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Sustainable production of WMA with pine gum wax modification

İslam GÖKALP[a,*], Ramazan YANİ[b]

[a]*Batman University, Faculty of Engineering and Architecture, Department of Civil Engineering, Batman, Turkey*
[b]*Batman University, Institute of Graduate Studies, Department of Civil Engineering, Batman, Turkey*

Highlights

- Lower stripping resistance caused by the S® was increases by addiction of PGW
- PGW modified samples perform rutting resistance better than S®-WMA samples
- PGW modified S®-WMA reduced fatigue resistance more than S® samples
- Better low temperature cracking resistance performance by addiction of PGW

Abstract

Warm mix asphalt (WMA) (S®-WMA) produced with Sasobit® (S®), a widely used organic admixture, shows a significant increase in softening point value and a decrease in stripping resistance. These two characteristics of S®-WMA are considered as some problems to be solved. Therefore, this study was established to evaluate possible solutions to these two problems through another modification process. In this study, it was investigated whether modifying S®-WMA using a previously unstudied product, pine gum wax (PGW), could be a solution to the problem. In this context, WMA was produced with S® at 1, 2 and 3 in percent by mass of bitumen. As PGW has not been previously used as an additive to modify S®-WMA, it was added within a limit of 1% (by bitumen mass) for initial investigation. Physical and rheological standard tests were performed on each sample to demonstrate the change in properties of S®-WMA produced with 1% PGW compared to S®-WMA. The results indicated that the addition of 1% PGW to S®-WMA resulted in a significant reduction in softening point and an improvement in stripping resistance compared to S®-WMA. Thus, it seems that the use of PGW could be a potential solution for the two mentioned problems. It can also be pointed out that modifying the S®-WMA specimen with PGW without compromising its properties can help in an efficient, economical and environmentally friendly solution. However, due to the use of PGW, more in-depth research is required.

Information

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1. Introduction

Hot mix asphalt (HMA) generates high amounts of greenhouse gas emissions during the production and construction process, causing environmental pollution [1]. It also endangers occupational health and safety due to its high working temperature. During the production and construction processes, HMA requires high temperatures of 150 °C and higher and a significant amount of heat energy, usually derived from fossil fuels [2]. Environmental contamination caused by the high amount of emission gases from fossil fuels, a major cause of global warming, has motivated researchers to search for solutions for high-temperature HMA production. In addition, growing social environmental awareness and sensitivity has also accelerated this solution process. In this context, research scientists have long been interested in the production of HMAs at lower temperatures on the principles of energy efficiency, cost effectiveness and environmental friendliness [3]. Therefore, researchers have developed a new asphalt technology known as warm mix asphalt (WMA) for the production and construction of a sustainable, cost-effective, ecological and energy-efficient flexible pavement as well as providing technical advantages. Over time, the use of HMA technology is gradually being replaced by WMA technology [4].

WMA technology intends to improve workability by reducing viscosity and decrease fuel consumption by lowering the processing temperature. As compared to HMA, WMA enables processing at lower mixing and compression temperatures of 100-135 °C or lower [5]. Thus, it preserves the environment by minimizing greenhouse gas emissions and contributes to

*Corresponding author: islamgokalp@gmail.com (İ. Gökalp), +90 488 217 2156

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occupational health and safety by ensuring a lower working temperature in the workplace [6].

Nowadays, there are a number of widely used WMA technologies with different levels of efficiency for a common objective [4]. These are categorized as (1) chemical, (2) foaming and (3) organic hot mix technologies [7]. There are many additives developed for use in WMA production. Some of them are Revix, Evotherm, Cecabase RT, Interlow, ZycothermTM Rediset ® LQ for chemical techniques; Licomont ® BS, Asphaltan A, Asphalt B, Sasobit ® and Ecoflex for organic hot mix; and Advera®, Zeolite, Aspha-min for foaming [8]. The organic WMA technology used in this study is mostly used in the transportation sector [9, 10]. This technology aims to significantly decreases the viscosity of the binder by blending WMA additives with some modified bitumen or HMA [11-13]. In this technology, after the additive is added to the bitumen, it is crystallized during cooling, leading to a harder binder. The best known and commonly used organic additive used in this study is Sasobit® produced by the Fisher-Tropsch (FT) process [14], where FT is produced by a process using natural gas [15].

Instead of presenting a detailed individual literature summary, it is important to highlight the outputs of the review articles in which hundreds of studies are cited to provide a broad perspective on WMA characteristic characteristics. Some of the reviewed review papers can be illustrated as those presented by Cheraghian et al [16], Diab et al [17] Sukhija and Saboo [18], Prakash and Suman [19], Abdullah et al [20], Guo et al [21]. All of them presented the advantages and disadvantages of WMA technology and categorized the advantages into environmental, economic and technical categories. The use of WMA technology is mainly attributed to environmental benefits, which include the minimization of energy use and the amount of greenhouse gases due to the lower thermal treatment than with HMA. It has been reported that the use of WMA can result in a reduction of greenhouse gases (Carbon dioxide, Sulphur dioxide, Nitrogen dioxide, Carbon monoxide, etc.) from the use of fossil fuels by between 10% and 70% and the amount of energy used in WMA production can be up to 20% less than that used in HMA processes. A reduction in WMA production and coating construction costs of between 10% and 40% can therefore be achieved, depending on parameters such as technology, product type and quantity. Cost savings can be achieved by improving occupational health and safety by working at lower temperatures and then improving the performance of workers. Apart from the environmental benefits, there are also some technical benefits that can be expressed as the follows: (1) Enhanced workability throughout the entire construction phase. (2) Extending the construction season and enabling work in cold weather conditions. (3) Reducing the time required to open the pavement to traffic by enabling faster curing following pavement construction. (4) Permitting longer distances and transportation times between the construction site and bitumen plants due to low temperature operation; (5) Demonstrating a significant increase or decrease in binder and mixture performance.

Excessive increase in softening point and low moisture resistance are two problems for (S®-WMA). In the perspective of numerous existing studies [9, 22-31], solutions can be on the basis of modifying the WMA with supplemental additives. To address these problems, S®-WMA was prepared with S® with a 1% increase from 1% to 3% (by weight of bitumen). For the modification of S®-WMA, Pine Gum Wax (PGW) was used, which has not been used in any WMA preparation before. Apart from being an organic product like S®, the main reason behind the use of PGW is due to the fact that it exhibits some physical and mechanical properties similar to bitumen. Consequently, as a preliminary preparation for the use of PGW, only 1% (by weight of bitumen) was added to each S®-WMA produced at each S® ratio for modification purposes. Last but not least, the properties of the new S®-WMA were analysed through a series of physical and rheological test methods.

2. Material and Method

2.1. Material

In the production of S®-WMA and PGW modified S®-WMA specimens, 70/100 penetration grade base bitumen obtained from Batman refinery was used. Also, the aggregate used in this study to investigate the peel resistance of the produced WMA specimens was basalt. The reason for using basalt is its wide use in the region and the stripping resistance can be seen more clearly. Table 1 and Table 2 show the properties of the base bitumen and aggregate.

Table 1. Properties of bitumen

Test methods	Unit	Standard	Results
Penetration (at 25°C)	0.01 mm	EN 1426	85.7
Softening Point	°C	EN 1427	50.5
Flashing Point	°C	EN 22592-b	240.0
Ductility (at 25°C)	cm	EN 13589	103.8
Viscosity (135 °C)	cP	ASTM D4402	405.6
Viscosity (165 °C)			112.3
Solubility	%	EN 12592	99.4
Density	g/cm³	EN 1087	1.032

All the results are presented for each test specimen, taking into account a series of nomenclature according to the principle given below.

- Base Bitumen: BB
- Sasobit® S
- Pine Gum Warm Wax: PGW
- Modified Bitumen with S + X (%): S-X
- Modified Bitumen with PGW + X (%): PGW -X

- Modified Bitumen with combination of S and PGW + X (%): SPGW-X

where, X is the rate of Sasobit® as a percentage by mass of bitumen.

Table 2. Properties of bitumen

Test methods	Unit	Standard	Results
Water Absorption (Coarse)	%	EN 1097-6	2.22
Water Absorption (Fine)		EN 1097-6	8.79
Unit Weight (Coarse)	g/cm3	EN 1097-6	2.63
Unit Weight (Fine)		EN 1097-6	3.08
Unit Weight (Filler)		EN 1097-7	2.72
LA Fragmentation Resistance	%	EN 1097-2	18.95
Weathering Resistance		EN 1367-1	8.15
MD Abrasion Resistance		EN 1097-1	9.94
Polish Stone Value	PSV	EN 1097-8	56.79
Flakiness Index	%	EN 933-3	11.92
Mytilene Blue	MB	EN 933-9	1.75
Stripping Test	%	EN 12697-11	40-45

3. Method

3.1. Physical test methods

Within the scope of physical test methods, penetration, softening point, rotational viscometer tests and Nicholson stripping tests were performed on bitumen samples. In addition, the thermal sensitivity of each sample was determined using the penetration index developed based on the penetration and softening point test results.

Penetration test method: The test helps researchers to ensure adequate consistency of bituminous binder. It is used to determine the hardness or consistency of bitumen. The EN 1426 standard [32] was followed to perform the test on test samples in this study.

Softening point test method: The test helps researchers to ensure adequate fluidity before it is used for a variety of road applications. It is used to determine the temperature at which the bituminous binder reaches a certain softening phase. EN 1427 standard [33] was followed to test samples in this study.

Rotational viscometer test method: The test helps researchers to measure the apparent viscosity of bituminous binders at different temperatures. The test is applied under controlled temperature provided by thermal chamber and rotational rate. The tests were conducted on the samples according to ASTM D 4402 standard [34]. Considering the viscosity data provided at different temperatures, usually 135 and 165°C, it is possible to estimate the mixing and compaction temperatures of asphalt binders. In this study, viscosity tests were carried out for each unaged sample at the specified temperatures. The viscosity values defined for the determination of mixing and compression temperatures of bitumen samples are 170 ± 20 centipoise (cP) and 280 ± 30 cP, respectively [35].

Nicholson stripping test method: This test is based on coating a defined size and quantity of aggregate with a certain amount of bitumen and keeping the test specimens prepared in this way in water at a constant temperature for a certain period of time to determine whether they strip or not by visual analysis. The EN 12697-11 standard [36] was followed for implementing the test in this study.

3.2. Rheological test methods

The rheological properties of bituminous binders are presented in the Superior Performance Asphalt Pavements (SuperPAVE) system developed by the Strategic Highway Research Program [37]. Within the scope of testing defined in the SuperPAVE system, there are test methods that measure the rheological properties of bitumen samples. These test methods are as follows: (1) Dynamic Shear Rheometer (DSR), (2) Bending Beam Rheometer (BBR), (3) Rolling Thin Film Oven (RTFO) for short-term aged samples and (4) Pressure Aging Vessel (PAV) for long-term aged samples [38, 39].

Dynamic Shear Rheometer-DSR: It is one of the main machine systems used in SuperPAVE. The test is performed within a uniquely designed logical framework and analyzed with a dedicated software program. The test is performed on unaged, short- and long-term aged bitumen samples. Complex shear modulus (G*) and phase angle (δ°) are the two parameters obtained from the test. The phase angle means that bitumen specimens are elastic as they approach 0° and viscous as they approach 90°. The interrelationships developed between G* and δ° provide information on the structural performance of the bituminous binder, including rutting and fatigue resistance. The rutting factor is determined by G*/Sin(δ°), while the fatigue factor is calculated by G*. It is calculated with Sin(δ°). The SuperPAVE system determines limit values taking into account different aging conditions. These limit values are defined as 1000 Pa (1 kPa) and 2200 Pa (2.2 kPa) in the G*/Sin(δ°) for non-aged and short-term aged specimens, respectively, and 5 × 106 Pa (5000 kPa) for G*.Sin(δ°) for long-term aged specimens. Testing of DSR was performed on the specimens in accordance with EN 14770 [40].

Bending Beam Rheometer-BBR: The system of the machine is used to determine low temperature properties through thermal cracking and relaxation. The test is applied to long-term aged bitumen samples. The BBR test has two test parameters, creep stiffness (St) and m-value. St is the quotient of the bending stress and bending strain under the specified load, while m-value is the slope of the creep stiffness at 60 seconds. As with the DSR, the limit values are defined as a maximum of 300 MPa for St and a minimum of 0.300 for m-value. The test temperature that satisfies both two limit values is labelled low temperature resistance or thermal cracking. Testing is performed on specimens following the EN 14771 standard [41].

Table 3. Physical test result

Test method	BB	S-1	S-2	S-3	PGW	SPGW-1	SPGW-2	SPGW-3
Penetration (0.01. mm)	85.7	58.1	49.5	49.0	81.5	58.0	51.8	50.8
Softening Point (℃)	50.5	62.5	67.1	69.0	51.4	61.8	65.6	67.7
Penetration (dmm)	70.2	50.0	44.8	30.6	45.7	36.3	35.7	29.2
Softening Point (℃) (After short-term Aging)	59.3	65.5	69.6	71.0	62.0	62.3	67.7	69.2
Mass Loss (%)	0.45	0.25	0.30	0.40	0.60	0.71	0.43	0.60

Rolling Thin Film Oven-RTFO: Bitumen during mixing, transportation and compaction undergoes some aging process, referred to as short-term aging. It is possible to simulate aging under laboratory conditions using the Rolling Thin Film Oven (RTFO) test method. The test system consists of a specially designed rotary oven, standard open-end glasses and an air supply compressor. The testing occurs under a defined speed and temperature by blowing hot dry air from the open end into the specimen, which exposes the specimens spread as a thin film on the glass. The test is conducted according to the EN 12607-1 standard [42].

Pressure Aging Vessel-PAV: Long-term aging is the aging process occurring from the production of bitumen samples until the end of their service life. As with RTFO, it is possible to collect long-term aged bitumen samples under laboratory conditions. The testing systems consist of a container, a dry air supply compressor and a set of sample plates. The test can be at 90, 100, 110 °C under a pressure of 2.1 kPa. In this study, the testing was carried out according to EN 14769 standard [43].

3.3. Modification process

The bitumen modification procedure below was determined in the light of previous studies [9, 17, 26-29, 44-46].

Step 1. The bitumen needs to be liquefied and in this respect the base bitumen was heated at 160 ± 5 °C for 60 minutes.
Step 2. 500 grams of heated bitumen was then transferred into metal cans.
Step 3. The cans (filled with bitumen samples) were placed one by one on a heater running at 140 ± 5 °C.
Step 4. S® and PGW additives were mixed into the bitumen in the specified proportion and mixed for 30 minutes with a propeller mixer at 500 rpm.
Step 5. The modified bitumen was mixed again for the next 10 minutes with the mixer running at 100 rpm to ensure successful bitumen modification through homogeneity.
Step 6. Finally, the modified bitumen samples were transferred for testing.

4. Results and Discussion

4.1. Physical test methods

Results of physical tests of bitumen including penetration, softening point tests and analysis of the penetration index based on these tests. Also, the mass loss of the base and short-term aged specimens is presented in Table 3.

From the results shown in Table 3, the penetration value of the specimens decreased as the S® content increased significantly. Though the addition of PGW at 1% (by weight of bitumen) decreases the penetration, the rate of decrease is much lower than that with added S®. Together, the use of both additives in the bitumen resulted in a slight increase in the penetration of the samples. However, the addition of PGW to the base bitumen increases the softening point value. PGW addition, compared to the other, results in a significant decrease. This prevents the excessive rise of the softening point observed in the S®-WMA specimens. For all specimens, the mass loss after short-term aging is below the allowable limit of 1%. Short-term aging leads to an increase in hardness, which results in a decrease in the penetration value and an increase in the softening point value.

The rotational viscosity test results were determined at two temperatures, 135℃ and 165℃, and the calculated mixing and compression temperature ranges are given in Table 4.

Table 4. Viscosity and mixing-compaction temperature results

Samples	Viscosity (cP) 135 °C	Viscosity (cP) 165 °C	Mixing Temperature (℃)	Compaction Temperature (℃)
BB	405.6	112.3	157.1-161.1	144.8-150.9
S-1	345.4	101.1	154.1-159.0	139.3-146.7
S-2	284.8	95.5	150.0-156.4	131.0-140.5
S-3	252.5	81.6	146.0-153.0	124.9-135.4
PGW	366.4	100.8	154.9-159.4	141.4-148.1
SPGW-1	309.6	96.1	151.8-157.4	134.9-143.4
SPGW-2	344.8	89.5	153.2-157.9	139.1-146.1
SPGW-3	355.6	88.3	153.6-158.1	140.1-146.9

As mentioned earlier, the secondary motivation that led to the establishment of this work was to overcome the problem of stripping of S®-WMA specimens. To overcome this, 1% of PGW was added to S®-WMA due to some of the inherent properties of the bitumen such as stickiness, oily structure and temperature sensitivity. The stripping resistances of both S®-WMA and SPGW bitumen samples are reported, and the results and post-test sample images are shown in Figure 1. One can see from Figure 1 that the S® modification reduces the stripping resistance of the base bitumen as the rate increases. On the other hand, the addition of PGW significantly increases the stripping resistance of the base bitumen.

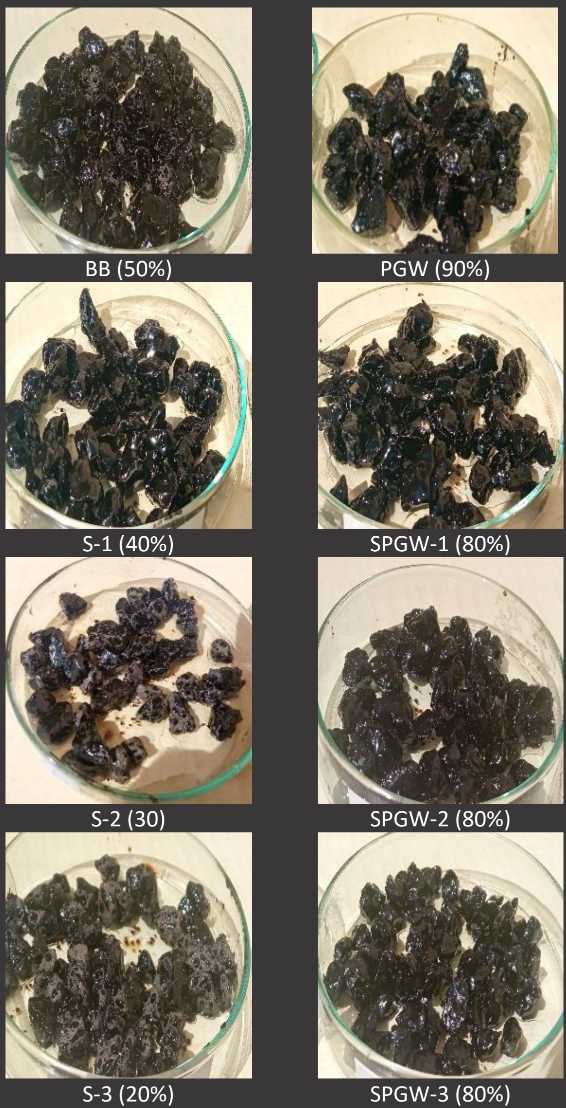

Figure 1. Nicholson stripping resistance results

4.2. Rheological test methods

Under constant and/or variable loading and high temperature conditions, the prepared untreated, i.e. unaged, short-term and long-term aged specimens were analysed by DSR. Furthermore, the long-term aged specimens are subjected to BBR tests at the specified temperature. Besides, RTFO and PAV tests are used to provide short- and long-term aged specimens, respectively. DSR test results for all specimens under different aging conditions are given in Figures 2 to 4.

The DSR test results presented in Figures 2 and 3 show that the rutting resistance of the bitumen samples increased with S® modification. As the S® content increases, the rutting resistance also increases. In this sense, the highest value was determined for the highest content ratio in the additive, whereas the addition of PGW decreases the rutting resistance. The lowest rutting resistance performance of SPGW bitumen samples is seen for SPGW-1.

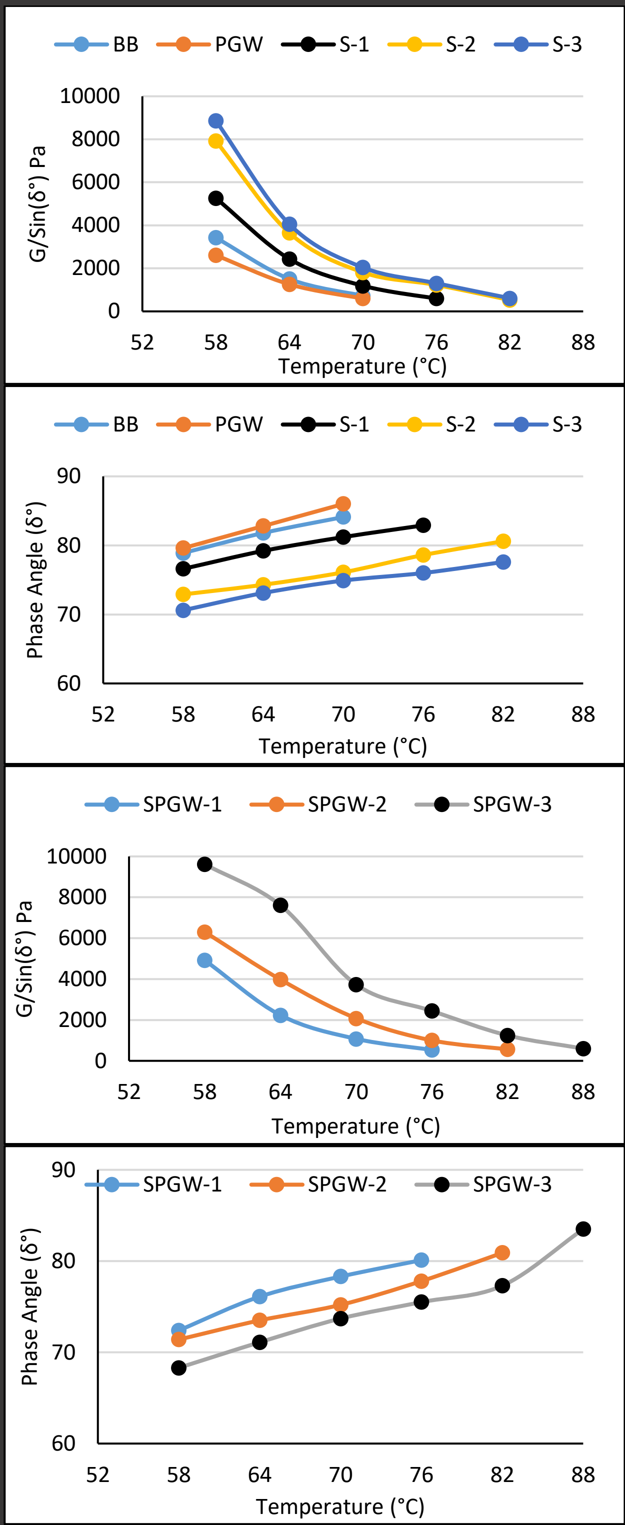

Figure 2. DSR results for unaged bitumen samples

The DSR results given in Figure 4 show the fatigue resistance of the specimens. The fatigue resistance of the modified bitumen decreases with the addition of S® and this becomes more pronounced as the rate increases. There is, however, a modification that does not significantly change the performance of the PGW modified one. The reduction in the fatigue performance of the bitumen, on the other hand, is more pronounced for the SPGW specimens.

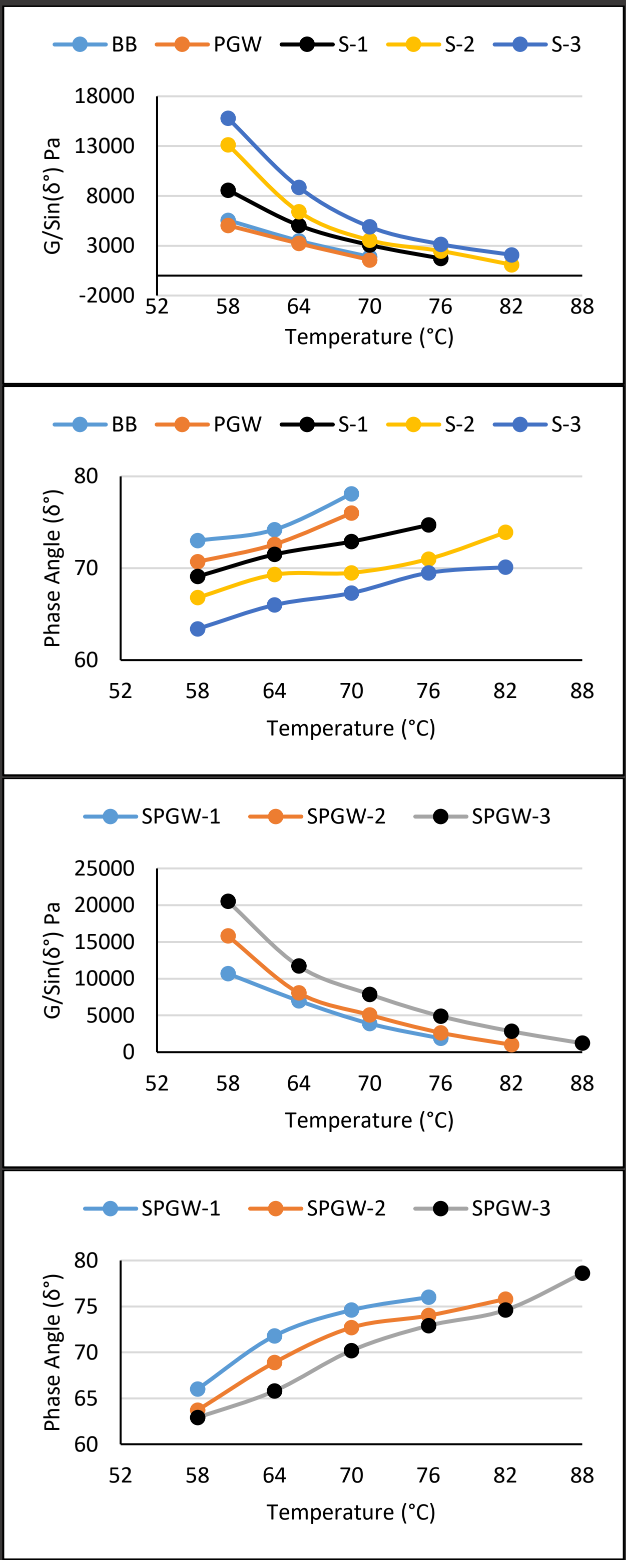

Figure 3. DSR results for short-term aged bitumen samples

For the whole study, BBR tests were only carried out at -18°C. Because, the temperature at which all specimens satisfy the requirements (the limit values are defined as a maximum of 300 MPa for St and a minimum of 0.300 for m-value) as specified in the SuperPAVE system. Therefore, no further BBR testing at different temperatures was required to ensure an adequate test temperature that met both parameters. It is seen from the test results presented in Figure 5 that all specimens met the minimum St and m-value criteria.

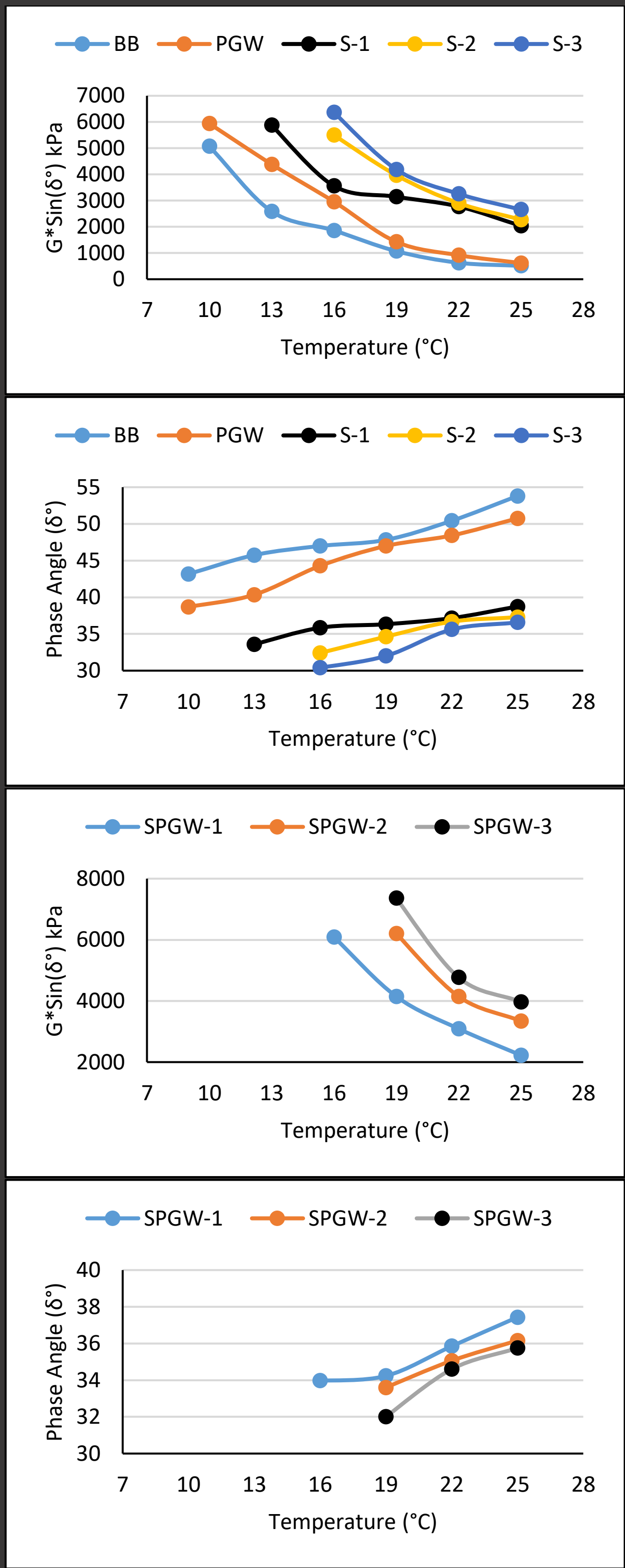

Figure 4. DSR results for long-term aged bitumen samples

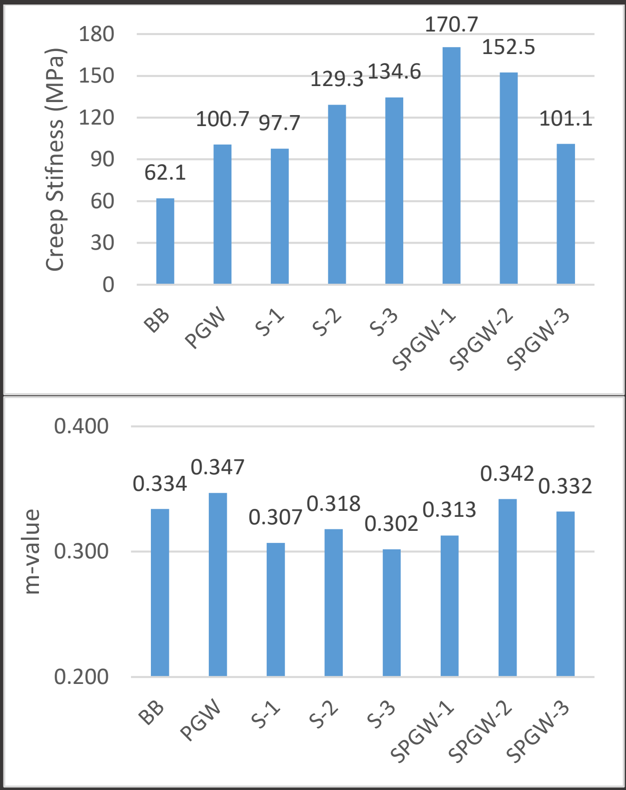

Figure 5. BBR test results

The classification based on the SuperPAVE system is determined by the degradation temperatures determined by the DSR test applied to unaged and short-term aged bitumen samples, as well as the temperature determined by the BBR test applied to long-term aged samples. In this context, Table 5 is provided to illustrate the SuperPAVE-based classification of each bitumen sample.

Table 5. SuperPAVE classification for bitumen samples

Samples	High temperature Grade	Low Temperature Grade	SuperPAVE Classification
BB	64	- 18	64-18
S-1	70	- 18	70-18
S-2	76	- 18	76-18
S-3	76	- 18	76-18
PGW	64	- 18	64-18
SPGW-1	70	- 18	70-18
SPGW-2	76	- 18	76-18
SPGW-3	82	- 18	82-18

5. Conclusions

In the present study, a possible solution to the two common problems of S®-WMA bitumen (excessive increase in softening point and decrease in peel resistance) was investigated. In this context, S®-WMA bitumen samples prepared with different proportions of Sasobit® additives were modified using 1% PGW, a commercial industrial product that has not been studied before, as far as can be seen from the available literature. To this end, Sasobit® additives were used in 1% increments up to 3% (by bitumen weight) to modify 70/100 penetration base bitumen for WMA production. A series of physical and rheological properties characteristics of the bitumen samples were determined by the standard test methods. Consequently, the following can be pointed out.

- A significant decrease in penetration results occurred with the S® modification, but the decrease in penetration was realized with the addition of PGW.
- The softening point showed an excessive increase with the S® modification, but a significant decrease in softening point was obtained with the addition of PGW.
- The low stripping resistance caused by the S® modification increased with the addition of PGW.
- The specimens with PGW showed less rutting resistance than the S®-WMA specimens.
- The fatigue resistance of PGW modified S®-WMA decreased more than that of Sasobit® modification.
- The low temperature cracking resistance performance of SPGW specimens is better than that of S®-WMA specimens.

Altogether, this study shows that adding 1% PGW additive to S®-WMA can help to produce an innovative, effective, environmentally friendly and sustainable product. Overall, this study shows that the addition of 1% PGW additive to S®-WMA may help an innovative, effective, environmentally friendly and economical solution to overcome the excessive increase in softening point and low stripping resistance.

From existing studies, it can be seen that PGW has not been used before in WMA production, especially not in WMA produced with S®. For this reason, as a preliminary study, the PGW utilization rate was kept low at 1%. As such, the scope of future studies may include the use of PGW at higher rates. The analyses in the study were limited to a series of physical and rheological tests and analysis methods. It is suggested that the study be expanded in the future to include chemical, image processing and/or microscopic analyses to gain a deeper understanding of the relationship between the materials. Furthermore, the production of WMA blend samples was beyond the scope of the present study. Therefore, future studies are needed to investigate the structural and functional performance of WMA blend samples within the parameters defined in this study.

Declaration of Interest Statement

The authors declare that they have no known competing for financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contribution Statement

İ. Gökalp: Conceptualization, Formal analysis, Methodology, Resources, Supervision, Validation, Writing – Original Draft, Writing – Review & Editing; **R. Yani:** Data curation, Formal analysis, Investigation, Resources, Visualization, Writing – Original Draft.

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