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# Performance Investigation of Diatomite Modified Asphalt Mixtures for Different Diatomite Ratios and Grinding Sizes

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## ABSTRACT

Modification of asphalt mixtures has become almost mandatory today due to increased stresses in pavements, shortening of load cycle times, and decreases in binder quality. For this reason, many additives can be added to bitumen or asphalt mixture. Industrial material wastes can also be among these additives. When diatomite material is used as a performance enhancer in asphalt mixtures, it significantly improves the main performance indicators of the asphalt mixture. However, low temperature cracking of diatomite-modified asphalt mixtures is still controversial in the literature. This study evaluated the asphalt mixture in terms of low-temperature cracking, water damage, and rutting, depending on the diatomite grinding size (gradation) and addition ratio. Three different sizes of diatomite additives (106, 212 and 300-micron maximum diameter) were used at three addition ratios (5, 10 and 15% by weight of bitumen). According to the test results, it was seen that the mechanical properties of asphalt mixtures were significantly affected by the addition ratios and diatomite sizes, and the use of 300-micron maximum diameter diatomite at the rate of 10% and 15% was more effective. However, according to the BBR test results, the use of diatomite additives with a maximum size of 106 μm at 5% slightly increased the low temperature cracking resistance.

**Keywords:** Asphalt mixture, diatomite, grinding size, cracking, low temperature cracking.

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## 1. INTRODUCTION

Diatomite is a mineral that is a low-cost mineral, easy to produce, and does not cause environmental pollution. The European region is very rich in diatomite reserves. Turkey is the country with the highest diatomite reserves after America and China. The diatomite reserves in the United States, China and Turkey represented around 250, 150 and 44 million metric tons, respectively, in 2022 [1].

The distinctive characteristics of diatomite have resulted in its adoption in various industrial uses, with the primary applications encompassing liquid filtration and serving as a bulking agent, as seen in the context of paint production. Additionally, diatomite finds utility in insulation, particularly in the production of fire-resistant bricks, as well as in fine abrasion applications, such as toothpaste and various polishing products. Furthermore, it is employed as an effective pesticide in certain scenarios [2].

One of the applications of diatomite is in the asphalt pavement industry. Incorporating diatomite into asphalt can have multiple advantages. This method can utilize natural resources to their fullest potential and enhance the asphalt's overall performance. Additionally, preparing a diatomite-modified asphalt mixture is a straightforward process that doesn't require any specialized equipment. Moreover, the construction process for this type of pavement is identical to that of regular asphalt, making it an economically feasible option [3].

Relevant research has indicated that the incorporation of diatomite can greatly enhance the resistance of bitumen to high-temperature rutting and thermal oxidative aging [4,5]. Furthermore, it is stated that diatomite exhibits superior performance compared to hydrated lime and fly ash in enhancing asphalt mixture properties [6–9]. The improvement of asphalt performance through diatomite is primarily attributed to physical adsorption, as the exceptional adsorption capacity of diatomite effectively inhibits the thermal and oxidative aging of asphalt binders [10]. Diatomite effectively adsorbs low molecular weight compounds and low polarity aromatic molecules in bitumen, while exhibiting limited adsorption capacity towards asphaltene and resin [11]. Diatomite possesses robust adsorption and hardening characteristics on bitumen leading to significant enhancement in bonding performance, strength, stiffness, and anti-aging properties of asphalt mixtures [12].

In research conducted on diatomite-modified asphalt mixtures, emphasis has been placed on the high-performance characteristics of the modified mixtures. The study showed that diatomite outperformed higher performance than kaolin, Na-bentonite, and Ca-bentonite in view of the high-temperature performance, low-temperature performance, and temperature sensitivity of asphalt mixtures [13]. Also, diatomite-modified asphalt mortar exhibited excellent rutting and water damage resistance [14,15]. In another study, it was stated that diatomite had the potential to enhance the water damage resistance of asphalt mixtures based on freeze-thaw splitting tests [16]. Diatomite possessed strong adsorption capacity, which reduced the contact and binding opportunities of water with aggregates, consequently improving the water stability of asphalt mixtures [3]. The impact of diatomite on the low-temperature performance of asphalt mixtures using various testing methods was studied and it was concluded that diatomite-modified asphalt mixtures exhibited better low-temperature performance compared to matrix asphalt mixtures [17]. In another investigation, it was highlighted that the addition of diatomite improved the crack resistance and fatigue properties of asphalt mixtures based on beam bending tests and four-point bending fatigue tests [18].

Diatomite can be used in combination with other additives, fibers, or fillers to improve the final mechanical properties, aging properties, and anti-icing performance of the asphalt mixture [7, 19-24]. It is understood from the literature that the general performance of diatomite-modified asphalt mixtures is high. Therefore, diatomite has the potential to be used as an additive in asphalt mixtures. However, some studies have observed that diatomite modification reduces the asphalt mixture's fatigue performance [25] and low-temperature cracking resistance [17]. Some studies have stated that the diatomite additive is almost ineffective in terms of low-temperature cracking [4].

In a study by Wang et al., a clean asphalt production method was investigated to reduce the damage to the environment and human body. In this context, diatomite and modified-attapulgite were multi-modified. The results of the BBR test on asphalt mixtures proved that the additives slightly reduced the low temperature properties of asphalt pavements [26].

In a study, it was found that rubber and diatomite modification of asphalt mixtures can improve the high temperature stability and low temperature cracking resistance of asphalt mixtures, but especially the effect of improving the low temperature cracking resistance is weaker than that of SBS modified ones. This was attributed to the fact that rubber and diatomite particles mainly form a physical bond in asphalt mixtures and the effects of this bond are weaker than the chemical modification of SBS [27].

In a study where basalt fiber was used to overcome the inadequacies of diatomite modified asphalt mixtures in low temperature performances, in addition to the control (unmodified) mixture, the mixture modified with diatomite at 15% of the asphalt binder ratio; asphalt mixture modified with basalt fiber additive at the rate of 0.3% of the mixture weight by dry method; diatomite modified with 15% of the bitumen by wet method and basalt fiber modified with 0.3% of the asphalt mixture by dry method. To mix diatomite into bitumen, the bitumen was heated at 135°C for 4 hours and then mixed in a high-speed mixer for 15 minutes. Basalt fiber was added to the heated aggregate and mixed for 5 minutes. Diatomite-modified bitumen was added to the basalt aggregate mixture and mixed thoroughly. The asphalt mixture samples were subjected to low temperature cracking test at -5°C and 4-point beam bending test at 20°C. The results showed that the combination of diatomite and basalt fiber compensated well for the vulnerability of asphalt mixtures to low temperatures [24].

In a study by Li et al., surface modified diatomite and bio-oil were used at 0, 5, 10, 15% by mass of bitumen and the modification processes were carried out with a high-speed mixer at 155°C at a shearing speed of 4500 rpm for 60 minutes. As a result of the BBR test, it was observed that hybrid modification can improve the low temperature behaviour of asphalt mixtures [28].

In a study carried out by Aksoy et al., it was aimed to find the optimum ratios of diatomite filler additive, which is extracted as waste for a sustainable future and the environment, that could potentially be used in asphalt plants, and therefore modification processes were carried out at different ratios. Asphalt mixtures were modified by wet method. Diatomite additives were used at 5% and 10% by weight of bitumen. Asphalt mixture samples were evaluated in terms of rutting resistance by repeated load creep test and the highest rutting resistance was obtained with 5% diatomite ratio in unconditioned samples and 10% diatomite ratio in conditioned samples [29].

The main uses of diatomite additives were summarised above, and it was observed that diatomite modified asphalt mixtures were effective in terms of high temperature, rutting and water damage resistance, but ineffective in terms of low temperature cracking resistance. The reason for this weakness of diatomite modified asphalt pavements in low temperature performance is thought to be the physical bonding of diatomite with the asphalt mixture. It was observed that the use of diatomite-basalt fiber increased the resistance of the asphalt mixture to low temperature cracking. As seen in the literature research, it is important to determine the optimum diatomite ratio. In addition, no study investigating the size effect using diatomite at different grinding sizes has been found in the literature and it is thought that the size effect of diatomite additives used in this study will contribute to the literature.

### 1.1. Objectives and Scope

More current and precise information is needed on the effects of diatomite modification on low-temperature cracking. The overall performance of diatomite-modified asphalts depends on many factors such as the type of diatomite used, mixing ratio, other additives and conditions of use. The potential of diatomite modification to improve the performance of asphalt mixtures has been demonstrated in many studies. However, these studies generally did not distinguish between different types of diatomite or focused on a limited number of properties. This deficiency raises the possibility that different diatomite types or gradations may have different effects on asphalt mixture performance. The main purpose of this study is to evaluate the effects of grinding size and addition ratio of diatomite on asphalt mixture performance. For this purpose, diatomite additives obtained in three different grinding sizes were added to bitumen at three different addition ratios and the asphalt mixture samples produced were evaluated by repeated creep test, water damage test and indirect tensile test. The modified binders were evaluated by bending beam rheometer (BBR) test, penetration, and softening point tests.

## 2. MATERIAL AND METHOD

### 2.1. Material

The study used 50-70 penetration grade asphalt cement, whose properties are shown in Table 1, and limestone aggregate in Table 2. According to the Turkish Highways Technical Specification [30], aggregate gradation suitable to produce dense-graded asphalt concrete was determined. Aggregate gradation curve is given in Figure 1.

*Table 1 - Pure bitumen properties*

| Test | Test method | Result | Specification limits |
|---|---|---|---|
| Specific gravity (25°C) | ASTM D70 | 1.011 | |
| Softening point (°C) | TS EN 1427 | 52 | 46-54 |
| Flash point (°C) | TS EN ISO 2592 | 240 | ≥ 230 |
| Penetration (25°C) 0.1mm | TS EN 1426 | 63 | 50-70 |
| Ductility (25°C) | ASTM D-113 | 100+ (cm) | |
| Solubility (%) | TS EN 12592 | 99.8 | ≥ 99 |

*Table 2 - Limestone Aggregate Properties*

| Aggregate properties | Test method | Result | Specification limit |
|---|---|---|---|
| Specific gravity (coarse aggregate) | ASTM C 127 | | |
| Bulk | | 2.707 | |
| Apparent | | 2.733 | |
| Specific gravity (fine aggregate) | ASTM C 128 | | |
| Bulk | | 2.763 | |
| Apparent | | 2.776 | |
| Los Angeles abrasion (%) | TS EN 1097-2 | 26.70 | ≤27 |
| Water absorption (%) | TS EN 1097-6 | 0.90 | ≤2 |
| Soundness (MgSO4) (%) | TS EN 1367-2 | 2.4 | ≤16 |
| Flakiness (%) | BS 812 | 9.71 | ≤25 |
| Stripping resistance (no additive) (%) | TS EN 12697-11 | 60-65 | ≥60 |
| Plasticity index for sandy aggregate | TS-1900-1 | NP | NP |
| Organic matter for sandy aggregate | TS EN 1744-1 | Negative | Negative |

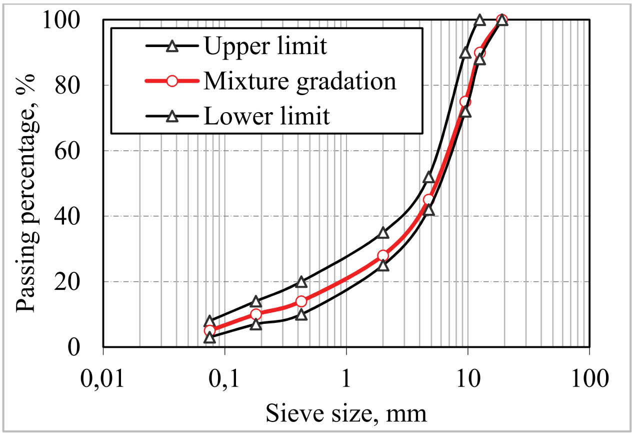


*Figure 1 - Aggregate gradation curve*

Three diatomite additives obtained from Bentaş Bentonit Mining Industry and Trade Joint Stock Company and coded as D1 (First type diatomite), D2 (Second type diatomite), and D3 (Third type diatomite) were used in the study. Properties of agents were given in section 2.2.2 in detail.

## 2.2. Method

### *2.2.1. Preparation of Diatomite Modified Bitumen*

Direct and indirect (wet) methods can be used to prepare the diatomite-modified asphalt mixture. When the direct blending method is used, diatomite is added to the asphalt and aggregate mixture as mineral powder. When the indirect method is used, diatomite-modified asphalt binder is prepared before preparing the mixture [31]. It is stated that the two blending methods lead to approximately the same blending effect [32]. The wet method was used in this study. Diatomite additives were added to the bitumen at the rates of 5%, 10%, and 15% by weight (according to the asphalt mixture weight, respectively: 0.243%; 0.485%; 0.728%), and asphalt mixtures were prepared with diatomite-modified bitumen.

There have been some studies which were researched the diatomite-bitumen modification; in a study, 50/70 penetration grade bitumen was used and diatomite was added to the bitumen at the rates of 5%, 10%, 15% and 20% by weight. For the preparation of diatomite modified asphalt, the shearing temperature was chosen as 150°C, the shearing speed was 3000 rpm, and the shearing time was 120 minutes for the preparation of diatomite-modified asphalt [4]. In another study, 70/100 penetration grade bitumen was used and diatomite was added to the bitumen as 0.1%, 0.2% and 0.3% according to the total asphalt mixture weight. Mixture preparation temperature was taken as 150°C, the shearing speed was 4000 rpm, and the shearing time was 40 minutes [33]. In another study, 80/100 penetration grade bitumen was used in the asphalt mixture and 14% diatomite was added to the asphalt cement. The shearing temperature was 160°C, the shearing speed was 5000 rpm, and the shearing time was 40 minutes [17].

In the current study, diatomite-modified bitumen was processed at 4000 rpm at 160 °C. It was prepared with a mixing time of 40 minutes at shear speed.

### *2.2.2. Chemical Properties*

XRF (X-ray fluorescence) main oxide analysis results of diatomite additives are shown in Table 3. XRD (X-ray diffractometer) graphs of diatomite additives are given in Figure 2, 3, 4. EDS (Energy Dispersive Spectrometry) layered images of diatomite additives are presented in Figures 5, 6 and 7. EDS Map sum spectra of the additives are presented in Figures 8, 9 and 10; and SEM (Scanning Electron Microscopy) images are presented in Figures 11, 12 and 13. When Figures 2, 3 and 4 are analysed; it is clear that; the densest diatomite contribution in terms of minerals according to XRD results of diatomite additives is in D1 diatomite with 5000 levels. In Figure 3, it is observed that the quartz peak and anorthite peak are intense. It is also understood that the amount of paragonite is also high. It is understood that D2 diatomite has low anorthite content compared to D3 diatomite; but it is rich in Na mineral. Among the 3 samples, it is observed that the amount of quartz and anorthite in D1 is high and D1 is the richest contribution in terms of mineral. As the maximum grain size decreased, different residual phases in the samples appeared. The SEM/EDS data showed that the elements distribution in D1, D2, D3 are mostly rich in terms of C, O, Si however the Fe, Ca, K, Mg contents showed differences on the diatomite samples due to the particle size difference. The overall contents of element distribution can be estimated by D1, which has a finest particle size with 1.5wt% Mg, 3.8wt% Al, 1.0wt% Ca and 1.4 wt% Fe. The surface of all samples has a non-uniform microstructure with voids and pores. The density of the D1 surface is higher than the other samples since the particle size of D3 is the lowest.

*Table 3 - Main oxide analysis results of diatomite additives*

| | Unit | D1 | D2 | D3 |
|---|---|---|---|---|
| $SiO_2$ | % | 73.955 | 74.099 | 74.199 |
| $Al_2O_3$ | % | 8.989 | 8.437 | 8.832 |
| $Fe_2O_3$ | % | 2.899 | 2.839 | 3.032 |
| MgO | % | 2.485 | 3.049 | 2.649 |
| CaO | % | 2.270 | 1.852 | 2.434 |
| $Na_2O$ | % | 0.207 | 0.109 | 0.164 |
| $K_2O$ | % | 1.256 | 1.002 | 1.274 |
| $TiO_2$ | % | 0.511 | 0.460 | 0.518 |
| $P_2O_5$ | PPM | 0.206 | 0.139 | 0.199 |
| MnO | % | 0.102 | 0.054 | 0.149 |
| $SO_3$ | % | 0.014 | 0.016 | 0.018 |
| Cl | PPM | 94 | 83 | - |
| BaO | % | 0.032 | 0.017 | 0.041 |
| CuO | % | 0.194 | 0.319 | 0.294 |
| NiO | % | 0.227 | - | 0.009 |
| SrO | % | 0.038 | 0.029 | 0.046 |
| $V_2O_5$ | % | 0.0739 | 0.077 | 0.072 |
| ZnO | % | - | - | 0.008 |
| $ZrO_2$ | % | 0.028 | 0.018 | 0.033 |

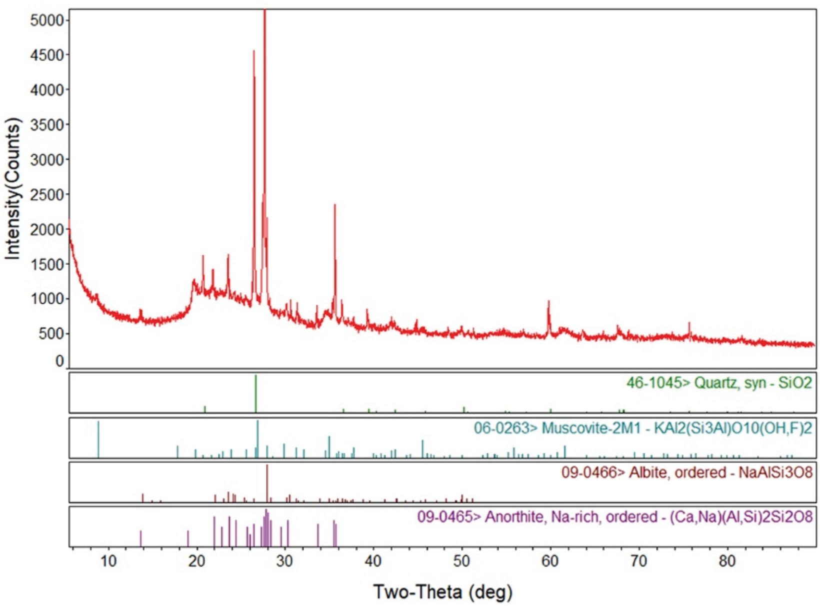


*Figure 2 - XRD test result of D1 diatomite*

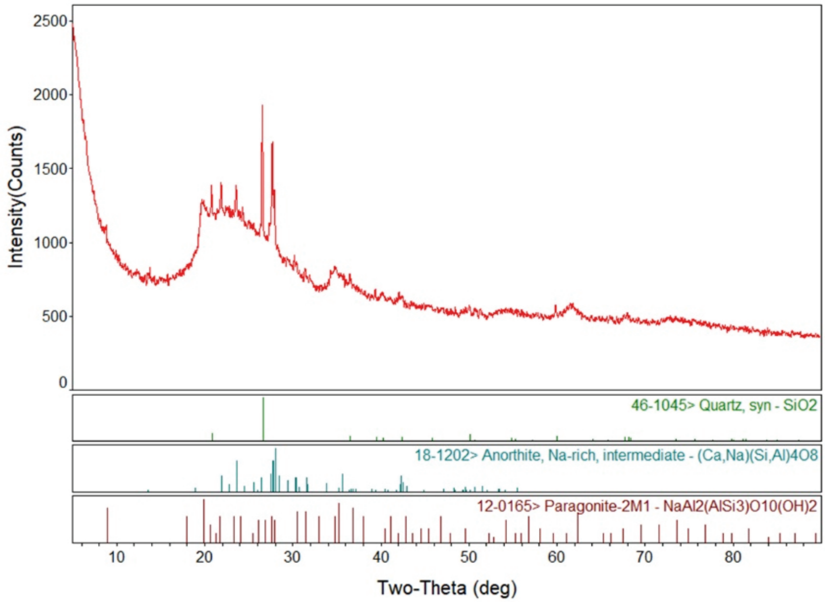


*Figure 3 - XRD test result of D2 diatomite*

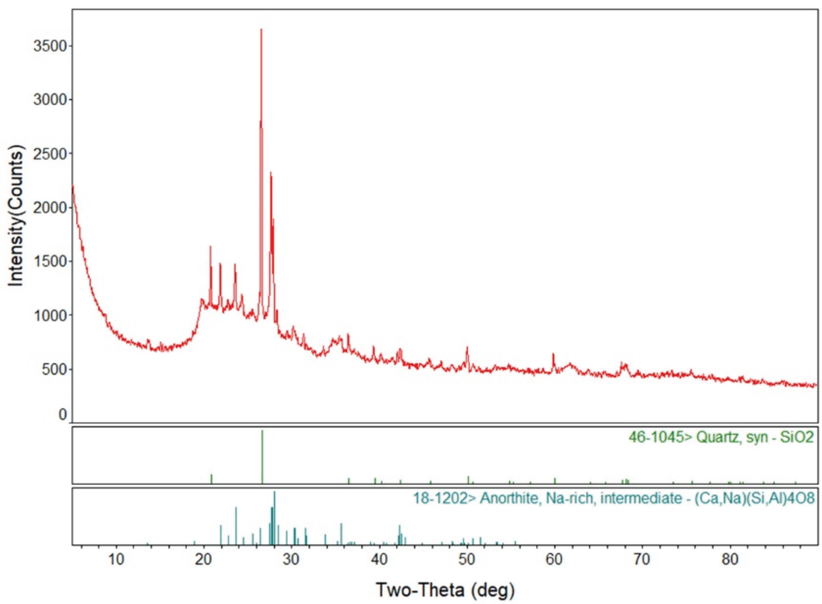


*Figure 4 - XRD test result of D3 diatomite*

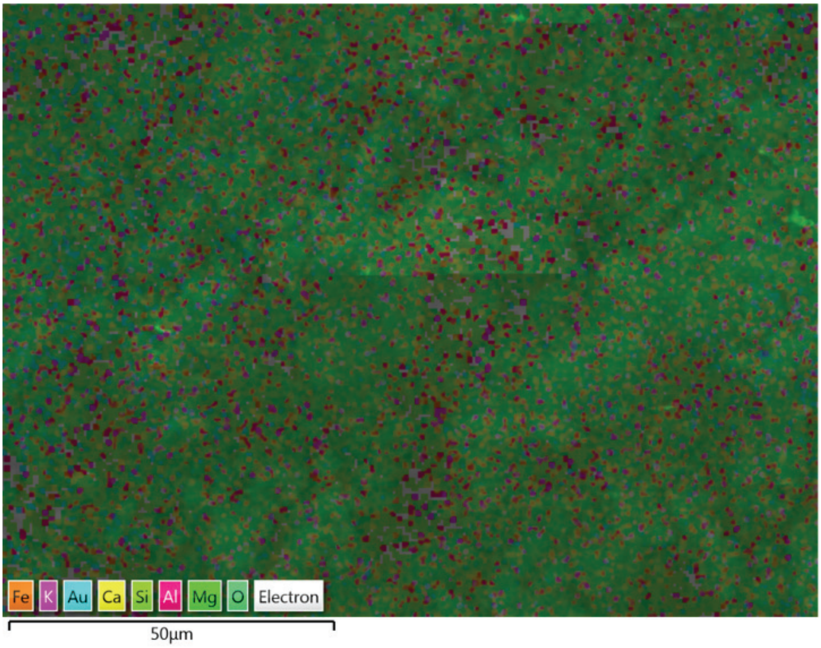


*Figure 5 - EDS layered image of D1 diatomite*


*Figure 6 - EDS layered image of D2 diatomite*

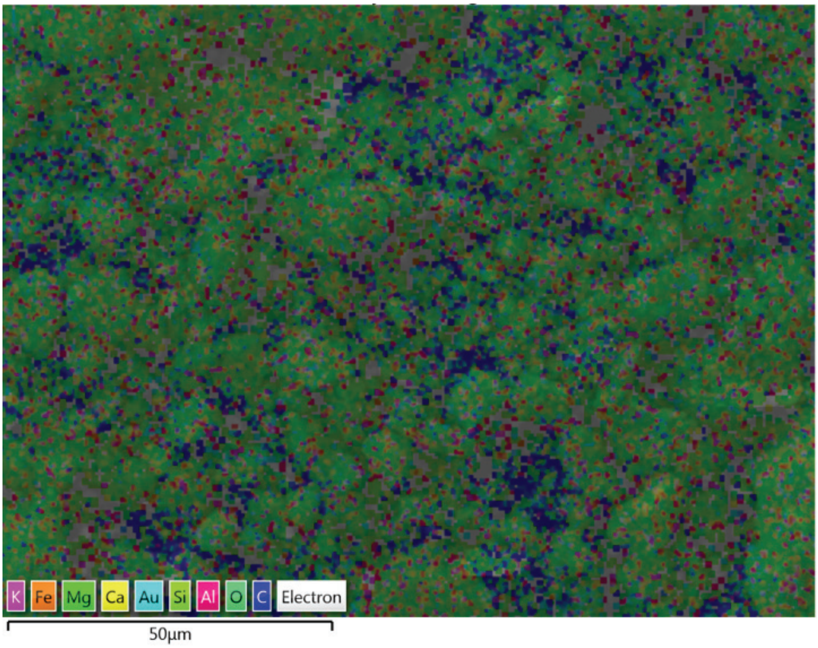


*Figure 7 - EDS layered image of D3 diatomite*

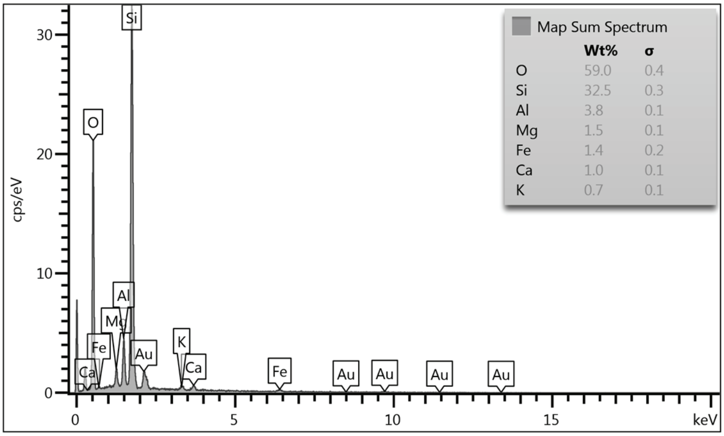


*Figure 8 - EDS map sum spectrum of D1 diatomite*

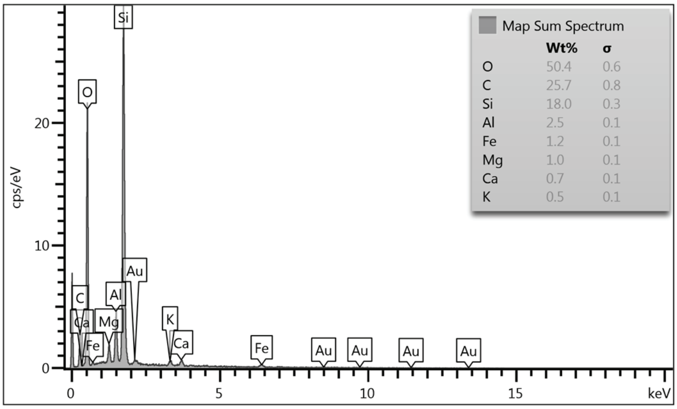


*Figure 9 - EDS map sum spectrum of D2 diatomite*

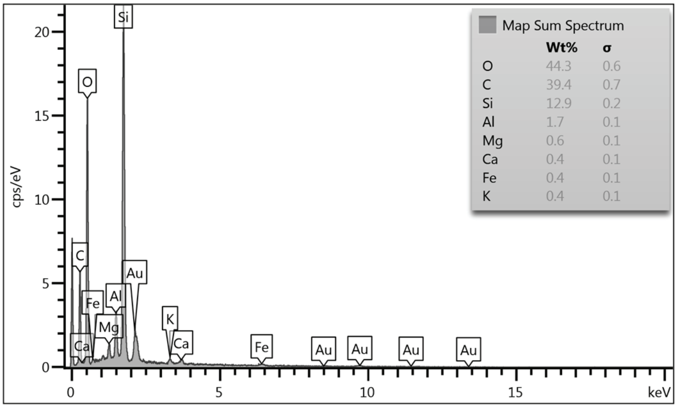


*Figure 10 - EDS map sum spectrum of D3 diatomite*

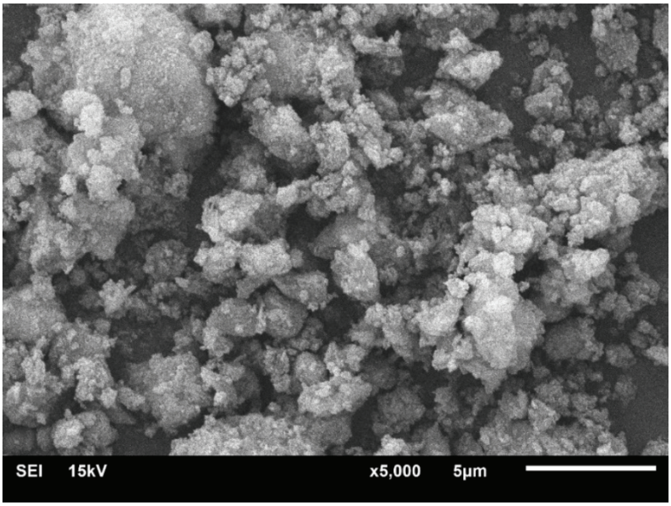


*Figure 11 - SEM image of D1 diatomite*

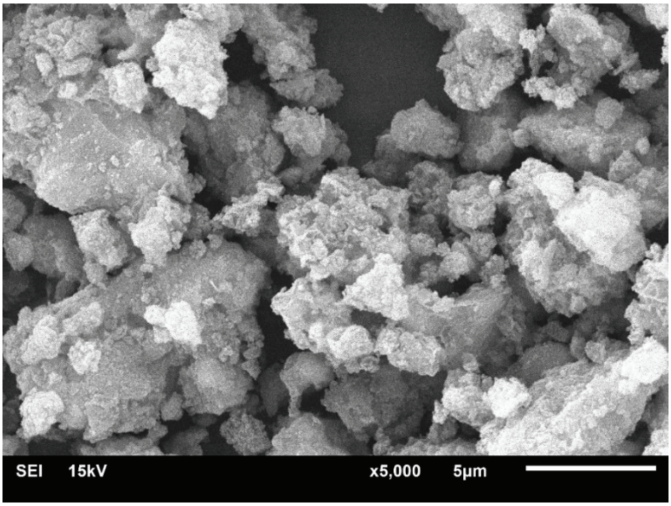


*Figure 12 - SEM image of D2 diatomite*

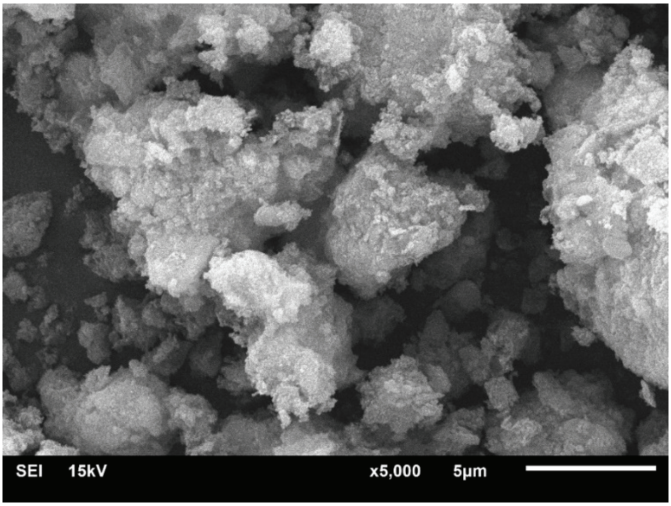


*Figure 13 - SEM image of D3 diatomite*

### *2.2.3. Mix Design Properties*

Gradation analysis results of diatomite additives are shown in Table 4. From Table 4, it can be seen that D1 diatomite is in the finest gradation and D3 diatomite is in the coarsest gradation. 50% of D1, D2 and D3 diatomite are thinner than 9.46 microns, 111.35 microns, and 190 microns, respectively. At the same time, the maximum diatomite size is also in the same order. The low-temperature tensile strengths of D1-modified asphalt mixtures were lower than those of D2 and D3 diatomite used at the same inclusion rates. The higher surface area of D1 diatomite may have increased the stiffness of the mixture by absorbing more asphalt cement. This may have had a negative impact on the low temperature strength of asphalt mixtures. The higher tensile strength of D2 and D3 diatomite confirms this idea.

In accordance with the Highway Technical Specification [30], the dense graded asphalt mixture design was made with pure bitumen according to the Marshall method. The bitumen content giving 4% air void was taken as the optimum bitumen content and calculated as 5.10%. It was observed that other mixture properties also met the specification limits in the calculated bitumen content (Table 5). Control mixtures and diatomite-modified mixtures were produced at the calculated bitumen content.

Control and diatomite-modified asphalt mixtures were investigated for low-temperature cracking, water damage, and rutting problems. Conditioned and unconditioned samples were used in the experimental processes.

*Table 4 - Gradation analysis results of diatomite additives*

| D1 diatomite additive material | | D2 diatomite additive material | | D3 diatomite additive material | |
|---|---|---|---|---|---|
| Sieve size, μm | Gradation, (% Passing) | Sieve size, μm | Gradation, (%Passing) | Sieve size, μm | Gradation, (%Passing) |
| 106 | 100 | 212 | 100 | 300 | 100 |
| 80 | 98.4 | 150 | 99.1 | 212 | 68.2 |
| 60 | 97.1 | 106 | 95.1 | 206 | 54.6 |
| 40 | 94.3 | 80 | 88.4 | 150 | 52.1 |
| 30 | 90 | 60 | 79.6 | 106 | 44.5 |
| 20 | 85.4 | 40 | 68.8 | 80 | 31.4 |
| 10 | 76.2 | 30 | 51.9 | 60 | 23.0 |
| 5 | 52.2 | 20 | 42.1 | 40 | 17.2 |
| 2 | 25.1 | 10 | 30.9 | 30 | 12.6 |
| 2 | 4.7 | 5 | 18.7 | 20 | 11.1 |
| | | 2 | 10.1 | 10 | 9.2 |
| | | 2 | 2.9 | 5 | 6.6 |
| | | | | 2 | 3.7 |
| | | | | 2 | 1.9 |

Asphalt mix design was made according to 4% air voids and the suitability of parameters such as VMA and VFA was checked. The air voids of the asphalt mix briquettes produced for the experimental studies were calculated by density measurements and given in Table 6 as the average of 15 samples. The air voids of diatomite modified asphalt mixtures increased by 0.07% - 0.13% compared to control mixtures. However, the air voids of diatomite modified asphalt mixtures varied slightly among themselves.

*Table 5 - Asphalt concrete design results*

| Design parameters | Specification limits | Mixture values |
|---|---|---|
| Number of blows per face | 75 | 75.00 |
| Density, gr/cm3 | | 2.44 |
| Marshall stability, kg | Least 900 | 1189 |
| Air voids, $V_h$, % | 3-5 | 4.00 |
| Voids filled with asphalt, $V_f$, % | 65-75 | 71.64 |
| Flow, mm | 2-4 | 3.48 |
| Bitumen content, % | 4-7 | 5.10 |
| VMA (Voids in Mineral aggregate), % | 14-16 | 14.37 |

*Table 6 - Air void ratios of asphalt mixture briquettes*

| Mixture | Control | D1 | | | D2 | | | D3 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 5% | 10% | 15% | 5% | 10% | 15% | 5% | 10% | 15% |
| Air voids (%) | 3.99 | 4.08 | 4.10 | 4.12 | 4.07 | 4.07 | 4.11 | 4.06 | 4.09 | 4.11 |

### *2.2.4. Penetration and Softening Point Tests*

Penetration test determines the consistency of asphalt as well as the class of solid asphalt. As penetration increases, adhesion increases, viscosity and solidity decrease. Asphalt cement is semi-solid at room temperature. It is not possible to predict viscosity at high viscosities. However, the binding ability of asphalt on the road surface depends on the consistency. As the consistency of the asphalt cement increases, the aggregates in the mixture bind more strongly to each other. The penetration of bitumen is defined as the distance in 0.01 mm at which a standard needle penetrates vertically into asphalt cement for a certain time (5 s) under a certain load (100 g) at room temperature [34]. Penetration test standards of bituminous binders are determined by TS EN 1426 [35].

The sensitivity of asphalt to temperature changes can be determined most simply by the "ring and ball" method. Penetration values of some asphalt types at 25°C may be the same. However, when the temperature is increased, a difference in properties can be observed. This is determined by the softening point test. The asphalt sample is placed in a ring of standard thickness and diameter. A ball of standard diameter and weight is placed on the sample and heated rapidly in water. The temperature at which the asphalt in the ring collapses to a certain depth (when it touches the bottom) with the weight of the ball is considered as the temperature at which the asphalt softens (softening point). The softening point test of bituminous binders is determined by TS EN 1427 [36].

The penetration index (PI) is used to get an idea of the temperature sensitivity of bitumen. An increase in the penetration index indicates a decreased sensitivity of the bitumen sample to temperature. The PI value of bitumen is usually between -2 and +2. If this value is close to +2, the temperature sensitivity of bitumen is considered to be low, and if it is close to -2, it is considered to be high [37]. Asphalt mixtures containing bitumen with higher PI have a higher resistance to low temperature cracking and permanent deformation [38]. PI is usually determined by the classical method given in Eq. 1 [39].

$$PI = \frac{1952-500\times\log(Pen25)-20\times SP}{50\times\log(Pen25)-SP-120} \tag{1}$$

where Pen25 is the penetration value of bitumen at 25°C and SP is the softening point of bitumen.

### *2.2.5. Bending Beam Rheometer (BBR) Test*

BBR is performed on aged bitumen samples according to ASTM D6648 [40] to determine the behaviour of bitumen at low temperature. BBR simply indicates the amount of creep or deflection the binder undergoes at a given temperature and under a constant load. Asphalt becomes too hard at low temperatures to rely on the results obtained with DSR. Therefore, a bending beam rheometer has been developed to monitor asphalt at low temperatures. The test is usually carried out at sub-zero temperatures (the test criterion is usually taken as the temperature at which the stiffness of the binder is 300 MPa) and the deflection of a beam-shaped asphalt bar under constant load is monitored over time. In this test, bitumen samples aged by RTFO and PAV tests are used. Thus, the asphalt is tested after mixing and paving operations. The test is carried out under computer control and the instantaneous time-deformation and time-creep stiffness graphs are plotted by the computer to calculate the creep stiffness "S" and creep rate "m" at the end of 60 seconds [41, 34]. The deflection of a bitumen beam of a given size at a given time ($t$) is measured and the $(t)$ of the bitumen is calculated from the classical beam theory using Equation (2). On the other hand, the creep rate is related to the variation of $(t)$ with time $(t)$. For practical reasons, the software of the BBR device establishes a logarithmic relationship between $(t)$ and $t$ in the form of Equation (3). The m-value, which is the slope of the graph of log $(t)$ - log $t$ at a given time $t$, can be calculated using Equation (4), which is the derivative of Equation (3) with respect to log $t$ [42].

$$S_{(t)} = \frac{P\times L^3}{4\times b\times h^3\times\delta_{(t)}} \tag{2}$$

$$logS_{(t)} = A + Blogt + C(logt)^2 \tag{3}$$

$$m(t) = \left|\frac{d[\log S_{(t)}]}{d(logt)}\right| = B + 2C\log t \tag{4}$$

where: $S(t)$ is the flexural creep stiffness at time $t$in MPa, $m(t)$ is the creep rate at time $t$, $P$ is the measured test load in mN, $L$ is the span length in mm, $h$ is the depth of the specimen in mm, $\delta(t)$ is the deflection of test specimen at time $t$, and $A$, $B$, $C$ are regression coefficients.

In the Superpave bitumen specification, an upper limit of 300 MPa is imposed on $(t)$ in order to keep the bitumen hardness below a certain level [43]. However, an increase in the m-value means that the hardness of the bitumen changes quite rapidly and therefore shows better stress relaxation capability [44]. Due to this fact, a lower limit of 0.300 for the m-value was introduced in the Superpave bitumen specification to ensure that the bitumen has an adequate stress relaxation capability [43]. It is assumed that bitumen with an m-value above this limit can quickly dissipate stresses caused by thermal changes [42].

### *2.2.6. Conditioning Method*

Performance tests of asphalt mixtures were conducted on conditioned and unconditioned-control (unmodified) and diatomite-modified asphalt mixtures. The conditioning system was applied according to the AASHTO T 283 [45] method. Three identical Marshall briquettes were used for each mixture option created according to additive type, ratio, and conditioning. According to the method, the samples are divided into two groups. While the samples in one of the groups are not conditioned, the samples in the other group are first saturated with water in the range of 70% to 80% with a vacuum pycnometer. The water saturated samples are tightly wrapped with plastic film and placed in a plastic ziplock bag containing 10±5ml water. Then the samples are kept at -18±3°C for at least 16 hours. The samples taken out of the freezer are placed in a water bath at 60±1°C for 24±1 hours without waiting. After these procedures, the samples are considered conditioned, and the plastic bag and film are removed [45].

### *2.2.7. Indirect tensile strength (ITS) test*

The indirect tensile strength (ITS) test is among the most widely accepted tests for investigating the behaviour of asphalt mixtures at low temperature [46]. The ITS test was developed by the Strategic Highway Research Program (SHRP). Because the test is simple, can be done with inexpensive equipment, and gives essential information, it has been adopted by researchers and practitioners. With the test, creep stiffness can also be obtained besides the low-temperature resistance of asphalt mixtures. The ITS values obtained when the test was performed at low temperatures represent the low-temperature resistance of the pavement under real road conditions [47]. Samples are placed horizontally between two loading bars that can move parallel to each other in the vertical plane. While the bars compress the briquette at a constant speed of 50 mm/min, the sample cracks in the vertical plane due to the indirect tensile stresses. The largest load causing cracking is recorded in the device as tensile strength. At the same time, the deformation of the sample can be recorded until it cracks. The test can be performed at different temperatures. This study selected a temperature of 0°C, and the test was repeated with three identical samples. The failure strength (ITS) was calculated using equation (5) by using the recorded maximum load that caused crack formation in the sample during the test.

$$ITS = \frac{2000\,P}{\pi\,D\,t} \tag{5}$$

where ITS = indirect tensile strength (kPa), $P$ = maximum load (N),$t$ = thickness of specimen (mm), and$D$ = diameter of specimen (mm).

### 2.2.8. Moisture Damage Test

The weakening or breaking of the bonds between the aggregate surface and the bituminous binder due to water or moisture is called water damage or moisture susceptibility [48]. The fact that asphalt pavement is sensitive to moisture; in other words, its low water damage resistance reduces the adhesion and cohesion resistance of the pavement, may cause significant deterioration in the pavement such as stripping, ravelling, water infiltration, and may trigger other types of deterioration [49].

Moisture susceptibility of asphalt mixture samples can be evaluated with tensile strength ratio (TSR) test. TSR, determined according to the AASHTO T 283 standard, is the most accepted test method used to determine the resistance of compacted asphalt mixture samples against deterioration caused by water. The test aims to determine the indirect tensile strength of unconditioned and conditioned that have undergone a series of conditioning processes, compacted asphalt mixture samples. TSR is the ratio of ITS of conditioned and unconditioned samples and is calculated according to Equation 6 [50]. If the tensile strength ratio of the samples is 80% or more, it is resistant to moisture susceptibility [51, 52].

$$TSR = \frac{ITS_{conditioned}}{ITS_{unconditioned}} \tag{6}$$

### 2.2.9. Repeated Creep Test

The repeated creep test offers valuable information about the susceptibility of asphalt mixtures to rutting when changes in temperature occur [53,54]. The test system allows the simulation of application conditions by providing options such as variable temperature, stress or load, loading period, and conditioning loading. The repeated application of load to the asphalt mixture sample (loaded and unloaded periods) allows for the advantages provided by the elasticity properties of the mixture to be observed (the effect of returning deformations can also be taken into account). The loading number or cumulative deformation that ends the test can be selected. During the test, the number of loadings and the amount of deformation are automatically recorded [55].

It is recommended that the repeated creep test be performed at relatively low-stress levels (cannot usually exceed 206.9 kPa) and low temperature (cannot usually exceed 40˚C); otherwise, it is stated that the sample will fail prematurely [56].

In the research, repeated creep tests were performed on conditioned and unconditioned samples according to EN-12697-25 (A) [57] test standard. In this study, three samples from each mixture were tested. The conditioning was carried out following the AASHTO T 283 method. The tests were conducted at 40°C temperature, under the tension of 95 kPa, at a frequency of 0.5 Hz and 20000 loading cycles. In this test, the conditioning stress was 3 kPa; conditioning stress time 2 min; loading duration was 500 milliseconds and unloading duration was 500 milliseconds.

## 3. TEST RESULTS AND EVALUATION

### 3.1. Penetration and Softening Point Tests Evaluation

Table 7 shows the penetration, softening point and penetration index values on control and diatomite-modified asphalts. When all penetration values were analysed, it was observed that they were within the specification limits. It was observed that the penetration values decreased by minimum 9.36% and maximum 21.81% in diatomite modified asphalt mixtures compared to control (virgin) asphalt mixtures. It was observed that the penetration value decreased as the diatomite content increased in all diatomite modified asphalt mixtures. When the softening point values given in Table 7 are analysed, it is seen that all of the diatomite modified asphalts are higher than the control (virgin) mixtures. According to the penetration and softening point values, it was observed that the high temperature performance of diatomite modified asphalt mixtures improved due to the porous structure of diatomite. This result was confirmed in a study by Wang et al. [26], which showed that the softening point values of diatomite modified asphalt mixtures increased and penetration values decreased compared to control (virgin) asphalt mixtures. This was attributed to the decrease in the overall fluidity of the modified asphalt due to the absorption of the light components of asphalt into the porous structure of diatomite.

When the PI values in Table 7 are analysed, it is seen that the PI values of all diatomite modified asphalt mixtures are higher than the control (virgin) bitumen, therefore, the modification of diatomite additive to asphalt increases the temperature sensitivity of the mixtures. It was observed that the highest PI value and the least sensitive mixture to temperature was observed in 15% D3 mixtures with 1.681.

*Table 7 - Penetration and softening point test results of control and diatomite-modified bitumen*

| | Control | D1 | | | D2 | | | D3 | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 5% | 10% | 15% | 5% | 10% | 15% | 5% | 10% | 15% |
| Penetration 25°C, 100 g, 5 s (0.1 mm) | 63.1 | 57.7 | 53.2 | 52.8 | 55.6 | 53.5 | 52.5 | 54 | 53.2 | 51.8 |
| Softening point (°C) | 52.4 | 57.1 | 58.6 | 60.2 | 57.7 | 58.7 | 61.2 | 60.4 | 61.5 | 62.9 |
| PI | -0.043 | 0.796 | 0.902 | 1.207 | 0.828 | 0.937 | 1.390 | 1.306 | 1.483 | 1.681 |

### 3.2. Bending Beam Rheometer (BBR) Test Evaluation

The S value obtained by the BBR test is shown in Table 8 and the m value is shown in Table 9. When S values are analysed, it is observed that all values are within the specification limits (≤ 300 MPa) at -12°C. However, S values at -18°C were not within the specification limits. It was observed that the S value increased as the diatomite additive ratio increased. When the m values in Table 9 are analysed, it is seen that the specification limit value at -18°C is met only for 5% D3 modification; it is not met for samples with other ratios and control (virgin) asphalt. At -12°C, it was observed that all asphalt binders met the specification limits. When Table 8 and Table 9 are analysed together, it is observed that the S value increases and the m value decreases as the diatomite additive ratio increases at -12°C. This result shows that modification of diatomite alone is ineffective in the resistance to low temperature cracking.

In a study by Li et al., it was confirmed that diatomite modified asphalt mixtures may weaken the low temperature properties of asphalt mixtures, but their low temperature behaviour can be significantly improved when used with bio-oil [28].

*Table 8 - Creep stiffness values of control and diatomite-modified asphalts*

| Diatomite content (%) | 0 | | 5 | | 10 | | 15 | |
|---|---|---|---|---|---|---|---|---|
| Temperature (°C) | -12 | -18 | -12 | -18 | -12 | -18 | -12 | -18 |
| Control (MPa) | 118.0 | 324.1 | | | | | | |
| D1 (MPa) | | | 121.8 | 325.7 | 149.7 | 353.2 | 160.5 | 439.5 |
| D2 (MPa) | | | 122.6 | 327.2 | 146.9 | 353.4 | 161.3 | 446.4 |
| D3 (MPa) | | | 124.7 | 320.1 | 146.5 | 349.8 | 165.1 | 451.5 |

*Table 9 - Creep rate values of control and diatomite-modified asphalts*

| Diatomite content (%) | 0 | | 5 | | 10 | | 15 | |
|---|---|---|---|---|---|---|---|---|
| Temperature (°C) | -12 | -18 | -12 | -18 | -12 | -18 | -12 | -18 |
| Control (m-value) | 0.382 | 0.283 | | | | | | |
| D1 (m-value) | | | 0.390 | 0.275 | 0.388 | 0.271 | 0.381 | 0.267 |
| D2 (m-value) | | | 0.396 | 0.291 | 0.390 | 0.277 | 0.382 | 0.273 |
| D3 (m-value) | | | 0.401 | 0.305 | 0.394 | 0.278 | 0.388 | 0.281 |

### 3.3. Strength of Mixtures at Low Temperature

The strength of asphalt mixtures at low temperatures was determined by indirect tensile strength test at 0°C. Experimental results are given in Figure 14. The test results showed that increasing the diatomite ratio increased the tensile strength of the modified mixtures. The smallest tensile strengths were obtained with D1 diatomite at a 15% addition rate (2494 kPa). Increasing the diatomite ratio in D1 and D2, diatomite caused a decrease in their tensile strength. Compared to the control mixture, it was observed that 5% diatomite modification reduced the tensile strength of the asphalt mixture, resulting in mixtures that are more sensitive to low temperatures.

Indirect tensile strength (ITS) test is currently the most widely used method to characterise thermal cracking susceptibility in asphalt pavements [58]. When the diatomite ratio was increased to 10%, a 9.7% improvement in the cracking resistance of D3 modified mixtures was observed compared to the control mixtures. Mixtures with D1 diatomite showed lower low temperature resistance than control mixtures. D2 modified mixtures showed tensile strengths equal to or lower than the control mixtures, while D3-modified mixtures showed tensile strengths equal to or higher than the control mixtures.

### 3.4. Moisture Damage Evaluation

The resistance of the produced diatomite-modified asphalt mixtures to water damage was determined according to the AASHTO T283 method. The indirect tensile strength test was performed on three identical briquettes at 25°C, and the tensile strength values were averaged and given in Figure 15 for unconditioned samples and Figure 16 for conditioned samples.

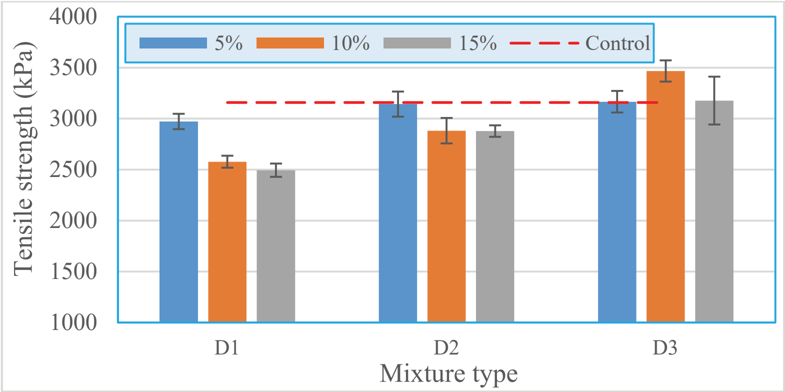


*Figure 14 - Comparison of tensile strength of control and diatomite modified asphalt mixes*

At 25°C, the tensile strength of diatomite-modified mixtures was generally higher than the control mixture. The conditioned diatomite-modified asphalt mixture samples showed higher tensile strength than the conditioned control samples. In unconditioned samples, D2 diatomite was equivalent to the control mixtures, while D1 and D3 diatomite showed a higher performance than the control mixtures. Increasing the diatomite ratio in the asphalt mixture generally led to a decreasing trend in tensile strength. This trend could be seen more clearly in the conditioned mixtures (Figure 16).

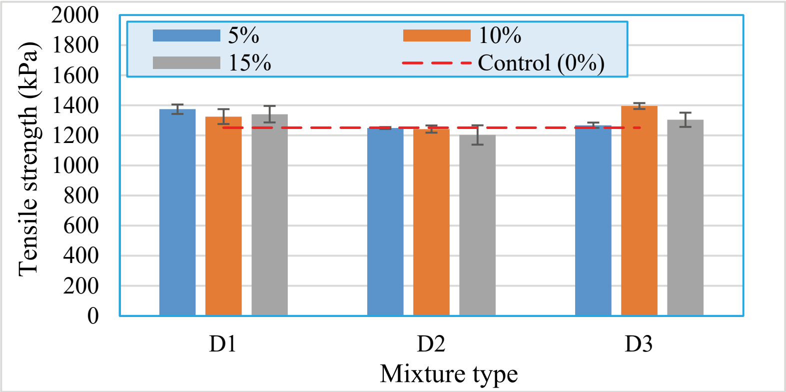


*Figure 15 - Tensile strength of unconditioned asphalt mixtures at 25°C*

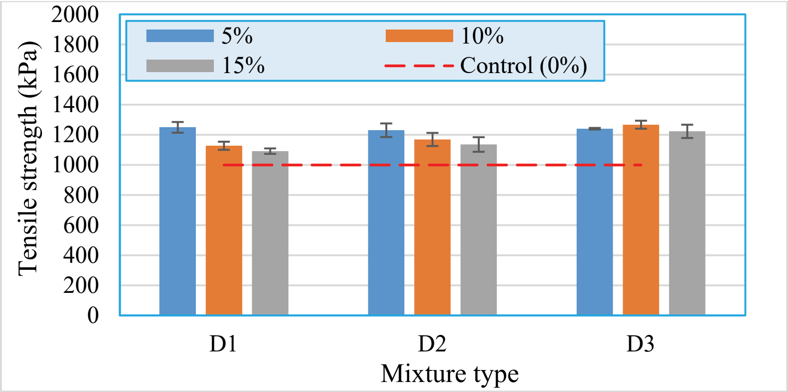


*Figure 16 - Tensile strength of conditioned asphalt mixture at 25°C*

While higher tensile strengths were obtained in low-concentration modifications (5% inclusion rate), lower tensile strengths were observed in high concentrations (15%). Although there is no significant difference between diatomite types in terms of tensile strength, D3-modified asphalt mixture samples showed the highest tensile strength in both conditioned and unconditioned samples at 10% inclusion rate. In conditioned mixtures, the highest tensile strength was obtained with D3 diatomite and the lowest tensile strength with D1 diatomite at the same inclusion rates. When the conditioned mixtures were evaluated, it was seen that the tensile strength of D3-modified asphalt mixtures increased by 22% or more at all inclusion rates compared to the control mixtures. This increase remained at 13% in D2-modified asphalt mixtures.

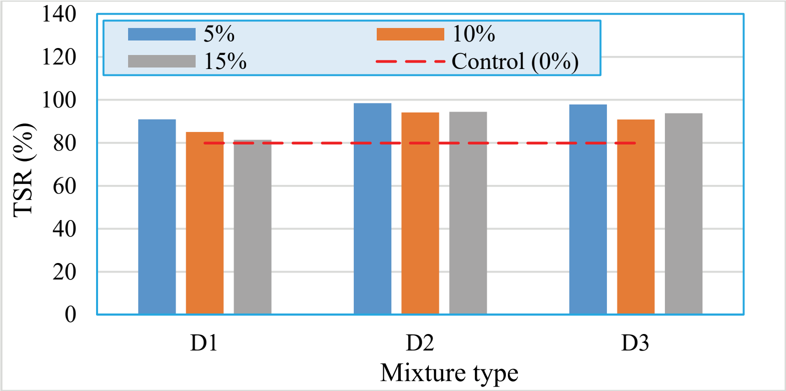


*Figure 17 - Tensile strength ratio*

The tensile strength ratios of asphalt mixture samples are given in Figure 17. All of the diatomite modified asphalt mixtures showed higher tensile strength than the control mixtures and 80%. It has been observed that the TSR value of the asphalt mixture can be increased by 22% with diatomite modification. There was a tendency for the water damage resistance of diatomite modified asphalt mixtures to decrease as the diatomite ratio increased. Higher TSR values were obtained at 5% diatomite ratio compared to 10% and 15% adding ratios. D2 and D3 diatomites showed higher water damage resistance compared to D1 diatomite.

### 3.5. Rutting Evaluation

The rutting behavior of control and diatomite modified asphalt mixtures was investigated by repeated creep tests at 40°C. The deformation curves of unconditioned samples are shown in Figure 18, and those of conditioned samples are shown in Figure 19. It was observed that D3 diatomite showed the highest deformation in unconditioned mixtures and suffered higher deformation compared to the control mixture. As the diatomite inclusion rate increased, the deformation resistance of asphalt mixtures decreased, and the highest deformation resistance was obtained with D1 diatomite at a 5% inclusion rate. With 5% D1 modification, a 17% decrease in permanent deformation was observed compared to the control mixture. It was understood that as the diatomite ratio increased, the slopes in the second region of the creep curves of the asphalt mixture samples increased; however, the tertiary region did not form in any sample in the number of load repetitions applied.

The positive effect of diatomite modification could be better observed in conditioned mixtures. Unlike unconditioned mixtures, as the diatomite ratio increased, the amount of deformation decreased. While the highest deformation resistance was achieved with D2 diatomite at a low inclusion rate (5%), the highest deformation resistance was achieved with D3 diatomite at medium (10%) and high (15%) inclusion rates. With D3 diatomite, a 37% reduction in deformation was achieved compared to the control mixtures at a 15% usage rate.

Considering the experimental results of both unconditioned and conditioned mixtures, it is understood that diatomite modification can have a positive effect on increasing the deformation resistance of asphalt mixtures. Since asphalt mixtures are exposed to natural conditioning under application conditions, the positive effect of diatomite modification in terms of rut resistance may be better revealed. Based on earlier research, it has been confirmed that diatomite can enhance the performance of asphalt mixes at elevated temperatures because of its extensive surface area and porous structure. Furthermore, it can adsorb small amounts of bitumen, leading to an overall increase in the complex shear modulus of the asphalt. This, in turn, enhances the mixture's resistance to rutting [59].

The optimum diatomite content in the modified asphalt mixture rises as the amount of diatomite increases. This is due to the diatomite's capacity to absorb light oil in the asphalt, leading to an increase in the stickiness of the asphalt. The energy required for bending strain encompasses both stress and strain, making it a more appropriate measure for assessing the cold-weather performance of the asphalt mixture. Based on the results of the low-temperature bending test, the most favourable diatomite dosage is achieved at 13-14%. This is when the bending stiffness modulus and bending strain energy reach their peak levels. Considering the equivalent brittle point, the optimum diatomite dosage is 13% [60].

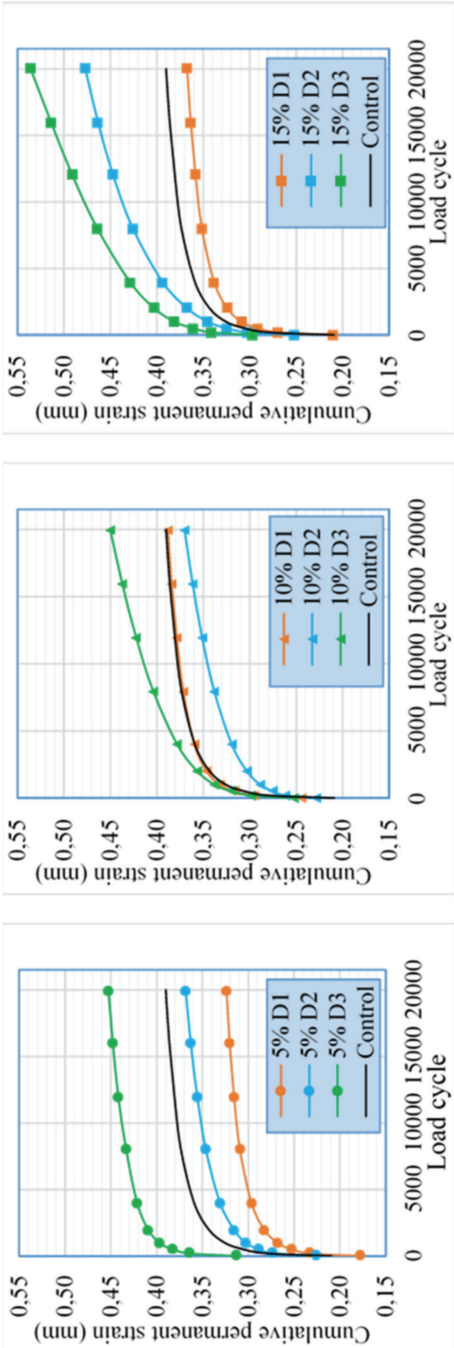


*Figure 18 - Creep curves of unconditioned asphalt mixture samples*

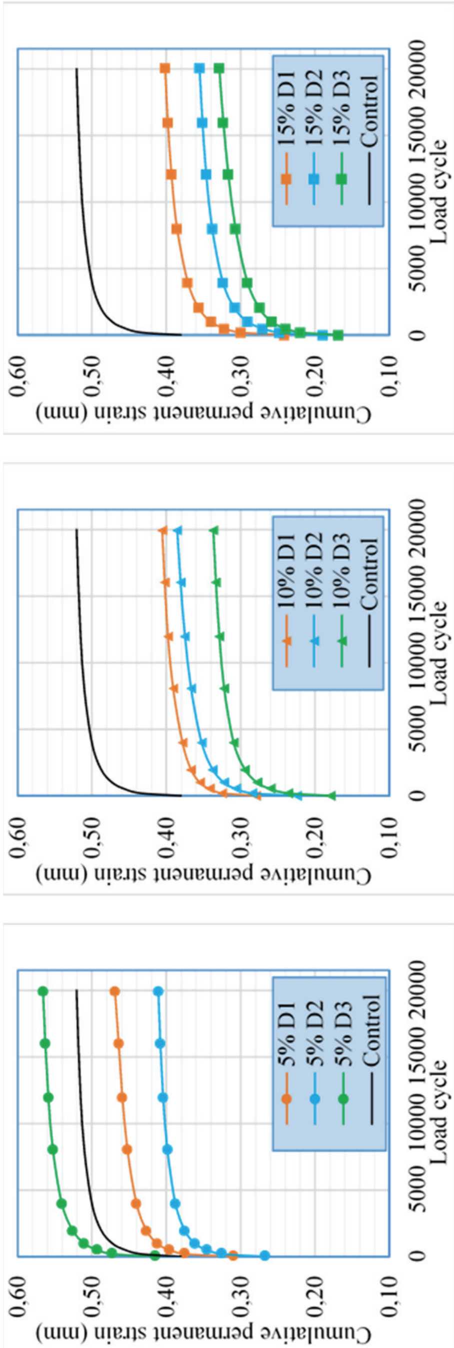


*Figure 19 - Creep curves of conditioned asphalt mixture samples*

D1, D2, and D3 diatomites used in this study show similar properties in terms of their chemical composition. However, D1 diatomite was ground in the finest gradation, and D3 diatomite was ground in the coarsest gradation. When the mechanical test results of asphalt mixtures are evaluated, it is understood that D2 and D3 diatomites show better results than D1 diatomites. D1 diatomite creates a larger surface area with its fine structure and requires a higher amount of bitumen to cover the particles. The bitumen film around the particles becomes thinner for the same amount of bitumen. The thinness of the bitumen film reduces the flexibility of the mixture and causes harder mixtures to form. It is thought that the lower low-temperature cracking resistance of the D1-modified asphalt mixture is related to the surface area size of the diatomite. As the diatomite ratio increases, the surface area increases, and the tensile strength decreases slightly, confirming this idea.

It is thought that fiber addition may be effective in increasing tensile strength at low temperatures more significantly. The previous studies have emphasized that there is an improvement in the tensile strength of diatomite-fiber modified asphalt mixtures [61]. It is also stated that there is a significant increase in the fatigue life of basalt fiber-diatomite-modified asphalt mixtures compared to diatomite-modified asphalt mixtures [62].

The porous structure of diatomite helps to form a stronger adhesion between the bitumen film and diatomite particles. This strong adhesion increases the asphalt mixture's resistance to water damage. However, when the bitumen film thickness decreases too much, the effect of adhesion may decrease. Therefore, the maximum size and gradation of diatomite have a significant impact in terms of water damage as well as in low temperatures.

When all performance test results are evaluated, it is understood that the use of D3 diatomite in the range of 10-15% provides higher mechanical properties. If thinner form diatomites such as D1 are to be used, the additive ratio should be chosen lower.

It is considered that the ineffective/low-effective results emphasized in the evaluation of low-temperature performances of diatomite-modified asphalt mixtures in previous studies may be related to the gradation of the diatomite used. It becomes clear that the diatomite ratio must be determined meticulously according to gradation.

## 4. CONCLUSION

The study investigated basic performance indicators such as low-temperature cracking, water damage, and deformation behaviors of asphalt mixtures at different grinding and inclusion rates of diatomite additive. Three grinding sizes and three inclusion ratios were selected. As a result of the study, the following results were obtained.

Increasing the inclusion rate of diatomite negatively affected the low-temperature cracking resistance of the asphalt mixture. This result coincides with a study by Liu et al. [63], which proved that 15% diatomite utilisation rate showed higher cracking resistance than 10%. In general, as the inclusion rate increased, the tensile strength of the asphalt mixture decreased. The increase in the maximum size of diatomite and the coarsening of its gradation positively affected the cracking resistance of the asphalt mixture. The highest tensile strength was obtained with D3 diatomite whose maximum size is 300 microns. However, D1 diatomite-added asphalt mixtures with a maximum size of 106 microns showed lower cracking resistance than the control mixtures.

Diatomite-modified asphalt mixtures showed higher water damage resistance than control mixtures at all sizes and inclusion rates of diatomite additive. Although there is no significant difference, increasing the maximum size of diatomite increased the TSR value. However, increasing the addition rate tended to decrease the TSR value, and the highest TSR values were obtained at a 5% addition rate.

The deformation behaviour of diatomite-modified asphalt mixtures could not be clearly observed in unconditioned mixtures, and misleading results emerged. In conditioned mixtures, high deformation resistance of diatomite modified mixtures was revealed. Increasing the diatomite ratio and maximum size increased the deformation resistance. The highest deformation resistance was achieved at a 15% inclusion rate of D3 diatomite.

This result coincides with the finding of Du et al. [64] on SBS and diatomite modified asphalt mixtures that the temperature stability of the asphalt pavement increased more than the SBS modified ones at an optimum diatomite usage rate of 13% and the rutting resistance of the asphalt pavement improved more.

When all tests are evaluated, it is understood that it would be appropriate to use D2 and D3 diatomites at rates of 10%-15%. In the evaluations made within the scope of the study, it was concluded that the maximum size of diatomite above 200 microns was more effective. In the literature search, no studies on diatomite modified asphalt pavements with different grinding sizes and different types were found.

It is recommended to investigate compatible fiber types, sizes and ratios to improve low temperature cracking. In future studies, the effect of diatomite grind size on fiber-diatomite multiple modification can be investigated. It would also be useful to evaluate the effect of diatomite modification on the fatigue resistance of asphalt and to investigate the effect of surface modified diatomite additives on asphalt pavement performance.

**Symbols**

| | |
|---|---|
| **D** | Diameter of specimen (mm) |
| **D1** | First type diatomite |
| **D2** | Second type diatomite |
| **D3** | Third type diatomite |
| **EDS** | Energy dispersive spectrometry |
| **ITS** | Indirect tensile strength |
| **NP** | Non-plastic |
| **P** | Maximum load (N) |
| **PPM** | Parts per million |
| **Rpm** | Revolutions per minute |
| **SEM** | Scanning electron microscopy |

**SHRP** Strategic highway research program

**TSR** Tensile strength ratio

**XRD** X- Ray diffractometer

**XRF** X- Ray fluorescence

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