

# Investigation of the Effects of Different Modified Asphalts on Storage Stability

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**Abstract** - In this study, the storage stability of modified bitumen prepared with bitumen additives styrene-butadiene-styrene (SBS) and American Gilsonite (AG) was evaluated. For this purpose, modified bitumen was prepared by using different ratios of each additive type. The prepared modified bitumens were subjected to storage stability test according to EN 13399 standard. Penetration, softening point and rotational viscometry tests were performed on the samples subjected to storage stability test. As a result of the penetration and softening point tests of the samples prepared with SBS after storage stability, it was determined that the penetration and softening point values of the samples obtained from the upper part of the tube were higher. This is thought to be due to the accumulation of SBS, which has a lower specific gravity than bitumen, in the upper part of the tube during the storage stability test. Considering the viscosity test results, it was determined that the viscosity values of the modified bitumen samples obtained from the upper part of the tubes were higher than the samples obtained from the lower part of the tubes.

**Keywords:** Storage stability, Bitumen, Modification, Styrene-butadiene-styrene, American Gilsonite.

## 1. Introduction

Bituminous hot mixtures (BSK), which are used on medium and heavy traffic roads to carry traffic loads and to protect other layers in the superstructure from the negative effects of environmental conditions, are obtained by heating and mixing bitumen and aggregate. Bituminous hot mixtures have a composite structure consisting of aggregate, bituminous binder and air space dispersed in the mixture. Depending on the material, the properties of bituminous hot mixes vary according to the use of different binder-aggregate combinations, aggregate gradations and modification techniques [1].

Deterioration such as rutting, moisture damage, low temperature and fatigue cracks occur in bituminous hot mixtures due to traffic and environmental conditions [2]. Additives are used to extend the service life of bitumen and bituminous hot mixtures by increasing their resistance and performance against heat and traffic loads [3].

In practice, modified bitumen is prepared in a separate plant and then pumped to the main plant, mixed with aggregate and bituminous hot mixtures containing modified bitumen are prepared. Although it is desired to consume the prepared modified bitumen on the same working day, it is necessary to store the modified bitumen as a result of problems encountered in practice such as technical failures that may occur in other equipment [4]. Due to storage, decomposition may occur in the modified bitumen in the plant. When such a situation is encountered, storage stability test is performed according to EN 13399 standard in order to determine how the rheological structure of modified bitumen will change.

In this study, the storage stability of modified bitumen prepared with one type of polymer (SBS) and one type of natural asphalt (AG) was investigated. Penetration, softening point and rotational viscometry tests were used to evaluate the storage stability of the binders. Thus, it was tried to determine the most suitable additive in terms of storability.

## 2. Materials and Methods

In the study, PG 52-28 class pure binder obtained from Batman TÜPRAŞ refinery was used as the main binder. SBS and American Gilsonite were used as additives. SBS (Kraton D 1101) produced by Shell Company was used as an additive. The specific gravity of gilsonite varies between 1.03 and 1.10 and its hardness is 2 according to Mohs scale. It

contains 10-20% fixed C, 0-2% O<sub>2</sub> and trace mineral matter. Melts between 120°C and 175°C. The degree of solubility in carbon sulphide is 98-100%. Penetration is 0. The features that distinguish gilsonite from other natural asphalts are its high asphaltene content, high solubility in organic solvents, high purity, high molecular weight and high nitrogen content [5].

### 2.1. Preparation of Modified Asphalt

According to the previous study, it was decided to use 2%, 3%, 4% and 5% SBS (MB5%SBS), 18% American Gilsonite (MB18%AG), 2% SBS + 13% AG (MB2%SBS+13%AG), 3% SBS + 10% AG (MB3%SBS+10%AG) and 4% SBS + 6% AG (MB4%SBS+6%AG).

First, the pure asphalt was heated in a vacuum furnace at  $180 \pm 5$  °C for 30 min and rendered fluid. The fluidized asphalt specimens were poured into the metal chamber of the mixer in 500 g. In order to provide homogeneous thermal sources, the asphalt specimens poured in the metal chamber were put down in the thermal jacket on the heater, which was conditioned at  $180 \pm 5$  °C. Then, the polymers were poured into the hot asphalt at the defined percentage by weight of the asphalt. Finally, asphalts with polymers were created by mixing with a mechanical mixer at 1000 rpm for 1 h. In addition, the mixing procedure was employed for the pure binders to eliminate the oxidation effect [6].

### 2.2. Storage Stability Test

In this experiment, homogeneously prepared modified bitumen is kept in a vertical tube at 180°C for 3 days. After cooling, the sample is cut into three equal parts. The top and bottom parts of the tube are analysed in order to better evaluate the differences of the sample due to phase separation.

The tubes to be used in the experiment must be made of unpolished aluminium. They should be at least 160 mm in length, 30-40 mm in diameter and closed on one side. These tubes are typical toothpaste tubes. The oven in which the tubes will be placed must be able to maintain a temperature of  $180 \pm 5$  °C for 3 consecutive days. The heated and homogeneously mixed sample is filled into the tube at a height of 100-120 mm. Air bubbles should be avoided during the filling process. After the sample is filled into the tubes, the tube should be tightly sealed to prevent any reaction of the sample with air during storage at high temperature. The tube should be slightly compressed due to the possibility of thermal expansion. Once the tubes are filled, they are placed upright in the preheated oven and stored upright in the oven at  $180 \pm 5$  °C for  $72 \pm 1$  hours.

After the tubes are removed from the oven, they are cooled in the same upright position and at room temperature. The aluminium tube is then removed to obtain the modified bitumen sample. The cylindrical sample removed from the tube is placed horizontally on a clean surface and cut into 3 equal parts with a heated cutter (Figure 2.1). The top and bottom pieces are marked and stored in separate containers, while the middle piece is not used. Binder tests are performed on the obtained samples to evaluate the storability of the additives.



Fig. 2.1: Removal of the tested samples from the tubes.

### 2.3. Penetration Test

Penetration is the vertical penetration length of a standard needle into a binder at a certain temperature under a certain load and within a certain time. The unit of penetration is 0.01 cm. Each division on the penetration device

display is equal to 0.1 mm. For bituminous binders with a penetration value up to 500, a load of 100 g is applied to the sample at 25°C for 5 seconds.

### 2.4. Softening Point Test

The softening point test is an experiment to measure the sensitivity of asphalt cements to temperature. The softening point is the temperature read from the thermometer at the moment when the softened material touches the base by heating the bituminous material in a standard ring with a ball on it, placed in a water bath at a certain speed. In the experiment, 2 standard rings made of brass, 2 standard steel balls, a heat-resistant 600 or 800 cm<sup>3</sup> glass beaker with an inner diameter of 8.5 cm and a height of not less than 12 cm and a thermometer are used.

### 2.5. Rotational Viscometer (RV) Test

The rotational viscometer test is used to measure the fluidity of the asphalt binder at high temperatures to determine the workability and pumpability of the binder in the hot mix plant. Some asphalt organisations refer to this measurement as "Brookfield viscosity". The method of measuring Brookfield viscosity of bituminous binders using Brookfield thermocell instrument is described and detailed in ASTM D 4402 standard [7-10]. According to the Superpave specification, the Pascal-second (Pa.s) unit is used in binder classification and the rotational viscosity value of the binder measured at 135°C should not exceed 3 Pa.s (3000 cP) [7-9].

## 3. Results and Discussion

After the storage stability test, penetration, softening point and viscosity tests were performed on the binders.

### 3.1. Penetration Test Results

As a result of the storage stability tests applied to the modified binders, the penetration test results of the samples obtained from the bottom of the tubes are given in Figure 3.1 and the penetration test results of the samples obtained from the top of the tubes are given in Figure 3.2. The penetration test results of all specimens are shown in Figure 3.3.

As can be seen in Figure 3.3, the penetration values of the samples obtained from the top of the tubes with the use of additives were higher than the bottom of the tubes. As the SBS content increased at the bottom of the tubes, the penetration value decreased and therefore the consistency increased. In the upper part of the tubes, the penetration values varied between 85-96. In addition, the penetration values of binders containing 3%, 4% and 5% SBS were higher than 2% SBS modified bitumen. It was determined that the penetration values of the sample obtained from the top of the tube after storage stability in 2%, 3%, 4% and 5% SBS modified bitumen were 1.38, 1.84, 2.73 and 3.47 times higher than the penetration values of the sample obtained from the bottom of the tube, respectively. This situation shows that as the SBS content increases, the separation between bitumen and polymer increases in terms of storage stability.

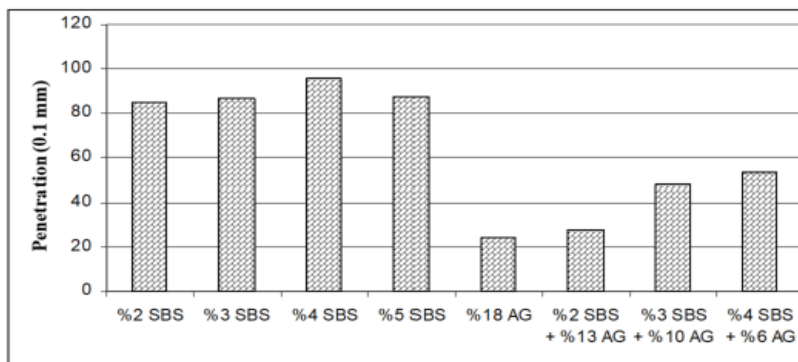


Fig. 3.1: Penetration test results of samples obtained from the top of the storage tubes.

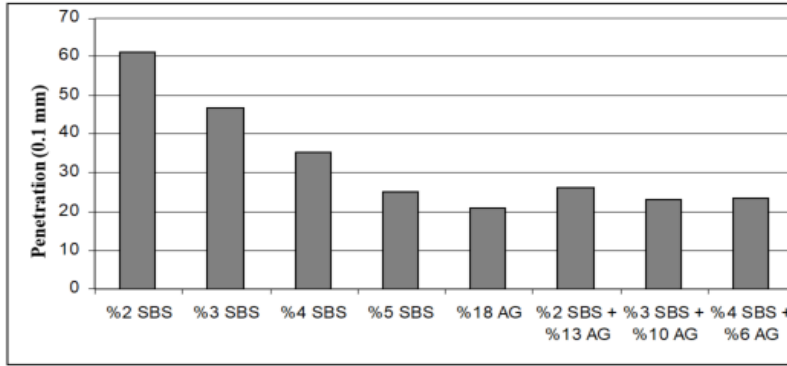


Figure 3.2: Penetration test results of samples obtained from the bottom of the storage tubes.

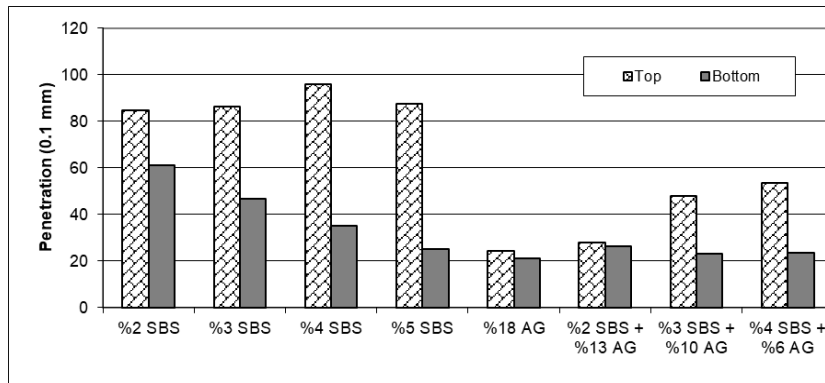


Fig. 3.3: Penetration test results after storage stability.

### 3.2. Softening Point Experiment Results

The softening points obtained from the top of the tubes as a result of the storage stability tests on different modified bitumen are given in Figure 3.4 and the samples obtained from the bottom of the tubes are given in Figure 3.5 All softening point experiments are given in Figure 3.6.

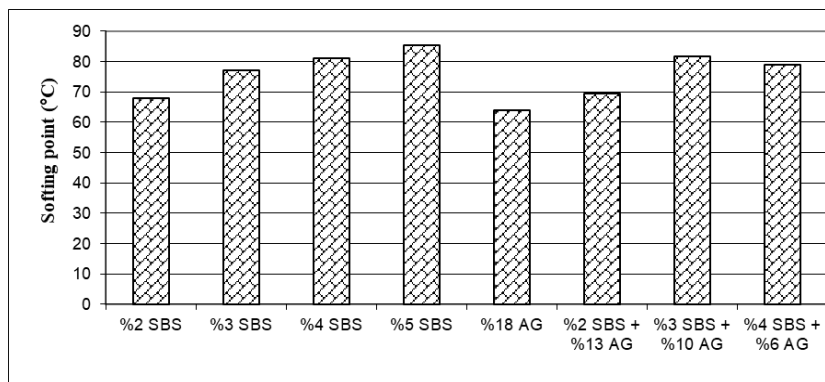


Fig. 3.4: Softening point test results obtained from the top of the tubes as a result of storage stability tests.

As seen in Figure 3.5, the softening point values of the samples obtained from the upper part of the tubes with the use of additives were higher than the lower part of the tubes. As the SBS content increased at the bottom of the tubes, the softening point values increased. Since the SBS density of the binder was higher in the upper part of the tubes, the softening point values increased more. In addition, the softening point values of binders containing 3%, 4% and 5% SBS were higher than 2% SBS modified bitumen. It was determined that the softening point values of the sample obtained from the top of the tube after storage stability in 2%, 3%, 4% and 5% SBS modified bitumen

were 1.34, 1.42, 1.44 and 1.42 times higher than the softening point values of the sample obtained from the bottom of the tube, respectively.

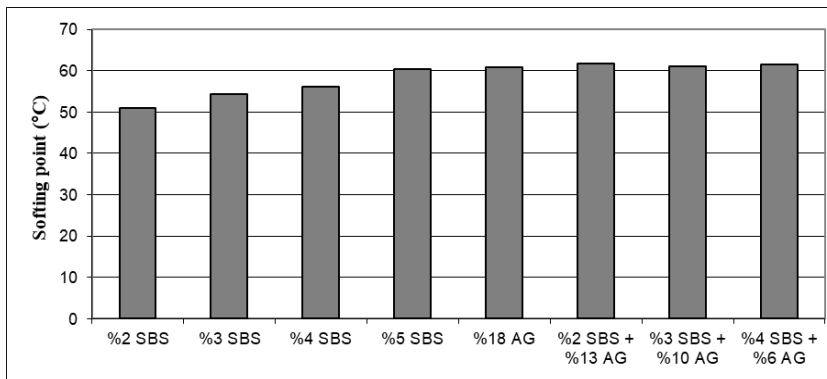


Fig. 3.5: Softening point test results obtained from the bottom of the tubes as a result of storage stability tests.

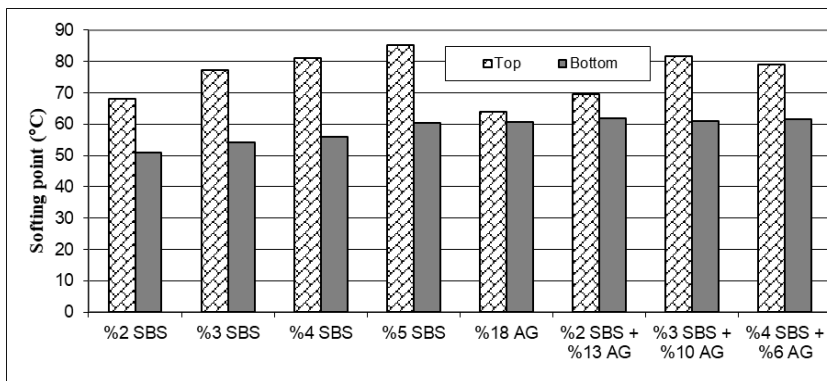
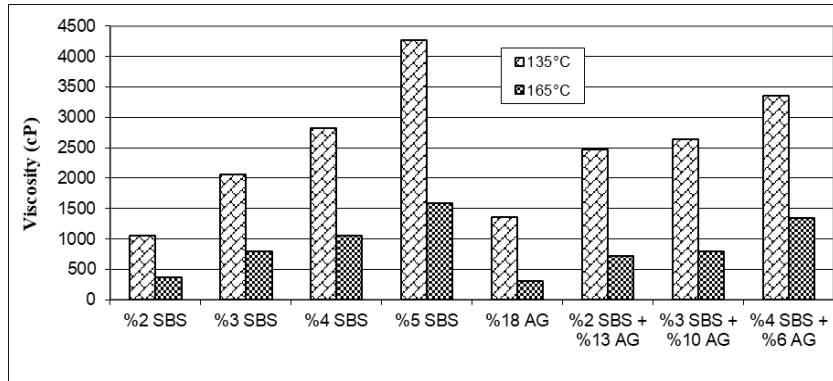


Fig. 3.6: Softening point test results.

AG modified bitumen was the most stable binder in terms of softening point values in terms of storage among all modified bitumens. It was determined that the softening point values of the sample obtained from the upper part of the tube were higher than the lower part. When the binders containing SBS and AG and having the same performance level with the modified bitumen containing 5% SBS (2%SBS+13%AG, 3%SBS+10%AG and 4%SBS+6%AG) were compared, it was determined that the difference between the softening point values after storage stability increased as the SBS content increased in the modified bitumen containing SBS and AG, but the difference between the softening point values of all SBS and AG containing binders decreased compared to the use of 5% SBS. In modified bitumen containing SBS and AG, the binder containing 3% SBS and 10% AG gave the best result in terms of softening point at the top of the tube. In modified bitumen containing 2%SBS+13%AG, 3%SBS+10%AG and 4%SBS+6%AG, the softening point values of the samples obtained from the top of the tubes after the storage stability test were 1.12, 1.34 and 1.29 times higher than the softening point values of the samples obtained from the bottom of the tubes, respectively. When compared with modified bitumen containing 5% SBS, it was determined that the softening point values of modified bitumen prepared with 2%SBS+13%AG, 3%SBS+10%AG and 4%SBS+6%AG decreased by 0.79, 0.94 and 0.91 times, respectively, compared to modified bitumen containing 5% SBS.

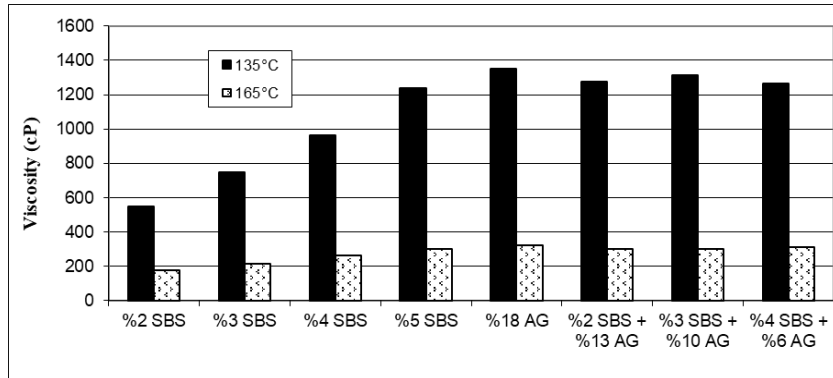
### 3.3. Viscosity Test Results

As a result of the storage stability tests performed on different modified bitumens, the results of the rotational viscosimetry tests obtained from the top of the tubes are given in Figure 3.7 and the test results of the samples obtained from the bottom of the tubes are given in Figure 3.8. All of the rotational viscosimetry test results are given in Figure 3.9.



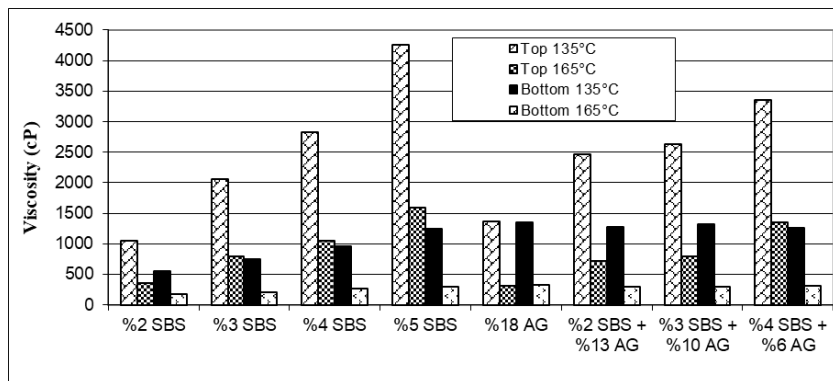
**Fig. 3.7:** Viscosity test results of binders obtained from the tops of storage stability tubes.

As seen in Figure 3.8, the viscosities of the samples obtained from the upper parts of the storage tubes decreased with increasing temperature. As the SBS content increased, viscosity values increased. It was determined that viscosity values increased as the SBS content increased in binders with the same performance level. Among the modified bitumen with the same performance level, it was determined that the modified bitumen containing 5% SBS had the highest viscosity value and the modified bitumen containing 18% AG had the lowest viscosity value.



**Fig. 3.8:** Viscosity test results of binders obtained from the lower parts of storage stability tubes.

When the viscosity values of the lower part samples obtained from the modified bitumen samples applied in the storage stability test were analysed, it was determined that 18% AG modified binder had the highest value. Since the polymer accumulated at the top of the tube in SBS modified binders, it was determined that the viscosity values of the samples obtained from the lower parts of the storage stability tubes of the binders with the same performance level were close to each other.



**Fig. 3.9:** Viscosity values of storage stabilised binders.

When the viscosity test results after the storage stability test of all binders shown in Figure 4.10 were analysed, it was determined that the viscosity values decreased with increasing temperature. When only modified bitumen containing SBS was analysed, it was determined that viscosity values increased both at the top and bottom of the tubes as the SBS content increased. However, when the upper parts of the tubes were evaluated, it was determined that the viscosity value increased by 117% when the SBS content increased from 2% to 3%, while the viscosity values increased by 189% and 338% when the SBS content increased to 4% and 5%, respectively. In the lower parts of the tubes, it was determined that the viscosity values increased by 21%, 50% and 71% with the increase of the additive content from 2% to 3%, 4% and 5%, respectively. This was due to the accumulation of polymer in the upper part of the tube after the storage stability test in SBS modified bitumen.

When the viscosity values of the modified bitumen with the same performance level after storage stability were analysed, it was determined that the viscosity values of the samples obtained from the upper part of the tubes were higher than the samples obtained from the lower part of the tubes. It was determined that 5% SBS modified binder had the highest viscosity value and 18% AG modified binder had the lowest viscosity value. At 135°C, it was determined that there was a difference of 3.44 times between the viscosity values of the samples obtained from the lower and upper parts of the tubes after the storage stability tests in 5% SBS modified bitumen, 1.01 times in 18% AG modified bitumen (0.96% increase), 1.91 times in 2% SBS + 13% AG modified bitumen, 2.01 times in 3% SBS + 10% AG modified bitumen and 2.65 times in 4% SBS + 6% AG modified bitumen. At 165°C, it was determined that there was more difference between the viscosity values of the samples obtained from the bottom and top of the tubes after storage stability compared to 135°C. This shows that there is more difference between the samples obtained from the top and bottom of the tubes after storage stability at high temperatures.

When the viscosity values of the binders with the same performance level were analysed after the storage stability tests, it was determined that 18% AG modified bitumen gave the best result in terms of storage and 5% SBS modified bitumen gave the worst result. It was determined that as the SBS content in the modified bitumen increased, the unfavourability in terms of storage increased and the storage stability decreased. This situation shows that it is more advantageous to use AG together with SBS in modified bitumen production in case of storage of modified bitumen.

#### **4. Conclusion**

In this study, the storage stability of modified bitumen prepared with one type of polymer (SBS) and one type of natural asphalt (AG) was investigated. Penetration, softening point and rotational viscometry tests were used to evaluate the storage stability of the binders.

As a result of the penetration tests applied to the binders, the penetration values of the samples obtained from the upper part of the tubes with the use of additives were higher than the lower part of the tubes. As the SBS content increased at the bottom of the tubes, the penetration value decreased and therefore the consistency increased. AG modified bitumen was the most stable binder in terms of penetration values in terms of storage among all modified bitumens. As a result of the penetration tests, it was determined that the most stable binder in terms of storage stability was the modified bitumen prepared with 2% SBS + 13% AG and the weakest binder in terms of storage was the modified bitumen containing 5% SBS.

Considering the softening point test results, the softening point values of the samples obtained from the upper part of the tubes with the use of additives were higher than the lower part of the tubes. As the SBS content increased in the lower part of the tubes, the softening point values increased. Since the SBS density of the binder was higher in the upper part of the tubes, the softening point values increased more. When the binders containing SBS and AG and having the same performance level with the modified bitumen containing 5% SBS (2% SBS+13% AG, 3% SBS+10% AG and 4% SBS+6% AG) were compared, it was determined that the difference between the softening point values after storage stability increased as the SBS content increased in the modified bitumen containing SBS and AG, but the difference between the softening point values of all SBS and AG containing binders decreased compared to the use of 5% SBS.

When the viscosity values of the modified bitumen after storage stability were analysed, it was determined that the viscosity values of the samples obtained from the upper part of the tubes were higher than the samples obtained from the lower part of the tubes. It was determined that 5% SBS modified binder had the highest viscosity value and 18% AG modified binder had the lowest viscosity value. When the viscosity values of the binders with the same performance level were analysed after the storage stability tests, it was determined that 18% AG modified bitumen gave the best result in terms of storage and 5% SBS modified bitumen gave the worst result. It was determined that as the SBS content

in the modified bitumen increased, the unfavourability in terms of storage increased and the storage stability decreased. This situation shows that it is more advantageous to use AG instead of SBS in modified bitumen production in case of storage of modified bitumen. The results are reliable because each experiment was performed in three repetitions.

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