualization, C.X. and M.L.; methodology, C.X. and M.L.; formal analysis, C.X., T.J. and J.Q.; data curation, C.X., M.L. and T.J.; writing—original draft preparation, C.X. and J.Q. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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