



Article Rheological Behavior of Warm Mix Asphalt Modified with Foaming Process and Surfactant Additive

Guoyang Lu^{1,2}, Shaowei Zhang ³, Shaofeng Xu⁴, Niya Dong ^{3,*} and Huayang Yu³

- ¹ Research and Development Centre of Transport Industry of Technologies, Materials and Equipment of Highway Construction and Maintenance, Gansu Road and Bridge Group Co. Ltd., Lanzhou 730050, China; guoyang.lu@polyu.edu.hk
- ² Department of Civil and Environmental Engineering, The Hong Kong Polytechnic University, Kowloon, Hong Kong, China
- ³ School of Civil Engineering and Transportation, South China University of Technology, Wushan Road, Tianhe District, Guangzhou 510000, China; ctzsw@mail.scut.edu.cn (S.Z.); huayangyu@scut.edu.cn (H.Y.)
- ⁴ Department of Civil and Environmental Engineering, University of Macau, Macao 999078, China; db92762@umac.mo
- * Correspondence: dny1987@scut.edu.cn; Tel.: +86-137-7073-2002

Abstract: Surfactants are frequently used to improve the engineering performances of foamed bitumen. Additionally, the foaming process can also perform a significant influence on the foam characteristics and rheological properties of foamed bitumen. However, rare research investigates the synergistic effect of both surfactant and foaming process on the engineering properties of foamed bitumen. To fill the gap, this research investigated the synergistic effect of surfactant and foaming process on the foaming characteristics and rheological properties of foamed bitumen. Based on the experimental results, the synergistic effect shows a significant effect on improving the half-life of foamed bitumen, which reached up to 69 s when 6% foaming Evotherm-DAT content was used. In addition, the foaming temperature also has a significant effect on the foaming characteristics. This study shows that the best foaming conditions can be achieved when the foaming temperature and Evotherm-DAT content are 170 °C and 8%, respectively. Based on the study of synergistic effect, the engineering performances of surfactant foamed bitumen were further characterized in this research, for instance, the enhancement in high-temperature performance and fatigue resistance, and the improvement in workability. Generally, the results of this study have greatly promoted the application of surfactant foam bitumen in the engineering practice.

Keywords: GHG emissions; foamed bitumen; surfactant additives; rheological properties; SBS modified bitumen

1. Introduction

Asphalt pavement has a significant impact on the environment, because during construction, asphalt needs to be heated to a higher temperature, which can lead to huge energy consumption and greenhouse gas emission. Therefore, environmental problems and risks caused by asphalt pavement in the production process have attracted increasing attention [1,2].

A huge amount of research has been devoted to the cleaner production of asphalt mixtures, and various technologies have been used to reduce the manufacturing temperature [3–6]. Warm mix asphalt (WMA), as one of the technologies that can reduce the impact on the environment during the asphalt production process, has been greatly developed over the past few decades. This technology can generally make bituminous mixtures that are produced at temperatures at least 20 °C to 40 °C lower than typical Hot Mix Asphalt (HMA) [7]. It is reported that by decreasing the mixing temperature from 20 °C to 40 °C of asphalt mixtures, the energy consumption and greenhouse gas (GHG) emissions can be saved up to 20% to 70% when compared to HMA [8]. In addition to the environmental



Citation: Lu, G.; Zhang, S.; Xu, S.; Dong, N.; Yu, H. Rheological Behavior of Warm Mix Asphalt Modified with Foaming Process and Surfactant Additive. *Crystals* **2021**, *11*, 410. https://doi.org/10.3390/ cryst11040410

Academic Editor: Edyta Pawluczuk

Received: 15 March 2021 Accepted: 8 April 2021 Published: 12 April 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). benefits, WMA can also bring other benefits, such as slowing down the aging of the asphalt, extending the hauling distance, better working conditions for road workers because of the decrease of asphalt fume, quicker turnover to traffic, extending paving window, etc. [9,10].

One of the WMA techniques that are available to reduce asphalt production and construction temperatures is the application of foam bitumen, which is increasingly popular due to the low investment and production costs [3,11,12]. Conventional foam bitumen is the mixture of bitumen, water, and air, which can be produced by injecting a certain amount of water into the bitumen in the expansion chamber at a higher temperature. During this process, liquid water is converted into vapor, and the vapor will expand the original volume of bitumen to form foamed bitumen. Eventually, these foams will burst, and most of the injected moisture will be dissipated in the form of vapor so that the remaining bitumen has properties similar to the original one. Due to the cost-effectivity, environmental benefits, and high field-performance properties, foam bitumen is increasingly getting recognized in cold-in-place recycling [13–16] and base course stabilization [17–19].

In recent years, surfactants have been frequently used to improve the performance of foamed bitumen, because they can reduce the surface tension of the liquid bitumen, providing a more comprehensive coating of the aggregate at low temperatures without changing the workability of the asphalt mixture at reduced pavement laying temperatures [20,21]. Therefore, surfactant also acts as an anti-stripping agent, increasing the adhesion between the aggregate and the bitumen, thereby reducing the related problems of foamed bitumen in the application, such as raveling and shredding [22,23]. Apart from the surfactant additives, the foaming process also presents a significant impact on the foaming characteristics and engineering performances of the foamed bitumen [24]. Temperature is one of the most important factors that influence the engineering properties of foamed bitumen. The optimum mixing temperature of the aggregates for foamed asphalt mixes lies in a certain range depending on the type of aggregate [25]. Temperatures below this range result in poor-quality mixes. Foamed asphalt mixes may also be prepared with heated aggregates, which will increase the binder dispersion within the mix and aid in the coating of the larger aggregates [26].

Given this, though there have been various studies investigating the surfactant and foaming process on the foaming characteristics of the foamed bitumen, the research of the synergistic effect of surfactant and foaming process is still scarce and lacks a comprehensive investigation and comparison on the binder properties. In this study, foamed bitumen was investigated on its engineering property with different contents of surfactant and foaming processes. Meanwhile, a series of laboratory tests were performed to evaluate and compare the foam characteristic and rheological properties of surfactant-foamed bitumen (SFB) and non-foamed bitumen binders.

2. Materials and Sample Preparation

2.1. Materials

The SBS modified bitumen (shortly named as B) was selected in this research and its fundamental properties are presented in Table 1. A typical chemical WMA additive with surfactant properties, Evotherm-DAT, was chosen as a foaming agent for this study, while a non-foaming organic additive, Sasobit, was selected for comparison [27]. Table 2 compiles the properties of Evotherm-DAT and Sasobit. In the current study, the surfactant was manufactured by blending the raw material of water-free Evotherm-DAT (viscous liquid) with water at a ratio of 1:20 under ambient temperature. The well-blended Evotherm-DAT surfactant was then directly injected into the hot bitumen to form SFB. To ensure consistency, all materials used in this study were from the same production lot.

Properties		Standard	Value	Specification Limits
	Softening point (°C)	ASTM D36	89	≥75
	Ductility (5 °C, cm)	ASTM D113	32	≥ 20
Unaged	Penetration (25 $^\circ$ C, 0.1 mm)	ASTM D5	54	40~60
	Viscosity (135 °C, mPa·s)	AASHTO T316	2.38	3
	$G^*/\sin\delta$ (82 °C, kPa)	AASHTO M320	1.24	-
RTFO aged	G*/sinδ (82 °C, kPa)	AASHTO M320	1.75	-
	G*sinδ (25 °C, kPa)	AASHTO M320	2675	-
PAV aged	Creep stiffness (-12 °C, MPa)	AASHTO T313	147	\leq 300
	Creep rate m-value (-12 °C)	AASHTO T313	0.322	≥ 0.3

Table 1. Properties of SBS modified bitumen.

Table 2. Properties of Evotherm-DAT and Sasobit.

Properties	Evotherm-DAT	Sasobit
Appearance		
Ingredients	Fatty amine derivatives, Alkylamines	Solid saturated hydrocarbons
State	Liquid	Solid
Color	Caramel	Milky-white
Odor	Amine-like	None
Density	$>1.0 \text{ g/cm}^{3}$	0.622 g/cm^3
PH value	9–10	N/A
Boiling point	150–170 °C	N/A
Melting point	N/A	105–110 °C
Water solubility	Partially soluble	Insoluble

2.2. Sample Preparation

2.2.1. Preparation of Non-Foamed Bitumen

Non-foamed bitumen binder was prepared by mixing Evotherm-DAT (6%, 8%, and 10% by weight of B) or Sasobit (3% by weight of B) with B (160 °C) in the laboratory. The mixing speed and time were set to 1500 rpm and 10 minutes, respectively, ensuring that the additive was properly integrated into the binder. The mixing equipment consists of an electric heating jacket with a temperature probe, a paddle mixer and a 2-liter metal bucket (13 cm in diameter). During the mixing process, a temperature probe was inserted into the bucket to monitor the temperature of the binder, in which case the temperature was to be controlled precisely. In the current study, five different non-foamed bitumen binder samples were prepared; the sample IDs and the detailed preparation procedures are summarized in Table 3. It should be noted that the volume of the bitumen at high temperature could expand by the addition of water in surfactant; hence, the water-free Evotherm-DAT was added to manufacture the non-foamed bitumen.

2.2.2. Preparation of SFB

Foamed bitumen binder was prepared using an iFoam foaming machine improved from the WLB10 laboratory machine. Through extensive laboratory trials and the validation of iFoam and WLB10, it had been proven that iFoam was feasible and reliable for producing the foamed bitumen. Therefore, iFoam was applied to prepare the SFB. In the current study, three types of SFB binders, EFB6, EFB8, and EFB10 (Table 3), were prepared under the optimal foaming condition obtained from followed foaming tests.

Table 3. Summary of sample IDs and the detailed preparation procedures.

Sample ID	Preparing Process		
В	Directly use SBS modified asphalt		
BS	Directly mixing Sasobit and SBS modified asphalt together (3% of asphalt) at 160 °C for 10 min		
BE6	Directly mixing water-free Evotherm-DAT (6% of asphalt) and SBS modified asphalt together at 160 $^\circ$ C for 10 min		
BE8	Directly mixing water-free Evotherm-DAT (8% of asphalt) and SBS modified asphalt together at 160 °C for 10 min		
BE10	Directly mixing water-free Evotherm-DAT (10% of asphalt) and SBS modified asphalt together at 160 °C for 10 min		
EFB6	Directly inject Evotherm-DAT (6% of asphalt) into SBS modified asphalt (170 °C) to produce the surfactant-foamed asphalt using a foaming machine		
EFB8	Directly inject Evotherm-DAT (8% of asphalt) into SBS modified asphalt (170 °C) to produce the surfactant-foamed asphalt using a foaming machine		
EFB10	Directly inject Evotherm-DAT (10% of asphalt) into SBS modified asphalt (170 $^\circ$ C) to produce the surfactant-foamed asphalt using a foaming machine		

3. Experimental Program

To systematically evaluate the properties of SFB with different foaming Evotherm-DAT contents (FEC) at different temperatures, both the binder foaming tests and rheological tests were conducted. The performance of SFB was compared to that of B and BE.

3.1. Foaming Tests

The iFoam foaming machine (Hengtong Asphalt Testing Co. Ltd., Guangzhou, Guangdong, China) was used to conduct the foaming tests under a variety of bitumen temperatures (160 °C, 170 °C, and 180 °C) and FEC (6%, 8%, 10% by weight of B). The quality of foamed bitumen under different foaming conditions and the optimum foaming conditions were evaluated and determined using expansion ratio (ER), half-life (HL), and foam index (FI). FI is a parameter developed for characterizing and optimizing foaming properties [28], and it can be calculated by the following equation:

$$FI = -\frac{\tau_{1/2}}{\ln 2} (4 - ER_m - 4\ln(\frac{4}{ER_m})) + (\frac{1+c}{2c}) * ER_m * t_s$$
(1)

where, c is the correction factor (Erm/Era); ts is bitumen spraying time (s); $\tau_{1/2}$ denotes the half-life period; Erm is the measured maximum expansion ratio; and ERa is the actual expansion ratio.

3.2. Rheological Tests

The softening point and rotational viscosity tests were firstly performed according to ASTM D36 and AASHTO T316 [29,30]. The softening point represents the high-temperature performance of the binder. A Brookfield RV–DVIII Ultra rotational viscometer (Brookfield testing company, US) was utilized to capture the viscosity of all binders at three temperatures (135 °C, 155 °C, and 175 °C) and the workability of binders was then evaluated. Dynamic shear rheometer (DSR) tests were performed to characterize the rheological properties of the binder at high and intermediate temperatures according to AASHTO T315-12 [31]. In the DSR tests, a dynamic shear rheometer (Malvern Kinexus Lab+, Malvern

analytical Company, UK) was utilized to measure the rutting factor ($G^*/\sin\delta$), failure temperature, non-recoverable creep compliance (J_{nr}) and fatigue factor ($G^*\cdot\sin\delta$). To evaluate the low-temperature performance of surfactant-foamed and non-foamed bitumen binders, bending beam rheometer (BBR) tests were also performed to measure creep stiffness and m-value.

3.2.1. High-Temperature Performance Tests

The high-temperature performance of surfactant-foamed and non-foamed bitumen binders was evaluated by two parameters: the rutting factor and non-recoverable creep compliance (J_{nr}). For the rutting factor G*/sin δ , it was obtained by the rutting factor test using a DSR. In this test, unaged and rolling thin film oven (RTFO) aged binder samples were prepared with 25 mm diameter and 1 mm gap parallel plate, and two replicates of each type of binder were tested. A variety of high temperatures from 76 °C for unaged binders and 70 °C for RTFO aged binders with a 6 °C increment were applied to binders until the compliance was fulfilled. The failure temperatures can be obtained according to AASHTO M320 [32], i.e., 1.0 kPa for unaged binder and 2.2 kPa for RTFO binder.

For J_{nr}, it was obtained by the multiple stress creep recovery (MSCR) test according to AASHTO MP19 [33]. In this test, only RTFO aged binder samples were prepared and two replicates of each type of binder were tested. A creep load and a temperature of 64 $^{\circ}$ C were applied to each sample (10 times at creep stress level of 0.1 kPa and 3.2 kPa respectively with 1 s loading and 9 s recovery).

3.2.2. Fatigue Resistance Tests

The intermediate-temperature performance was evaluated by the fatigue factor test. In this test, pressure aging vessel (PAV) aged binders were prepared with the 8 mm diameter and 2 mm gap parallel plate. From the test, $G^*sin\delta$ can be obtained, which is a parameter of the fatigue resistance of the binders. The test temperature started at 25 °C, and the test temperature was reduced by 3 °C each time, until the fatigue resistance factor was greater than 5000 kPa, the test was stopped [33]. Two replicates of each type of binder were tested.

3.2.3. Low-Temperature Performance Test

The low-temperature performance of bitumen binders was evaluated by two parameters, i.e., the creep stiffness and m-value, which were obtained by conducting a BBR test in accordance with AASHTO T313 [34]. In the BBR test, pressure aging vessel (PAV) aged binders were tested at temperatures of -6 °C, -12 °C, and -18 °C. Two parallel samples for each kind of binder were tested.

4. Results and Discussion

4.1. Foaming Conditions

Figure 1 shows the results of maximum expansion ratio (ER_m) and HL of SFB tested at three temperatures and three FEC levels. As can be seen from the figure, ER_m gradually increased with the increase of FEC, but at the same time the HL gradually decreased, indicating the gradual deterioration of the stability for foamed bitumen, which is detrimental to the effective dispersion and adhesion of foamed bitumen in the mixture. The test results imply that the properties were significantly sensitive to FEC. Specifically, at 160 °C, 170 °C, and 180 °C, as the FEC increased from 6% to 10%, the ER increased by 46%, 35%, and 20%, respectively, and the HL decreased by 75%, 73%, and 65%, respectively. Despite a 75% reduction of HL found at 10% FEC and 160 °C, the HL could still reach 17.5 s. It should be noted that the maximum HL of SFB could reach 69 s when 6% FEC was used, demonstrating the HL of foamed bitumen can be significantly increased by adding the surfactant, Evotherm-DAT. The maximum HL of SFB at 170 °C and 180 °C were 50 s and 48 s, respectively, at which point the minimum expansion ratio (ER_{min}) was 10.



Figure 1. The ERm and HL variations with FEC at different temperatures: (a) 160 °C; (b) 170 °C; (c) 180 °C.

The Wirtgen approach recommended the $\text{ER}_{\min} = 8$ and $\text{HL}_{\min} = 6$ [35]. In this research, a higher recommendation ($\text{ER}_{\min} = 10$ and $\text{HL}_{\min} = 12$) was taken into consideration according to the Council for Scientific and Industrial Research [36]. Nevertheless, both the ER_{\min} and HL_{\min} of 170 °C and 180 °C met the requirements, showing the positive effect of surfactant on improving HL and stability of SBS modified bitumen. However, at 160 °C, only 10% FEC met the requirements with the $\text{ER}_{m} = 12$, which is greater than the recommended value of 10. Hence, the optimum FEC was determined by taking the average of the two FEC required to meet the recommended values of ER_{\min} and HL_{\min} . Following this approach, as shown in Figure 1b,c, the optimum FEC was determined as 8% at 170 °C and 180 °C.

To further investigate the influence of temperature on the foaming properties, a line graph of ER_m and HL can be plotted at three temperatures (based on the data in Figure 1) in Figure 2 by using the ER_m as horizontal coordinate and the HL as vertical coordinates. It should be noted that upward line position indicates a better foaming condition, i.e., a greater ER_m value at the same HL level. Compared to 170 °C and 180 °C, the line position of 160 °C was lowest, indicating the worst foaming properties. At the same time, the foaming line at 170 °C was the highest, which showed the best foaming effect. Therefore, the analysis further proved that the bitumen temperature also had a significant influence on the foaming properties. It can be explained that a certain increase in bitumen temperature can transfer more heat energy to help the formation of vapors, which may make the bitumen to be foamed more efficiently. However, it was not the case that the higher the temperature, the better the foaming effect, because too high temperature can also be detrimental to the foaming properties. One of the reasons can be explained as that if the equilibrium



Figure 2. Relationship between ERm and HL of SFB at 160 °C, 170 °C and 180 °C.

Figure 3 shows the relationships between FEC and FI at different temperatures. As an indicator of foam characteristics, FI considers the combined effects of ER and HL of foamed asphalt, which is very suitable for judging the applicability of foamed asphalt in dispersing and mixing with mineral aggregates. Therefore, FI was often used as the target of characterizing and optimizing foaming properties, where higher FI represents better foam characteristics. As presented in Figure 3, the FI showed a continuous improvement with the increase of FEC when the temperature was 160 °C, whereas at 180 °C the opposite trend was found, the FI decreased as the FEC increased. The maximum FI of 321 was observed when the temperature was 170 °C and FEC was 8%. The FI at 170 °C shows the largest value at three different FECs, which are 314, 321, and 287, respectively. The results of FI were consistent with ER and HL due to the regular variations mentioned earlier. To sum up, the optimum foaming conditions for SFB were determined to be 170 °C foaming temperature and 8% FEC.



Figure 3. Relationship between FEC and FI at three temperatures.

4.2. Workability Analysis

The rotational viscosity is considered as one of the most significant parameters for characterizing the workability of bitumen binders. Generally, the lower the viscosity, the better the binder can provide processability with aggregates and better mixing and compaction capabilities. In the viscosity tests, all types of binders were tested at three temperatures, 135 °C, 155 °C, and 175 °C. The results of rotational viscosity tests are presented in Figure 4, showing that the viscosities of all binders decreased with the increase of temperature. As expected, both surfactant and Sasobit were effective in reducing the viscosities of SBS modified bitumen within a temperature range of 135-175 °C. The viscosity of both SFB and BE showed a downward trend by increasing the FEC from 6% to 10%, and SFB had slightly lower viscosities compared with BE at all levels of FEC, exhibiting better viscosity reduction. However, SFB did not show the expected significant reduction in viscosity because the correct measurement of fluid viscosity required a steady-state shearing motion, which is impossible for foamed bitumen that collapses over time [37]. As can be seen from Figure 4, it also demonstrated that the addition of 3 wt% of Sasobit can reduce viscosity dramatically. One reason should be attributed to the fact that the addition of Sasobit was much larger than the addition of water-free Evotherm-DAT. Another reason may be due to the type of additives. Based on research [7], it is found that organic WMA additives (such as Sasobit) mainly affect viscosity, while chemical WMA additives (such as Evotherm) change the lubrication/friction properties of the adhesive, even if the viscosity does not change; it can also improve the workability of asphalt mixtures.



Figure 4. Rotational viscosity test results.

4.3. High-Temperature Performance

Figure 5 shows the results of softening point tests, and it can be observed that all SFB, BE, and BS binders had higher softening point values than B, indicating better performances at high temperatures. In addition, with the increase in FEC, the softening point of SFB binders decreased and BE showed similar softening point values. However, the softening point values of SFB are slightly higher than those of BE. When FEC was 10%, SFB had the minimum value of 82.3 °C, which is still higher than the maximum value of 81.4 °C for BE when 6% Evotherm-DAT was added. Therefore, the synergistic effect of the foaming process and surfactant enhanced high-temperature performance. Apart from that, the organic WMA additives, Sasobit, showed a more significant effect on the rising softening point of binders compared with SFB and BE due to the higher amount added.



Figure 5. Test results: softening point.

The results of the rutting factor $G^*/\sin\delta$ were used to evaluate rutting resistance, and the higher value of $G^*/\sin\delta$ means the greater rutting resistance of the binder. The failure temperature is determined as the temperature value when $G^*/\sin\delta$ equals 1.0 kPa for unaged binder and 2.2 kPa for RTFO aged binder. Figure 6 shows the rutting factor results of unaged binders (a) and RTFO aged binders (b). As expected, the $G^*/\sin\delta$ decreased as the temperature increased, which is because bitumen binders softened and the viscosity reduced as the temperature increased. For unaged binders, SFB and BE had higher $G^*/\sin\delta$ values than B. With the content of Evotherm-DAT increasing, the $G^*/\sin\delta$ values of BE increased while the $G^*/\sin\delta$ values of SFB decreased (Figure 6a). However, with the exception of BS, SFB still had higher $G^*/\sin\delta$ values than BE, indicating the better rutting resistance, which is consistent with the results of softening point test. For RTFO aged binders, SFB and BE had lower $G^*/\sin\delta$ values than B. With the content of Evotherm-DAT increasing, the $G^*/\sin\delta$ values of both SFB and BE decreased (Figure 6b), indicating the decline of high-temperature performance. This is consistent with the findings in previous research [38].

The failure temperature test results are illustrated in Figure 6c, which shows the difference of failure temperatures between the binders before and after RTFO ageing. It can be seen from the figure that all binders presented lower failure temperatures after RTFO ageing. It is worth noting that SFB had a higher failure temperature than BE when the same Evotherm-DAT content was added before ageing, but after RTFO ageing, SFB had a lower failure temperature than BE, indicating that SFB is more negatively affected by short-term ageing. Among those binders, BS had the highest failure temperatures before and after RTFO ageing, with 95.2 °C and 87.2 °C respectively. Hence, in the current study, only Sasobit helped to further increase the failure temperature of B after RTFO ageing, indicating the best rutting resistance.

To further evaluate the high-temperature performance of the foamed and non-foamed bitumen including the elastic response and the change in elastic response, the MSCR test was also performed at two different stress levels (0.1 kPa and 3.2 kPa). The J_{nr} and percentage recovery (%R) of bitumen binders were then determined by the test. The maximum allowable J_{nr} difference is 75% according to the requirement of AASHTO tp70-13. The test results are summarized in Table 4. It can be seen that all binders except BS satisfied the maximum allowable J_{nr} difference, and it is because the J_{nr} values of BS at 0.1 kPa are extremely low [39]. According to the J_{nr} values at 3.2 kPa, the surfactant Evotherm-DAT had a negative influence on the high-temperature performance while Sasobit showed a positive effect in improving the performance. Additionally, the J_{nr} values of SFB at 3.2 kPa were higher than those of BE, showing the foaming process also had a negative effect on the high-temperature performance of the binders. However, the J_{nr} values of both SFB

and BE can still meet the requirements for the highest traffic level "E" [40]. The results are consistent with the rutting factor test, the J_{nr} values of both SFB and BE at 3.2 kPa increased with the increase of Evotherm-DAT content. Similarly, the BS exhibited the lowest J_{nr} value at 3.2 kPa, which indicates the best high-temperature performance among all eight binders.



Figure 6. High-temperature performances: (**a**) rutting factors for unaged binders; (**b**) rutting factors for RTFO aged binders; (**c**) failure temperature of unaged binders and RTFO aged binders.

Table 4.	MSCR test results.	

	Jnr		% Recovery (%)		
Sample ID	0.1 kPa (kPa ⁻¹)	3.2 kPa (kPa ⁻¹)	J _{nr} % Diff	0.1 kPa (%)	3.2 kPa (%)
В	0.133 ± 0.009	0.148 ± 0.002	11.9 ± 5.7	75.0 ± 0.2	73.3 ± 1.0
BS	0.015 ± 0.005	0.049 ± 0.013	230.2 ± 29.8	94.3 ± 1.7	86.1 ± 3.4
BE6	0.166 ± 0.006	0.182 ± 0.004	11.9 ± 1.8	77.8 ± 0.6	76.7 ± 0.2
EFB6	0.251 ± 0.009	0.294 ± 0.005	17.0 ± 2.2	69.0 ± 0.1	66.5 ± 0.6
BE8	0.176 ± 0.005	0.186 ± 0.002	3.6 ± 1.9	76.1 ± 0.5	76.6 ± 0.4
EFB8	0.255 ± 0.007	0.308 ± 0.011	20.1 ± 0.9	69.2 ± 1.6	66.0 ± 2.0
BE10	0.182 ± 0.004	0.197 ± 0.000	8.6 ± 2.5	78.0 ± 0.6	77.5 ± 0.3
EFB10	0.263 ± 0.011	0.319 ± 0.019	21.5 ± 2.4	69.8 ± 1.4	66.4 ± 2.0

4.4. Fatigue Resistance

The fatigue factor, $G^*sin\delta$, is commonly utilized to evaluate the fatigue properties of binders at intermediate temperature, and the long-term aged binders by RTFO and PAV

were prepared and tested. Figure 7a presents the relationship between the logarithms of $G^*sin\delta$ (lg $G^*sin\delta$) and temperatures. It is worth noting that the logarithms of $G^*sin\delta$ of all binders increase proportionally with the temperature decrease. The threshold temperature for all binders was less than 22 °C except for BS, which had a threshold temperature of less than 25 °C. Thus, surfactant had a positive effect on improving the fatigue resistance of the binder, while Sasobit had a notable negative effect. After long-term ageing, as can be seen in Figure 7a, the lg $G^*sin\delta$ value of SFB was slightly improved compared to B and the gap between the three SFB binders were not evident, which means the foaming process led to a similar fatigue behavior of SFB.



Figure 7. Fatigue performance: (a) fatigue factor versus temperature and (b) failure temperatures.

The failure temperature of each binder when fatigue factor G*sin δ equals 5000 kPa was also presented in Figure 7b. The lower the fatigue failure temperature means the better fatigue resistance of the binder. Hence, Figure 7 shows the surfactant content has a positive effect on the fatigue resistance of BE binders, but a negative effect was found in SFB binders. This is because the failure temperatures of SFB increased while that of BE decreased as the surfactant content increased at the same temperature. Among various binders, BE10 showed the best fatigue resistance, and the incorporation of 10 % Evotherm-DAT enhanced the fatigue resistance by decreasing 1.3 °C failure temperature. However, the BS showed the worst fatigue resistance, which noticeably impaired the fatigue resistance by increasing the failure temperature by 4.4 °C.

4.5. Low-Temperature Performance

The stiffness and m-value results obtained by the BBR test are presented in Figure 8. On the basis of AASHTO T313, the stiffness should not exceed 300 MPa while the m-value should be larger than 0.3 for one specific low-temperature grade. As shown in Figure 8, with the exception of BS, which only meets the requirements at -6 °C, the other binders achieved the requirements both at -12 °C and -6 °C. In the current test, lower stiffness and higher m-values can indicate better resistance to low-temperature cracking. It can be seen from the figure that with the temperature decrease, the stiffness increased, and the m-value decreased, which indicates that bitumen binder could be more prone to cracking or creeping at a lower temperature. Moreover, SFB and BE displayed an increasing trend in creep stiffness compared to B, which was detrimental to the cracking resistance. It can be concluded that the incorporation of surfactant exhibited a negative influence on the low-temperature performance, and the foaming process further exacerbated this negative effect. However, the surfactant and foaming process seemed to have no considerable effect on the m-value. As the surfactant content increased, the m-values of both SFB and BE increased slightly. In summary, the low-temperature performance of SFB and BE binders was lower than that of B, whereas BS exhibited the most significant negative effect on the low-temperature performance.





Figure 8. BBR test results: Creep stiffness and m-value of all binders at different temperatures.

5. Conclusions

This research investigated the synergistic effect of surfactant and foaming process on the foam characteristics and rheological properties of SBS modified bitumen. A series of laboratory tests were performed to evaluate and compare the foam characteristic and rheological properties of surfactant-foamed bitumen and non-foamed bitumen binders. On the basis of the experimental results, the following conclusions can be drawn:

- 1. The synergistic effect of surfactant and foaming process on the foam characteristics of SBS modified bitumen is significant; especially significant was the improvement in the half-life of foamed bitumen with the addition of surfactant.
- 2. Both foaming temperature and foaming Evotherm-DAT contents significantly influence the expansion ratio and half-life of foamed bitumen. However, the higher temperature did not show the better foaming effect. On the contrary, if the temperature is too high, it can also be detrimental to foaming properties. The optimum foaming conditions are finally determined when the foaming temperature is 170 °C and the Evotherm-DAT content is 8%.
- 3. The synergy of the surfactant and foaming process leads to a slightly lower viscosity than that of SBS modified bitumen, which could improve the workability of binders.
- 4. The combination of surfactant and foaming process enhances the high-temperature performance of SBS modified bitumen before ageing, but it also leads a negative effect on the high-temperature performance after RTFO ageing. For unaged binders, surfactant foamed bitumen had higher G*/sin δ values than SBS modified bitumen. However, for RTFO aged binders, surfactant foamed bitumen showed lower G*/sin δ values than SBS modified bitumen. With the content of Evotherm-DAT increasing, the G*/sin δ values of surfactant foamed bitumen canfurther decrease, indicating the decline of high-temperature performance.
- 5. The incorporation of surfactant improves the high-temperature performance and fatigue resistance. However, the increase of Evotherm-DAT content reduces the high-temperature performance and fatigue resistance. Compared with SBS modified asphalt, formed asphalt or Evotherm modified asphalt exhibits slightly lower resistance to low-temperature cracking, and BS exhibits the most significant negative impact on low-temperature performance.

6.

It can be also found in the study that the incorporation of Sasobit can significantly enhance the workability and high-temperature performance of SBS modified bitumen, but it also has noticeable negative effects on its fatigue resistance and low-temperature performance.

In general, the combination of surfactants and foaming technology is feasible to achieve foamed SBS modified asphalt with both high-level foaming characteristics and good road performance. However, this research only focused on the rheological properties of the foamed and non-foamed bitumen, and the forming mechanism or the further feasibility in producing mixtures is still unclear. Hence, future research will focus on the engineering performances of foamed asphalt mixture and the reaction mechanisms of formed bitumen.

Author Contributions: Data curation, S.Z. and G.L.; funding acquisition, H.Y.; investigation, G.L., S.X., and N.D.; writing—original draft preparation, G.L.; writing—review and editing, S.X., and H.Y. All authors have read and agreed to the published version of the manuscript.

Funding: The Natural Science Foundation of Guangdong Provinence [2020A1515110900], and The open funding of Research and Development Center of Transport Industry of Technologies, Materials and Equipment of Highway Construction and Maintenance, Gansu Road & Bridge Group [No. GLKF201801].

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

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

References

- 1. Cui, P.; Schito, G.; Cui, Q.B. VOC emissions from asphalt pavement and health risks to construction workers. *J. Clean. Prod.* 2020, 244, 118757. [CrossRef]
- 2. Kim, B.; Lee, H.; Park, H.; Kim, H. Framework for Estimating Greenhouse Gas Emissions Due to Asphalt Pavement Construction. *J. Constr. Eng. Manag.* **2012**, *138*, 1312–1321. [CrossRef]
- 3. Yu, H.; Deng, G.; Wang, D.; Zhang, Z.; Oeser, M. Warm asphalt rubber: A sustainable way for waste tire rubber recycling. *J. Cent. South. Univ.* **2020**, *27*, 3477–3498. [CrossRef]
- 4. Kheradmand, B.; Muniandy, R.; Hua, L.T.; Yunus, R.B.; Solouki, A. An overview of the emerging warm mix asphalt technology. *Int. J. Pavement Eng.* **2014**, *15*, 79–94. [CrossRef]
- Ebels, L.J. Characterisation of Material Properties and Behaviour of Cold Bituminous Mixtures for Road Pavements. Ph.D. Dissertation, Stellenbosch University, Stellenbosch, South Africa, March 2008.
- 6. Lu, G.Y.; Liu, P.F.; Wang, Y.H.; Faßbender, S.; Wang, D.W.; Oeser, M. Development of a sustainable pervious pavement material using recycled ceramic aggregate and bio-based polyurethane binder. *J. Clean. Prod.* **2019**, 220, 1052–1060. [CrossRef]
- 7. Ingrassia, L.P.; Lu, X.; Canestrari, F.; Ferrotti, G. Tribological characterization of bituminous binders with Warm Mix Asphalt additives. *Constr. Build. Mater.* **2018**, 172, 309–318. [CrossRef]
- 8. Ma, F.; Sha, A.M.; Lin, R.Y.; Huang, Y.; Wang, C. Greenhouse Gas Emissions from Asphalt Pavement Construction: A Case Study in China. *Int. J. Environ. Res. Public Health* **2016**, *13*, 351. [CrossRef] [PubMed]
- 9. Yu, H.; Zhu, Z.; Zhang, Z.; Yu, J.; Oeser, M.; Wang, D. Recycling waste packaging tape into bituminous mixtures towards enhanced mechanical properties and environmental benefits. *J. Clean. Prod.* **2019**, 229, 22–31. [CrossRef]
- 10. Yu, H.; Deng, G.; Zhang, Z.; Zhu, M.; Gong, M.; Oeser, M. Workability of rubberized asphalt from a perspective of particle effect. *Transp. Res. D-Transp. Environ.* **2021**, *91*, 102712. [CrossRef]
- 11. Jin, J.; Gao, Y.; Wu, Y.; Li, R.; Liu, R.; Wei, H.; Qian, G.; Zheng, J. Performance evaluation of surface-organic grafting on the palygorskite nanofiber for the modification of asphalt. *Constr. Build. Mater.* **2021**, *268*, 121072. [CrossRef]
- 12. Jin, J.; Gao, Y.; Wu, Y.; Liu, S.; Liu, R.; Wei, H.; Qian, G.; Zheng, J. Rheological and adhesion properties of nano-organic palygorskite and linear SBS on the composite modified asphalt. *Powder Technol.* **2021**, *377*, 212–221. [CrossRef]
- 13. Hailesilassie, B.W.; Jerjen, I.; Griffa, M.; Partl, M.N. A closer scientific look at foam bitumen. *Road Mater. Pavement* 2017, 18, 362–375. [CrossRef]
- 14. Kim, Y.; Lee, H.D. Development of mix design procedure for cold in-place recycling with foamed asphalt. *J. Mater. Civ. Eng.* **2006**, *18*, 116–124. [CrossRef]
- 15. Turk, J.; Pranjić, A.M.; Mladenovič, A.; Cotič, Z.; Jurjavčič, P. Environmental comparison of two alternative road pavement rehabilitation techniques: Cold-in-place-recycling versus traditional reconstruction. J. Clean. Prod. 2016, 121, 45–55. [CrossRef]

- 16. Sánchez, D.B.; Airey, G.; Caro, S.; Grenfell, J. Effect of foaming technique and mixing temperature on the rheological characteristics of fine RAP-foamed bitumen mixtures. *Road Mater. Pavement* 2020, *21*, 2143–2159. [CrossRef]
- 17. Saleh, M.F. Effect of Aggregate Gradation, Mineral Fillers, Bitumen Grade, and Source on Mechanical Properties of Foamed Bitumen–Stabilized Mixes. *Transp. Res. Rec.* **2006**, *1952*, 90–100. [CrossRef]
- 18. Saleh, M.F. Cost evaluation of foam bitumen and other stabilisation alternatives. Int. J. Pavement Eng. 2007, 8, 157–161. [CrossRef]
- 19. Bowering, R.H. Properties and behavior of foamed bitumen mixtures for road building. In Proceedings of the 5th Australian Road Research Board Conference, Canberra, Australia, 6–8 April 1970; pp. 38–57.
- 20. Yu, H.; Leng, Z.; Dong, Z.; Tan, Z.; Guo, F.; Yan, J. Workability and mechanical property characterization of asphalt rubber mixtures modified with various warm mix asphalt additives. *Constr. Build. Mater.* **2018**, 175, 392–401. [CrossRef]
- Kakar, M.R.; Hamzah, M.O.; Akhtar, M.N.; Woodward, D. Surface free energy and moisture susceptibility evaluation of asphalt binders modified with surfactant-based chemical additive. J. Clean. Prod. 2016, 112, 2342–2353. [CrossRef]
- Chomicz-Kowalska, A.; Mrugała, J.; Maciejewski, K. Evaluation of Foaming Performance of Bitumen Modified with the Addition of Surface Active Agent. In *IOP Conference Series: Materials Science and Engineering*; IOP Publishing: Bristol, UK, 2017; Volume 245, p. 032086. [CrossRef]
- 23. Yu, H.Y.; Zhu, Z.H.; Wang, D.Y. Evaluation and Validation of Fatigue Testing Methods for Rubberized Bituminous Specimens. *Transp. Res. Rec.* **2019**, *2673*, 603–610. [CrossRef]
- 24. Dong, F.Q.; Yu, X.; Wang, T.Y.; Yin, L.; Li, N.; Si, J.J.; Li, J. Influence of base asphalt aging levels on the foaming characteristics and rheological properties of foamed asphalt. *Constr. Build. Mater.* **2018**, 177, 43–50. [CrossRef]
- He, G.P.; Wong, W.G. Laboratory study on permanent deformation of foamed asphalt mix incorporating reclaimed asphalt pavement materials. *Constr. Build. Mater.* 2007, 21, 1809–1819. [CrossRef]
- Bowering, R.H.; Martin, C.L. Foamed bitumen production and application of mixtures evaluation and performance of pavements. In Association of Asphalt Paving Technologists Proc, Proceedings of Technical Session; Taylor & Francis Group: New Orleans, LA, USA, 1976; Volume 45, pp. 453–477.
- Yu, H.; Chen, Y.; Wu, Q.; Zhang, L.; Zhang, Z.; Zhang, J.; Miljković, M.; Oeser, M. Decision support for selecting optimal method of recycling waste tire rubber into wax-based warm mix asphalt based on fuzzy comprehensive evaluation. *J. Clean. Prod.* 2020, 265, 121781. [CrossRef]
- 28. Jenkins, K.J. Mix Design Considerations for Cold and Half-Cold Bituminous Mixes with Emphasis on Foamed Bitumen. Ph.D. Thesis, University of Stellenbosch, Stellenbosch, South Africa, 2000.
- 29. ASTM Standard D36. *Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus);* American Society for Testing and Materials: West Conshohocken, PA, USA, 2006.
- AASHTO Standard T316. Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer; American Association of State and Highway Transportation Officials: Washington, DC, USA, 2010.
- 31. AASHTO T315. Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR); American Association of State and Highway Transportation Officials: Washington, DC, USA, 2012.
- 32. AASHTO Standard M320. *Standard Specification for Performance-Graded Asphalt Binder;* American Association of State and Highway Transportation Officials: Washington, DC, USA, 2010.
- AASHTO Standard MP19. Standard Specification for Performance-Graded Asphalt Binder Using Multiple Stress Creep Recovery (MSCR) Test; American Association of State and Highway Transportation Officials: Washington, DC, USA, 2010.
- 34. AASHTO Standard T313. Standard Method of Test for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR); American Association of State and Highway Transportation Officials: Washington, DC, USA, 2012.
- 35. Muthen, K.M. Foamed asphalt mixes and mix design procedure. In Proceedings of the 7th Conference on Asphalt Pavements for Southern Africa, Johannesburg, South Africa, 7 June 1999.
- Hailesilassie, B.W.; Hugener, M.M.; Partl, N. Influence of foaming water content on foam asphalt mixtures. *Constr. Build. Mater.* 2015, 85, 65–77. [CrossRef]
- Ozturk, H.I.; Kutay, M.E. Novel Testing Procedure for Assessment of Quality of Foamed Warm Mix Asphalt Binders. J. Mater. Civ. Eng. 2014, 26, 04014042. [CrossRef]
- 38. Yu, H.; Leng, Z.; Zhou, Z.; Shih, K.; Xiao, F.; Gao, Z. Optimization of preparation procedure of liquid warm mix additive modified asphalt rubber. *J. Clean. Prod.* 2017, 141, 336–345. [CrossRef]
- 39. Yu, H.; Zhu, Z.; Leng, Z.; Wu, C.; Zhang, Z.; Wang, D.; Oeser, M. Effect of mixing sequence on asphalt mixtures containing waste tire rubber and warm mix surfactants. *J. Clean. Prod.* **2019**, 246, 119008. [CrossRef]
- 40. Yang, Z.; Zhang, X.; Zhang, Z.; Zou, B.; Zhu, Z.; Lu, G.; Xu, W.; Yu, J.; Yu, H. Effect of Aging on Chemical and Rheological Properties of Bitumen. *Polymers* **2018**, *10*, 1345. [CrossRef] [PubMed]