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Article

# Investigation on the Performance of Fire and Smoke Suppressing Asphalt Materials for Tunnels

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**Abstract:** Variety of harmful gases are produced in asphalt mixture after mixing, paving and rolling process. Effective measures must be taken to suppress the asphalt pavement in the tunnel due to fire accidents and other toxic gases and fumes, reducing the human health during the construction process. In this study, a flame retardant and smoke suppressant (compound) with Mg(OH)<sub>2</sub> as the main component was developed, the flame retardant asphalt mixture and asphalt mastics were prepared to evaluate the flame retardant-smoke suppressant properties and performance effects. Firstly, its low and high temperature performances were investigated with the bending beam rheometer (BBR) and dynamic shear rheological (DSR), respectively. Then, the indoor combustion test and the cone calorimeter test were used to evaluate the fire retardant smoke suppression effect of the asphalt mastic. Thirdly, the flame retardant effect of asphalt mastic mixed with the compound was further analyzed by thermogravimetric (TG) test and scanning electron microscopy (SEM). The pyrolysis temperature, mass loss and microscopic state of asphalt surface were used to verify and explain the flame retardant reaction effect and process of the compound. Finally, the asphalt mixture performance was evaluated, as well as the flame retardant smoke suppression effect was verified by asphalt mixture combustion tests. The results showed that the flame retardant smoke suppression time of the flame retardant asphalt mixture was reduced by 66% and the smoke emission area was reduced by 20%. The flame retardant smoke suppression effect of the asphalt mixture was improved by 44%. The flame-retardant and smoke-suppressing compound and the asphalt mixture with the compound prepared in this study meet the asphalt mixture performance and flame retardant smoke suppression function, providing an option for application of fire retardant and smoke-suppressing asphalt pavement materials in tunnels.

**Keywords:** asphalt mastic; asphalt mixture; flame retardant - smoke suppressant; performance

## 1. Introduction

The tunnel is characterized as poor lighting, large longitudinal slopes, poor ventilation, and small space for activities, so the toxic fumes generated by asphalt mixture in paving will gather in large quantities, which seriously affects the health of construction workers and the quality of asphalt pavement paving<sup>1</sup>, and asphalt fumes mainly contain polycyclic aromatic hydrocarbons and a small amount of oxygen, nitrogen, and sulfur heterocyclic compounds, as well as more than 100 kinds of naphthalene, phenanthrene, phenol, carbazole, pyridine, pyrrole, indole and indene, and so on. Organic substances. Benzo(a)pyrene and other compounds are not only strong carcinogens, but also cause different degrees of pollution and damage to the natural environment such as surface water, groundwater, atmosphere and soil<sup>2</sup>. Asphalt pavement ignited by traffic accidents in tunnels is not only fiery, but also produces a large amount of asphalt smoke. In fires caused by asphalt combustion, 85% of the casualties die from asphyxiation due to inhalation of noxious gases rather than being burned to death<sup>34</sup>. Therefore, research and development of fire-retardant asphalt pavement materials for pavements is of great significance in terms of construction aspects and preventive functions. Liu et al<sup>5</sup> found that flame retardants reduced combustion and smoke, and the structural integrity of

flame retarded asphalt mixtures after combustion was superior to that of virgin asphalt mixtures<sup>6</sup>. Liu et al.<sup>7</sup> prepared flame retardant modified styrene-butadiene rubber (SBS) asphalt, and the results showed that the peak exothermic rate was reduced by 4.02 kw/m<sup>2</sup> with the addition of 12% of the flame retardant compared to that without the flame retardant. Qiu et al.<sup>8</sup> found that the Asphalt pavement temperatures above 330 °C promote the development of combustion. The experimental results of Yuan et al.<sup>9</sup> showed that the burning degree of fire-retardant asphalt mixtures was lower than that of virgin asphalt mixtures. Li et al.<sup>10</sup> demonstrated that the fire retardant effectively shortened the combustion time of the asphalt mixtures, and the surface temperatures and the stability of the residue after combustion were also reduced. Qin et al.<sup>11</sup> found that the split strength ratios were the same before and after combustion, while the freeze-thaw split strength ratios were reduced after combustion, which verified the fire retardant properties of fire retardant asphalt mixtures.

Inorganic phosphorus-based flame retardants are efficient and non-toxic but without well compatible with water molecule and prone to spontaneous combustion<sup>12</sup>. Organic halogen-based flame retardants emit toxic and corrosive gases during the flame retardant process and were proved not environmentally friendly, while nitrogen-based flame retardants have poor flame retardant effects<sup>13</sup>. The organosilicon and organophosphorus systems mostly perform as modifiers and plasticizers, but not used widely as flame retardant components in asphalt mixtures<sup>14</sup>. Magnesium hydroxide has become one of the most popular halogenated flame retardant alternatives due to its good stability, non-toxicity and smoke inhibition. There is a trend to develop low-dose flame retardants to balance the flame retardant properties with the pavement performance of asphalt mixtures<sup>15</sup>. Hu et al.<sup>16</sup> found that the alkaline mixture has good smoke suppression effect and does not affect the performance and construction process of asphalt pavement and is suitable for pavement materials. Qin et al.<sup>17</sup> and Li et al.<sup>18</sup> found that the incorporation of magnesium hydroxide flame retardant can improve the high temperature performance and reduce the low temperature performance of asphalt mixtures by conventional performance tests and rheological tests, but have little effect on water stability. Thus, they demonstrated that the use of flame retardant in asphalt mixtures is feasible. Cong et al.<sup>19</sup> studied the effect of adding of flame retardants on the physicochemical properties of asphalt and found that the addition of 6% flame retardants could reduce the heat released from asphalt in the temperature range of 195 °C to 600 °C. Tan et al.<sup>20</sup> used 1% kaolin and 8% traditional flame retardants to enhance flame retardant efficiency of traditional flame retardants, while weakening low temperature performance asphalt mixtures. Yu et al.<sup>21</sup> evaluated the stability, indirect tensile strength and rutting resistance and concluded that a certain hydroxide content had the most significant effect on improving the flame retardancy of asphalt. Bonati et al.<sup>22</sup> found that the use of magnesium hydroxide flame-retardant filler instead of conventional mineral powders had a significant flame-retardant effect on the heat-absorption decomposition and cooling of asphalt substrate at 200 °C and 300 °C. Zhu et al.<sup>23</sup> found that low doses of layered hydroxide can be well dispersed in asphalt, improving the resistance to thermal oxidation and densification of the coke layer and reducing the burning rate of asphalt. Sheng et al.<sup>24</sup> studied composite halogen-free flame retardant asphalt mixes with magnesium hydroxide and found that the flame retardant not only reduced the amount of heat and carbon monoxide released during burning, but also validated the application of such materials in tunnels. From the asphalt mixture performance, Xu et al.<sup>25</sup> found that using magnesium hydroxide flame retardant to replace part of the mineral powder improved the thermal stability of asphalt and reduced the burning level of asphalt from FH-3 to FH-1 (burning difficulty level) without significantly reducing the original road performance of the asphalt mixture. Xia et al.<sup>26</sup> found that the molecular weight of the characteristic substances of soot and residue of asphalt mixtures after combustion was smaller after doping with fire retardant, and the effect of fire retardant was significant.

According to the above investigation on the performance of flame retardant asphalt and asphalt mixture, the development and utilization of hydroxide flame retardants were better than other types of flame retardants in terms of preparation process, and environmental protection. However, in this study, not only the fire retardant asphalt mastic properties but also the mechanical properties of fire

retardant asphalt mixtures were investigated. The objective of this study is to evaluate the flame retardant smoke suppression effect, performance of asphalt mastic and asphalt mixtures using magnesium hydroxide-based composite flame retardants. Compared with the previous studies, the study on the performance of fire-retardant asphalt mixtures in addition.

Firstly, the conventional performance of virgin asphalt and asphalt mastic with flame retardant were tested. Secondly, the indoor combustion test, cone calorimeter test, thermogravimetric analysis, scanning electron microscopy (SEM) and low-high temperature performance of the flame retardant asphalt mastic were studied. Thirdly, for the optimal flame retardants content, the performances of asphalt mixture, including water stability, high-temperature stability, low-temperature performance and combustibility were evaluated. This research aims to provide technical support and application possibilities for the solution of fire retardant smoke suppression asphalt pavement materials in tunnels.

## 2. Raw Materials and Mix Proportioning

### 2.1. Asphalt

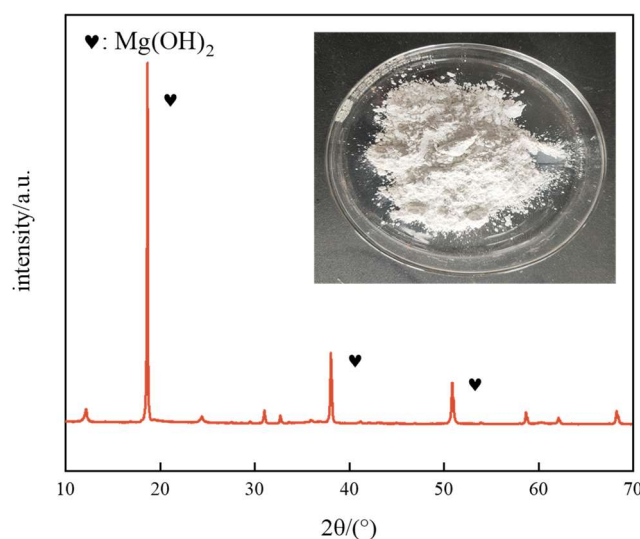
Panjin #90 virgin asphalt binder was used. Asphalt penetration test, ductility test and softening point test were carried out with reference to "Standard Test Methods for Asphalt and Double-layer Asphalt Mixture in Highway Engineering JTG E20-2011". The test methods referred to T0604-2011, T0605-2011 and T0606-2011, respectively. The technical indicators are shown in Table 1.

**Table 1.** Technical specifications of virgin asphalt.

Testing items	Specification requirements	Test results
Penetration (25 °C, 100 g, 5 s)/0.1 mm	80-100	89
Ductility (15 °C, 5 cm/min)/cm	≥100	≥150
Softening point / °C	≥44	45

### 2.2. Flame retardant-smoke suppressant

The flame retardant-smoke suppressant was obtained from the Road Engineering Laboratory of Dalian University of Technology. X-ray diffraction (XRD) and X-ray Fluorescence (XRF) results of the specimens are shown in Figure 1 and Table 2.



**Figure 1.** XRD diffraction pattern of flame retardant smoke suppressant.

**Table 2.** Quantitative and qualitative analysis by XRF.

Compositions	Percentage (%)
MgO	77.6
SiO <sub>2</sub>	14.8
CaO	4
Other	3.6

The specimen was dried and then sieved through a 80 micron square hole sieve 5 g of powder was taken. For different crystals, the XRD spectra have diffraction peaks of different intensities at different angles  $\theta$  or energies  $E$ . Moreover, the number, position, intensity and profile of the main diffraction peaks are the main information extracted and referenced when analyzing and comparing XRD spectra<sup>27</sup>, and the three intensity peaks of this XRD diffraction pattern are all from Mg(OH)<sub>2</sub> crystals. For XRF quantitative analysis shows that more than 77% of the component is the Mg.

### 2.3. Mineral powder

In this study, three types of mineral powders were selected. A is clayey mineral powder, B is limestone mineral powder, and C is granite mineral powder. Limestone is a carbonate rock with calcite as the main component and is not very stiff, kaolinite is mainly a product of natural alteration of feldspar and other silicate minerals, the main minerals in granite are quartz, potassium feldspar and acidic plagioclase. The samples after sieving are shown in Figure 2.

**Figure 2.** Three types of mineral powder samples: (a) Clayey, (b) Limestone, and (c) Granite.

### 2.4. Aggregate gradation

In this study, AC-16 with 4.93% asphalt content in Lvshun Midway was used. The mineral powder for three types was A, B and C. The coarse basalt aggregates were used. The grading of mineral powders and aggregates is shown in Table 3 and the synthetic grading curves are shown in Figure 3.

**Table 3.** AC16 asphalt mixture ratio of each material grade.

Material grade (mm)	Percentage (%)
10-20	22.6
5-10	10
0-5	62
Mineral powder	5.4

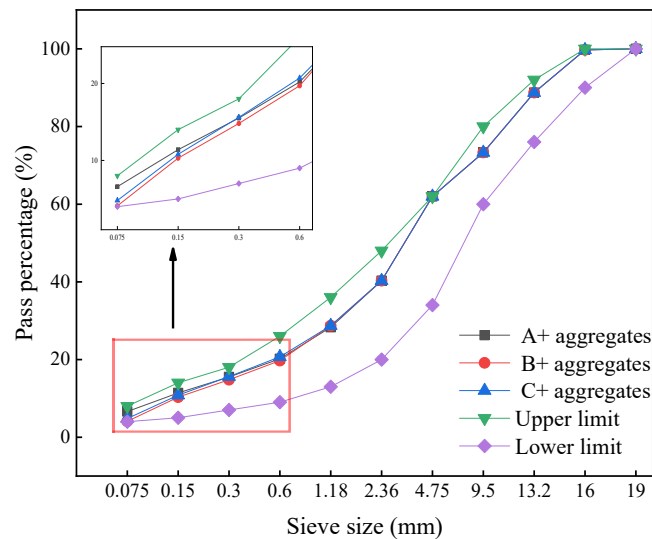


Figure 3. Aggregate gradation curves of AC-16 asphalt mixture.

### 3. Experimental methods

#### 3.1. Asphalt mastics

The filler was sieved to less than 0.075 mm and then mixed with asphalt to obtain asphalt mastic. Two filler/asphalt ratios of 1.0 and 1.1<sup>28</sup>, and five flame retardant contents of 6%, 7%, 8%, 10%, and 15% (the proportion of replacement mineral powder) were selected. To prepare specimens and carry out the following studies in accordance with the "Test Procedure for Asphalt and Asphalt Mixture for Highway Engineering JTG E20-2011".

The method was referred to Huang et al.<sup>29</sup> and Hu et al.<sup>30</sup> in the preparation of hydroxide flame retardant asphalt mastic. It was combined with the actual preparation method in this study as follows:

① The flame retardant and the mineral powder were dried in an oven at  $105 \pm 5$  °C for about 1 h each. After weighing the test dosage, the rest was kept sealed. The virgin asphalt<sup>31</sup> was dehydrated in an oven at  $150 \pm 5$  °C for 1 h. ② Flame retardants were added to the mineral powders at dosages of 6%, 7%, 8%, 10% and 15% by mass of the mineral powders. The dried flame retardant and mineral powder mixtures were mixed into virgin bitumen at  $160 \pm 5$  °C with reference to filler/bitumen ratios of 1.0 and 1.1. A test group was formed. ③ Start the high speed shear machine at 500 r/min agitation. After mixing, continuous shearing was carried out at 3000 r/min for 1 h. Then shear at 500 r/min for 5 mins to remove air bubbles, during which the asphalt temperature is controlled at  $160 \pm 5$  °C. ④ During the cooling process, the prepared flame retardant asphalt mastic was continued to be stirred manually with a stirring rod to prevent segregation during the cooling process. Then, the molds were poured to test the three main indicators of asphalt mastic.

#### 3.1.1. Asphalt mixture performance test methods

##### (1) Conventional performance tests

The conventional performance test referred to "Standard Test Methods for Asphalt and Asphalt Mixture in Highway Engineering JTG E20-2011". Test methods refer to T0604-2011, T0605-2011 and T0606-2011 respectively.

##### a. Penetration test

The mixed asphalt binder was poured into a small sample dish with inner diameter of 55 mm and depth of 35 mm. The sample dish containing the specimen was cooled at room temperature of  $15$  °C ~  $30$  °C for not less than 1.5 h, and then held in a constant temperature water bath at  $25$  °C  $\pm$  0.1

°C for not less than 1.5 h. SYD-2801F penetration degree tester and HWY-3 low temperature constant temperature water bath tester were used. Read the scale, and the numerical accuracy of 0.1 mm.

#### b. Ductility test

For each type of asphalt mastic, three specimens were prepared. Firstly, the mix asphalt mastic was slowly injected into the standard test mold, the height of the specimen should be higher than test mold. The specimen was cooled at room temperature for at least 1.5 h, and then it was scraped flat. Then put the test piece into the water tank at 25 °C for 1.5 h, the test piece was tested by SYD-4508G-1 asphalt ductility tester. The distance of specimen until broken was defined as the ductility value

#### c. Softening point (universal method) test

For each type of asphalt mastic, two specimens were prepared. The asphalt mastic was slowly injected into the standard mold, the height of specimen higher than the mold. The specimen was cooled at room temperature for 30 mins, and then scraped flat. The specimen was tested by SYD-2806F instrument. The specimens gradually soften until they touched the bottom surface, the average temperature (accurate to 0.5 °C) was regarded as the softening point indicator.

### (2) Low-temperature rheological properties

Low-temperature rheological properties test referred to the “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011” with T0627-2011.

The filler/asphalt ratio was determined to be 1.1 by the conventional tests described above. The amount of fire retardant and smoke inhibitor was determined by indoor combustion testing of fire retardant asphalt mastic. For each type of asphalt mastic, three specimens were prepared. The filler/asphalt ratio was 1.1. Two kinds of asphalt mastic were made with reference to the proportion of 0% and 7% fire retardant smoke suppressant, and then mix asphalt mastic was slowly injected into the standard mold, the height of specimen higher than the mold. The size of the test piece is 6.35 mm × 12.7 mm × 127 mm, the test temperature is -6 °C, -12 °C and -18 °C, and specimens need to be kept at the test temperature for 60 ± 5 min. The bending creep stiffness  $s$  and creep rate  $m$  of the asphalt mastic were tested using a bending beam rheometer (BBR). The contact load of the specimen was 35 ± 5 mN, and the test load was 980 ± 50 mN.

### (3) High-temperature rheological properties

High-temperature rheological properties test referred to the “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011” with T0628-2011.

Both the temperature sweep test and the frequency sweep test were performed using a dynamic shear rheometer (DSR). The filler/asphalt ratio was 1.1. Two types of asphalt mastics were prepared with 0% and 7% fire retardant and smoke suppressant ratios, respectively. And the standard specimens of asphalt mastic were poured on silicone sheet molds with diameters of 25 mm and 8 mm, respectively.

The temperature sweep test was used to evaluate the high temperature rheological properties of asphalt. The test was conducted over a temperature range of 46 °C to 82 °C, at a frequency of 10 rad/s, in a strain-controlled loading mode with a strain level of 12% and a rotor diameter of 25 mm with a pitch of 1 mm.

The frequency sweep test is used to characterize the rheological properties of asphalt materials. Test conditions: the test temperature range is 4~64 °C, with 12 °C as the interval, and the frequency range is 0.1~60 Hz. The loading mode was strain-controlled: at high temperatures (above and including 40 °C), using 25 mm rotors with a pitch of 1 mm, the strain level is 0.1%; at low temperatures (below 40 °C), using 8 mm rotors with a pitch of 2 mm, the strain level is 0.05%. The low frequency (high temperature) section was selected, and then dynamic shear modulus was fitted with the 2S2P1D model. Expression for the complex shear modulus ( $G^*$ ) is given as follows in Eq. (1):

$$G^* = G_e + \frac{G_g - G_e}{1 + \alpha(i\omega\tau_0)^{-k} + (i\omega\tau_0)^{-h} + (i\omega\beta\tau_0)^{-1}} \quad (1)$$

where

$G_e$  represents the equilibrium modulus,  $\omega$  converges to 0;  $G_g$  represents the glassy modulus,  $\omega$  converges to  $\infty$ ;

$\alpha$ ,  $k$ , and  $h$  are three constants,  $0 < k < h < 1$ ;  $\beta$  is a constant;  $\tau_0$  represents the characteristic time.

### 3.1.2. Evaluation of flame retardant and smoke suppressing effect

#### (1) Indoor combustion test

In order to determine the dosage of flame retardant and smoke suppressant, an indoor combustion test was used. The fire-retardant and smoke-suppressing effects of virgin asphalt, virgin asphalt mastic and fire-retardant asphalt mastic with different dosages of fire-retardant and smoke-suppressing agents were investigated separately. The mixed flame retardant asphalt mastic was poured into a small sample tray with an inner diameter of 55 mm and a depth of 35 mm at a height of 2/3. Flame retardant asphalt mastic samples were made at 6%, 7%, 8%, 10%, and 15% mineral powder replacement ratios at a filler/asphalt of 1.1.

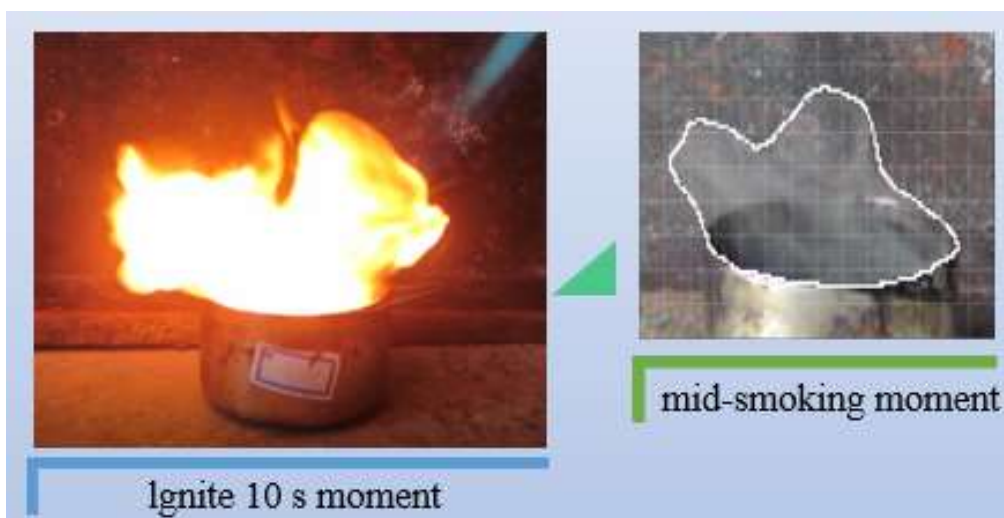


Figure 4. Indoor combustion test and image processing.

The indoor combustion was ignited by gas burner<sup>32</sup>, the ignition time of each group of asphalt mastic was 10 s, the mass of asphalt mastic was 25 g, and the combustion exposure area was consistent. In image processing, set the time point 10 seconds after ignition to 0 time point. Set the time interval to 1 or 2 seconds until no more smoke is produced. The image processing software unified the image specification as 3.49×2.25 pixels/inch, and set the grid division as 10 mm×10 mm, so that the smoke area of a certain moment can be obtained through software processing. Through the image equipment, the whole burning process of flame retardant asphalt mastic was captured.

#### (2) Cone calorimeter test

The smoke production rate of asphalt mastic containing 7% and 0% flame retardant smoke suppressant was further tested by cone calorimeter. Two separate asphalt mastic specimens were prepared according to the filler/asphalt ratio of 1.1. The size was 100 mm × 100 mm × 5 mm, and the specimens were placed in the center of the conical calorimeter specimen table, and the conical heater was lowered onto the specimens. The parameters set by the console were generally 35 kW/m<sup>2</sup> heat flow, corresponding to a temperature range of 300 °C to 500 °C. The most important parameter index was tested, namely: smoke production rate (SPR).

#### (3) Thermogravimetric analysis

Thermogravimetric (TG) tests were conducted for two doses of asphalt mastic with the determined amount of 7% flame retardant smoke suppressant and 0% flame retardant smoke suppressant. TG data can be analyzed for heat-absorption temperature characteristics of flame

retardants in fire-retardant asphalt mastics, residuals, and mass loss rates during the combustion phase. TG test temperature was 30 °C-800 °C, and the heating rate was 10 °C/min. 1 g of each of the two doping asphalt mastic was taken as a specimen

#### (4) Microscopic analysis of flame retardant asphalt mastic.

SEM was used to observe the asphalt mastic mixed with two fire retardant and smoke suppressant compounds from a microscopic point of view. Totally four samples were divided into two groups: one group of surfaces treated by flame (about 300 °C) combustion, and the other group of samples tested by TG (max. temperature 800 °C). At different temperatures, changes in the state of the flame retardant smoke suppressant compound particles encapsulated on the surface of the asphalt mastic were observed, verifying the flame retardant smoke suppressant effect of the flame retardant asphalt mastic.

### 3.2. Asphalt mixture

The asphalt mixes in this study were prepared as follows: ① Placed the virgin asphalt into an electric oven and kept it at  $140 \pm 5$  °C for 1.5 h. And mineral powder and flame retardant smoke suppressant compound were kept dry at 105 °C. ② Used the ratio of aggregate, put the prepared aggregate into the asphalt aggregate mixer and mixed it well, maintained at  $150 \pm 5$  °C for 1 h. ③ Asphalt content was 4.93%, took a certain amount of virgin asphalt, poured it into the mixer and mixed it fully for 90 s. ④ Then the mineral powder and the corresponding proportion of the flame retardant and smoke suppressant compound were poured into the mixing pot and mixed thoroughly for 90 s. ⑤ Specimens were made under mixing temperature conditions.

#### 3.2.1. Asphalt mixture performance test

This section for flame retardant asphalt mixture molding, testing and other requirements refer to the "Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011" regulations.

##### (1) Marshall stability and flow value

For mixing temperature was  $150 \pm 5$  °C, asphalt content was 4.93%, filler/asphalt was 1.1, mineral powder and aggregate were mixed well. Each mineral powder was 1 group, each group was 4 specimens, a total of three groups were prepared. Each specimen was taken about 1250 g into the preheated mold and sleeve, and weighed to ensure the height of the molded Marshall specimen meets the requirement of  $63.5 \pm 1.3$  mm. Automatic Marshall testing machine was used in the test. The test referred to the "Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011" with T0709-2011.

##### (2) Water stability

Water stability test referred to the "Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011" with T0729-2000. Asphalt mixture freeze-thaw splitting test was used to evaluate the water stability of asphalt mixture. Marshall compaction method was used to form the 8 standard duplicated specimens: the number of compactions for each side of 50 times, asphalt content was 4.93%, filler/asphalt was 1.1, and flame retardant alternative mineral powder content was 7%. The specimens were randomly divided into control group and test group, each group were 4 duplicated specimens. The test group was completely immersed in water filled with water under vacuum conditions. The vacuum was maintained at 97.3-98.7 kPa for 15 min, and the specimens were immersed in water for 0.5 h after pressure was restored. The specimens were tested into a plastic bag, about 10 mL of water was added, the bag was tightened. The packaged specimens were then placed in a refrigerator at a freezing temperature of  $-18 \pm 2$  °C for  $16 \pm 1$  hours. And then specimens were placed in a thermostatic water bath at  $60 \pm 0.5$  °C for 24 h. The two groups of specimens were immersed in a constant temperature bath of  $25 \pm 0.5$  °C over 2 h. The splitting test was carried out by UTM-100. Water stability was calculated by the following Eq. (2):

$$TSR = \frac{\bar{R}_{T2}}{\bar{R}_{T1}} \times 100 \quad (2)$$

where,  $TSR$  represents the ratio of freeze-thaw splitting strength at T1 and T2(%);

$\bar{R}_{T2}$  represents average value of splitting tensile strength (MPa) of the second group of valid specimens after freeze-thaw cycles;

$\bar{R}_{T1}$  represents average value of splitting tensile strength (MPa) of the first group of valid specimens without freeze-thaw cycles.

### (3) High-temperature stability

High temperature stability of this asphalt mixture was tested by asphalt mixture rutting test. The method was referred to the "Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011" with T0719-2011.

The protocol of asphalt mixture with rutting test was used to evaluate the high temperature stability of asphalt mixture. In this study, the size of the rutted plate is 300 mm×300 mm×50 mm, and the compaction density of the mixture should meet the requirements of the Marshall standard specimen. Then the specimens were placed in a constant temperature box at  $60 \pm 5$  °C for 3 h. The contact pressure between the test wheel and the specimen was adjusted to  $0.7 \pm 0.05$  MPa, then starting the automatic rutting tester. The rutting deformation  $d_1$  and  $d_2$  at 45 min ( $t_1$ ) and 60 min ( $t_2$ ) were measured after heat preservation as required (accurate to 0.01 mm). Dynamic stability was calculated by the following Eq. (3):

$$DS = \frac{(t_1 - t_2) \times N}{d_1 - d_2} \times C_1 \times C_2 \quad (3)$$

where,  $DS$  represents dynamic stability (times/mm);  $d_1, d_2$  represent deformation (mm) corresponding to time  $t_1, t_2$ , respectively;  $C_1$  and  $C_2$  equal to 1.0;  $N$  is 42 (times/mm).

### (4) Low-temperature stretchability

Low temperature stability of this asphalt mixture was tested by asphalt mixture bending test. The method was referred to the "Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering JTG E20-2011" with T0715-2011. Asphalt mixture low-temperature bending beam test was used to evaluate the mechanical properties of hot mix asphalt mixture bending damage at the specified temperature and loading rate. The experimental conditions were as follows: the test temperature was  $-10 \pm 0.5$  °C, the loading speed was 50 mm/min, and the specimen dimensions were  $250 \pm 2.0$  mm in length,  $30 \pm 2.0$  mm in width, and  $35 \pm 2.0$  mm in height. After the specimens were made, specimens were kept at a constant temperature of  $-10 \pm 0.5$  °C for 45 mins, and then placed on the test platform for testing. The low-temperature cracking resistance of asphalt mixtures was comprehensively evaluated by the basis of fracture strength, fracture strain, and fracture stiffness modulus. Formed rutting specimens were cut according to the above dimensions, and the number of duplicated specimens is 4. After meeting the test conditions, the test was completed on UTM-100 until the specimens were broken. The modulus of bending strength was calculated by the following Eq. (4):

$$S_B = \frac{R_B}{\varepsilon_B} \quad (4)$$

where,  $S_B$  represents modulus of bending strength (MPa);  $R_B$  represents bending and tensile strength (MPa);  $\varepsilon_B$  represents maximum bending tensile strain ( $\mu\varepsilon$ ).

### 3.2.2. Evaluation tests for flame retardant and smoke suppressing effect

Two Marshall specimens containing different amounts of flame retardant and smoke suppressant compounds were made separately for combustion tests. 30 g of gasoline were poured into each specimen. Mass loss was recorded at 30 s, 60 s, 90 s, 120 s and 150 s until the end of combustion. The flame retardant and smoke suppressant effect of the flame retardant and smoke

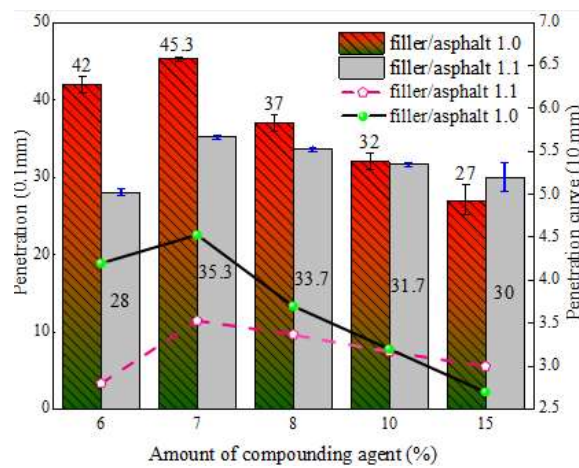
suppressant compound into the asphalt mixture was verified according to the mass loss at different moments.

## 4. Results and discussion

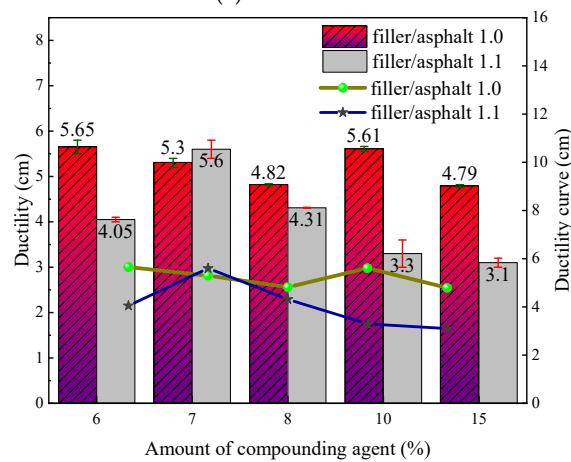
### 4.1. Asphalt mastic

#### 4.1.1. Conventional performance

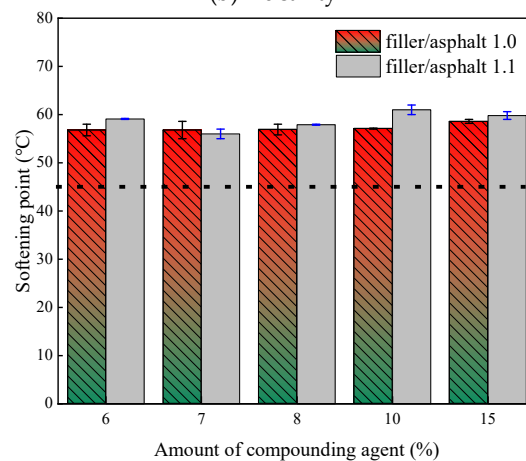
The asphalt mastic with different amounts of flame retardant and smoke suppressant compound was tested for conventional performance, including penetration ductility and softening point. In this way, the basic properties of this material were studied, and the results are shown in Figure 5.



(a) Penetration



(b) Ductility



(c) Softening point

**Figure 5.** Conventional performance of flame retardant asphalt mastic with different filler/asphalt: (a) Penetration, (b) Ductility, and (c) Softening point.

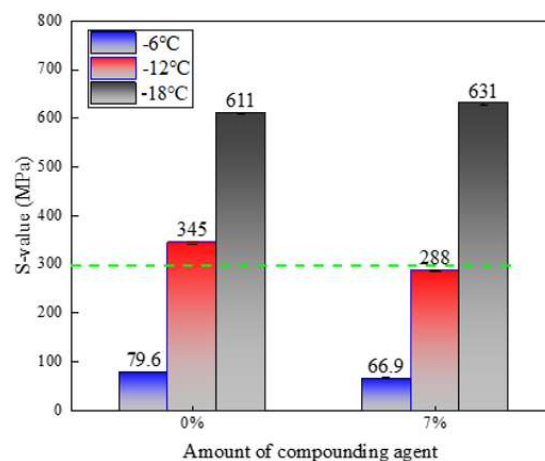
In Figure 5(a), the penetration of these two asphalt mixtures with different fillers/asphalt showed an increasing and then decreasing trend. The maximum flame retardant was found at 7% with different filler/asphalt. The standard deviations of penetration for the two different filler/asphalt ratios were 7.3 and 2.9, respectively. The penetration dispersion degree of asphalt mastic with filler/asphalt ratio of 1.1 was reduced by 55% compared with that of filler/asphalt of 1.0 under different dosing of flame retardant; Figure 5(b), the maximum ductility for both filler/asphalt ratios was about 5.6 cm. However, when filler/asphalt ratio was less than 1.1, the ductility showed the law of increasing and then decreasing with the increase of flame retardant, and the change was not obvious at the filler/asphalt ratio of 1.0; From Figure 5(c), the softening points for different filler/asphalt ratios were 24% higher than the standard values for virgin asphalt. However, the lowest softening point was found at 7% flame retardant addition. At a filler/asphalt ratio of 1.1, the softening point showed a trend of decreasing and then increasing with the increase of flame retardant dosage. However, this trend was not obvious in the case of filler/asphalt ratio of 1.0.

It should be noted that the asphalt mastic formed by adding mineral powder and fire retardant to asphalt was used in this test. Accordingly, the overall penetration compared with 90# virgin asphalt, the value decreased to more than 50%. The reason for this is that the virgin asphalt hardens with the addition of mineral powder. For both filler/asphalt ratios, the difference in penetration was the largest at 6% flame retardant dosage, with a difference of about 33%. It was also found that the difference in penetration at these two filler/asphalt ratios gradually decreased with the increase in the content of flame retardant and smoke suppressant. It also showed that mineral dust hardens virgin asphalt; By the same principle, the plasticity of the virgin asphalt after the addition of mineral powder was reduced in the ductility test. The maximum value of ductility was about 5.6 cm. Unlike the above, the softening point had little effect and to some extent was slightly increased.

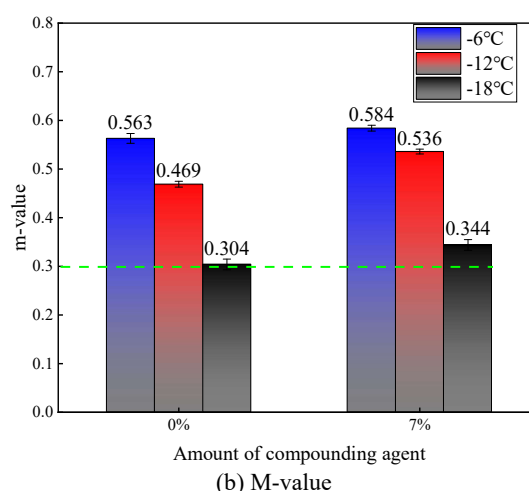
In the case of filler/asphalt ratio of 1.1, the general trend of permeability, ductility and softening point: the permeability and ductility increased and then decreased with the increase of flame retardant dosage, and the softening point decreased and then increased with the increase of flame retardant dosage. The inflection point in the above analysis occurs at 7% flame retardant dosage. Therefore, the dosage of flame retardant smoke suppressant was determined to be 7%.

#### 4.1.2. Low-temperature rheological properties

The low-temperature rheological properties were tested by BBR to simulate the mechanical response of asphalt under pavement temperature stress. Creep stiffness modulus  $s$  and creep rate  $m$  were derived for evaluating the deformation resistance and low-temperature relaxation properties of asphalt materials, respectively. The creep stiffness modulus  $s$  and creep rate  $m$  at 60 s were chosen for the tests at  $-6\text{ }^{\circ}\text{C}$ ,  $-12\text{ }^{\circ}\text{C}$  and  $-18\text{ }^{\circ}\text{C}$ . The results are shown in Figure 6.



(a) Creep stiffness modulus



**Figure 6.** Different flame retardant and smoke suppressing compound dose of asphalt mastic BBR test: (a) creep stiffness modulus, (b) m-value.

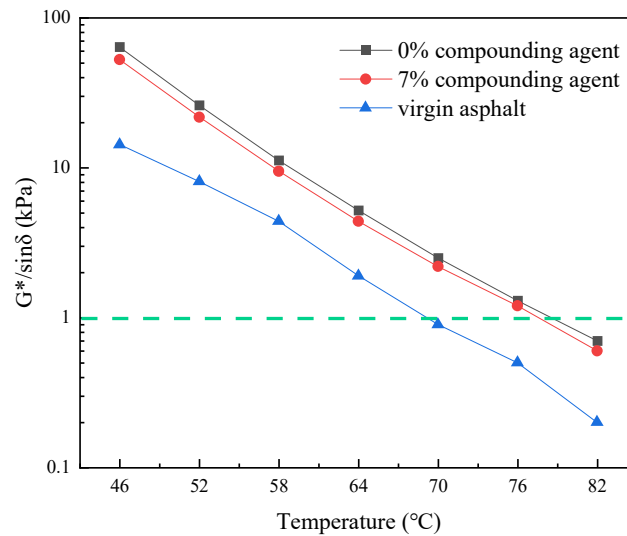
Cracks occurred in asphalt pavements in low temperature environments due to the inadequate low temperature crack resistance of asphalt<sup>33</sup>. The low-temperature rheological properties of asphalt mastic with two kinds of flame retardant and smoke suppressant compounds were studied, and the creep stiffness  $s$  and creep rate  $m$  values are shown in Figure 6. Creep stiffness  $s$  can evaluate the temperature shrinkage of asphalt materials, the greater the creep stiffness indicates that the higher the temperature stress, resulting in a greater likelihood of cracking; And  $m$  value can characterize the rate of change of the creep stiffness of asphalt materials under low-temperature conditions, the greater the value of  $m$  indicates that the material has a higher ability to relax stresses<sup>34</sup>. According to the Superior Performing Asphalt Pavement (abbreviated as “Superpave”), in order to ensure the low temperature performance of asphalt, it is necessary to ensure that  $s \leq 300$  MPa and  $m \geq 0.30$ . Firstly, in Figure 6(a), stiffness modulus of asphalt mastic with two different blends showed an increasing trend with decrease of temperature. However, 7% content of flame retardant smoke suppression compound asphalt mastic at  $-6$  °C and  $-12$  °C did not reach the upper limit of 300 MPa, it’s low temperature performance was significantly better than 0% content of asphalt mastic. From the creep rate index analysis in Figure 6(b), the creep rates of the two different blends of asphalt mastic showed a decrease with temperature. Further analysis revealed that the  $m$ -value of the asphalt mastic with 7% fire retardant and smoke suppressant compounds complied with the lower limit at  $-6$  °C,  $-12$  °C, and  $-18$  °C, while the  $m$ -value at  $-12$  °C exceeded the lower limit by 30%. It fully illustrates that the asphalt mastic containing 7% flame retardant inhibitor compound has superior low temperature rheology. Fire retardant and smoke suppressant can not only achieve the effect of fire retardant and smoke suppressant, but also improve the low temperature performance of asphalt mastic.

The  $S$  value at the three temperatures did not differ by more than 20% for two different flame retardant and smoke suppressant dosages (0%, 7%). 7% fire retardant smoke suppressant dosage of asphalt mastic, its low temperature performance was better than other dosage of asphalt mastic. However, at  $-18$ °C, the  $S$  value (611,631) did not differ much, which to some extent reflects that the low-temperature performance of asphalt mastic had little relationship with the dosage of fire retardant and smoke suppressant.

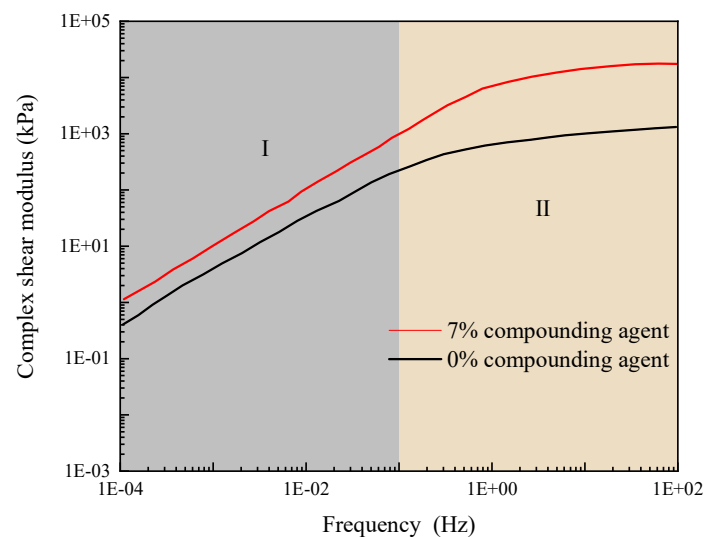
#### 4.1.3. High-temperature rheological properties

Temperature sweep tests were used to evaluate the high-temperature rheological properties of asphalt, whereas in this study,  $G^*/\sin\delta$  was used to characterize the high temperature resistance to permanent deformation of asphalt mastic. The results are shown in Figure 7. The frequency sweep test was also used to characterize the rheological properties of the asphalt material. Using the time-temperature superposition principle, the viscoelastic parameters at different temperatures were transformed into the viscoelastic parameters at the base temperature of 28 °C Thus, the master

curves of the asphalt material were obtained, and the low-frequency (high temperature) part was chosen to be analyzed in this study. The results are shown in Figure 8.



**Figure 7.**  $G^*/\sin\delta$  for different asphalt mastics.



**Figure 8.** Master curves of low-frequency complex shear modulus for different types of asphalt.

In Figure 7, the rutting factor ( $G^*/\sin\delta$ ) in the Superpave system was used to evaluate the high temperature resistance of asphalt to permanent deformation. It represents the modulus of energy dissipation during creep. At the same temperature, the value of  $G^*/\sin\delta$  becomes larger, it indicated that energy dissipation value becomes smaller, flow deformation becomes smaller, and rutting resistance becomes stronger, which proves that high-temperature performance becomes better<sup>35</sup>. Firstly,  $G^*/\sin\delta$  shows a good linear correlation with temperature in semi-log-temperature coordinates. The rutting factor of the asphalt mastic was greatly improved by the addition of mineral powder, which enhanced the rutting resistance of the asphalt mastic. However, the rutting factor was reduced with the addition of 7% flame retardant smoke suppressant. Between 70 and 82°C, the rutting resistance of the 7% fire-retardant and smoke-retardant asphalt mastics was close to that of the 0% fire-retardant and smoke-retardant asphalt mastics. The critical temperature was the lowest temperature corresponding to  $G^*/\sin\delta \geq 1.0$  kPa. In terms of critical temperature, the critical temperature of asphalt mastic containing 7% flame retardant and smoke suppressant was 77 °C. The difference between the critical temperatures of asphalt mastic with 7% fire retardant content and asphalt mastic with 0% fire retardant was 2 °C, which was not significant.

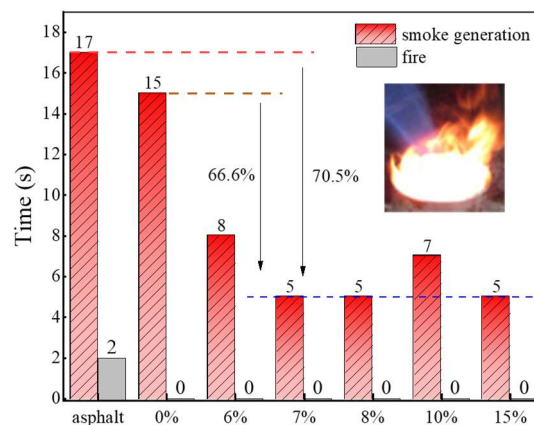
From Figure 7, the high-temperature critical temperature of asphalt mastic with 7% flame retardant and smoke suppressant compound was 77 °C, which exceeded the high temperature critical temperature of the original asphalt by 10%. The increase in critical temperature compared to virgin asphalt somewhat showed that the fire retardant smoke suppressant improved the high temperature performance of the asphalt mastic.

In Figure 8, in the same frequency range, in phase I, although the two asphalt mastics were approximately the same magnitude of change in complex shear modulus, in a certain sense with lower temperature sensitivity. However, in the low frequency (I, II) zone, the asphalt mastic mixed with 7% flame retardant smoke suppressant compound has the largest complex shear modulus, so it has better high temperature performance. The same conclusions from the high temperature rheological performance and low temperature rheological performance study were obtained.

#### 4.2. Analysis of flame retardant and smoke suppression effect of asphalt mastic

##### 4.2.1. Indoor combustion and cone calorimetry analysis

Through the smoke generation time and smoke area, the flame retardant smoke suppression performance of asphalt mastic was evaluated, the results are shown in Figures 9 and 10. The dosage of flame retardant and smoke suppressant with significant effect was determined based on indoor combustion test. And then conical calorimeter was used for precise analysis, the results are shown in Figure 11.



Virgin asphalt and asphalt mastic with different compound dosing

Figure 9. Indoor combustion effect.

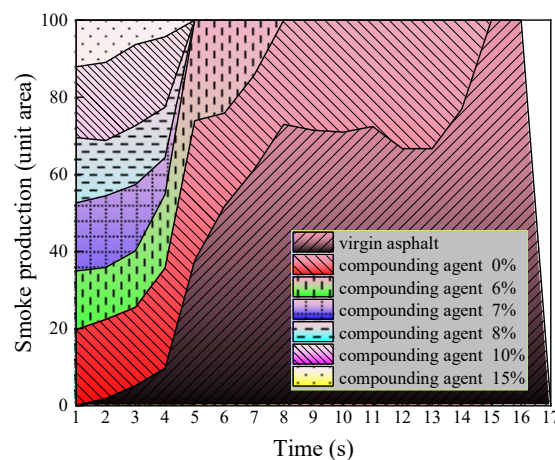
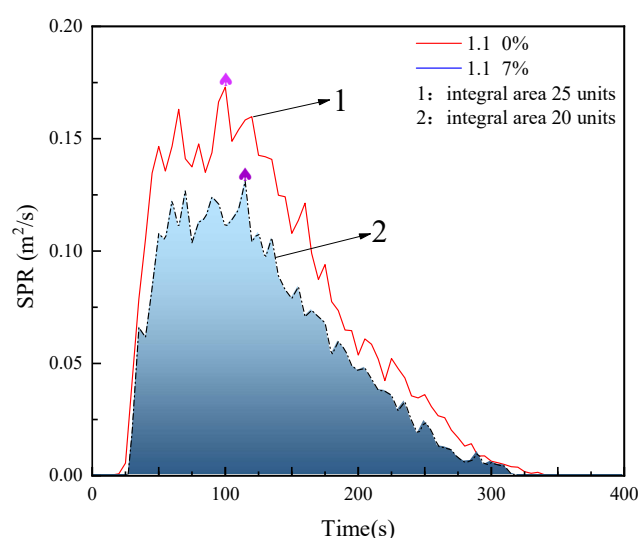


Figure 10. Smoke-generating area of asphalt mastic with different amounts of compounding agents.

In Figures 9 and 10, the fire retardant effect of the asphalt mastic mixed with the compound can be observed after 10 s from ignition. The virgin asphalt had about 2 s of open flame burning, which

was not seen in other test groups. By comparing the burning area at intermediate moments, it was found that the burning time tended to shorten as the amount of flame retardant and smoke suppressant increased. The smoke duration of the flame retardant and smoke suppressant (6%, 7%, 8%, 10%, 15%) was less than 10 s, which showed the obvious effect of flame retardant and smoke suppressant. Notably in Figure 9, the amount of smoke doped with 10% fire retardant smoke suppressant is higher than the neighboring group, which is considered to be generated by the stability of the asphalt mastic in the test. Overall, it does not affect the trend of the effect of flame retardant smoke suppression with flame retardant doping. The presence or absence of flames from the virgin asphalt and other controls side-steps the fact that the mineral powders also produced some fire retardant effect. In terms of smoke generation time, the asphalt mastic with 7% fire retardant and smoke suppressant shortened the smoke generation time by 70.5% and 66.6% compared with virgin asphalt and asphalt mastic with 0% fire retardant and smoke suppressant, respectively. The flame retardant and smoke suppressant effect of the flame retardant and smoke suppressant was fully demonstrated, and 7% was the optimal dosage.

Through comprehensive analysis, the dosage of flame retardant and smoke suppressant was confirmed to be 7%. In particular, the determination of the mineral powder category used in this subsection was determined in the Marshall flow value stability test.



**Figure 11.** Conical calorimetric-smoke generation rate diagram.

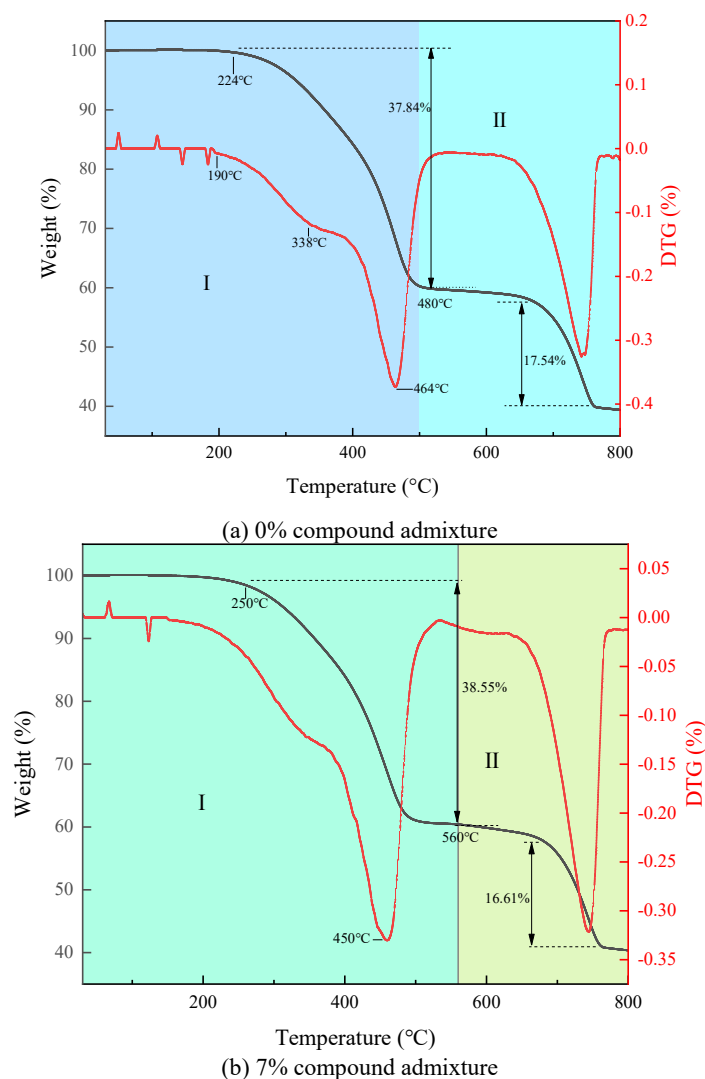
The specimens of 0% and 7% of two flame retardant and smoke suppressant compound doping were tested by the conical calorimeter, as shown in Figure 11. The smoke rate of the test showed that the smoke moment of the asphalt mastic without flame retardant and smoke suppressant was at least 20 s earlier than that of the asphalt mastic with 7% flame retardant and smoke suppressant, and the extinguishing time was at least 25 s later.

The difference in peak smoke emission between the sample without FRS and the sample with 7% FRS was more than 40%. It showed that the flame retardant smoke suppression effect of compounding agent with 7% was obvious; From the point of view of smoke integral area: after mathematical integration, the integral area of the unadulterated composite sample was 25 units; the integral area of the 7% flame retardant and smoke suppressant addition was 20 units. The amount of smoke was reduced by 20%. In contrast, asphalt mastic with 7% flame retardant and smoke suppressant achieved a significant flame retardant and smoke suppressant effect.

#### 4.2.2. Thermogravimetric analysis of flame retardant asphalt mastic

The TG tests were conducted at high temperatures for asphalt mastics. The amounts of flame retardant and smoke suppressant were 0% and 7%, respectively. The pyrolysis temperature and mass changes of asphalt mastic were obtained under different temperature change conditions. Tests were

conducted to characterize the flame retardant effect of fire retardant smoke suppressants in asphalt. The results are shown in Figure 12.



**Figure 12.** TG analysis graph: (a) 0% compound admixture, (b) 7% compound admixture.

In the TG analysis, it was found that the fire-retardant asphalt mastic was basically divided into two pyrolysis segments, which developed in a roughly cyclical manner (part I and II), with part I being the most dominant pyrolysis stage. Meanwhile, 7% flame retardant compared to 0% flame retardant smoke suppressant asphalt mastic: the thermal decomposition temperature increased by 25 °C at the beginning of the first stage and 80 °C at the end of the first stage, and the mass loss in the first stage was about 38%. It can be seen that the flame retardant smoke suppressant has a significant flame retardant effect in asphalt mastic. From the conductivity thermogravimetric analysis (DTG) curve, the asphalt mastic mixed with 7% compound started to absorb heat after 250 °C. The flame retardant and smoke suppressant delays the volatilization of the burning material of the asphalt mastic, and the magnesium oxide produced by pyrolysis promotes the rapid dehydration and carbonization of the polymer surface<sup>36</sup>, forming a carbonized layer, which achieves the flame retardant effect. The maximum flame retardant effect was achieved at about 450 °C. Comparing the residual amount of fire-retardant asphalt mastic at the two dosages, the residual amount of fire-retardant asphalt mastic at a dosage of 7% remained above 40% in the TG analysis.

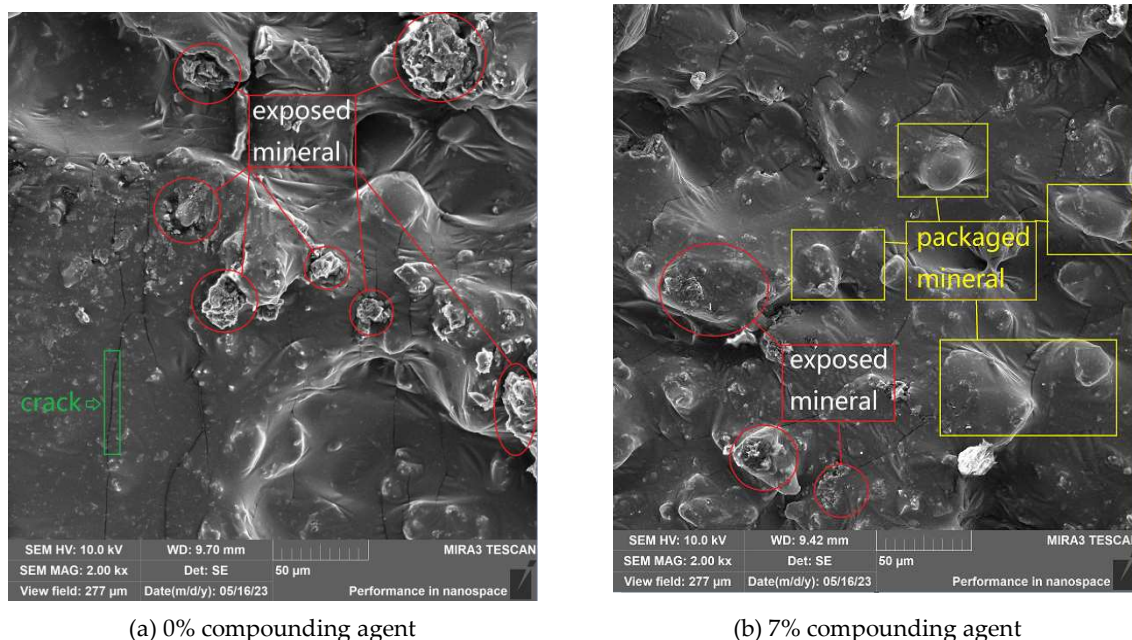
#### 4.2.3. Microscopic analysis of asphalt mastic

The specimens were taken for flame burning (about 300 °C) and TG test (about 800 °C). The surfaces observed by SEM was to study the state of asphalt<sup>37</sup>.

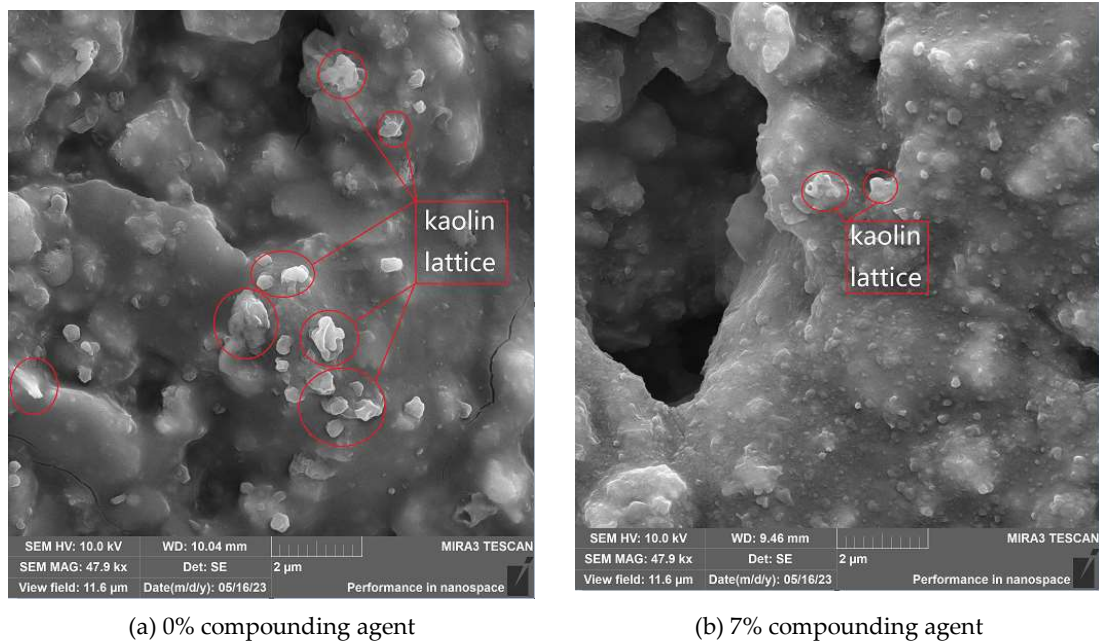
The surface of asphalt mastic doped with flame retardant and smoke suppressant compound was analyzed by SEM on the reaction surface at two temperatures (ordinary flame temperature at about 300°C and the highest temperature of TG test at about 800 °C).

In Figure 13(a), the asphalt layer adhering to mineral powder surface first contacted with the flame, and the asphalt on the surface pyrolytically was evaporated (the surface of the mineral powder was exposed) without flame retardant smoke suppressant effect. On the contrary, in Figure 13(b), the asphalt layer adhering to the surface of the mineral powder does not evaporate thermally in the presence of the flame retardant smoke suppressant (the mineral powder is wrapped by the asphalt). SEM observation at flame burning temperature (approx. 300 °C) showed that asphalt pastes containing 7% flame retardant and smoke suppressant has a significant flame retardant and smoke suppressant effect after flame burning.

Figure 14 shows the surface of the residual specimen after the TG test. In Figure 14(a), the asphalt bonded to the surface of the mineral powder was completely pyrolytically volatilized (lamellar lattices appear). The surface of the mineral powder showed distinct angularity, and the asphalt around the mineral powder showed pyrolytic deficiency. In Figure 14(b), the surface of the asphalt mastic showed pyrolytic softening, with some particles on the surface showing pyrolysis (small amounts of lattice bodies). However, most of the asphalt mastic adhering to the surface of the mineral powder did not pyrolyze. In contrast, in Figure 14(a), it was found that the asphalt on the surface of the mineral powder was pyrolyzed and appeared as a laminated lattice. The reason is that the mineral powders are joined together in the form of flaky particles after being subjected to a high temperature of 800 °C. Due to the distortion of the lattice during calcination, the increase in aggregates as hydroxyl groups are removed. In contrast, asphalt with 7% flame retardant and smoke suppressant did not show similar phenomena. Thus, the change in the surface state of asphalt proves the important role and applicability of fire retardant inhibitors.



**Figure 13.** SEM microscopic images after flame-burning (about 300 °C): (a) 0% compounding agent, (b) 7% compounding agent.

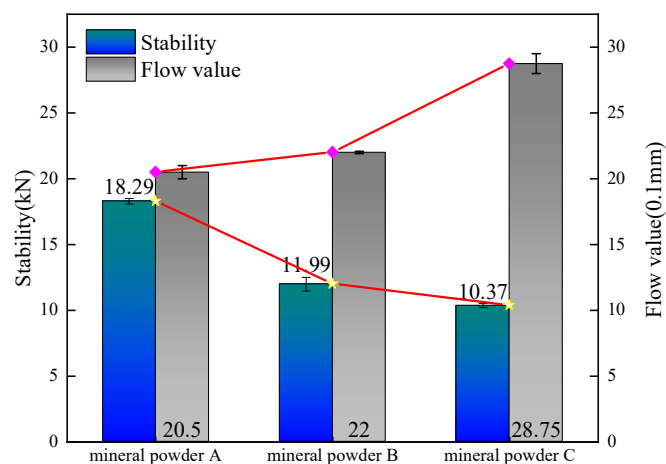


**Figure 14.** SEM microscopic images of the samples tested by TG (about 800 °C): (a) 0% compounding agent, (b) 7% compounding agent.

#### 4.3. Analysis of performance of asphalt mixtures

##### 4.3.1. Flow value and stability of asphalt

The optimal type of mineral powder (as one of the three mineral powders from the Dalian area) was determined by stability and flow value. The results are shown in Figure 15.

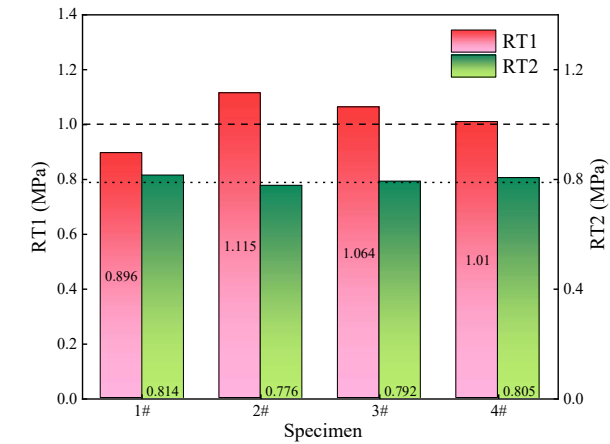


**Figure 15.** Comparison of Marshall stability and flow value of different mineral powders.

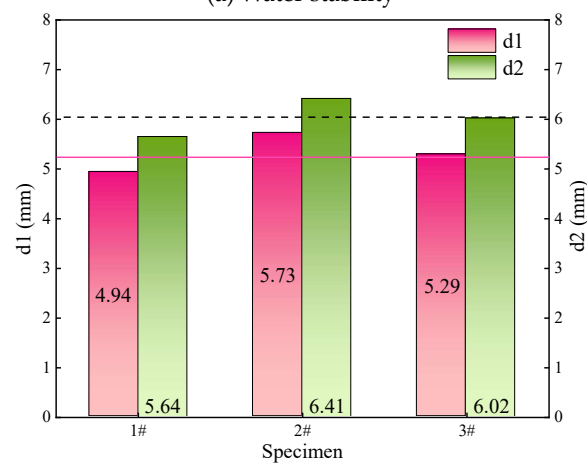
In Figure 15, the specimen with A mineral powder showed a maximum value of 18.29 kN of stability and also a minimum value (20.5 mm) of flow. Stability and small deformation were good, indicating that the asphalt mixture strength was higher. Therefore, A mineral powder was selected for the subsequent index test.

##### 4.3.2. Asphalt mixture performance analysis

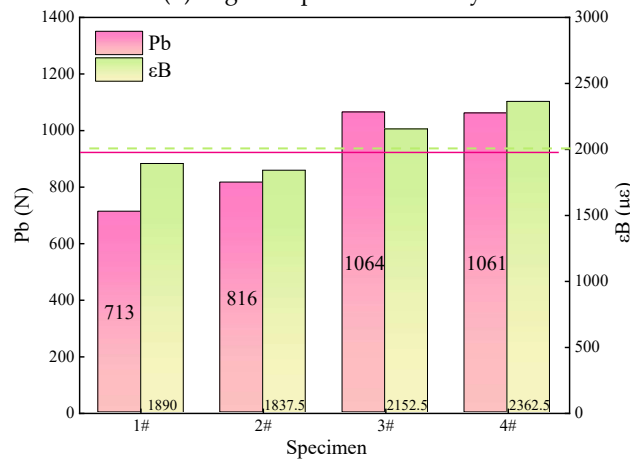
On the basis of research on fire retardant asphalt mastics, further investigate the effect of flame retardant and smoke suppressant compound on the asphalt mixture performance. The results are shown in Figure 16.



(a) Water stability



(b) High-temperature stability



(c) Low-temperature stability

**Figure 16.** Performance of asphalt mixtures: (a) Water stability, (b) High-temperature stability, and (c) Low-temperature stability.

In Figure 16(a), the water stability of fire retardant asphalt mixtures (7%) was investigated in asphalt mixture performance tests. The freeze-thaw split test was used and the calculated freeze-thaw split strength ratio (TSR) was 78%, which exceeded the standard value by 4%. In Figure 16(b), the dynamic stability (DS) of the rutting plate calculated in the high temperature stability test was 896 (number of times/mm), which exceeded the standard value by more than 12%. The coefficient of variation was 3.5%, which met the requirements of asphalt pavement. In Figure 16(c), the maximum bending tensile strain calculated in the low temperature stability( $\mu\epsilon$ ) experiments was 2060 which exceeded the standard value of winter temperature zone and winter cold zone by 3%. The addition

of such flame retardant asphalt mixture fully meet the asphalt mixture performance should be verified by the experiments more from the perspective of practical.

#### 4.4. Flame retardant smoke suppression analysis of asphalt mixture

Indoor laboratory combustion tests were conducted on Marshall specimens containing different dosage of flame retardant and smoke suppressant (0%, 7%). The mass loss of each group was recorded at an interval of 30 s to analyze the flame retardant and smoke suppression performance of asphalt mixture with two different amounts of flame retardant compound. The weight loss variations were analyzed, as shown in Figure 17.

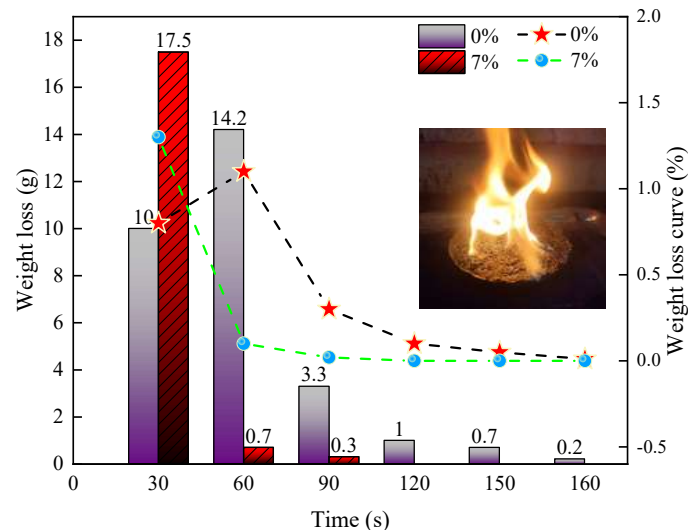


Figure 17. Specimen burning weight loss.

In Figure 17, In terms of weight loss: burning time of specimens containing 7% flame retardant smoke suppressant up to 90 s. At the 30 s instant, although the weight loss of the asphalt mixture containing 7% flame retardant and smoke suppressant was maximized, the burning time was reduced by 43% compared to the asphalt mixture without flame retardant and smoke suppressant. In addition, the total weight loss of the specimen with 7% flame retardant was 37% less than that of the specimen with 0% flame retardant during the whole combustion process.

From the analysis of weight loss rate: in 30-60 s, although the weight loss rate of 7% flame retardant smoke suppressant specimen was the largest, after 60 s, there was basically no significant change in the weight loss rate. It means that the flame retardant and smoke suppression effect is played quickly in the shortest time. On the contrary, the weight loss of specimens with 0% flame retardant and smoke suppressant showed a pattern of increasing and then decreasing, and the weight loss of burning time over 1 g lasted until 120 s. Through the above analysis, asphalt mixtures containing fire retardant and smoke suppressant have significant fire retardant and smoke suppressant effects.

## 5. Conclusions

This paper addressed the engineering problem of flame retardant smoke suppression in asphalt pavements in tunnels, developing a compound with  $Mg(OH)_2$  as the main component, and conducted a series of studies on asphalt mastic and asphalt mixes with it for the flame retardant smoke suppression performance. The conclusions were drawn as follow:

- (1) The asphalt mastic doped with fire retardant and smoke suppressant was improved the low temperature rheology and reduced the high temperature rheology to varying degrees.
- (2) Asphalt mastic and asphalt mixtures containing 7% fire retardant and smoke suppressant had the best fire retardant and smoke suppressant effect.

(3) Asphalt mixtures containing fire retardant smoke suppressant had reliable water stability, high temperature performance and low temperature tensile properties for asphalt pavement in Chinese tunnels.

(4) The pyrolysis initiation and termination temperatures of the asphalt mastic containing fire retardant and smoke suppressant were substantially increased during the main combustion phase.

(5) Fire retardant smoke suppressant asphalt mastic had excellent low temperature rheological properties.

(6) After burning at 800 °C, a small amount of pyrolysis occurred in the asphalt layer containing fire retardant and smoke suppressant. The flame retardant effect is obvious.

(7) Burning time, smoke emission time and weight loss of asphalt mixtures containing fire retardant and smoke suppressant were significantly reduced.

**Author Contributions:** Jiaquan Li: Conceptualization, Methodology, Writing-original draft, Software, Data curation; Mingjun Hu: Supervision, Visualization; Changjun Zhou: Supervision, Visualization, Funding acquisition, Writing-Review & editing; Fei Liu: Visualization, Writing-Review & editing, Software; Liujiyuan Su: Writing-Review & editing, Data curation; Peng Cao: Supervision, Investigation.

**Declaration of Competing Interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

**Data Availability Statement:** Some or all of the data or code supporting the results of this study are available from the corresponding authors upon request.

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