

EFFECTS OF GRAPHITE ON MECHANICAL PROPERTIES OF STONE MASTIC ASPHALT PAVEMENT

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Abstract. Hot mix asphalt needs to be developed to resist particular permanent deformations like rutting and thermal cracking due to increased traffic volumes and vehicle loads. Additives such as carbon black, graphite, carbon fibers are used in the mixture or the binder for improving the mechanical features of asphalt. In this article, the effects of graphite used for developing the mechanical properties of asphalt have been investigated in mixtures tests. Therefore, Marshall stability, indirect tensile stiffness modulus and indirect tensile fatigue tests were performed to bituminous mixtures modified with three different proportions of graphite by weight of bitumen. Stone mastic asphalt mixtures which were manufactured with pure and modified bitumen were aged in different time intervals in the oven. In conclusion, it has defined that Marshall stability values have declined. It has been determined that there is no significant difference in the time-dependent deformation behavior of the original and aged samples in pure and different graphite content although the stiffness modulus and load repeat number of the samples increased with the rise of the aging time. These results shown that graphite generally used for improving the thermal properties in literature were also determined to contribute to mechanical properties of mixtures.

Keywords: graphite, stone mastic asphalt, aging, mechanical properties, bitumen, pavement.

Introduction

Large parts of transport in the world are made with highways and heavy traffic increases day by day thereby the roads are structurally damaged. Heavy vehicle traffic and overmuch load repetition, as well as environmental factors, cause major disruptions on the roads. There are various ways of preventing these disruptions. One of these ways is to obtain more powerful mixtures by changing the aggregate gradation (Liu *et al.* 2012). In practice, the most obvious example of this is stone mastic asphalt (SMA). Using of stone mastic asphalt earns high durability, low permeability, low traffic noise pollution, high strength against reflective cracks and high strength against rutting properties to asphalt layer in the road (Austrroads Technical Report 2002). It was also determined that elastic property of SMA could be improved by adding waste polyethylene terephthalate and styrene-butadiene-styrene (Moghaddam *et al.* 2012; Mokhtari, Nejad 2012). Another way to prevent of these distortions is to modify the bitumen. Due to viscoelastic behavior of bituminous binder, mechanical features of asphalt pavement vary significantly because of daily and seasonal temperature changes. Asphalt concrete temperature can ascend to 70 °C in summer owing to it's over absorp-

tion parameter (Van Bijsterveld *et al.* 2001). Furthermore, this incident eventually deteriorates the durability of pavement. It will trigger the permanent deformations of pavement with the impact of loads (Tongyan *et al.* 2012). It was determined that thermal expansion were slightly larger than the thermal contraction (Mamlouk *et al.* 2005) There are various ways to prevent deteriorations occurred from high temperatures in the pavement. One of them is the use of thermally conductive materials in order to improve the resistance of pavement to the adverse effect of high temperature. Du and Wang (2015) showed that using graphite in asphalt mixture can successfully transmit the temperature to the bottom layers. It was determined that the temperature decreased 6.5 °C at the 4 cm depth from the surface which induced 43.5% decrease in rutting by using graphite as an additive. Liu *et al.* (2014) determined that using 40% graphite with 0.3% carbon fiber induces 78%, 15% and 4% increase in dynamic modulus, indirect tensile strength and Marshall stability of the bituminous mixtures respectively. Lu *et al.* (2008) produced conductive asphalt concrete using steel slag as aggregate and graphite in bituminous mixtures. They have concluded that Marshall stability and dynamic

stability values of mixtures declined a bit with the rise of graphite percentage, but these mechanical properties values still compensate the standard requirements. The authors recommended constructing thermal-modified pavement structure in order to improve the rutting and fatigue cracking resistance (Chen *et al.* 2016).

Another important issue about the deterioration of bituminous mixture is aging processes. Firstly the bitumen used asphalt mixtures are subjected to short-term aging during production and paving processes and then exposed to long-term aging throughout service life. The chemical change due to oxidation results in a harder, more brittle asphalt mixture, which induces low temperature cracking, fatigue cracking or another mode of distress. It was determined that mixing time has an effect on the mechanical properties of SMA during the production. It was also stated that different batching technologies impact an unstable bitumen short-term aging (Bražiūnas *et al.* 2013). Researchers have been studying on mitigating the adverse effects of aging by using various additives and also investigates new technics to evaluate the aging processes. Warm-mix-asphalt additives which induced a reduction in mixing and compaction temperature was reported to be used successfully for the stone mastic asphalt (Kim *et al.* 2015). The influence of the additives especially after long-term aging should be observed in order to make a comprehensive evaluation about the service life of hot mixtures. It was determined that aging induces more stiffness for the unmodified mixtures than for polymer or rubber modified mixtures (Radziszewski 2007). Wang *et al.* (2015) showed that Styrene-butadiene-styrene (SBS) which is a widely used binder additive and significantly contribute to the performance of unaged binder may lose its modifying function after long-time aging. FT paraffin wax was found to be effective in enhancing the resistance to aging (Aghazadeh Dokandari *et al.* 2014). The improvement effects of crumb rubber on short-term aging properties of asphalt binder are reported by the researchers (Dong, Tan 2011; Reed 2010; Shatnawi 2012). Conversely it was determined that the asphalt binder source rather than the nanoparticle content plays an important role in determining the influence of long-term ageing process (Xiao *et al.* 2011). In another study, the thermal properties of graphite added binders are examined. It is found that graphite improves the anti-aging properties of the bitumen (Pan *et al.* 2014). Yao

et al. (2016) investigated the effects of 1–2% graphite nanoplatelets on asphalt binder properties. It was determined that aging groups increases in the graphite modified binder which results in an increased resistance to rutting and moisture damage. Graphite nanoplatelets also improve the resistance to cracking.

Although there are some studies about the thermal properties of bituminous mixtures containing graphite, there are limited studies about the effect of graphite on mechanical properties of stone mastic asphalt mixtures. In this study, the effects of the different amount of graphite on the mechanical properties of mixtures were investigated in large-scale.

1. Materials

In this article, the aggregate and the filler used to manufacture of the asphalt mixtures are chosen as the limestone. The bitumen (B 50/70) supplied from TÜPRAŞ refinery was utilized for the produce of modified binders. The properties of the bitumen are given in Table 1. Graphite powder passing the No. 200 sieve (0.075 mm) has a carbon content of 85.7%, an electrical resistivity of $10^{-4} \Omega \cdot m$ and a density of about 2.15 g/cm³. Elemental analysis of graphite used in bitumen modification is given in Table 2. SEM image of graphite powder is also shown in Figure 1. In the mixture samples, the limestone's aggregate characteristics were given in Table 3 was used.

2. Test sample preparation

Bituminous hot mixture samples were prepared as stone mastic asphalt, which has high resistance to the rutting,

Table 1. The properties of the bitumen

Properties	Value
Penetration (25 °C, 100 g, 5 s, 0.1 mm)	51.2
Softening point (°C)	52.2
Penetration indeks	-0.6
Density (g/cm ³)	1.013
Viscosity (cP, 135 °C)	600
Viscosity (cP, 165 °C)	175

Table 2. The elemental analysis of the graphite

C (%)	N (%)	H (%)	S (%)	O (%)	Moisture (%)	Ash (%)
85.7	-	-	0.1	12.9	0.4	0.6

Table 3. Physical properties of aggregate

Properties	Standard	Limits	Coarse	Fine	Filler
Abrasion loss (%) (Los Angeles)	ASTM C 131 (2014)	Max 30	25	-	-
Frost action (%) (Na ₂ SO ₄)	ASTM C 88 (2013)	Max 10	4.5	-	-
Flat and elongated particle (%)	ASTM D 4791 (2010)	Max 10	4	-	-
Water absorbtion (%)	ASTM C127 (2015)	Max 2	1.37	-	-
Specific gravity (Gsb)	ASTM C127 (2015)		2.613	-	-
Specific gravity (Gsb)	ASTM C128 (2015)		-	2.622	-
Specific gravity (Gsa)	ASTM D854 (2014)		-	-	2.711

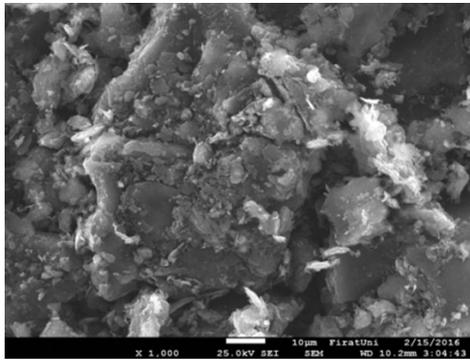


Fig. 1. SEM image of graphite

has a high durability due to the high bitumen content and has recently been widely used in high standard roads. Gradation prepared according to Turkish Highway Technical Specification stone mastic wear layer Type-1 is shown in Table 4 and was designed in accordance with standard Marshall design method. In order to compare the effects of graphite powder on mechanical performance of asphalt mixture, all the mixture samples were prepared with the same gradation and same asphalt content.

Table 4. The gradation of the stone mastic asphalt

Sieve size (mm)	19	12.5	9.5	4.75	2.0	0.425	0.180	0.075
Passing (%)	100	95	62.5	37.5	25	17	13	10

Asphalt binder (500±5 g) was firstly heated to 165±5 °C in the container. Then, graphite was added gingerly within 10 min, while the shear speed was kept at 1000 rpm. Hot mix asphalt samples were prepared with the bituminous binders. Graphite was added into pure bitumen in 3 different ratios as 10%, 15% and 20% by weight of bitumen used in mixtures and they were represented as 0% (G0), 10% (G10), 15% (G15) and 20% (G20), respectively. The stone mastic asphalt mixtures obtained with pure and modified binders were aged for 1, 2 and 3 weeks in the oven at 50 °C. In the study totally 4 different mixtures were evaluated with aging. The effects of graphite on mixture's properties were examined by Marshall stability, indirect tensile fatigue and indirect tensile stiffness modulus tests with cylindrical shape specimens prepared 101 mm diameter and 65 mm height.

3. Test methods

3.1. Marshall stability test

Marshall stability test on hot mix asphalt specimen was applied according to TS EN 12697-34 (2004). Stability is described as the maximum strength against deformation. Flow is also the vertical deformation that occurs in time which reaches the maximum load of the sample in the standard. The samples were stored in a water bath at 60±1 °C for 40 minutes. The samples were loaded at a speed of 50±2 mm/min. In the experiment, the maximum load and deformation occurred at the maximum load were recorded.

3.2. Indirect tensile stiffness modulus test

The stiffness modulus which is a measure of the load distribution capability of bituminous pavement is one of the most important performance characteristics of bituminous hot mixtures (Zoorob, Suparma 2000). The indirect tensile stiffness modulus (ITSM) test, a non-destructive and deformation-controlled test, described in BS DD 213:1993 standard and it is carried out by using the UMATTA (Universal Material Testing Apparatus). The test apparatus are shown in Figure 2. The test was carried out at 20 °C. Before the experiment, the samples were kept at the test temperature for at least 3 hours. The values such as sample height, diameter, estimated Poisson ratio (0.35), target horizontal deformation (6.5 µm), load application time (3000 ms) and load rise (124 ms) times were entered the software. ITSM values S_m (MPa) were calculated according to following formula:

$$S_m = F(R + 0.27) / LH, \quad (1)$$

where F is the maximum vertical load (N); H is the average horizontal deformation (µm) occurring after 5 load repetition; L is the average sample thickness (mm) and the R is the Poisson ratio (0.35).

3.3. Indirect tensile fatigue test

The bituminous materials used on the roads are exposed to a short time load during the passage of each vehicle.

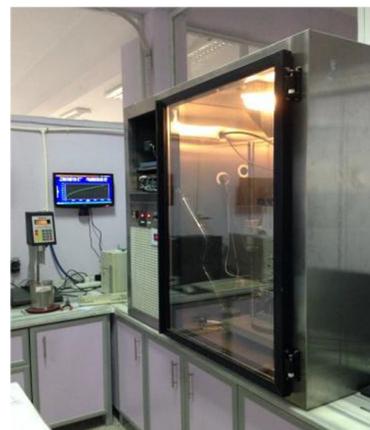


Fig. 2. The indirect tensile stiffness modulus test apparatus

These loads lead to micro-damage which reduces the rigidity of the material. These micro-damages cause pavement deterioration called fatigue cracks in long-term (Francken 1998). Fatigue cracks commonly occurred at bituminous hot mixtures, are the type of deterioration caused by the load. Fatigue cracks are gradually increasing by following the formation of the crack with repetitive loads. The cylindrical test specimens are applied to repeated pressure loads in the vertical diametric plane. This loading creates tensile stresses relatively uniform in the horizontal diametrical direction and vertical to the applied load direction. The tensile stresses cause to split in the middle at the vertical direction of the specimen. The test apparatus are shown in Figure 3. The test was carried out at stress controlled condition by applying cyclic constant loads of 350 kPa with a 0.1-s loading followed by a 1.4-s rest period. The test was carried out at 20 °C. The experiment continued until the specimens completely collapsed.

4. Results and discussions

4.1. Marshall stability test results

Marshall specimens were prepared by applying 50 blow on both sides due to determine the optimum bitumen ratio of the asphalt mixtures prepared with the pure binder. The bituminous content was selected with 0.5% increments from 5% to 7%. The fiber which is used for stabilizing the high amount of binder was used as 0.5% by weight of the mixture. The volumetric properties such as bulk specific gravity (G_{mb}), air void (V_a), voids in mineral aggregate (VMA), voids filled with asphalt (V_{fa}) and the stability-flow values of the samples prepared in different bitumen contents were determined. The optimum bituminous content of the pure mixture was determined as 6.5%. The graphite-added samples were prepared in the same bitumen content. The average values of the characteristics of all samples with 6.5% bitumen content are given in Table 5. It was found that Marshall stability values declined with the increasing of graphite content. This reduction can be thought to be due to the oily properties of graphite. The maximum usage of graphite (20%) induces only 6% reduction in stability. All of the mixtures provide the minimum VMA and air voids requirements which are 16% and 3–4% respectively. Stone mastic asphalt mixtures give high flow values compared to dense graded mixtures. An example of a stability-flow relation

Table 5. The average values of the characteristics of the mixtures

Specimen Type	G_{mb}	V_a (%)	VMA (%)	V_{fa} (%)	Stability (kgf)	Flow (mm)
G0	2.307	3.59	17.07	79.91	962.55	6.80
G10	2.313	3.34	16.86	80.10	938.23	6.33
G15	2.306	3.59	17.08	78.96	929.40	6.62
G20	2.308	3.53	17.02	79.27	905.18	6.86



Fig. 3. The indirect tensile fatigue test apparatus

is given in Figure 4 (Kök *et al.* 2014). As it is seen here that SMA mixtures hold the load in its body longer than that of the dense graded mixtures due to having a coarse gradation. Therefore SMA mixtures do not show a peak stability value, it bears the load longer than the dense graded mixture in the meantime it exhibits high flow values.

4.2. Indirect tensile stiffness modulus test results

For this experiment, three samples were prepared for each sample type. Each sample was subjected to loading at three different locations and 9 values were obtained for a sample type. ITSM values were determined by calculating the averages of the seven values outside the largest and the smallest of these values. The test was carried out at 20 °C with deformation control. The target deformation was set to 6.5 microns.

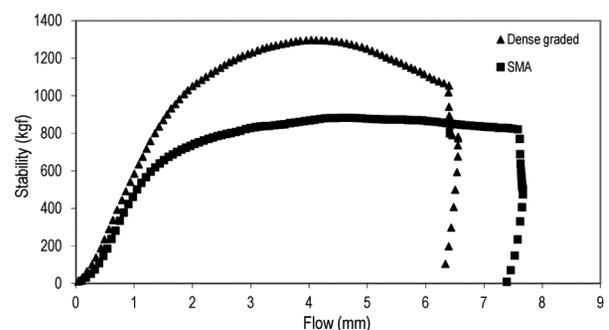


Fig. 4. The stability-flow curves of SMA and dense graded asphalt (Kök *et al.* 2014)

At first, the deformation-time characteristics of the specimens were examined. The deformation-time relation of the original and aged samples is given for pure and 20% graphite added samples in Figure 5. At the representation of the specimen the first number indicate the graphite content and the second one presents the aging time in a week. G20-3 stand for 20% graphite added samples aged 3 weeks at 50 °C in the oven. It is seen from the figures that the deformations return since the load is removed after the sample reaches to the maximum target deformation at about 150 milliseconds. It was determined that the deformations at 400th milliseconds are between 2 and 3 microns in all graphite contents. Even at the highest graphite level (20%), there is no significant difference either positively or negatively in the elasticity properties compared to the pure mixture. The effects of aging time on deformation characteristics do not so important for both pure and graphite added mixtures.

The area under the stress-deformation curves is considered as a measure of the load distribution capability of the sample. Stress-deformation relations of the unaged and three-week aged specimens are given as depending on the graphite due to see the effects of the graphite rate on the areas in Figure 6. While there is no significant difference between the unaged pure and graphite added samples, 20% graphite added mixture exhibits different performance compared to others after the three-week aging conditions. Since this test is performed at elastic region by considering the 6.5 deformations as target deformation, it enables to compare the flexibility properties of the samples in the linear viscoelastic region. In this

respect, it can be concluded that the graphite added mixtures up to 15% graphite content do not have a significant performance compared to the pure mixture. Besides the high amount of graphite (20%) take effect after 3-week aging conditions.

Figure 7 shows the effect of aging time on the stress-deformation relation of pure and G20 mixtures. It is seen that the areas under the curves change at a considerable degree depending on the aging period. In all samples, it was determined that the areas rise with the increase of the aging time indicating an improved ability to absorb the elastic energy, at medium temperatures.

Stiffness modulus (S_m) of all mixtures types in all aging conditions are given in Figure 8. It is seen that graphite content does not have any influence on stiffness modulus at the unaged condition. Pure and 10% graphite added mixtures exhibit similar performance at all aging time. S_m values of these mixtures increase gradually with the aging time. However, the mixtures show different performance after 15% graphite content depending on the aging time. The aging time loses its significance for the 15% and 20% graphite added mixtures. The S_m values of the G15 and G20 mixtures after 1, 2 and 3 weeks are close to each other but 49–58% higher than that of the unaged ones. 20% graphite content also induces 44% increment in the S_m values of the pure mixture.

4.3. Indirect tensile fatigue test results

In the fatigue test, three samples experimented to each type of mixture. The load repetition number – deformation relations of the pure and 20% graphite-added speci-

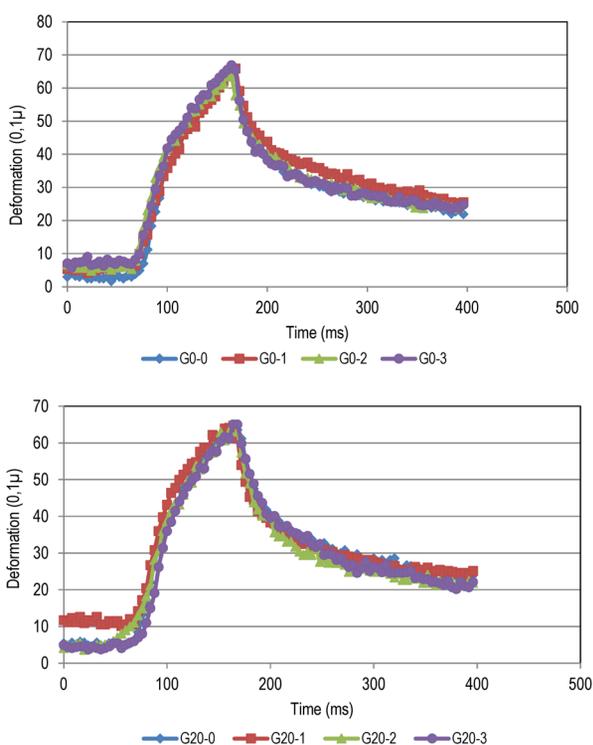


Fig. 5. The deformation-time relation of the asphalt mixtures

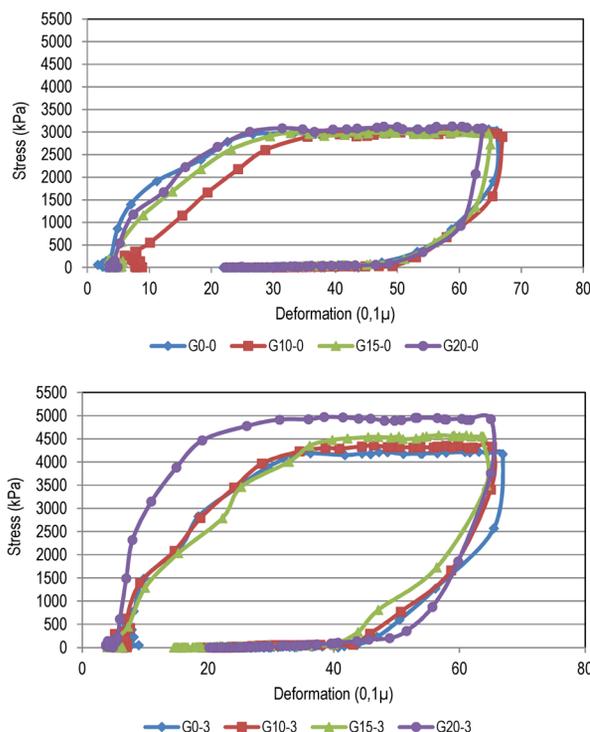


Fig. 6. The stress-deformation relation of the asphalt mixtures with graphite ratio

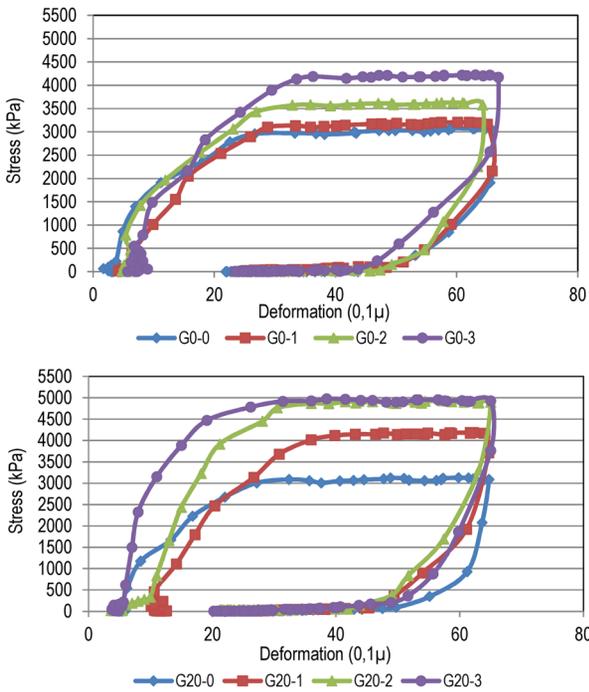


Fig. 7. The stress-deformation relation of the asphalt mixtures with aging

mens at different aging times are given in Figure 9. These curves belong to a nearest average of the three samples tested for each type of mixture. As it can be seen from the figures, all samples show a rapid deformation at the beginning and the increase of deformation trend continues linearly and then increases rapidly again. The rapid deformation increase of the curves at the beginning is caused by the decreasing of the air volume due to compression in the samples. The specimens exhibit elastic behavior for certain time by inducing a linear increase in deformations. After the initial crack in the specimen, the deformation increase accelerates again. In the phase after the beginning of the crack, which is called as the third region in the pure mixture, it is seen that the samples do not take up much load and break even before reaching the third region. In graphite-added samples, the third region is obviously formed. In other words, after the beginning

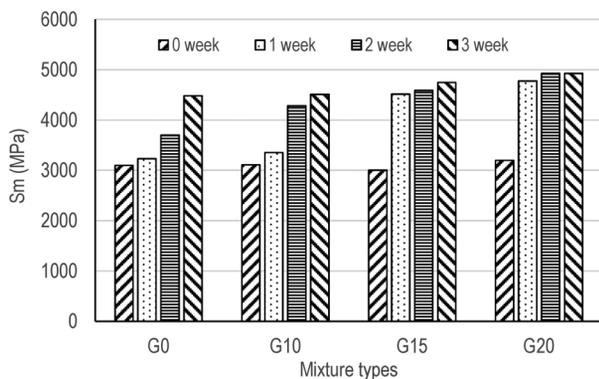


Fig. 8. The stiffness modulus (Sm) values of mixtures in aging conditions

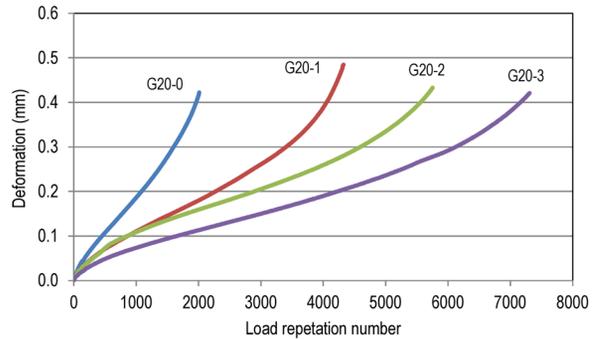
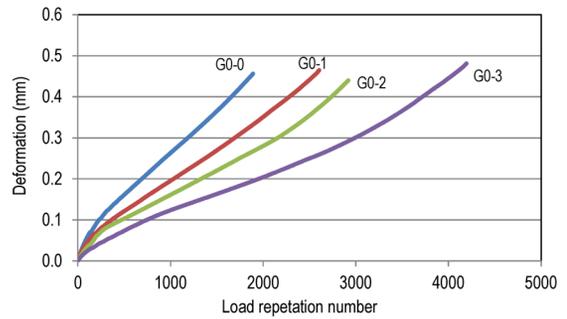


Fig. 9. The load repetition number-deformation relation of the mixtures with aging

of the crack, the graphite-added specimens show more resistance to the progress of the crack.

The load repetition number of all sample types increases significantly with the increase of aging time. Since the samples were collapsed at different deformation levels, 4 mm deformation was selected as the threshold value, which is the lowest deformation level reached by all samples in order to make a correct comparison between them. The average load repetition numbers of the specimens at 4 mm deformation are given Figure 10. The figure provides for assessing the aging effect on mixture by considering the slope of the curves. Pure and G10 mixtures have the lowest slope, G15 and G20 have the highest. Effects of aging time on load repetition number is more pronounced after the 15% graphite content. G10, G15 and G20 mixtures have 7%, 65% and 78% higher load repetition number compared to the pure mixture after 3-week aging condition.

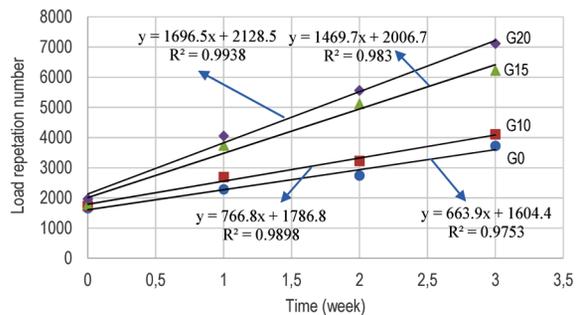


Fig. 10. The load repetition numbers of the mixtures at 4 mm deformation

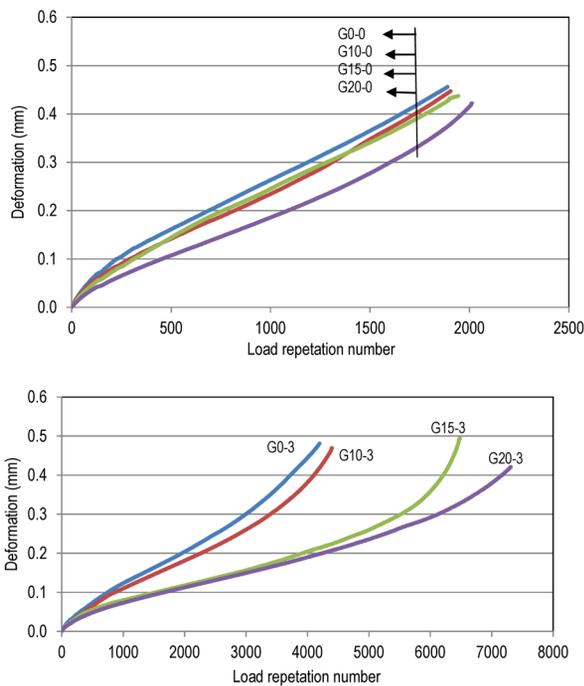


Fig. 11. The load repetition number-deformation relation of the mixtures

The deformation-load repetition number relationship of samples in the different graphite contents is given Figure 11 for the unaged and 3-week aged conditions. The graphite content in the unaged samples has no significant effect on the load repetition number but the increase of the graphite content with aging time has a significant effect on the load repetition number.

The variation of load repetition number versus graphite content for original and aged mixtures are given in Figure 12. Here, it can be said that graphite-added samples are more aged, in other words, they are more affected from aging. However, graphite-added mixtures which take more load repetition number than the pure mixture in the fatigue test will be resistant to repeated loads of traffic for a longer time without cracking at medium temperatures. The curves are become upright and become decumbent at the left and the right side of the 15% graphite content at the aged situations. Hence the 15% graphite content can be assumed as the most effective value in terms of fatigue life.

Conclusions

In this study, the effects of graphite on mechanical features of stone mastic asphalt pavement were researched. Mechanical tests such as Marshall stability and flow, indirect tensile stiffness modulus and indirect tensile fatigue tests were applied to bituminous mixtures at three different proportions of graphite by weight 10%, 15% and 20% of bitumen. It was determined that there is no significant difference in mechanic properties of graphite added mixtures compared to the pure mixture at the unaged condition. The aging time considerably effects the performance

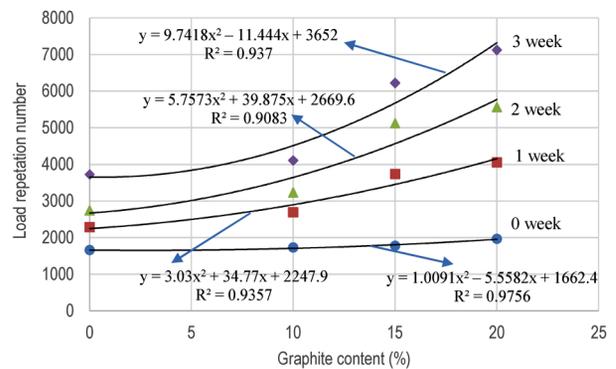


Fig. 12. The load repetition number-graphite ratio relation of the mixtures

of pure and graphite added mixtures. The area under the stress-deformation curves of aged mixtures indicates an improved ability to absorb the elastic energy, at medium temperatures. After 15% graphite content the aging time lose its significance in terms of stiffness modulus. The fatigue test results are also similar to the stiffness modulus' test results. While the load repetition number of all mixture closes each other, graphite added mixture exhibits a superior performance after aging. Effects of aging time on load repetition number are more pronounced after the 15% graphite content. G10, G15 and G20 mixtures have 7%, 65% and 78% higher load repetition number compared to the pure mixture after 3-week aging condition.

Finally, it was found, 15% graphite content is an effective ratio when considering the elastic response, the absorbed energy, the deformation behavior after the initial crack and load repetition number induced to deterioration. According to these results, graphite generally used for improving the thermal properties in literature were also determined to contribute to mechanical properties of mixtures at medium temperatures.

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