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# Effect of Silicon Carbide Nanotube (SiCNT) on the Mechanical Properties and Moisture Susceptibility of Hot Mix Asphalt

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Indirect tensile strength,
Moisture susceptibility

**Abstract:** Asphalt pavements are damaged by moisture. Strength loss is caused by moisture damage. Anti-stripping agent is used to address this damage in asphalt mixtures. Bitumen modification is also effective method to prevent moisture damage. To this aim, in this study, bitumen was modified by silicon carbide nanotube (SiCNT) in three percentages (1%, 3% and 5%) by weight. After the modification effort was performed, asphalt mixtures were designed according to Superpave™ procedure. Indirect Tensile (IDT) Strength test was employed determining moisture damage in asphalt mixtures modified by SiCNT. As a result, moisture susceptibility of two percentages (3% and 5%) of SiCNT ensured the specification limit.

# Silikon Karbür Nanotüplerin (SICNT) Sıcak Karışım Asfaltın Suya Duyarlılığına ve Mekanik Özelliklerine Etkisi

**Anahtar Kelimeler**
Silikon karbür nanotüp,
Bitüm modifikasyonu,
İndirekt çekme dayanımı,
Nem hassasiyeti

**Özet:** Asfalt kaplamalar nemin etkisiyle zarar görmektedir. Sudan kaynaklanan bozulmalar ile dayanım kaybı oluşmaktadır. Asfalt karışımlarda sudan kaynaklanan bozulmaların önüne geçebilmek için soyulma önleyici malzemeler kullanılmaktadır. Bitüm modifikasyonu da sudan kaynaklanan bozulmaları önlemede etkili bir metottur. Bu amaçla, bu çalışmada, bitüm silikon karbür nanotüplerle (SiCNT) üç farklı oranda (ağırlıkça %1, %3 ve %5) modifiye edilmiştir. Modifikasyon gerçekleştirildikten sonra asfalt karışımlar Superpave tasarım yöntemine göre tasarlanmıştır. Silikon karbür ile modifiye edilmiş asfalt karışımların suya karşı duyarlılığı İndirekt Çekme Dayanımı testi ile belirlenmiştir. Sonuç olarak, SiCNT modifikasyonu iki oranda (%3 ve %5) şartname limitini sağlamıştır.

## 1. Introduction

Materials exhibit significant properties at the nano level owing to its large surface area and it is possible to apply in the field [1]. Asphalt-like materials having a complex structure with better engineering at nano level resulted in some novel smart characteristics. The service life of a pavement depends on the structure conditions, material properties, thickness and accepted damage criterion. To prevent moisture deteriorations, nanomaterials are novel in the literature such as polymer-modified materials, nano silica, nano carbon fiber. To this aim, in this study, SiCNT was chosen to improve moisture susceptibility and indirect tensile strength of asphalt mixtures. Nanomaterials with unique properties such as large surface area, surface atoms have a wide fraction, quantum effects, structural properties are growing up rapidly. Benefits of nano modified asphalt have improved rutting resistance, cracking, and fatigue life [2]. Silicone and carbon nanotubes have been generally adopted as an agent to improve bitumen characteristics [3, 4].

Silica as an additive is used in three percent (2%, 4%, 6%) for modification of bitumen to improve some properties of warm mix asphalt (WMA) containing sasobit (2%). Resilient modulus of modified WMA was increased [3]. Properties of modified bitumen containing 1, 3, 5, 7 and 9% nano silica and 0.01, 0.1, 0.5 and 1% nano carbon are investigated in terms of penetration, viscosity and softening point. Increment of softening point and decrement of penetration values were obtained according to test results [2]. Nano silica as an anti-aging agent is used in three percent (2%, 4% and 6%). Short term aging was applied to bitumen by rolling thin film oven (RTFO). After aging, complex modulus (G*) and complex

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viscosity (η*) parameters of bitumen was increased by nano silica modification [4]. Nano silica have been added into bitumen in three percentages (2%, 4%, 6%) to modify WMA containing sasobit in 2% by weight. Complex modulus increment has been obtained [5]. Generally, nano silica used as an inorganic agent to modify bitumen [6]. Modified asphalt mixtures by steel slag, $TiO_2$ and $SiO_2$ were examined by Shafabaksh and Ani. Improvement of resistance of modified asphalt mixture was aimed by modification with nano particles. As a result, $TiO_2$ and $SiO_2$ nano particles boost the characteristics of bitumen. Rutting and fatigue life of bitumen were also increased [7].

Rutting performance of asphalt pavements by modifying bitumen with nano-$TiO_2$ is examined by Tanzadeh et. al. [8] and the results illustrated that modifying bitumen with nano-$TiO_2$ improved rutting depth when compared to base mixtures. Polymer modified bitumen containing styrene-butadiene-styrene (SBS) was used to boost visco-elastic characteristics of the mixture. Fatigue life was increased when the additive increased [9]. Bitumen and mixture characteristics were examined by Mothlagh et. al. [10] using carbon nanotube, and minimal changes were obtained by results on physical characteristics.

Moisture damage is occurred by the bond loss between the bitumen and the fine and coarse aggregates. Moisture damage speeds up as moisture permeates and weakens the mastic during repetitive traffic loading. Stripping, bleeding, rutting etc. distresses were observed by the results [11]. The common method for eliminating the moisture damage is using antistrip additives. Hamedi et. al. [12] examined the moisture damage of hot mix asphalt (HMA) using nano- particles as an antistrip agent. The results showed indirect tensile strength ratio for the mixtures modified with nano ZnO have better performance. Nano-$TiO_2$ were used by Azarhoosh et. al. [13] has used the surface free energy method. The results showed the nano-$TiO_2$ increases the wettability of the asphalt binder and strengthen the bond between the asphalt binder and the aggregate.

Nabiun and Khabiri [14] examined ferrite filler in different combinations to address mechanical properties and moisture resistance of asphalt mixtures. Marshall resistance, indirect tensile strength (ITS) and resilient modulus were conducted. Results showed that the higher ferrite percentages proved high performance. Hossain et. al. [15] examined the effect of two different nanoclays on moisture resistance of modified binders. As a result, nanoclay modified binders were more resistive against moisture damage than the base binder. Behbahani et. al. [16] evaluated that the glasphalt mixture modified with zycosoil as an anti-stripping agent to determine the moisture sensitivity. Results showed the zycosoil improved the moisture sensitivity of mixture.

Yao et. al. [17] investigated that the moisture susceptibility of nano hydrated lime modification of asphalt mixtures. Tensile Strength Ratio (TSR) test was used to explore moisture susceptibility. After TSR test, polar groups were extracted and analyzed from tested asphalt mixtures by Fourier Transform Infrared Spectroscopy (FTIR). As a result, moisture damage is reduced. Hamedi et. al. [18] investigated the effects of nano-$CaCO_3$ on moisture resistance of asphalt mixtures. Surface Free Energy (SFE) method and modified Lottman test was used to determine the moisture susceptibility. Modified Lottman test results showed that the nano modified mixtures were high resistive to moisture. SFE results indicated that the nano material decrease the moisture susceptibility. Iskender [19] examined that the performance of nano clay modified asphalt mixtures against moisture damage. The results showed higher performance against moisture damage. Hamedi [20], evaluated the effects of two types of nano materials on moisture susceptibility. Nano materials have decreased moisture susceptibility. Ziari et. al. [21] investigated that the effect of nano-organosilane modification on water damage of asphalt mixtures. Nano modified asphalt mixtures have improved resistance against water damage.

Babagoli et. al. [22] investigated that the performance of SMA mixtures modified with styrene-butadiene-styrene (SBS) and nanoclay. According to the results, nanoclay and SBS improved the moisture susceptibility of SMA mixtures. Zahedi and Baharvand [23] examined the effect of nano clay and crumb rubber on performance of hot mix asphalt. Results showed that nano clay and crumb rubber improved the mixture properties. Arabani and Hamedi [24] evaluated the susceptibility of asphalt binder with liquid antistrip against moisture damage. Based on the results, liquid antistrip agent decreases the stripping in presence of water.

SiCNT nanotube was chosen to improve the performance of modified asphalt mixtures against moisture. Base bitumen was mixed with nano materials in different ratios (1, 3 and 5% by weight). Rheological properties of nano-modified bitumen were analyzed using standard bitumen tests in terms of penetration, softening point and rotational viscosity (RV).

The mechanism of the SiCNT compound has been examined to determine the resistance of modified asphalt mixtures against moisture damage. The IDT test has been conducted in dry and wet conditions according to the Modified Lottman test procedure (AASHTO T283). The main objectives of the study are:

- Determining rheological characteristics of non-modified and SiCNT modified bitumen;

**Table 1.** Base bitumen properties

| Test | | Unit | Base Bitumen | Specification Limit |
|---|---|---|---|---|
| Penetration @25 °C | | 0.1 mm | 62.2 | 50-70 |
| Softening Point Ring&Ball | | °C | 49.9 | 46-54 |
| Ductility @25°C, 5 cm/min | | cm | >100 | >100 |
| RV @135 °C, | | Pa.s | 0.475 | ≤3Pa.s |
| RV @165 °C | | Pa.s | 0.15 | - |
| DSR @10 rad/s | Fail Temperature | °C | 67.9 | G*/sinδ>1 kPa |
| | Grade | °C | 64 | |
| Mass Loss | | % | 0 | <0.5 |
| Permanent Penetration | | % | 70.4 | >50 |
| Change in Softening Point | | °C | +3.2 | <9 |
| DSR @10 rad/s | Fail Temperature | °C | 67 | G*/sinδ>2.2 kPa |
| | Grade | °C | 64 | |
| DSR @10 rad/s | Fail Temperature | °C | 28.6 | G*.sinδ<5.000 kPa |
| | Grade | °C | 22 | |
| BBR @60 s | Temperature | °C | -12 | |
| | m-value | | 0.325 | m≥0.300 |
| | Stiffness | MPa | 213 | S≤300 MPa |
| | | Performance Grade | PG 64-22 | |

- Examining the effects of SiCNT on moisture susceptibility of asphalt mixtures; and
- Analyzing the dispersion of nano materials into bitumen using Scanning Electron Microscopy (SEM).

## 2. Materials and Methods

### 2.1. Aggregate and gradation

Hot mix asphalt (HMA) mixtures were prepared with limestone aggregate. Wearing course was adopted for preparation of HMA samples. 12.5 mm nominal maximum aggregate size was used. Dense graded HMA was prepared by Superpave™ guidance and gradation curve was selected as illustrated in Figure 1.

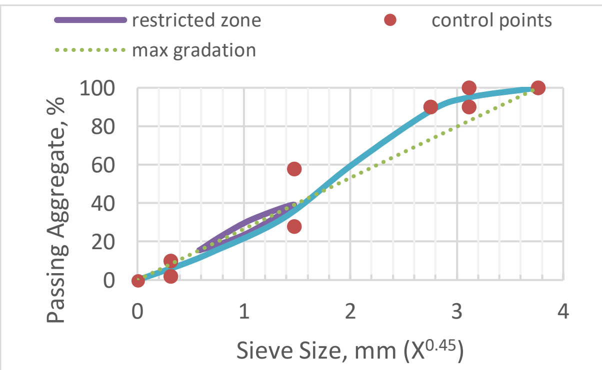


**Figure 1.** Gradation curve for HMA

### 2.2. Bitumen

Bitumen used in this study was PG 64-22 performance grade. Modified and non-modified bitumen characteristics were examined in accordance with the Superpave™ in terms of DSR, BBR, RTFOR and PAV. Rheological properties were determined in terms of penetration, ductility, rotational viscometer and softening point and the results were given in Table 1.

### 2.3. SiC nanotube

SiC nanotube was used as an antistripping agent. Silicon carbide is an inorganic nanotube with formula of Silica (Si) and Carbon (C). SiC nanotube is a grayish white, insoluble in water. SiC nanotube properties are given in Table 2.

Based on the literature, the dosage of nano materials are used generally 1%-10% by the weight of bitumen. 1, 3 and 5 percent were used by the weight of bitumen in this study.

**Table 2.** Properties of SiCNT

| | |
|---|---|
| Purity (%) | 99+% |
| Average Particle Size | <80nm |
| Morphology | Cubic |
| Surface Area | 25-50 $m^2/g$ |
| Color | Grayish white |
| Bulk Density | 0.05 $g/cm^3$ |
| True Density | 3.216 $g/cm^3$ |
| Free Si | 0.24% |
| Free C | 0.76% |

### 2.4. Experimental set up and procedure

Mixing the SiC nanotube with base bitumen was conducted by high shear mixer. DSR, BBR, RTFOT and PAV was adopted to determine the rheological properties of base bitumen. Compaction effort was performed with Superpave Gyratory Compactor (SGC) for the nano modified asphalt mixtures. Optimum bitumen content (OBC) was determined for nano modified mixtures separately. Modified Lottman procedure was adopted to determine the moisture susceptibility. Experimental setup is given in Figure 2.

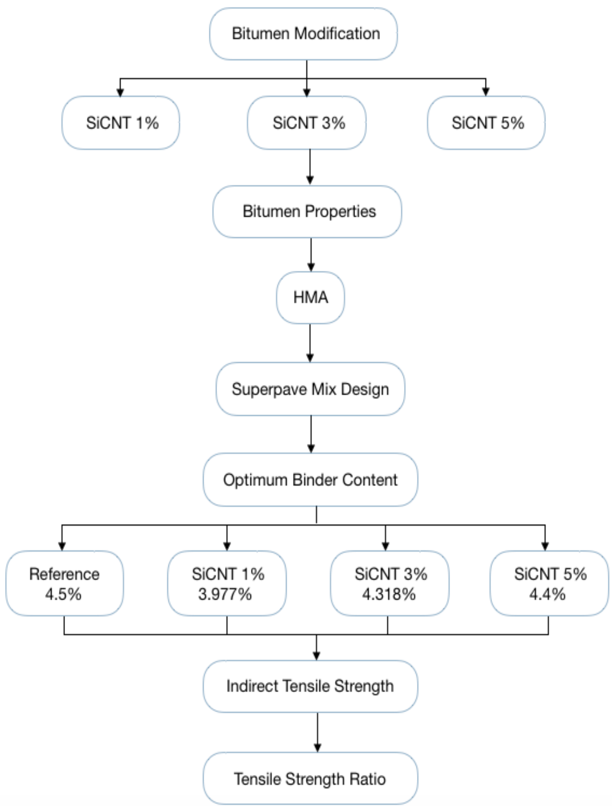


**Figure 2.** Experimental program

### 2.5. Preparation method of the modified bitumen with SiC nanotube

Modification effort was conducted with the high shear mixer. The mixer is capable of mixing 1.5 liter at 8000 rpm and 210°C. In this study, bitumen was mixed with nano materials at 4000 rpm. Temperature was set to 160°C and nano materials and bitumen was mixed for two hours in accordance with the literature review. Also, the contents of SiCNTs were chosen as 1%, 3% and 5% by weight of bitumen.

### 2.6. Mix design

OBCs of SiCNT modified mixtures were determined by Superpave™ mix design. %4 air void is aimed for compaction by SGC. Samples were compacted at 4% air void and in bitumen contents of 3.5%, 4%, 4.5% and 5% by SGC. OBC of base HMA was found as 4.5%. Air void content graphs of optimum binder were drawn from SGC compaction results. OBC was checked whether the limit value is ensured for VMA and VFA.

### 2.7. Tensile strength ratio (TSR)

Modified Lottman procedure is adopted to evaluate the moisture sensitivity. IDT strength and moisture susceptibilities of HMA was determined according to AASHTO T283 test procedure. First, samples are compacted a set of conditioned and unconditioned, three samples for dry and three samples for wet sets. Each sample is vacuum saturated to condition. Modified Lottman test includes short term aging, freeze thaw cycle, limits on air voids (6 to 8%) and saturation (55 to 80%). Saturation level is calculated. And then the Tensile strength and TSR is calculated.

## 3. Results and Discussion

### 3.1. Results of the modified bitumen test

Penetration results were decreased by SiCNT modification. Softening point was increased for 3% SiCNT modified bitumen. All results were given in Table 3.

**Table 3.** Bitumen results modified by SiCNT

| Test | Unit | Base | SiCNT 1% | SiCNT 3% | SiCNT 5% |
|---|---|---|---|---|---|
| Penetration | 0.1 mm | 62.2 | 56 | 50 | 53 |
| Softening Point | °C | 49.9 | 48.1 | 50.7 | 47.8 |
| Penetration Index | | -0.7 | -1.4 | -1.02 | -1.6 |
| Ductility | cm | >100 | 98 | 82 | 75 |
| RV @135 °C | Pa.s | 0.475 | 0.5 | 0.562 | 0.75 |
| RV @165 °C | Pa.s | 0.15 | 0.15 | 0.162 | 0.2 |

The viscosity of SiCNT modifications were increased when the content was increased. So that temperatures of mixing and compaction were higher than the base bitumen meaning that these modifications were less workable except SiCNT 1% modification (Figure 3). Mixing and compaction temperatures were given in Table 4.

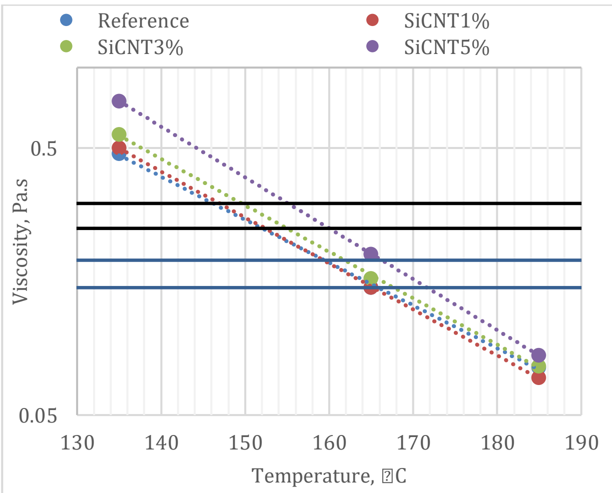


**Figure 3.** Rotational viscometer test results

**Table 4.** Compaction and mixing temperatures

| Sample* | Reference | SiCNT 1% | SiCNT 3% | SiCNT 5% |
|---|---|---|---|---|
| CI | 147-152 | 147-152 | 149-155 | 155-160 |
| CT | 149.5 | 149.5 | 152 | 157.5 |
| MI | 159-165.9 | 159-165 | 161.5-167.5 | 166-171.5 |
| MT | 162.45 | 162 | 164.5 | 168.75 |

*CI: compaction interval; CT: compaction temperature; MI: mixing interval; MT: mixing temperature

### 3.2. Results of the mix design

Optimum binder graphs were drawn and VMA and VFA was checked and ensured by 4.5% OBC of HMA (Figure 4). Same graphs were drawn for SiCNTs, separately and OBC was obtained for all content of SiCNT modified HMA as 3.977% for 1% modification, 4.318% for 3%modification and 4.4% for 5% modification (Figure 5). VMA and VFA was checked also for modified samples.

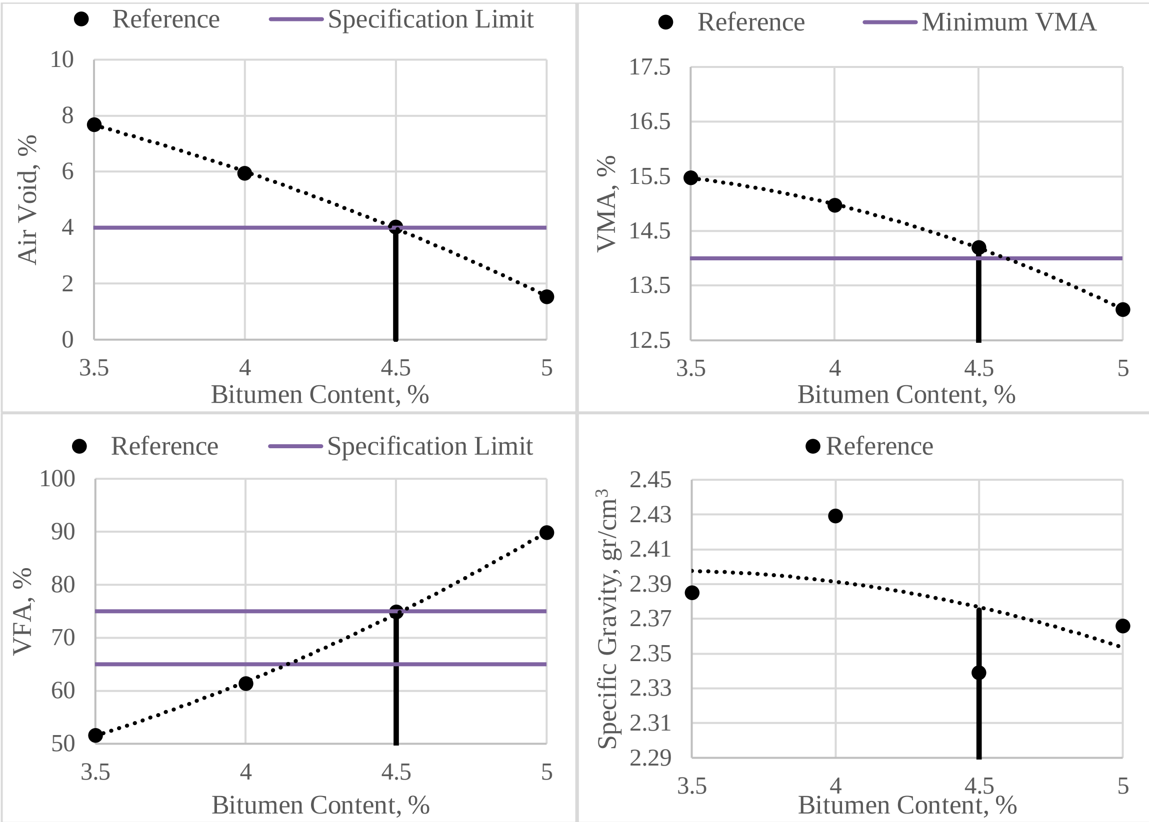


**Figure 4.** Optimum binder content for base HMA

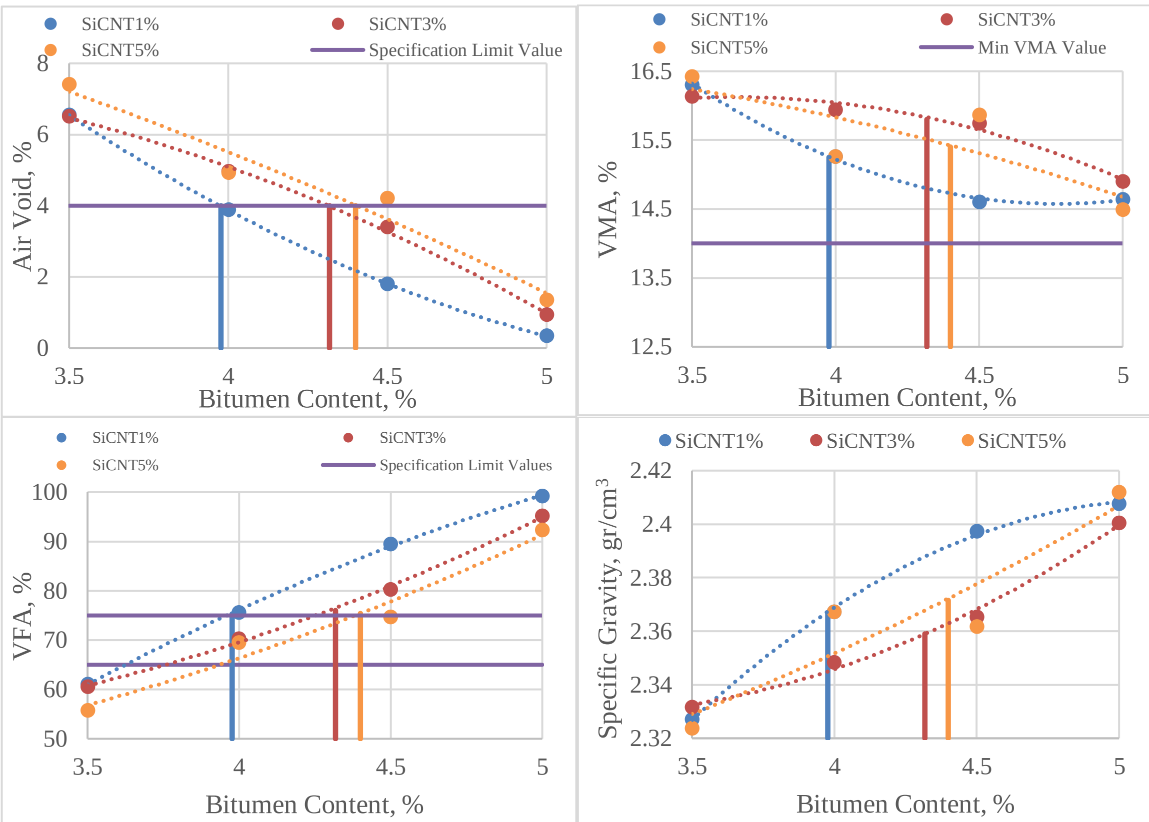


**Figure 5.** Optimum binder content for SiCNT

### 3.3. Results of the moisture susceptibility

IDT Strength test results were obtained as shown in Fig. 6. Indirect Tensile Strength of unconditioned sample ($IDT_u$) parameters were increased when the content of SiCNT increased for 1% and 3%. So, all modifications showed higher strength. $IDT_u$ parameters of all modifications were increased approximately 47%. High-performance was obtained by SiCNT modifications.

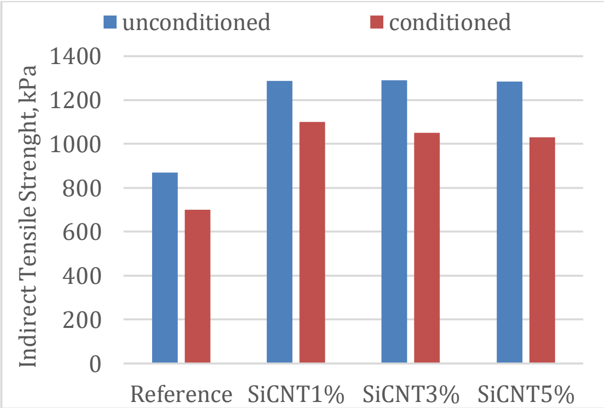


**Figure 6.** Indirect Tensile Strength

TSR was calculated for all modified samples (Fig. 7). The limit value should be 80% for TSR according to the specification. Specification limit was ensured for all modified HMA. TSR values were higher than the reference sample. As a result, bitumen modified samples have more resistance to the moisture.

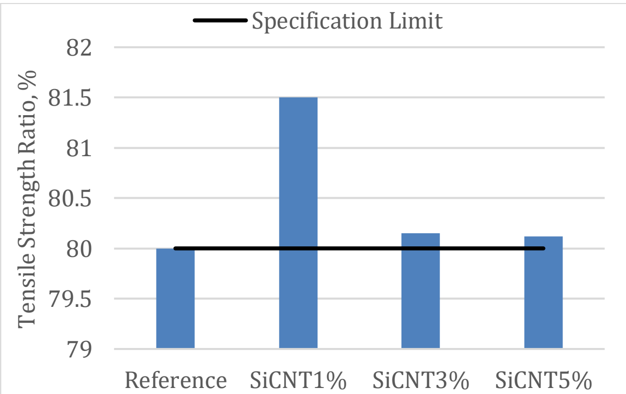


**Figure 7.** Tensile Strength Ratio

Results were given as improvement percentage in Table 5. Improvement percent was indicated as high (↑) having higher performance and low (↓) having lower performance. But, for penetration and OBC results, lower values mean high performance.

According to penetration index (PI) results, SiCNT modifications were found the less temperature susceptible. According to mixing and compaction temperatures only the SiCNT 1% modification was the most workable bitumen same as base bitumen. OBCs were decreased by modification. The highest moisture resistive sample was found the sample modified by SiCNT 1%.

**Table 5.** Comparison of all results

| *SiCNT modification (% improvement)* | | | | | |
|---|---|---|---|---|---|
| *Test* | Penetration | Softening Point | $IDT_U$ | TSR | Optimum Bitumen |
| *1%* | ↓9.9 | ↓3.6 | ↑47.8 | ↑1.875 | ↓11.6 |
| *3%* | ↓19.6 | ↑1.6 | ↑47.9 | ↑0.187 | ↓4 |
| *5%* | ↓14.7 | ↓4.2 | ↑47.5 | ↑0.15 | ↓2.2 |
| **↑Higher % when compared to reference sample* <br> **↓Lower % when compared to reference sample* | | | | | |

Economically, bitumen costs were big problems in terms of construction costs. Bitumen costs 2.135 TL/tons and additive costs increase these costs. However, additives are able to increase performance of the bitumen. So that, nanotubes can improve bitumen properties. According to the results, SiC nanotubes were found to be increase performance and decrease optimum bitumen contents. Decreasing optimum bitumen content provides less usage of bitumen content and less costs. In terms of economic evaluation SiC nanotubes provide decreasing costs.

### 4. Conclusion

Conclusions can be drawn as follows:

- Minimum 9.9% decrement was obtained by all modifications. The consistency was improved maximum 19.6% by SiCNT 3% modification.
- The mixing and compaction temperatures were same both for SiCNT 1% and base bitumen.
- The strength of all modifications was increased according to $IDT_u$ values.
- SiCNT 1% modified HMA had higher moisture resistance compared to reference HMA. All modifications have also high resistance against moisture according to TSR results.
- Optimum bitumen content was decreased by all modifications. Less bitumen usage provides less cost. Cost analysis could be conducted by these modifications for further studies.
- Best performance was obtained by SiCNT 3% evaluating whole test results which are given in Table 5.
- Bitumen properties should be investigated for further studies in accordance with the aging procedures.

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