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Effects of Gilsonite on Performance Properties of Bitumen

Perviz AHMEDZADE¹ Omar ALQUDAH² Taylan GUNAY³ Tacettin GECKIL⁴

ABSTRACT

The effectiveness of natural Gilsonite on the performance properties of modified bitumens was studied. Three samples having different ratios of Gilsonite that are 6%, 8% and 10% by weight of base bitumen which was PG 64-16 grade were prepared. The physical properties and storage stabilities of four samples were investigated by means of conventional tests. The short-term and long-term aging processes of bitumens were conducted with rolling thin oven and pressure aging vessel tests, respectively. Rheological properties of bitumens were examined by means of Superpave tests such as rotational viscosity, dynamic shear rheometer and bending beam rheometer. After the rheological study, the low and high temperature performance classes of bitumens were determined by the Superpave specification. The results showed that natural Gilsonite-modified bitumens provide improvement in rheological and physical properties of bitumen, and were found highly compatible with base bitumen. Gilsonite additive significantly increased stiffness and viscosity of bitumen, and improved the resistance against fatigue cracking and rutting of bitumen, without causing a reduction in thermal cracking resistance of bitumen.

Keywords: Bitumen, gilsonite, aging, rheological properties, performance grade.

1. INTRODUCTION

In the modern world, annually about 110 million metric tons of bitumen is needed. The manufacture, repair and maintenance of bituminous pavements require the large amount of

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fund and energy [1]. In addition to the high cost and inadequate resources of the bitumen used in road pavements, the aging due to transportation, storage, mixing, placement on the road surface and service life on the road also causes many problems. Moreover, after the application of bitumen, hot mix asphalt (HMA) pavements are also faced with various problems during their service life [2]. As is known, the main distresses occur in flexible pavements due to traffic loading or environmental effects or other factors, such as fatigue cracking, rutting, and low-temperature cracking [3].

The bitumen plays an important role in the performance properties of HMA during service life. However, many of the distresses are related to the characteristics of bitumen used in the pavement itself. Base binder is commonly used in most countries where some defections could be occurred early stages of service life. The rheological weakness of binder is the main reason of using modifiers to enhance properties of binder, and consequently make the performance of the pavements better. By the processes of mechanical mixing or chemical reaction, polymer modified bitumens (PMBs) are produced by adding polymers into bitumens. It is necessary appropriate compatibility between bitumen and polymer to obtain the optimum properties of PMBs. Poor storage stability occurs when the compatibility is poor. Thus, the separation of polymeric and bituminous phases or inconsistent bitumen quality takes place [4].

Bitumen modifiers could be natural or artificial. Many types of modifiers were used in this field, like rubber, crumb rubber, elvaloy, polypropylene, styrene-butadiene-rubber (SBR), and styrene butadiene styrene (SBS), and ethylene vinyl acetate (EVA) [3,5,6]. It is known that modifiers vary in effectiveness, workability, performance, availability, and cost. The cost efficiency as well as the degree of compatibility of modifier with the bitumen during storing and handling have been the biggest challenges in the industry of modified bitumens. Despite the fact that there is large number of modifiers, few of them are really appropriate for modification process. There have been many attempts within this scope to solve the problem related to lack of stability in polymer-modified bitumen. For example, the recycled polymers can be exposed to irradiation to get over this problem. In this process, functional groups and new bonds are formed in polymer chain and this provides chemical bonding with bitumen by preventing phase separation. The beneficial and significant enhancement of bitumen behavior and performance was obtained by the modification of bitumen with the electron irradiated recycled low-density polyethylene (e-LDPE_R) [7, 8]. The compatibility between SBS-g-M grafted with vinyl monomer under x-rays irradiation and bitumen, and thus the storage stability of bitumen was improved considerably as compared with SBS modified bitumen [9].

The main purpose of bitumen modification is to improve the main quality and properties of bitumen without negatively affecting the different properties of the bitumen or mixture [10]. In recent years, natural hydrocarbon-containing additives have begun to be used to improve the performance properties of bitumen. One of these additives is known in the market as Gilsonite, which is derived from naturally occurring bitumen resources, commonly referred to as asphaltite [11,12,13,14]. Discovered in the early 1860s, Gilsonite is a resinous hydrocarbons that has been used and evaluated in a variety of industrial applications [15]. Gilsonite, a crude petroleum-based by-product, contains natural hydrocarbons with a purity of about 99%, and 57–70% of asphaltene [14,16]. It is a mineral bitumen, black and fragile, and has a structure that can easily be crushed into powder [14,17]. It is also a fast-dissolving

additive in bitumen due to its easy using and good compatibility with bitumen [18]. The favorable properties of Gilsonite make it a good alternative to other commercially produced polymers, especially at high temperatures and high traffic volumes. In various studies, Gilsonite is generally used to improve high temperature performance properties due to the hardening effect of bitumen, but the hardening that occurs can affect the low and intermediate temperature performance characteristics of bitumen [13-15,19,20,21]. The use of Gilsonite is now considered very seriously in bitumen modification when cost and time effectiveness, high performance and storage stability are taken into consideration.

In this study, the Gilsonite, a natural hydrocarbon-containing additive, was used as a modifier for bitumen to examine its impact on the physical and rheological performance properties of the road bitumens.

Generally, in the studies carried out, the Gilsonite additive was used in bitumen modification in two different ways: the addition of Gilsonite into bitumen, or the addition of Gilsonite as a filler in the aggregate mixture [14]. In previous studies, it has been reported that the use of Gilsonite in bitumen modification has increased the viscosity and softening point of bitumen but decreased its ductility and penetration [13,14,22]. In some studies, it has been reported that Gilsonite reduced the temperature sensitivity by increasing the hardness of the bitumen, but it increased its elasticity [13,23]. In many studies it has been indicated that increasing the amount of Gilsonite in the bitumen increased the high temperature performance of the bitumen, but the tendency to fatigue and low-temperature cracking increased [11,12-15,21,24]. However, Feng and Ameri have reported that the intermediate and high temperature performance of the modified bitumen with Gilsonite has improved [23]. The results of another study showed that Gilsonite, as a modifier, could be used to improve its stiffness properties and rutting resistance of mixtures used in hot climates [14]. A study, in which two different types of bitumen were used, indicate that Gilsonite improved the high temperature performances of the bitumens, but decreased the low temperature performances [17]. Similarly, Anderson et al. also noted that Gilsonite modified bitumen showed a fragile behavior at low temperatures [19]. The results of a study showed that the Gilsonite-modified bitumens serve to prevent crack formation in the coating by forming a good bond between bitumen and aggregate [25]. In another study, it was found that the storage stability of Gilsonite-modified bitumen was better than SBS modified bitumen [13]. In addition, the results of a study showed that the use of Gilsonite in appropriate amount had no negative effect on the aging process of bitumen [26]. In many studies it has been reported that HMA modified with Gilsonite had higher stability, lower permanent deformation and better fatigue and moisture resistance [10,13,15,18,23]. Additionally, it has been determined that these mixtures showed more strength under dynamic loading [15,23]. Moreover, in some studies it has been stated that Gilsonite has many economic benefits of using it in pavement design [11,13,14,15,23]. Finally, it has been suggested that Gilsonite could be used as a modifier to improve the performance properties of HMA [11,13].

This research discusses the influence of Gilsonite as a modifier in bitumen. The primary objective of this research is to characterize the physical and rheological properties of Gilsonite modified bitumen. To this end, the test results of modified binders were discussed and compared with base (unmodified) bitumen.

2. MATERIALS

2.1. Materials

The base bitumen with a PG 64-16 performance grade used in this study was supplied from Izmit refinery in Turkey. The obtained physical properties of base bitumen were given in Table 1. The modifier used was natural Gilsonite material supplied by URAN Holding Ltd. Company in Turkey. The composition and physical properties of used Gilsonite are shown in Table 2.

Properties	Results
Penetration (25°C, 100 gr, 5 sec) 0.1 mm	51
Softening point, °C	46.5
Elastic recovery (25°C), %	88
Flash point, °C	265
Specific gravity (25°C), gr/cm ³	1.027

Table 1 - Physical properties of base bitumen (PG 64-16)

Table 2 - Composition and physical properties of used Gilsonite

Properties	Results
Carbon content (%)	85.22
Sulphur content (%)	3.09
Oxygen content (%)	1.49
Hydrogen content (%)	5.97
Nitrogen content (%)	0.77
Ash content (%)	2.24
Moisture content (%)	0.09
Solubility in CS2 (%)	41.74
Solubility in Toluene (%)	20.83
Softening point, °C	160-205
Specific gravity, at 25°C (gr/cm ³)	1.145

2.2. Preparation of Samples

The Gilsonite-modified bitumen were prepared by blending base bitumen with Gilsonite in powder form at three different percentages (6%, 8% and 10%) of Gilsonite by total weight of bitumen. For this purpose, blending process was accomplished via a laboratory high shear mixer. After heated in oven for 90 min at 163°C, the base bitumen was poured into a

temperature-controlled container of mixer rotating 500 rpm. Subsequently the Gilsonite was slowly added into bitumen by portions within the first 15 minutes of mixing, and then the mixing rate was adjusted to 1500 rpm. This mixing process continued for another 45 min. The mixing temperature was set at 170 °C and the temperature was checked with a thermometer at every 15 minutes.

Base and Gilsonite-modified bitumens used in this study were coded as B, B+6G, B+8G and B+10G, respectively.

3. TEST METHODS

3.1. Conventional Tests

Penetration, softening and flash point, elastic recovery, and specific gravity tests were performed on both original base and modified bitumens. After the thin film oven test (RTFOT), the mass losses, retained penetrations and softening points of the bitumen were established to understand the effects of the modifier on aging. All tests were conducted according to the requirements of (TS EN 14023) standard of polymer-modified bitumens (PMBs) [27].

3.2. Storage Stability Test

In order to provide the stability and decomposition resistance of the modified bitumens during handling, mixing and in-service life, the compatibility test was also performed throughout the storage stability test according to EN 13399 standard [28].

This test was conducted as follows. A glass tube consists of three symmetrical parts, 35 mm diameter, and 180 mm height, was used and filled with about 150 g of modified bitumen. The tubes containing the modified samples were tightly closed via glass hood with two hooks. Two small springs were used to close the tube and fasten the hood with the top part of the tube in order to ensure no vaporization problem would occur during the test. After that, the tubes were placed vertically in a preheated oven in 180 °C for 72 hr. The bitumen samples stored in oven were cooled at room temperature, and then cut into three parts. To compare the top and bottom parts of samples, these two parts were tested to evaluate the storage stability by measuring their penetration, softening point [29].

3.3. Rotational Viscosity Test

The viscosities of all bitumens were determined by the rotational viscometer (RV) to ensure the workability of the modified bitumens. The rotational viscometer as compared with the capillary viscometers used in the viscosity graded method can detect the viscosity of bitumen by measuring the torque required to provide a constant rotational speed (20 RPM) of a cylindrical spindle immersed into a binder sample held at a fixed temperature [29, 30].

The tests were conducted at 135 °C and 165 °C by means of a Brookfield viscometer (DVRV-II Pro) as described in AASHTO T316 standard [31]. The mixing and compacting process temperatures of base and modified bitumens to be used in HMA were also determined. In the

calculation, the suitable ranges of viscosities corresponding to the mixing and compacting temperatures were selected as 150-190 cP and 250-310 cP, respectively. The rotational viscosity of bitumen should not exceed 3000 cP for the tests at 135 °C [32].

3.4. Aging of Bitumen

The short-term aging operations of all bitumens were conducted with the rolling thin film oven test (RTFOT) according to EN 12607-1 standard [33]. The bitumen samples were aged for 75 min in an oven at a temperature of 163 °C. After RTFOT simulating changes in the physical properties of the bitumen during mixing and construction, the bitumens were subjected to penetration and softening point tests. In addition, the mass losses of the bitumens were also determined to understand the effects of the short-term aging. The Superpave specification for RTFOT aged bitumen implies that maximum mass loss after short-term aging process should not exceed 1% by weight of bitumen; for which TS EN 14023 standard is different depending on PG grade of tested bitumen [34]. For soft bitumens the maximum limit is 1%, and for harder bitumens it is 0.8% by weight of bitumen [27]. As is known, bitumen are exposed to heat and pressure to simulate the long-term aged bitumen in field during service; the pressure aging vessel (PAV) was used to simulate in-service aging over a 7 to 10-year period [32, 35].

3.5. Rheological Characterization

The viscous and elastic behavior of bitumen was determined by the dynamic shear rheometer (DSR) at medium to high temperatures. The complex shear modulus (G^*) and phase angle (δ) were measured by subjecting a small sample of bitumen to oscillatory shear stresses as sandwiched between two parallel plates in the DSR machine in stress controlled mode at a frequency of 10 rad/s. Non-aged and RTFOT samples were tested using 25 mm diameter plates and a gap of 1 mm. After RTFOT + PAV aging, samples are tested using 8 mm diameter plates and a 2 mm thickness sample. The rutting parameters ($G^*/sin\delta$) at high temperatures of the bitumen and the fatigue parameters ($G^*sin\delta$) at the intermediate temperatures are calculated for each sample. The $G^*/sin\delta$ of original bitumen should not exceed 1 kPa, and for RTFOT aged bitumen it should not exceed 2.2 kPa. Furthermore, the $G^*sin\delta$ of RTFOT+PAV aged bitumen should not exceed 5000 kPa [32, 36].

3.6. Bending Beam Rheometer (BBR) Test

The bending beam rheometer (BBR) test is used to determine the stiffness and consistency properties of bitumen at low temperatures. The obtained parameters are considered to be indicative of the ability of the bituminous binder to resist the low temperature cracking. The BBR test is also used to determine the performance grades (PG) of the bitumens at low temperatures. Properties of bitumen at low temperatures, the creep stiffness (s) and the creep rate (*m*-value), are measured using BBR. Superpave specification entails that the creep stiffness must not exceed 300 MPa, and creep rate should not be lower than 0.300 to avoid low temperature cracking [17, 22].

4. RESULTS AND DISCUSSIONS

The criterion applied in this research to evaluate Gilsonite effects on base bitumen was at first to determine the PG grade of the bitumens, and then to compare obtained conventional tests results and rheological properties with requirements of TS EN 14023 standard of polymer-modified bitumens (PMBs) at the specified PG grade of each bitumen. To evaluate the properties of the modified bitumen according to TS EN 14023 standard, the PG grade of this bitumen must be known in advance. For this reason, the conventional test results of modified bitumens including both before and after the short-term aging were intentionally given in Table 3.

			1	1
Tests / Binder types	В	B+6G	B+8G	B+10G
	(PG 64-16)	(PG70-16)	(PG76-16)	(PG82-16)
Penetration (25°C, 100gr, 5sec),	51	30*	28	22*
0.1mm	(50-70)	(45-80)	(25-55)	(25-55)
S-A-mine maint %C	46.5	52*	53*	53*
Soliening point, *C	(46-54)	(≥60)	(≥65)	(≥70)
Electic recovery (25°C) %		87	86	84.5
Elastic fectivery (25°C), 78	-	(≥60)	(≥60)	(≥60)
Flash point °C	250	255	255	260
Plash politi, C	(≥230)	(≥220)	(≥220)	(≥220)
Specific gravity (25°C) gr/cm ³	1.027	1.036	1.039	1.042
speenie gravity (25°C), gi/em	(1.0-1.1)	(1.0-1.1)	(1.0-1.1)	(1.0-1.1)
Storage stability:				
Difference in softening point °C		1.5	0.5	1.5
Difference in softening point, C	-	(≤5)	(≤5)	(≤5)
Difference in penetration 0.1mm		2	1	1
Difference in penetration, 0.1111		(≤13)	(≤9)	(≤9)
Dynamic shear rheometer (DSR)	68	74.7	77	82
$(G^*/sin\delta > 1$ kPa)	(>64)	(>70)	(>76)	(>82)
Failure temperature, (°C)	(≥04)	(≥70)	(≥70)	(≥82)
Rolling thin oven test (RTFOT):				
Weight loss %	0.295	0.036	0.031	0.029
weight loss, 78	(≤0.5)	(≤1)	(≤0.8)	(≤0.8)
Change in softening point:				
Increase, °C	5.5	7.5	7.5	7
	(≤9)	(≤8)	(≤8)	(≤8)
Decrease °C		-	-	-
	-	(≤5)	(≤5)	(≤2)
Retained penetration %	53	80	78.6	77.4
Retained penetration, 70	(≥50)	(≥50)	(≥45)	(≥40)

Table 3 - Evaluation of modified bitumen according to TS EN 14023 standard for (PMBs)

Tests / Binder types	B (PG 64-16)	B+6G (PG70-16)	B+8G (PG76-16)	B+10G (PG82-16)
Dynamic shear rheometer (DSR) RTFOT aged ($G^*/sin\delta > 2.2$ kPa) Failure temperature, (°C)	68.5 (≥64)	75.5 (≥70)	78.7 (≥76)	>82 (≥82)
Dynamic shear rheometer (DSR) RTFOT+PAV aged $(G^*sin\delta < 5000$ kPa) Failure temperature, (°C)	25 (≤31)	23.4 (≤31)	25.2 (≤34)	27.1 (≤37)
Bending beam rheometer (BBR) Stiffness (s≤300MPa, <i>m</i> ≥0.300) Failure temperature, (°C)	-20.5 (≤-16)	-19.9 (≤-16)	-19.3 (≤-16)	-18.3 (≤-16)

 Table 3 - Evaluation of modified bitumen according to TS EN 14023 standard for (PMBs) (continue)

() Requirements of binders according to standard.

* Result does not achieve the required specification.

4.1. Conventional Bitumen Tests Results

The results of penetration and softening point tests conducted on bitumens were given in Table 3. As seen in Table 3, the penetration was continuously in the decreasing trend with increasing Gilsonite additive, and therefore, B+10G had the lowest penetration with a value of 22 dmm. The softening point of the bitumen increased with increasing Gilsonite addition, confirming the results of the penetration test. These results mean a significant increase in the stiffness of the bitumen after Gilsonite addition. However, this is accompanied by a decrease in elastic recovery since bitumen becomes harder after the modification. However, Gilsonite had only a slight adverse effect on elastic properties of bitumen as the elastic recovery percentage decline to 84.5 (for B+10G) from 88 (for B). As for flash point tests, values for all the base and Gilsonite modified bitumen samples were found above the recommended minimum value of 220 °C.

As seen from Table 3, the tests performed on aged bitumens showed that the Gilsonite additive had a relatively positive effect on the aging potential of bitumens. The mass losses of modified bitumens were still very slight and its value was in the specified standard limits. The difference between softening point before and after aging within the specification for all bitumens. Additionally, after short-term aging, the retained penetrations of B, B+8G, and B+10G after RTFOT aging were (80, 78.6, and 77.4) % of the original bitumens, respectively. This showed that as the decrease in retained penetration was very slight while Gilsonite content is increased from 6 to 10 %. This also indicated that the increase in the modifier content had a positive effect on short term aging.

4.2. Storage Stability Test Results

A long period of storage for bitumen could lead to phase separation of the bitumen and poor stability problems. This situation is a major problem in the modified bitumen industry. The

results associated with storage stability was given in Table 4. Accordingly, both the softening point and the penetration results of the bottom and top parts of the Gilsonite-modified bitumen subject to the storage stability test are very close to each other. This result can be regarded as a good indicator of the homogeneity and consistency of Gilsonite modified bitumens.

Property / Bitumen types	B+6G	B+8G	B+10G
Penetration bottom part, 0.1mm	24	22	17
Penetration top part, 0.1mm	26	23	18
Difference in penetration between bottom and top part, 0.1mm	2	1	1
Softening point bottom part, °C	50.5	54	56.5
Softening point top part, °C	52	53.5	0.5
Difference in softening point between bottom and top part, °C	1.5	55	1.5

Table 4 - Storage stability test results of Gilsonite modified bitumens

4.3. Rotational Viscosity Test Results

The rotational viscosity (RV) of the bitumen was determined at 135 and 165 °C, and the results were summarized in Table 5. The mixing and compacting temperatures and the modification indexes ($\eta_{modified} / \eta_{base}$) of all bitumens to be used in HMA were also given in Table 5. Addition of Gilsonite to base bitumen increased the viscosity at 135 °C and 165 °C. This indicates that Gilsonite modification improved the stiffness of bitumen. For instance, at the heavy modification level of 10%, the viscosity increased from 443 cP to 1122.5 cP. On the other hand, both mixing and compacting temperatures increased as the content of Gilsonite was increased.

Binder Rotational viscosity (cP) Temperature range (°C) $\eta_{modified}$ / η_{base} types 135°C 165°C 135°C 165°C Mixing Compaction В 443 128 1.0 1.0 157-163 145-150 B+6G 715 170 1.61 1.33 162-167 152-157 B+8G 873 208 1.97 1.63 166-171 156-161 B+10G 1122.5 255 2.53 1.99 172-177 162-167

Table 5 - Rotational viscosity test results of the base and Gilsonite modified bitumens

The mixing and compacting temperatures of the bitumen were detected by means of Figure 1. The mixing and compacting temperature ranges increased from (157-163) °C and (145-150) °C for B, to (172-177) °C and (162-167) °C for B+10G, respectively. Although

the workability of modified bitumen decreased, the value of RV was less than 3000 cP and therefore still satisfied the ASTM D6373 standard criterion for bitumen workability [32, 38].



Figure 1 - Viscosity-temperature relationship for the base and Gilsonite modified bitumens

4.4. Rheological Characterization- Dynamic Shear Rheometer (DSR) Test Results

To determine the PG grade of each bitumen type and to study the effect of Gilsonite modification on rheological properties of bitumen, the DSR test was conducted. Viscoelastic parameters of all bitumens, such as complex modulus (G^*) and phase angle (δ) were obtained, and the effect of Gilsonite on G^* and δ for original and RTFOT-aged bitumens was shown in Figures 2 and 3, respectively. The rutting parameter ($G^*/sin\delta$) was calculated for each sample of base and modified bitumens and shown graphically in Figure 4.

As seen from Figure 2, the G^* parameter for original base bitumen (B) for example at 64 °C was 1.64 kPa and increased to 8.56 kPa for B+10G at the same temperature. Adding 10% of Gilsonite leads to an increase of bitumen's stiffness by 421%. It can be said that Gilsonite modification improved the stiffness of bitumen and rheological behavior especially under high temperatures.

As shown in Figure 3, the values of δ were decreased as Gilsonite content increased in original and aged bitumens. For example, δ at 64 °C for B was 87.2 and for B+10G was 78.7 of original case. This indicated that Gilsonite exhibited an improvement of elastic component of bitumen.

The bitumen used in the mixture provides its fair share of the overall resistance in the pavement in terms of permanent deformations by controlling stiffness of the mixture at high temperatures. The rutting parameters ($G^*/sin\delta$) of base and modified bitumens were found for original and RTFOT aged bitumen at high levels of temperature starting at 52 °C. While

 $G^*/sin\delta$ value increased, the bitumen was expected to be stiffer and as a result more rut resistant mixtures could be achieved.



Figure 2 - Effect of Gilsonite on complex shear modulus (G*) for original and RTFOT aged bitumens



Figure 3 - Effect of Gilsonite on phase angle (δ) for original and RTFOT aged bitumens



Figure 4 - Rutting performance of Gilsonite modified bitumens

Figure 4 showed the $G^*/sin\delta$ of original and RTFOT aged samples. B+10G has the highest G*/sin\delta value of 8.73 kPa compared to the rest of samples. This showed that the stiffness of bitumen increased as Gilsonite content increased. This result was compatible with conventional test data.

 $G^*/sin\delta$ of aged sample was higher than that of the original sample of the same bitumen type. For example, at 64 °C the $G^*/sin\delta$ for original B was 1.65 kPa and for aged B was 4.07 kPa. At the same temperature, for original B+10G the $G^*/sin\delta$ was also 8.73 kPa and for aged B+10G was 22.4 kPa.

 $G^*/sin\delta$ of the base and modified bitumens (seen in Figure 4) indicate that aged B+8G provides the maximum increase in stiffness between the samples which is around 182.7% compared to base bitumen, whereas the aged B, B+6G, and B+10G exhibited the increases of 146.7, 153.1, and 156.6% respectively. As expected, the increment in temperature leads to a decrease in G^* and an increase in δ . Moreover, the B+10G shows much more resistance against permanent deformation rather than B. As can be seen in Figure 4, B+10G achieved the specifications of rutting parameter in terms of both aged and unaged conditions up to 82°C, while B reaches only up to 64°C considering Superpave specifications.

To study the fatigue performance of Gilsonite-modified bitumen, complex shear modulus (G^*) and phase angle (δ) for PAV-aged bitumen at different temperatures and different Gilsonite contents were shown in Figure 5 and 6, respectively. The fatigue parameters $(G^*sin\delta)$ for all bitumen types were calculated, and fatigue performance of Gilsonite modification was shown in Figure 7. The effects of the Gilsonite additive on the rheological properties of bitumen are summarized in Table 3.



Figure 5 - Effect of Gilsonite on complex shear modulus (G^*) for PAV aged bitumens



Figure 6 - Effect of Gilsonite on phase angle (δ) for PAV aged bitumens



Figure 7 - Fatigue performance of Gilsonite modified bitumens

The fatigue parameters $(G^*sin\delta)$ of modified bitumens increased with increasing Gilsonite content compared to the base bitumen due to the increase of G^* and decrease in δ . It should be noted that fatigue parameter was determined at intermediate test temperature by adding the high-grade temperature to the low-grade temperature dividing the result by 2 and adding 4. Hence, the comparison between the base bitumen B and B+10G should be related to the PG grade of each bitumen since stiffness differs from one bitumen to another. The PG grade of B was PG 64-16, therefore, the intermediate test temperature of RTFOT+PAV aged bitumen used in DSR test was calculated as 28 °C (by using abovementioned formula which is correspond to (64-16)/2+4=28 in this case). Similarly, from Table 5 for B+10G which is PG 82-16 graded bitumen, the intermediate test temperature was found as 37 °C by using the same formula.

As can be seen in Figure 7, $G^{*sin\delta}$ of B was found as 2327 kPa at 28 °C, 2070 kPa at 31 °C for B+6G, 1858 kPa at 34 °C for B+8G, and 1610 kPa for B+10G at 37 °C, which indicated that Gilsonite modified bitumen achieved the standard requirement of fatigue parameter ($G^{*sin\delta}$ <5000 kPa). On the other hand, by comparing the intermediate temperature (28 °C) of RTFOT+PAV aged bitumens to base bitumen (Fig. 6), there was an improvement in δ as it goes down from 49.2 °C to 36.9 °C which corresponds to 25% decrement in phase angle after the modification. This is also applicable for other test temperatures. The decrease in δ value while G* value being increased by adding Gilsonite, caused the elastic behavior of Gilsonite-modified bitumens to prevail at the new achieved intermediate performance temperatures.

4.5. Bending Beam Rheometer (BBR) Test Results

Creep tests were performed at two different temperatures (-16 °C and -22 °C) to determine the performances of the bitumens at low temperatures. In Figures 8 and 9, the creep stiffness of all bitumens satisfied with the Superpave specification at -16 °C, and all bitumens showed higher m-values than the required minimum at this temperature. The creep stiffness of B+10G was higher than that of B while its creep rate as *m-value* was lower. Furthermore, the stiffness of samples increased with decreasing temperature while *m-value* decreased. In addition to this, the creep stiffness and *m-values* of bitumens were failed to meet the Superpave specification at -22 °C which means the low temperature PG grade of all bitumens was determined as PG X-16.

Ten percent addition of natural Gilsonite by weight of bitumen was enough for the beneficial use as it reaches to the highest (high temperature) performance grade of PG 82-16. Hence it is believed that the optimum usage of Gilsonite content is 10%, since the *m-value* of B+10G at -16 °C was 0.324 which was very close to 0.300. On the other hand, the Gilsonite modification did not contribute an enhancement to the low temperature PG grade. For example, *m-value* for B+6G was 0.271, and for B+10G it was 0.260. This indicated that with adding Gilsonite, the creep rate decreased, and the bitumen was more brittle and stiffer. At the same time, the PG grade of base and Gilsonite modified bitumen were given in Table 3.



Figure 8 - Creep stiffness of the base and Gilsonite modified bitumens

Block cracking is a low temperature failure in flexible pavement similar to thermal cracking which develops in both longitudinal and transverse directions. Block cracking generally occurs when flexible pavement is old (aged) and traffic volume is low. A parameter named critical temperature (ΔT_c) is relatively up to date for the evaluation of low temperature

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performance of binders against block cracking [39]. ΔT_c is obtained by means of the low continuous grade temperature for the stiffness criteria minus low continuous grade temperature for the m-value criteria (which are 10 °C lower than failure points determined in BBR test) and suggested to examine the ductility loss of aged bitumens leading to block cracking.

 ΔT_c of the binders was calculated as -9.9 °C, -9.5 °C, -9.7 °C, -9.9 °C for the base, %6, %8, and %10 Gilsonite modified binders, respectively. Lower ΔT_c means that binder is vulnerable to cracking at low temperature region. Generally, it is suggested that ΔT_c should not be lower than -5 °C which means that ΔT_c of base and Gilsonite modified binders are not within the acceptable level. However, it should be noted that base and Gilsonite modified binders have very similar ΔT_c which signifies that Gilsonite has no positive or negative effect on the loss of ductility of binder.



Figure 9 - The m-value of the base and Gilsonite modified bitumens

4.6. PG Grade and PMBs Requirements

The enhancement after Gilsonite modification was determined and mentioned in Section 4 as the base (B) is a PG64-16 graded bitumen (in Figs. 2-7) while grade of modified bitumens (B+6G, B+8G, and B+10G) are PG 70-16, PG 76-16, and PG 82-16, respectively.

Although the modified bitumens did not meet some of the specifications in TS EN 14023 standard, it is believed that the PG grade is a better indicator for bitumen performance, in which rheological parameters are taken into consideration, compared to penetration and softening point tests, in which only basic techniques are applied. Besides, the difference in softening point and penetration after storage stability tests indicated that all modified

bitumens with respect to their relative PG grades meet the requirements of storage stability in accordance with TS EN 14023 standard. This means that Gilsonite modification achieves the homogeneity and has no phase separation problems.

5. CONCLUSIONS

In this study, the effects of natural Gilsonite on the performance properties of bitumen were investigated, and the following conclusions were obtained.

- 1. The conventional test results showed that the addition of natural Gilsonite remarkably increased the viscosity and stiffness of the base bitumen. The RV results also confirm that Gilsonite had a stiffening effect on the bitumen. In spite of relatively high mixing and compacting temperature ranges of modified bitumens, it was assumed that using Gilsonite could be a suitable modifier taking into consideration the high cost of base bitumen and insufficient resources of this material.
- 2. The results obtained from elastic recovery showed a slight decrease in elastic recovery after the modification, yet it was considered acceptable.
- 3. The storage stability test showed that Gilsonite was fully compatible and highly stable with bitumen and had no phase separation in the bitumen during long time of storage process.
- 4. The natural Gilsonite has a considerable effect on G^* and δ of bitumen. Gilsonite modified bitumen had higher rutting parameter ($G^*/sin\delta$) than that of the base bitumen both before and after aging processes. In addition, Gilsonite modification had positive effect on fatigue resistance of bitumen. Moreover, the Gilsonite modification at three different levels of Gilsonite content (i.e, 6, 8, and 10%) provided high performance grade of bitumens of PG 70-Y, PG 76-Y, and PG 82-Y, respectively.
- 5. BBR test results showed that Gilsonite additive had no positive effect on lowtemperature cracking resistance of bitumen and caused the creep rate decrease. The low performance grade at three levels of Gilsonite modification of 6, 8, and 10 % were found as PG X-16, PG X-16, and PG X-16, respectively. This was attributed to the stiffening effect of Gilsonite additive which is also compatible with elastic recovery results.

According to the findings, Gilsonite has positive effects on the physical properties of bitumen such as stiffening meaning that the pavements become more resistant to permanent deformation after the modification. Although the Gilsonite contributes no enhancement against low-temperature cracking, it provides a positive effect on high temperatures. The use of 10 % of Gilsonite improves the performance grade of base bitumen from PG 64-16 up to PG 82-16 which corresponds to an enhancement of three levels. For this reason, it is advised to use Gilsonite at high temperature regions and/or very heavy loaded flexible pavement projects up to 10%. It should also be noted that light modification of Gilsonite is recommended in cold regions since the modifier increases the stiffness of bitumen.

Symbols

DSR	: Dynamic shear rheometer
G *	: Complex shear modulus
m-value	: Creep rate
PAV	: Pressure aging vessel
PG	: Performance grade
РМВ	: Polymer modified bitumen
RTFOT	: Thin film oven test
RV	: Rotational viscometer
S	: Creep stiffness
δ	: Phase angle
η base	: Viscosity of base bitumen
N modified	: Viscosity of modified bitumen

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