

Article

# Evaluation of the Rheological Property of Binder-Filler Systems after Oxidation Based on a Simple Film Oven Aging Method

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**Abstract:** Asphalt mastic is a combination of binder and filler. The binder-filler system within asphalt mixtures plays an important role in adhesion between mineral aggregates. The aging of binders in pavement always happens with fillers inside or contact with mineral aggregates, so it is critical to investigate the evolved rheological property of binder-filler systems during oxidative aging. In this study, simple film oven aging methods for the aging of mastics (binder-filler system) were conducted and verified by comparing the master-curves of aged mastics at different oven positions or different aging times. The frequency sweep test was performed to measure the changing stiffness of mastics with a different combination of binders and filler contents. Test results show that oven positions could influence the aging effect of the mastics significantly, given the influence of circulation. With increasing aging times, the complex modulus increases while phase angle decreases. Comparing the mastics which were first aged then mixed with mastics which were first mixed then aged, it was observed that fillers inside the binder could accelerate aging of the mastics. Additionally, the aging index of mastics with different combinations showed that both the modification of binders and filler contents could affect the aging rate of the mastics.

**Keywords:** asphalt mastics; rheological properties; oxidative aging; oven study; aging index

## 1. Introduction

The oxidative behavior of asphalt is thought to be a critical factor contributing to the performance of hot-mix asphalt (HMA) pavements [1,2]. During the oxidation, the ever-changing chemistry, kinetics, and mechanisms of the binder cause the deterioration of pavement performance, which has significant negative influence on the durability of asphalt mixtures, reducing the service life of asphalt pavement [3]. The process of the aging of binder could be divided into two parts. One is the short-term aging, which is caused by heating during the construction. The light oil within the binder may be evaporated and the oxidation is severe during the high-temperature mixing and compaction [4]. The other one is the long-term aging during the service time. Both the traffic effect and environmental condition could cause the aging or decay of the asphalt binder [5]. Factors affecting the aging of asphalt binders could fall into two types, including the internal factors from the material properties (such as *viscosity of binder*, *types of the modifier*, *air void*, *binder content*, and so on) and the external factors from the environment or traffics (such as *sunlight*, *air oxidation*, *rain soaking*, *traffic loading*, and so on) [6–9]. To evaluate or simulate the oxidation of binders in the laboratory, many binder tests have been calibrated and discussed in a previous study [10]. The rolling thin-film oven tests (RTFOT) play an important role in quantifying the extent of binder oxidation and hardening during the construction

process, which is called short-term aging [11]. For the long-term aging simulation, the short-term aged binder is conducted with the pressure aging vessel (PAV) procedure for more than 20 hours [12]. However, the effect of this 20H PAV procedure that corresponds to the in-service aging time is that it is unpredictable, since the climate (air or temperature) is not constant with the development of time or at different locations [13,14]. Additionally, the distributions of binder within the mixtures are varying. Therefore, it is also critical to consider the binder-filler system within the mixtures when it comes to the oxidation of binder or mastics. The conventional thin-film oven test (RTFOT) and pressure aging vessel (PAV) may not be suitable for the aging of blends of bitumen and filler due to the dramatically increased viscosity of the mastics [15]. Binder aging in pavement always occurs while binder is distributed around aggregates and mineral fillers with a certain distribution of thickness [16]. The catalytic or mitigating effect between the binder and mineral filler could influence the oxidation of the binder or mastics by changing the molecular size distribution of the aged binder [17,18]. Moraes and Bahia [19] found that during oxidative aging of asphalt binders and mastics, diffusion and adsorption mechanisms played a role in the rate of aging of asphaltic material. Filler concentration and mineralogy type could influence the oxidative process of the mastic significantly. Huang and Zeng have established the aging time-temperature effect for the rheological properties of asphalt mastics using PAV-aging [20]. Previous research has proven that the chemical components of binder, saturates, aromatics, resins, and asphaltenes are the key factors influencing the viscoelastic properties of asphalt binder [21,22]. Meanwhile, both the binder and the fillers scattered within the binder should also have an essential effect on the rheological properties of asphalt mastic [23].

Based on the above consideration, the main objectives of this study were as follows:

- (1) Develop a simple verified film oven aging method for the laboratory preparation of aged binder-filler systems.
- (2) Evaluate the effect of aging times and aging temperatures on the oxidation level.
- (3) Investigate the changing rheological property of binder-filler systems with different binders or filler contents during oxidation.

## 2. Materials and Design

Mastic samples with different binders and filler contents were prepared in this paper. One 70# neat binder meeting the superpave specification for PG58-22 and two styrene-butadiene-styrene (SBS) modified asphalt binders respectively meeting the superpave specification for PG70-22 and PG76-22 were selected for the fabrication of mastics. The PG70-22 binder has an SBS content of 3.5%, while the PG76-22 binder had an SBS content of 4%. The main rheological properties of the three asphalt binders are exhibited in Table 1. When it comes to the combination of both fillers and binders, the definition of the filler content is not unified. The mastics could be produced by certain filler/binder volume fractions or mass ratios. In this paper, similar to the mix design of asphalt mixtures, the mastics were designed by the mass ratio of the filler and binders. The filler content (F/B) was determined by Equation (1).

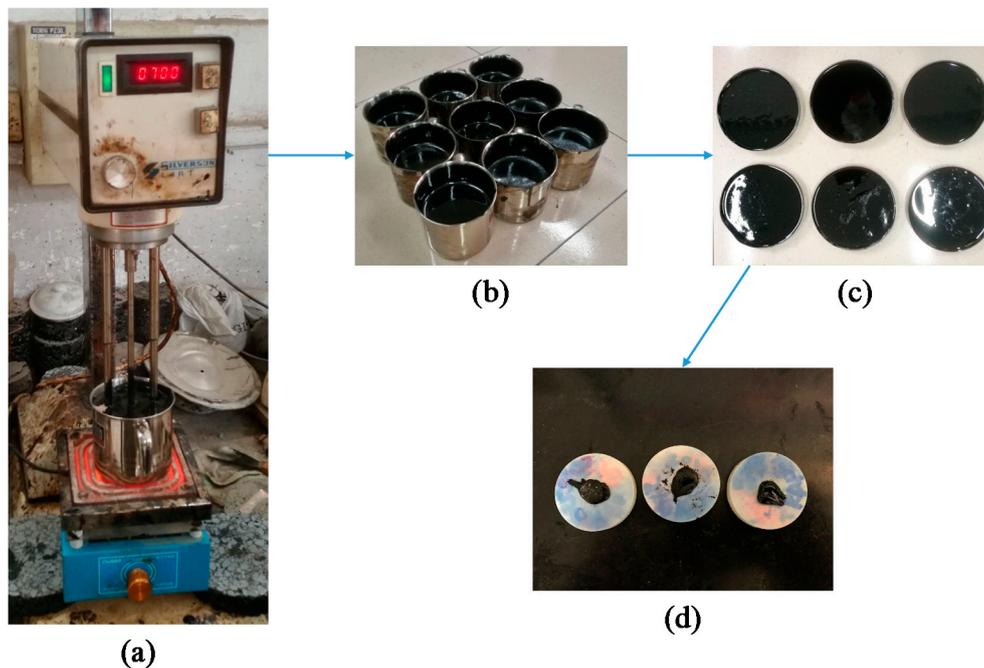
$$F/B = \frac{W_{filler}}{W_{binder}} \quad (1)$$

where:  $F/B$  = filler content, %;  $W_{filler}$  = mass of the filler, g;  $W_{binder}$  = mass of the binder, g.

A limestone mineral filler with a particle density of 2.695 g/cm<sup>3</sup> was used and sieved by the sieve size of 0.075 mm. The filler content was in the range of 20% to 180%, including 20%, 50%, 80%, 100%, 120%, 150%, and 180%. The mastics were produced by mixing the binder and filler using a homogenizer as Figure 1a illustrates. Before mixing, the binder and filler were firstly conditioned in an oven with a constant temperature of 165 °C for 30 min. Then the weighted binder was placed in a heated can and fillers were added into the binder slowly and blended by a high-speed mixer for 20 min to avoid filler segregation. During the mixing process, the mixing temperature was controlled within 165 ± 5 °C by an electric furnace control to guarantee the workability of the binder. Finally, mastics with different filler contents and binder types were fabricated as shown in Figure 1b.

**Table 1.** Rheological properties of three different binders.

| Properties                               | 70#           | PG70-22       | PG76-22       |
|--|---------------|---------------|---------------|
| Penetration (25 °C, 100 g, 5 s) (0.1 mm) | 70            | 65            | 51            |
| Ductility (cm, 5 cm/min, 5 °C)           | 25            | 38.1          | 33            |
| Softening point (°C)                     | 47            | 62.4          | 82            |
| G* /sinδ (Original sample)               | 1.513 (58 °C) | 1.991 (70 °C) | 1.880 (76 °C) |
|  | 0.773 (64 °C) | 0.956 (76 °C) | 0.652 (82 °C) |
| G* /sinδ (After RTFOT)                   | 5.027 (58 °C) | 2.881 (70 °C) | 2.831 (76 °C) |
|  | 1.923 (64 °C) | 1.194 (76 °C) | 1.321 (82 °C) |



**Figure 1.** Sample preparation procedures: (a) mixing by the homogenizer; (b) Completed asphalt mastics; (c) Mastic plates after oven aging; (d) Samples out of the silicon mold.

### 3. Test Setup

Similar to asphalt binder, the rheological properties of asphalt mastics also depend on temperature and loading frequency or time [24]. Therefore, the time-temperature sweep (TTS) test was carried out using the dynamic shear rheometer (DSR) to obtain the complex modulus ( $G^*$ ) and phase angle ( $\delta$ ) at different temperatures and loading frequencies. As Figure 1d shows, A 8 mm parallel plate geometry with a 2 mm gap was used in the TTS test at temperature of 5 °C, 15 °C, 25 °C, and 35 °C for the mastic samples. The loading frequencies for all test temperatures were the same, including 0.2, 0.4, 0.6, 0.8, 1, 2, 4, 6, 8, 10, 20 and 30 Hz. A constant shear strain amplitude of 0.01% was applied during the dynamic loading within the linear visco-elastic zone of the mastics. According to the time-temperature equivalence principle, the  $|G^*|$  at different temperatures and frequencies could be shifted to one master-curve under one reference temperature by the WLF function as Equation (2) shows. Given that, the composition of the mastics is complex compared to the neat asphalt binder, including different filler contents and different modified asphalt binders, the shape parameter  $m_e$  of the master-curve may not be equal to 1, so the CAM model [25] was used to fit the complex modulus mastercurve of the mastics as Equation (3) exhibits. Equation (4) shows the calculation of the parameter  $R$ .

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

$$G^* = G_e^* + \frac{G_g^* - G_e^*}{[1 + (f_c/f')^k]^{m_e/k}} \tag{3}$$

$$R = \log \frac{2^{m_e/k}}{1 + (2^{m_e/k} - 1)G_e^*/G_g^*} \tag{4}$$

where:  $T$  = test temperature, °C;  $\alpha_T$  = the shifted factors at temperature  $T$ ;  $C_1, C_2$  = fitting coefficient;  $T_0$  = the reference temperature, °C;  $G_g^*$  = glass modulus, Mpa;  $G_e^*$  = the modulus of equilibrium, Mpa;  $f_c$  = crossover frequency, Hz;  $m_e, k$  = shape parameters;  $f'$  = reduced frequency, Hz.  $G^*$  = predicted complex modulus, Mpa;  $R$  = the width of the relaxation spectrum.

#### 4. Discussion of Results

In order to investigate the rheological properties of aged asphalt mastics, the asphalt mastics were aged by a verified thin-film oven method firstly, and then mastics under different aging conditions with different compositions were tested by the DSR and the rheological parameters from the master-curve were analyzed and compared.

##### 4.1. Film Oven Aging Method

To simulate the oxidation aging of the mastics, they were placed in a steel plate with an identical thin mastic film thickness of 2 mm. As Figure 1c illustrates, the standard steel plate has an inner diameter of  $140 \pm 1$  mm, so the weight of the mastics with different filler content in one plate could be calculated by Equation (5); the results are exhibited in Table 2.

$$W_{mastic} = \pi r^2 \cdot t \cdot \left( \frac{1 + F/B}{\frac{1}{\gamma_b} + \frac{F/B}{\gamma_f}} \right) \tag{5}$$

where:  $W_{mastic}$  = the weight of the mastics, g;  $r$  = the radius of the plate, cm;  $t$  = the film thickness of the mastics, cm;  $F/B$  = the mass ratio of the filler and binder, %;  $\gamma_b$  = the specific density of the binder, g/cm<sup>3</sup>;  $\gamma_f$  = the specific density of the filler, g/cm<sup>3</sup>.

**Table 2.** The weight of the mastics with different filler contents in one plate.

| Filler content            | 20%  | 50%  | 80%  | 100% | 120% | 150% | 180% |
|---------------------------|------|------|------|------|------|------|------|
| Weight of the mastics (g) | 31.9 | 36.4 | 41.3 | 43.5 | 44.3 | 46.9 | 88.7 |

##### 4.1.1. Effects of Position

The asphalt mastics with certain weight were oxidized in a medium oven at 135 °C. The high temperature was created by a resistive heater at the right side of the oven. Because of the limitation of the recirculation within the oven, it is assumed that the temperature or air condition at different positions in the oven may vary, so mastics aged from different positions in the oven were compared by the TTS test. It could be seen from Figure 2 that there were 3 layers of mastic plates in Z-axis, 2 layers in Y-axis and 3 layers in X-axis. Testing results of the complex modulus  $G^*$  at different loading frequencies at a temperature of 5 °C are exhibited in a log-log coordinate system, as Figure 3 shows. A linear regression model could be used to fit the relation between  $\log(G^*)$  and  $\log(\text{frequency})$ , and it could be found that the aged mastics had higher  $G^*$  than the original mastics at different loading frequencies.

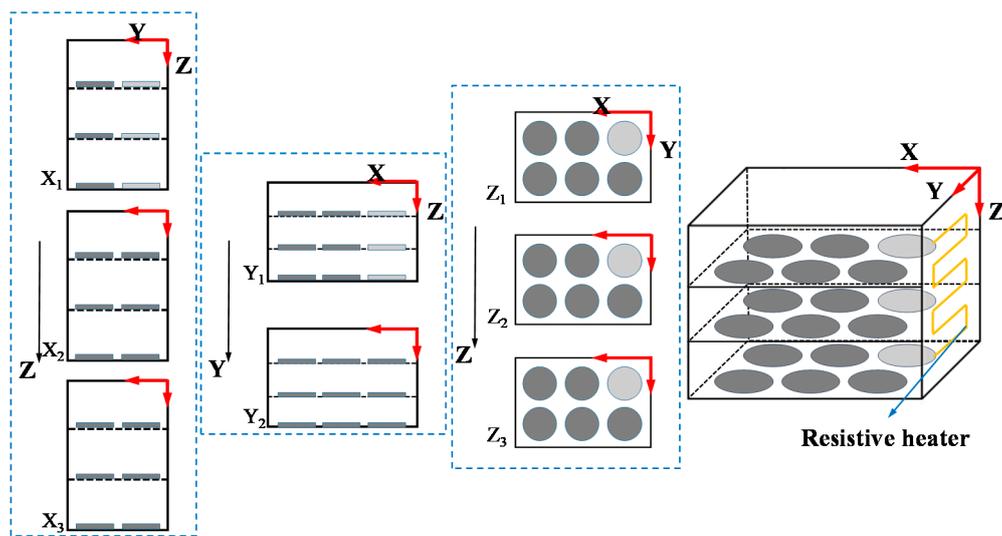


Figure 2. Schematic map of the positions of the mastic plates in the oven.

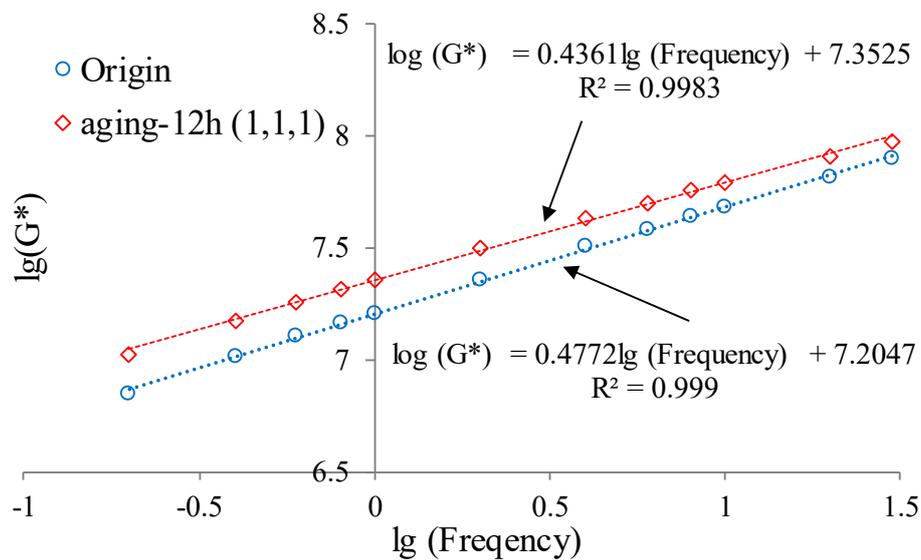


Figure 3. Complex modulus  $G^*$  versus loading frequency in a log-log coordinate system.

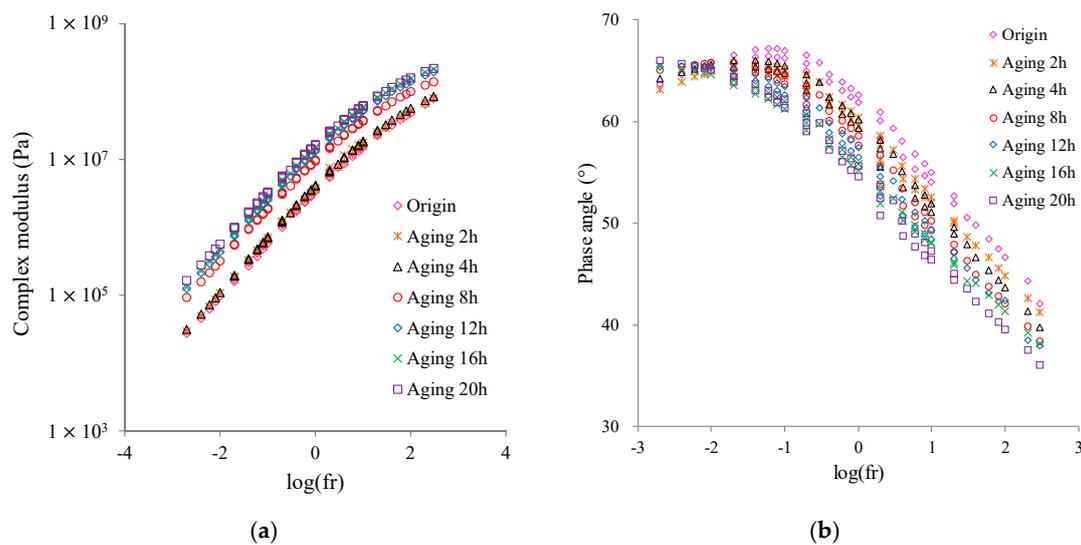
The parameter  $G^*$  of the mastics at temperature of  $5\text{ }^\circ\text{C}$  at a loading frequency of  $10\text{ Hz}$  from different positions are presented in Table 3. It could be found that position  $X_1$  has the highest  $G^*$  of  $6.21 \times 10^7\text{ Pa}$ , while position  $X_3$  has the lowest  $S$  of  $5.42 \times 10^7\text{ Pa}$ . Then, a one-way analysis of variance (ANOVA) was utilized to evaluate the sensitivity of different positions in the oven to the parameter  $G^*(5\text{ }^\circ\text{C} \& 10\text{ Hz})$ . The P-value was presented on the basis of an overall significant level fixed at 0.05. It is noted from the results that the effect of the position in X-axis on the parameter  $G^*(5\text{ }^\circ\text{C} \& 10\text{ Hz})$  is significant, which means that the oxidation level of the asphalt mastics in the oven is influenced significantly by the distance from the plate to the resistive heater in X-axis. Additionally, both the Y direction and Z direction have negligible effects on the aging of the mastics. Therefore, to minimize the influence of the position on the aging condition, only position  $X_1$  was used during the oven aging process for different mastics.

**Table 3.** Parameter  $G^*$  of the mastics at different positions and sensitivity analysis of different positions to  $G^*$ .

| Position ID  |         | $X_1$   | $X_2$ | $X_3$ | $Y_1$  | $Y_2$ | $Z_1$  | $Z_2$ | $Z_3$ |
|--|---------|---------|-------|-------|--------|-------|--------|-------|-------|
| $G^*(5\text{ }^\circ\text{C \& 10 Hz, } 10^7\text{ Pa})$ | Mean    | 6.21    | 5.86  | 5.42  | 5.97   | 5.85  | 5.79   | 5.72  | 5.74  |
|  | St.d    | 0.31    | 0.34  | 0.38  | 0.24   | 0.32  | 0.27   | 0.24  | 0.31  |
| St.d (multiple positions)                                |         | 0.062   |       |       | 0.031  |       | 0.026  |       |       |
| ANOVA  | P value | 0.0002* |       |       | 0.5288 |       | 0.6931 |       |       |

4.1.2. Effects of Time

Based on the film oven aging process, only  $X_1$  position was selected to remove the influence of oven position and the master-curves of asphalt mastics with a filler content of 80% after aging times from 0 h to 20 h were obtained to investigate the rheological properties of the mastics after different levels of oxidation. As Figure 4a shows, the complex modulus of the mastics increase when the aging time increases. Also, it could be found that the master-curves of the complex modulus from mastics after aging time from 0 to 4 h are similar, while there is a big gap between mastics with an aging time of 4 h and an aging time of 8 h, which indicates that 8 h of aging could change the rheological properties of the mastics significantly by visualization. As illustrated in Figure 4b, the phase angle of the mastics after different aging times also presents different patterns under different reduced frequencies. The phase angle may decrease when the reduced frequency larger than 0.1 Hz with the increasing aging time. However, when the reduced frequency is less than 0.1 Hz, the phased angle of the mastics after aging times from 0 to 4 h increase slowly with the reduced frequency, which may result from the increasing influence of the fillers when the binder goes into the flow state at a high temperature or low loading frequency. Table 4 exhibits the viscous-elastic parameters obtained from the CAM models of mastics after different aging times. Results show that when aging time increases, both the glass modulus ( $G_g$ ) and the modulus of equilibrium ( $G_e$ ) grow at a respectable pace, which indicates that the mastics become stiffer. The crossover frequency ( $f_c$ ), as well as the width of the relaxation spectrum R decrease, first sharply from 0 to 8 h aging time, then slowly with the increasing aging times. Lower crossover frequency ( $f_c$ ) comes from decreasing phase angles with the increasing aging times. Less parameter R means less gradual transition from the elastic behavior to the viscous behavior, which indicates more sensitivity to frequency changes.



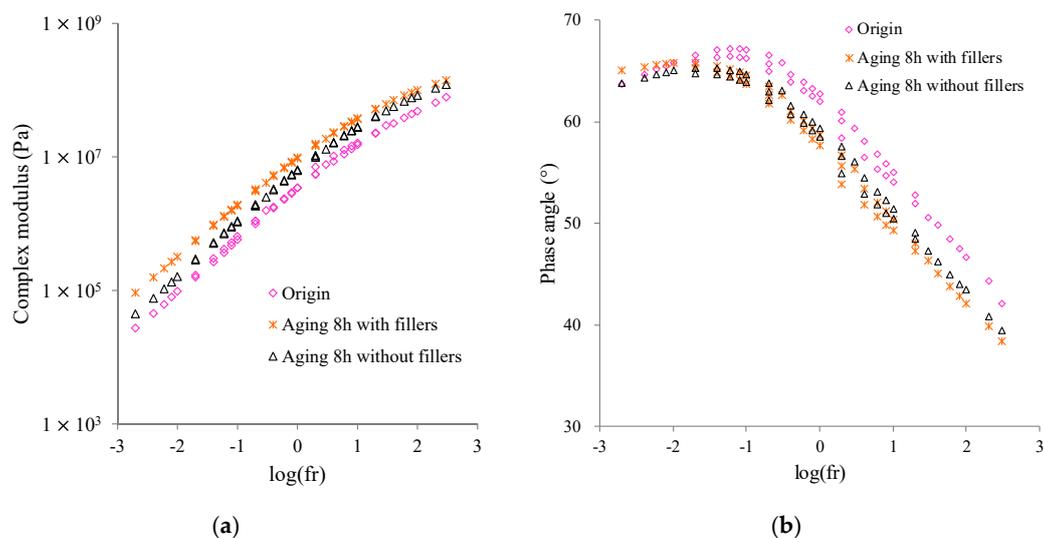
**Figure 4.** Master-curve of the mastics with filler content of 80% after different aging times: (a) complex modulus; (b) phase angle.

**Table 4.** CAM model fitting results of master-curves from mastics with different aging times.

| Parameters | Origin            | Aging 2 h         | Aging 4 h         | Aging 8 h         | Aging 12 h        | Aging 16 h        | Aging 20 h        |
|------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| $G_e$      | $3.9 \times 10^3$ | $4.3 \times 10^3$ | $5.5 \times 10^3$ | $6.5 \times 10^3$ | $9.2 \times 10^3$ | $9.5 \times 10^3$ | $9.8 \times 10^3$ |
| $G_g$      | $2.7 \times 10^8$ | $3.0 \times 10^8$ | $3.1 \times 10^8$ | $3.9 \times 10^8$ | $5.2 \times 10^8$ | $5.4 \times 10^8$ | $5.5 \times 10^8$ |
| $f_c$      | 55.50             | 56.58             | 50.28             | 31.01             | 30.88             | 29.86             | 28.55             |
| $k$        | 0.33              | 0.32              | 0.31              | 0.32              | 0.33              | 0.34              | 0.33              |
| $m_e$      | 0.95              | 0.93              | 0.91              | 0.85              | 0.85              | 0.85              | 0.84              |
| R          | 0.88              | 0.87              | 0.87              | 0.79              | 0.77              | 0.75              | 0.75              |

### 4.1.3. Effects of the Aging Process With or Without Fillers

There are two ways to fabricate the aged mastics. One is by aging the binder firstly, and then adding certain weight of fillers into the aged binder; the other way is to first add the fillers into the virgin binder, then aging the mastics to certain times. Figure 5 shows the different results of the master-curves obtained from two different aging processes after 8 h. From Figure 5a, it could be found that, compared with the original binder, when the mastics are aged with fillers inside, the complex modulus changes more than mastics aged without fillers inside. As can be seen in Figure 5b, mastics aged with fillers have a lower phase angle than aged without fillers. Therefore, it could be concluded that the fillers could accelerate the aging level of the mastics and aging mastics with fillers is a better way to produce the aged mastics, since it considers the influence of fillers.



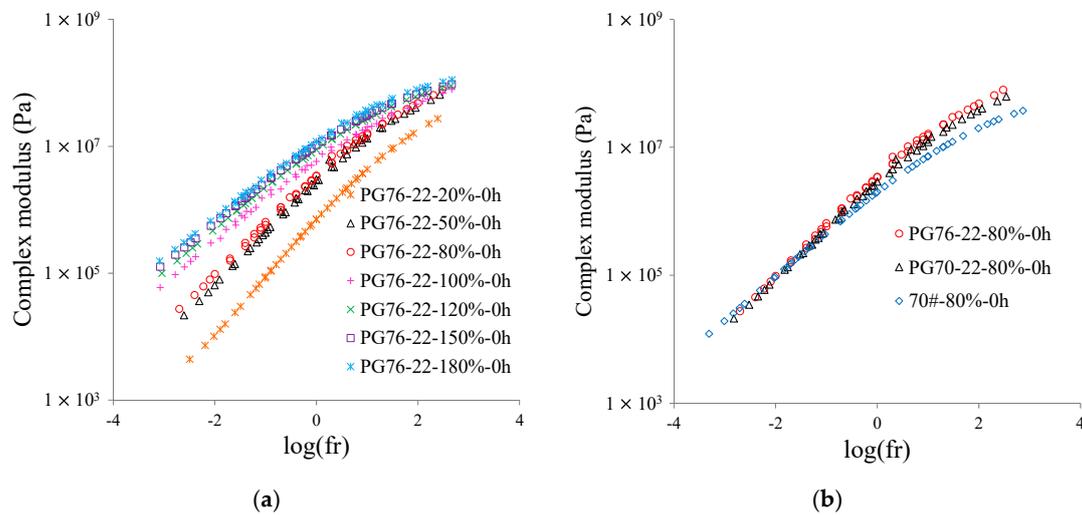
**Figure 5.** Master-curve of the mastics with filler content of 80% after aging 8 h with or without the fillers: (a) complex modulus; (b) phase angle.

## 4.2. Influence of Filler Contents and Binder Types

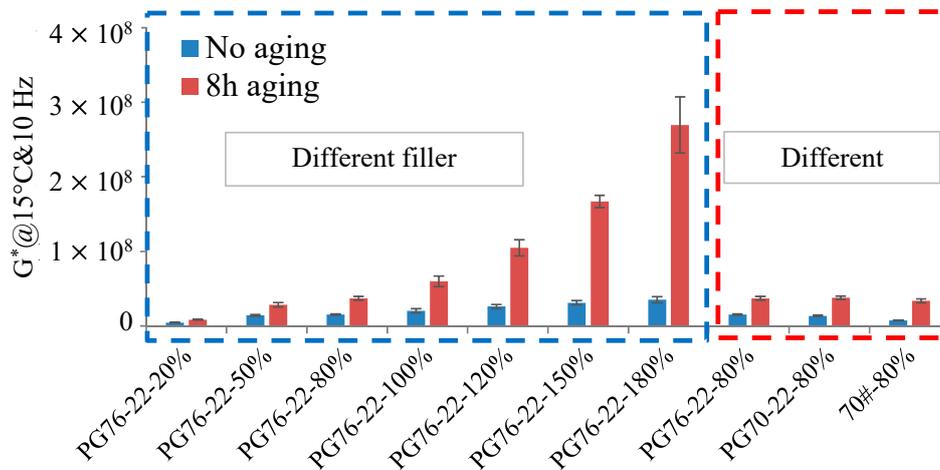
### 4.2.1. TTS Test Results

Mastics with different filler contents and binder types were prepared and the aging time was set to be 8 h. The TTS test was conducted on both virgin mastics and mastics after 8 h of aging. Figure 6 shows the master-curve of the complex modulus of different mastics. As Figure 6a illustrates, when the filler contents increase, the complex modulus of the mastics increase significantly. As Figure 6b shows, binder type could also affect the master-curve of the mastics. The complex modulus  $G^*$  at intermediate temperature of 15 °C and loading frequency of 10 Hz was proposed to evaluate the durability of the mastics. Figure 7 shows the  $G^* @ 15 \text{ }^\circ\text{C} \& 10 \text{ Hz}$  of different mastics before and after aging. It could

be observed that the  $G^*$  @ 15 °C & 10 Hz after 8 h of aging is much larger than no aging. The  $G^*$  @ 15 °C & 10 Hz increased with the increasing filler contents at both no-aging and 8 h aging conditions. The mastics with SBS modified binder had higher  $G^*$  @ 15 °C & 10 Hz than mastics with neat binder at both aging conditions.



**Figure 6.** Master-curve of the complex modulus of mastics with different compositions before aging: (a) different filler contents; (b) different binders.



**Figure 7.** Fatigue parameters ( $G^* \sin \delta$  @ 15 °C and 10 Hz) of different mastics with varying filler contents and binders before and after aging.

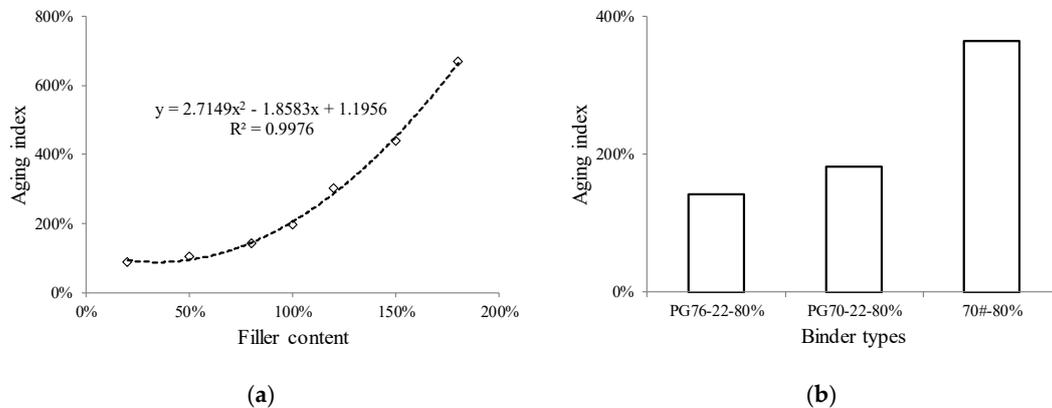
#### 4.2.2. Analysis of Aging Index

To exhibit the influence of aging on the mechanical properties of asphalt mastics, an aging index was proposed based on the changing ratio of the  $G^*$  at 15 °C and 10 Hz from un-aging to 8 h aging, which was in accordance with Equation (6).

$$Aging\ index = \frac{G_{8h}^* - G_0^*}{G_0^*} \tag{6}$$

As can be seen in Figure 8, both filler content and binder types could affect the aging index dramatically. In Figure 8a, the aging index increased with the increasing filler content in a quadratic function, and the  $R^2$  could reach 0.99. Since the oxidation of mastics is a diffusion mechanism [25,26], the effect of filler content on the aging index may come from the different binder film distributions

caused by different volume of fillers inside the binder. Additionally, as shown in Figure 8b, the aging index of the mastic with unmodified binder was larger than mastics with SBS modified binder, so the modification of the binder can also influence the aging properties of mastics.



**Figure 8.** The aging index of different mastics: (a) effect of filler contents on the PG76-22 mastics; (b) effect of binder types.

## 5. Conclusions

This study aimed to determine a simple way to age the binder-filler system within asphalt mixtures called mastics. The rheological properties and the rate of oxidation of the mastics with different combinations of binders and filler contents were then evaluated. Key findings were determined as follows:

- Based on the statistical analysis, the oven study shows that the oxidation level of the asphalt mastics in the oven is influenced significantly by the distance from the plate to the resistive heater in X-axis since the limitation of the circulation.
- The mastics could become stiffer, the phase angle and relaxation spectrum decreases with the increasing aging times, and it was observed that 8 h of aging could change the rheological properties of the mastics significantly by visualization.
- When compared to the aging process of mastics with and without fillers, it was found that the addition of fillers could accelerate the aging of mastics dramatically. The hypothesis is the addition of filler would shorten the binder film thickness and therefore speed up the aging process.
- From the analysis of aging index, it could be found that both the modification of binder and filler contents inside the mastics could influence the aging of mastics. The mastic produced by SBS modified asphalt present a lower aging rate when compared to the mastic from the neat binder. The aging index of the mastics increases with the filler content in a quadratic function, which may be attributed to the changing binder distribution within the mastics with a different volume of fillers.

This study could help to promote an understanding of the oxidative aging of the mastics, and a more precise evaluation and prediction of the performance of the mastics with different filler contents or modification after aging. But the influence of the interaction between the filler and binder on the aging properties of mastics needs to be further studied in the future by considering different mineral fillers and modification of binders.

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