Article

Performance of Warm-Mixed Flame Retardant Modified Asphalt Binder

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Abstract: In order to analyze the effect of flame retardant and warm mix asphalt (WMA) additives-Sasobit on the flame-retardant performance and pavement performance of asphalt binder, the limiting oxygen index test, conventional performance test, and Superpave evaluation index tests were performed on asphalt binders in the study. The test results show that flame retardant can effectively improve the flame resistance of asphalt binder, while Sasobit has a certain combustion-supporting effect. Therefore, when warm-mixed flame-retardant technology is applied, the concentration of Sasobit should be controlled appropriately. These two modifiers can significantly enhance the high-temperature performance of asphalt binder, but both of them have a slight negative influence on the low-temperature cracking resistance. Sasobit can substantially reduce the high-temperature viscosity of asphalt binder, which helps to improve the construction workability of asphalt binder, while the flame retardant adversely affects the viscosity reduction effect of Sasobit to a certain extent, but the overall impact is not large.

Keywords: asphalt binder; flame retardant; Sasobit; flame retardant performance; pavement performance; limiting oxygen index test; conventional performance test; superpave evaluation index

1. Introduction

In recent years, with the rapid development of China's economy, the country's pavement engineering has also developed very quickly, especially in Western China. A large number of roads and tunnels must be built due to complex terrain and geology conditions. At present, asphalt pavement is widely applied in tunnel engineering due to its superior pavement performance. However, because asphalt binder is flammable, it can easily burn in relatively closed tunnels in the event of a fire hazard. In this case, heat can't be expelled fast enough, and the diffuse smoke, in addition to high temperatures and toxic gas, can bring about huge challenges for safe excavation and rescue, thus seriously threatening the safety of human life and property. Furthermore, because of the narrow and closed tunnel space and poor atmospheric conditions, a great deal of heat and harmful smoke is generated in the process of paving construction, which leads to a terrible construction environment in the tunnel and severe damage to the health of on-site construction personnel. It is therefore imperative to carry out research on warm-mixed flame-retardant asphalt technology [1,2].

Research on warm mix and flame-retardant asphalt technology started earlier in foreign countries. In 1995, warm mix technology was first developed in Europe. Harrison and Christodoulaki first reported the technology in the German Asphalt Forum in 1997, the First International Asphalt Pavement Conference in 2000, and the Second European Asphalt Conference in Barcelona in 2000 [3]. Subsequently, Europe, Japan, Australia, and the United States began to use warm mix technology on a large scale [4]. In general, warm mix technology includes three major systems: Organic additives,

chemical additives, and foaming technologies. Sasobit is a typical viscosity-reducing organic additive, and the related research indicates that Sasobit has good pavement performance. For example, Wasiuddin N. and Zaman M. studied the rheological properties of Sasobit modified asphalt binder at different concentrations [5,6]. Results show that Sasobit can effectively improve the high temperature performance of the asphalt binder and improve the rutting resistance of asphalt mixtures. Hamzah M. O., Jamshidi A., and Shahadan Z. evaluated the CO₂ discharge and heat needed to heat the aggregate and asphalt binder from 25 °C to the mixing temperature at different Sasobit concentrations. The results show that 1% Sasobit can reduce CO₂ discharge by 3% and energy consumption by 2.8% [7]. Jamshidi A., Hamzah M. O., and You Z. focused on the rheological properties of Sasobit modified asphalt binder and proved that Sasobit could effectively improve the pavement performance of asphalt mixture. In addition, the life cycle of Sasobit modified asphalt binder was evaluated [8]. In some foreign countries, researchers have also focused on flame retardant agents. In 1986, the Asahi Rekisei company in Japan published a patent on the preparation of flame retardant and smoke suppression modified asphalt binder [9]. In 1991, Grube publicized the patent of flame retardant and smoke suppression modified asphalt binder, in which borate was applied as the flame retardant [10]. In 2011, Hull TR pointed out that the action process of inorganic flame retardants was more complex than that of halogen flame retardants, and the actions were different in different polymers [11]. In 2013, Bonina researched the flame retardant and smoke suppression effects of aluminum hydroxide, and combined it with nano composite adhesives to improve the flammability of asphalt mixtures [12]. Some Chinese scholars have also carried out a series of studies on the warm mixed and flame-retardant asphalt technology. Xiao F. studied the rutting resistance of asphalt mixtures at different conditions, including two kinds of moisture content (0% and 5%) of aggregate, two kinds of mineral powder content (1% and 2%), three kinds of warm mix asphalt (WMA) additives (Sasobit, Evotherm and Aspha-Min), and three kinds of aggregate sources. The results show that the mixture with Sasobit additive has the best rutting resistance [13]. Liu Zhanliang carried out rotational viscosity tests and dynamic shear rheological tests on Sasobit modified asphalt binder at different concentrations. The results indicate that the phase angle of asphalt binder grows slowly at first, then decreases with the addition of Sasobit [14]. Yue Jinzhao carried out conventional performance tests and Superpave Performance Graded (PG) grading tests respectively for Sasobit modified asphalt binder at different concentrations, and results show that Sasobit can significantly improve the high temperature performance and construction workability of asphalt binder. The study also found that Sasobit causes certain aging resistance performance and temperature susceptibility effects, but it has some adverse effects on low-temperature performance [15]. Li Xiaoling used magnesium hydroxide, melamine, ammonium polyphosphate, and titanate as raw materials to prepare flame retardants of different formulas [16]. Luo Jianping found that the compound of flame retardant and WMA additives reduces heat and smoke release and can increase the high-temperature performance of asphalt mixtures [17]. Wang Jialin believed that warm-mixed flame-retardant technology can improve the flame retardant properties of asphalt mixtures and reduce the mixing and paving temperatures, thus contributing to energy conservation, environmental protection and low-carbon construction [18].

In conclusion, although many studies have been made on warm mix technology and flame-retardant technology both in China and abroad, less are about the compounded technology of warm mix and flame retardants. In addition, the types of WMA additives and flame retardants are numerous, and there are differences in their research methods and evaluation means, and there are certain limitations in the conclusions of relevant research, so no corresponding technology specification and evaluation system have been formed in China at present, and it is necessary to conduct deeper and more comprehensive research on this synthesis technology so as to promote the extensive application of this technology in the large-scale construction of tunnel projects in China.

2. Materials and Testing Program

2.1. Materials

Styrene-Butadiene-Styrene (SBS) modified asphalt binder is used in the paper, the flame retardant is supplied by Shandong Dashan Road and Bridge Engineering Co., Ltd. (Jinan, Shandong, China, 2018), and Sasobit is supplied by Henan Lupeng Transportation Technology Co., Ltd (Zhengzhou, Henan, China, 2018). The properties of the SBS modified asphalt binder are shown in Table 1. After inspection, its technical indexes conform to the technical requirements of *Technical Specification for Construction of Highway Asphalt Pavements* (JTGF40-2004). In addition, the properties of Sasobit are based on the testing data provided by the manufacturer, as shown in Table 2.

Table 1. Properties of styrene-butadiene-styrene (SBS) modified asphalt binder.

Property		Units	Testing Value	Technical Requirements
Penetration (100 g, 5 s)	15 °C	0.1 mm	18.5	-
	25 °C	0.1 mm	44.8	30~60
	30 °C	0.1 mm	67.9	-
Penetration Index (PI)		-	0.38	≥0
T ₈₀₀		°C	58.2	-
T _{1.2}		°C	−16.5	-
Ductility	15 °C	cm	825	-
	5 °C	cm	252	≥20
Softening Point		°C	81.8	≥60

Note: “-” in the table means there are no requirements for the items in the technical specification.

Table 2. Properties of Sasobit.

Property	Melting Point/°C	Flashing Point/°C	Viscosity at 135 °C/(Pa·s)	Viscosity at 150 °C/(Pa·s)	Penetration at 25 °C/0.1 mm	Penetration at 60 °C/0.1 mm
Testing value	100	290	5.47×10^{-3}	3.26×10^{-3}	1	8

2.2. Testing Program

Referring to relevant research papers both in China and abroad, the warm-mixed flame retardant SBS modified asphalt binder with 1% Sasobit and 5%, 6%, 7%, and 8% flame retardant were prepared by additive mix method. The aforementioned concentrations refer to the percentage of Sasobit or flame retardant expressed as a percentage of SBS modified asphalt binder by weight. The flame retardant properties and pavement performance of warm-mixed flame retardant asphalt binder were evaluated by the limit oxygen index test, three-index test, Brookfield rotational viscosity test, dynamic shear rheological test and repeated creep recovery test, and simultaneously, a contrastive analysis was made by using SBS modified asphalt binder and Sasobit modified asphalt binder (the concentration of Sasobit is 1%).

2.3. Preparation Method of Warm-Mixed Flame Retardant Asphalt Binder

Heat the SBS modified asphalt binder up to 155 °C, then weigh the Sasobit and flame retardant by predetermined blending proportions, and put them into SBS modified asphalt binder in sequence. Mix them for around 30 min to make sure that Sasobit and flame retardant can evenly distribute in the SBS modified asphalt binder. Then warm-mixed flame-retardant asphalt binder can be obtained after keeping them in the constant temperature oven at 155 °C for 15–30 min.

3. Test Results and Analysis of the Limit Oxygen Index Test of Asphalt Binder

The limit oxygen index (LOI) was adopted to evaluate the flame-retardant properties of the asphalt binder in this study. Generally, it is determined using the oxygen index test method. LOI refers to the lowest oxygen concentration in the oxygen-nitrogen mixed gas inlet when it is just enough to keep the material burning under the required test condition. The calculation equation is shown as Equation (1).

$$\text{LOI} = \frac{[\text{O}]}{[\text{O}] + [\text{N}]} \times 100\% \quad (1)$$

In Equation (1), [O] indicates the volume flow of oxygen in mixed airflow at critical oxygen concentration; [N] indicates the volume flow of nitrogen in mixed airflow at critical oxygen concentration.

According to *Determination of the Combustion Performance of Rubber* (GB/T 10707-2008) [19], XZT-100A oxygen index determinator was adopted in this paper to determine the limit oxygen index (LOI) of SBS modified asphalt binder, Sasobit modified asphalt binder, and warm-mixed flame-retardant asphalt binder at different concentrations. In the test, the asphalt binder was poured evenly on the surfacing mat of fiberglass to make samples (100 mm × 5.5 mm). The apparatus of the oxygen index test is as shown in Figure 1, the samples are as shown in Figure 2, and the test results are as shown in Table 3.

Figure 1. Apparatus for oxygen index test.

Figure 2. Samples for the determination of limit oxygen index.

Table 3. Results of limit oxygen index test.

Types of Asphalt Binder	SBS	+1% Sasobit	+1% Sasobit +5% FR	+1% Sasobit +6% FR	+1% Sasobit +7% FR	+1% Sasobit +8% FR
LOI (%)	23.0	22.8	23.2	23.4	23.7	24.3

Note: FR is the acronym of “Flame Retardant”, similarly hereinafter.

As seen in Table 3: ① The limit oxygen index increased with the addition of flame retardant, which indicates that flame retardant can improve the flame resistance of asphalt binder, and the higher the concentration, the better the flame resistance. In addition, the limit oxygen indexes of warm-mixed flame retardant asphalt binder at different concentration all conform to the requirement of *Concrete of Flame Retardant Asphalt for Road Engineering* (GB/T 29051-2012) that the limit oxygen index of flame retardant asphalt binder shall not be less than 23% [20]; ② the limit oxygen index decreased from 23.0% to 22.8% after adding Sasobit into SBS modified asphalt binder, which means that Sasobit could have an adverse impact on the flame retardant properties, and according to the preliminary analysis, it is concluded that Sasobit has certain combustion-supporting effects on asphalt binder because it is a wax-based material.

4. Results and Analysis of Conventional Performance Test of Asphalt Binder

4.1. Penetration Test

The penetration numbers of different kinds of modified asphalt binders were tested at 15 °C, 25 °C, and 30 °C according to *Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering* (JTG E20-2011) (similarly hereinafter for the test methods standard in the paper) [21], and then the penetration index PI, equivalent softening point T_{800} , and equivalent brittle point $T_{1.2}$ were calculated accordingly. The test results are shown in Table 4.

Table 4. Results of the penetration test.

Types of Asphalt Binder	SBS	+1% Sasobit	+1% Sasobit +5% FR	+1% Sasobit +6% FR	+1% Sasobit +7% FR	+1% Sasobit +8% FR
Penetration (0.1 mm)	15 °C	18.7	18.0	17.8	17.5	17.1
	25 °C	44.8	44.6	42.7	43.6	42.0
	30 °C	67.9	65.3	64.8	63.2	61.9
T_{800}	58.2	58.7	58.7	59.1	59.4	59.7
$T_{1.2}$	−16.5	−16.4	−16.2	−16.1	−15.9	−15.6
PI	0.38	0.40	0.42	0.43	0.44	0.45

As seen in Table 4: ① The addition of flame retardant and Sasobit makes asphalt binder harder and the non-deformability higher, for example, for the penetration at 25 °C, Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder with different flame retardant concentration decreased by 0.45% to 9.15% compared to SBS modified asphalt binder, which indicates that flame retardant and Sasobit can improve the high temperature stability of asphalt binder; ② Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder exhibited PI that were 1.05–1.18 times that of SBS modified asphalt binder, indicating that both flame retardant and Sasobit can improve the temperature sensibility of asphalt binder; ③ with the addition of flame retardant or Sasobit, the equivalent brittle point of asphalt binder increased from −16.5 °C to −15.6 °C, which means that both of them produce certain negative effects on the low-temperature performance of asphalt binder, but the effect is not large.

4.2. Softening Point Test

The softening point of asphalt binder was determined using the ring and ball method, and the results are shown in Table 5.

Table 5. Results of the softening point test.

Types of Asphalt Binder	SBS	+1% Sasobit	+1% Sasobit +5% FR	+1% Sasobit +6% FR	+1% Sasobit +7% FR	+1% Sasobit +8% FR
Softening point/°C	81.8	82.5	83.6	85.2	87.3	90.4

It can be inferred from Table 5 that Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder with different flame retardant concentrations had softening points that were 1.00, 1.02, 1.04, 1.06 and 1.11 times that of SBS modified asphalt binder, respectively, which indicates that both flame retardant and Sasobit can effectively improve the high-temperature deformation resistance of asphalt binder, and for warm-mixed flame retardant asphalt binder, the higher the concentration, the better the effect, which is consistent with results based on the penetration test.

4.3. Ductility Test

The results of ductility test at 5 °C and 15 °C are shown in Table 6.

Table 6. Results of ductility test (mm).

Types of Asphalt Binder	SBS	+1% Sasobit	+1% Sasobit +5% FR	+1% Sasobit +6% FR	+1% Sasobit +7% FR	+1% Sasobit +8% FR
5 °C	252	250	248	230	215	210
15 °C	825	801	742	733	726	712

It can be seen in Table 6 that the addition of flame retardant or Sasobit reduced the ductility of the asphalt binder, which indicates that both of them have a negative influence on the low temperature cracking resistance of asphalt binder, while the overall effect is not large.

5. Results and Analysis of Superpave Evaluation Index Test of Asphalt Binder

5.1. Brookfield Rotational Viscosity Test

In order to evaluate the effect of flame retardant and Sasobit on the viscosity of asphalt binder, a Brookfield rotational viscosimeter was adopted in this paper to determine the Brookfield rotational viscosity of SBS modified asphalt binder, Sasobit modified asphalt binder, and warm-mixed flame retardant asphalt binder at 135 °C. Rotor (29#) and rotor speed (20 r/min) were selected according to the range of apparatus and test specifications [21]. The test results are shown in Table 7.

Table 7. Results of Brookfield rotational viscosity test (Pa·s).

Types of Asphalt Binder	SBS	+1% Sasobit	+1% Sasobit +5% FR	+1% Sasobit +6% FR	+1% Sasobit +7% FR	+1% Sasobit +8% FR
135 °C	2.749	1.895	2.214	2.337	2.392	2.469

It can be seen in Table 7 that ① the viscosity of Sasobit modified asphalt binder decreased by 31.07% compared to SBS modified asphalt binder, indicating that Sasobit can obviously reduce the high temperature viscosity of asphalt binder and increase its high temperature liquidity, thus improving the construction workability of asphalt binder and producing a good warm mix effect; ② the viscosity of warm-mixed flame retardant asphalt binder with different flame retardant concentration increased by 16.83% to 30.29% compared to Sasobit modified asphalt binder, suggesting that the addition of flame retardant improves the viscosity of asphalt binder, which adversely affects the viscosity reducing effect of Sasobit, but in general, the viscosity of warm-mixed flame retardant asphalt binder is lower than that of SBS modified asphalt binder, so it is believed that flame retardant has little impact on the warm mix effect of asphalt binder.

5.2. Dynamic Shear Rheological Test

The dynamic shear rheological test was employed in this paper to evaluate high temperature rheological properties of three kinds of asphalt binders. Original asphalt binder and rolling thin film oven tested (RTFOT) binder were studied, strain controlled mode was adopted, the frequency was 10rad/s, and the high-temperature performance was mainly evaluated with anti-rutting factor ($G^*/\sin\delta$). The test results are shown in Figures 3 and 4.

Figure 3. Results of the dynamic shear rheological test of original asphalt binder.

Figure 4. Results of the dynamic shear rheological test of rolling thin film oven tested (RTFOT) aged asphalt binder.

As seen in Figures 3 and 4, the addition of flame retardant and Sasobit can obviously improve the $G^*/\sin\delta$ of asphalt binder. For example, for the original asphalt binder, Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder with different flame retardant concentration exhibited $G^*/\sin\delta$ (at 64 °C) that were 1.10, 1.13, 1.19, 1.20, and 1.25 times that of SBS modified asphalt binder, respectively; for the RTFOT aged asphalt binder, Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder with different flame retardant concentration exhibited $G^*/\sin\delta$ (at 64 °C) that were 1.04, 1.10, 1.11, 1.13, and 1.22 times that of SBS modified asphalt binder, respectively. These results indicate that both flame retardant and Sasobit can significantly improve the high-temperature resistance deformability of asphalt binder; and as for warm-mixed flame retardant asphalt binder, $G^*/\sin\delta$ increased with the increase of flame retardant concentration, indicating that the concentration has a positive influence on the high-temperature performance, which is consistent with the penetration test results.

According to the above test results, the high-temperature PG grading made for SBS modified asphalt binder, Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder. The results are shown in Table 8.

Table 8. Summary of performance graded (PG) grading results.

Types of Asphalt Binder	SBS	+1% Sasobit	+1% Sasobit +5% FR	+1% Sasobit +6% FR	+1% Sasobit +7% FR	+1% Sasobit +8% FR
PG Grading	76	82	82	82	82	82

It can be inferred from Table 8 that the high temperature PG grading of asphalt binder has been improved by one grade after the addition of Sasobit and flame retardant, and as for the warm-mixed flame retardant asphalt binder, its high-temperature grading remains unchanged despite the increase of the concentration of flame retardant. The above test results indicate that the addition of flame retardant and Sasobit is beneficial for improving the high-temperature performance of asphalt binder.

5.3. Repeated Creep Recovery Test

For a long time, anti-rutting factor $G^*/\sin\delta$ has been considered an acclaimed index for the evaluation of the high temperature performance of asphalt binders, but there are some limitations in representing the high-temperature performance of modified asphalt binder [22,23]. Therefore, it is proposed in the NCHRP9-10 report of the U.S. to evaluate the high temperature performance of modified asphalt binder with repeated creep recovery tests [24]. This test method simulates the effect of load on pavement surfaces in the driving process by constantly loading and unloading the samples, which can effectively make up for the deficiency of only considering the elasticity and viscosity of asphalt binder in dynamic shear rheological tests, and more accurately reflect the creep and recovery ability of asphalt binder [25,26].

In view of the above reasons, in order to better evaluate the high-temperature performance of asphalt binder and the changes to the elasticity and viscosity of asphalt binder under long-term repeated loading, a dynamic shear rheometer (DSR) was employed in this paper to make a repeated creep recovery test on SBS modified asphalt binder, Sasobit modified asphalt binder, and warm-mixed flame retardant asphalt binder, and the accumulated strain γ was adopted as a high-temperature performance evaluation index. In this test, stress control mode was adopted, the stress level was 120 Pa, and a plate of 25 mm was used with a gap of 1mm. Three different test temperatures: 30 °C, 40 °C, and 50 °C were adopted, the loading duration for each cycle of creep was 1 s, unloading duration was 9 s, and the total cyclic loading was 100 times. The test loading pattern is shown in Figure 5.

In order to evaluate the high-temperature performance of the three kinds of modified asphalt binder, the cumulative strain development curves at three different temperatures from the 51st to 60th creep recovery cycles were selected to make analysis; see Figures 6–8.

Figure 5. Loading pattern of repeated creep recovery test.

Figure 6. Strain response of asphalt binder at 30 °C.

Figure 7. Strain response of asphalt binder at 40 °C.

Figure 8. Strain response of asphalt binder at 50 °C.

It can be seen from Figures 6–8 that ① during the 51th and 60th loading cycles, the accumulated strain value of asphalt binder tested at 50 °C is significantly greater than those tested at 40 °C and 30 °C, which shows that with the increase of the test temperature, the accumulated strain value of asphalt binder increased accordingly. ② At three different temperatures, the accumulated strain values of Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder are significantly lower than that of SBS modified asphalt binder, which indicates that both Sasobit and flame retardant can markedly enhance the deformation recovery ability of asphalt binder.

In addition, in order to better analyze the accumulated strain of asphalt binder, the accumulated strain values of asphalt binders under a stress level of 120 Pa, at three different temperatures of 30 °C, 40 °C, and 50 °C in the 1st, 10th, 20th, 30th, 40th, 50th, 60th, 70th, 80th, 90th and 100th creep recovery cycles were calculated in this paper. The strain values represent the accumulated results of all residual deformation after the 100th creep recovery cycle. The smaller the accumulated strain value, the stronger the deformation recovery ability of the asphalt binder. The testing results are shown in Figures 9–11.

Figure 9. The relationship between loading numbers and cumulative strain at 30 °C.

Figure 10. The relationship between loading numbers and cumulative strain at 40 °C.

Figure 11. The relationship between loading numbers and cumulative strain at 50 °C.

It can be seen in Figures 9–11 that ① at different temperatures, the accumulated strain values of modified asphalt binder increased with the increase of loading numbers, showing a curvilinear growth trend and viscoelastic rheological characteristics. ② The addition of flame retardant and Sasobit can effectively reduce accumulated strain value of asphalt binder, take the cumulative strain at 40 °C in the 50th creep recovery cycle for example, Sasobit modified asphalt binder and warm-mixed flame retardant asphalt binder with different flame retardant concentration decreased by 32.24%, 38.28%, 40.33%, 45.40%, and 51.50% compared to SBS modified asphalt binder, respectively, which suggests that both Sasobit and flame retardant can significantly improve the high-temperature stability of asphalt binder; and as for warm-mixed flame retardant asphalt binder, the accumulated strain decreased gradually with the increase of the concentration of flame retardant, indicating that the increase of the concentration is beneficial to enhancing the high-temperature deformation resistance of the asphalt binder.

6. Conclusions

(1) The flame retardant can effectively improve the flame resistance of the asphalt binder, while Sasobit has a certain negative effect on this performance. Therefore, the concentration of Sasobit should be properly controlled to ensure that the flame-retardant performance of asphalt binder will not be significantly affected when warm-mixed flame-retardant technology is adopted in tunnel engineering.

(2) Both the flame retardant and Sasobit can significantly strengthen the high-temperature stability of asphalt binder, but at the same time, the two have a slight adverse effect on the low-temperature cracking resistance.

(3) Sasobit can obviously reduce the high-temperature viscosity of SBS modified asphalt binder, and increase the high temperature fluidity, thus achieving ideal warm mixing effect. However, the flame retardant adversely affects the viscosity reducing effect of Sasobit, while the impact is generally not large.

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