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Moisture Susceptibility of Hot Mix Asphalt Mixtures Modified by Nano Silica and Subjected to Aging Process

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ABSTRACT

Moisture damage is described as a reduction in stiffness and strength durability in asphalt mixtures due to moisture. This study investigated the influence of adding nano silica (NS) to the Asphalt on the moisture susceptibility of hot-mix-asphalt (HMA) mixtures under different aging conditions. NS was mixed with asphalt binder at concentrations of 2%, 4%, and 6% by weight of the binder. To detect the microstructure changes of modified Asphalt and estimate the dispersion of NS within the Asphalt, the field emission scanning electron microscope (FE-SEM) was used. To examine the performance of Asphalt mixed with NS at different aging stages (short-term and long-term aging), asphalt mixture tests such as Marshall stability, flow, and Tensile Strength Ratio Test (TSR) were performed. According to the FESEM images, the NS particles in the mixture were sufficiently dispersed. The findings demonstrate that the NS enhances pavement performance by enhancing stability and volumetric characteristics and reducing susceptibility to moisture damage. Furthermore, TSR values of aged specimens show that increasing the NS content significantly reduces susceptibility to moisture and oxidative aging.

Keywords: Moisture Susceptibility, Nano Silica (NS), Tensile Strength Ratio (TSR), Aging

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تحسس الرطوبة لخلطات المزيج الاسفلتي الحار المعدلة بالنانو سيليكا و المعرضة لعملية التقادم

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الخلاصة

يوصف تلف الرطوبة بأنه انخفاض في الصلابة و ديمومة القوة في خلائط الأسفلت نتيجة للرطوبة. بحثت هذه الدراسة في تأثير إضافة النانو سيليكا (NS) إلى الأسفلت على حساسية خلائط الأسفلت الساخن لتلف الرطوبة في ظل ظروف التقادم المختلفة. تم خلط النانو سيليكا مع الأسفلت بتراكيز 2٪ و 4٪ و 6٪ كنسبة من وزن السمنت الأسفلتي. للكشف عن التغيرات في البنية المجهرية للأسفلت المعدل وتقدير توزيع النانو سيليكا داخل الأسفلت، تم استخدام المجهر الإلكتروني الماسح للانبعاثات الميدانية. تم إجراء اختبار مارشال واختبار نسبة مقاومة الشد لفحص أداء الأسفلت الممزوج بالنانو سيليكا في مراحل التقادم المختلفة. وفقا لصور المجهر الإلكتروني الماسح للانبعاثات الميدانية، كانت جزيئات النانو سيليكا موزعة في الاسفلت بشكل كافي. اظهرت النتائج أن النانو سيليكا يعزز أداء الرصيف من خلال تحسين خصائص مارشال وتقليل حساسية الخليط الإسفلتي للتلف الناتج عن الرطوبة. علاوة على ذلك، تظهر قيم نسبة مقاومة الشد للعينات المتقادمة أن زيادة محتوى النانو سيليكا يقلل بشكل كبير من الحساسية لتلف الرطوبة و التقادم الناتج عن الأكسدة.

الكلمات الرئيسية: تلف الرطوبة، النانو سيليكا، اختبار نسبة قوة الشد، التقادم

1. INTRODUCTION

Asphalt pavements should have sustainability and good durability during service to provide optimal performance, which depends on asphalt characteristics such as cohesion and adhesion between binder and aggregates, in addition to environmental factors like water, air, and temperature. Moisture in asphalt mixtures leads to a loss of bitumen cohesion or reduced adhesion between the asphalt binder and aggregates, resulting in bleeding and stripping in asphalt pavements **(Behiry, 2013; Mirabdolazimi et al., 2021).** The porosity, surface texture, and surface coatings of aggregates significantly affect the moisture sensitivity of asphalt mixtures **(Quipment, 2005; Ismael and Fattah, 2022)**.

Many pavement distress, such as raveling, cracking, and fatigue, are believed to be caused by bitumen aging. The oxidative aging process tends to make the asphalt mix susceptible to fracture and fatigue damage. When asphalt binder ages, it loses cohesiveness with the aggregate, resulting in rutting under traffic and moisture-related deterioration when exposed to water. Binder resources, mixing properties, and ambient conditions influence oxidative aging **(Wang et al., 2012).** Researchers used various types of modified bitumen to reduce the moisture sensitivity of asphalt mixtures **(Saleem and Ismael, 2020; Ali and Ismael, 2021)**. Researchers have recently incorporated nanotechnology into pavement construction by introducing various types of nanomaterials into the plain binder, such as nano silica, nanoclay, nanotubes, and nanofibers **(Khattak et al., 2013; Al-Sabaeei et al., 2021).** Nanomaterials' ability to enhance material performance is due to their uniquely large surface area, good dispersion ability, high stability, and chemical purity **(Bala et al., 2020)**. Cheng and his colleagues investigated the influence of nano and micro-sized hydrated lime on the performance of warm mixed Asphalt **(Cheng et al., 2011**). Regarding the Indirect Tensile Strength (ITS), flow number, and toughness in both dry and wet conditions, WMA mixtures with nano-size hydrated lime performed better when compared to mixtures with micro-size hydrated lime. Nanoclay is a nanomaterial that is frequently used for asphalt modification. It was observed that using nanoclay increases the softening point while decreasing the ductility and penetration values **(Ismael and Ismael, 2019).**

Nano silica is one of the nanomaterials widely used in asphalt mixtures. Previous studies have shown that adding NS to asphalt binder reduces penetration, increases viscosity, and increases softening point **(Shafabakhsh and Ani, 2015; Crucho et al., 2018)**. Alhamali and coworkers studied the effect of NS on storage stability and physical and rheological properties **(Alhamali et al., 2015)**. According to Dynamic Shear Rheometer (DSR) testing results, NS enhances the complex modulus compared to asphalt binder with polymer, resulting in improved rutting resistance. In contrast, the complex modulus was reduced, enhancing fatigue performance at temperatures below 40°C. Li and others revealed that NS has good self-healing and adhesive characteristics **(Li et al., 2017).** It has been demonstrated that including nanomaterials enhances the physical and chemical characteristics of the asphalt binder, resulting in nano-modified Asphalt with higher performance. According to **(Enieb and Diab, 2017)** asphalt stiffness, resistance to fatigue cracking, tensile strength, plastic deformation, and moisture damage were all enhanced by the NS modification. The influence of NS particles on the behavior of polymer-modified asphalt binders was investigated. According to the investigation's findings, NS particles improve the fatigue properties of a polymer-modified binder. This implies that NS particles significantly influence the performance properties of polymer-modified binders **(Bala et al., 2018)**. Al-Sabaeei and coworkers found an improvement in the performance of the NS-modified Asphalt in terms of rutting and aging **(Al-Sabaeei et al., 2020).** The exfoliated structure of the NS, which functions as a barrier to stop oxygen from entering the binder matrix and light components of the binder from evaporating, was found to be responsible for the aging resistance.

This work aims to study the effect of nano silica on the Marshall characteristics and moisture susceptibility of the modified asphalt mixture and compare it to a control mixture. Asphalt cement AC (40-50) was modified with NS at three concentrations (2%, 4%, and 6%) by weight of the binder. Asphalt mixture tests such as Marshall and Tensile Strength Ratio (TSR) were performed to assess the performance of modified mixtures. **Fig. 1** is the flowchart of the methodology opted for the present study.

2. MATERIALS and TESTING METHODS

2.1 Asphalt Cement

Asphalt cement with penetration grade 40/50, provided by the Al-Daurah refinery in Baghdad, was used as the base material. Asphalt cement's physical properties are listed in

Table 1. All test results meet the Iraqi State Corporation for Roads and Bridges **(SCRB/R9, 2003).**

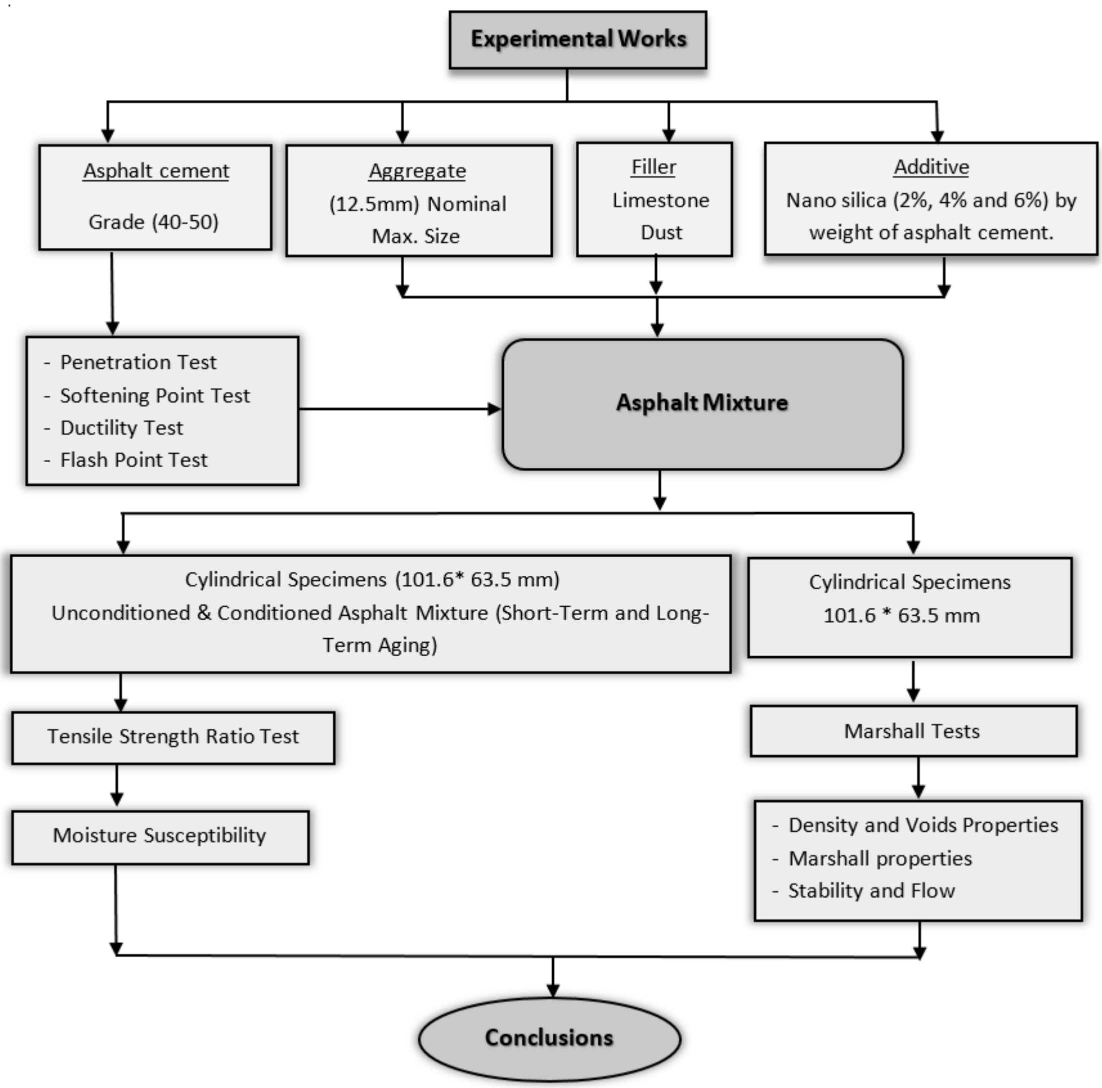

Figure 1. Flow chart for work program

Table 1. Asphalt cement's physical properties

Test	Results	SCRB 2003 Specification Limits	ASTM Specification No
Penetration (25 °C, 100g, 5 sec)	44	40 - 50	D-5
Ductility, (25°C, 5 cm/min)	168	≥ 100	D-113
Kinematics viscosity, at 135 °C	51	-----	D-36
Softening point (Ring & Ball)	1.046	-----	D-70
Flashpoint (Cleveland open cup)	272	>232	D-92

2.2 Aggregates

Coarse crushed aggregate was utilized in this study and supplied by Al-Obaidi Mix Plant. The coarse aggregate size for the Asphalt wearing course varied between 19 mm and 4.75 mm sieve sizes. According to Iraqi State Corporation for Roads and Bridges **(SCRB/R9, 2003)** requirements, fine aggregate has particle sizes ranging from No. 4 to No. 200.
The physical properties of fine and coarse aggregate are given in **Table 2**. Limestone dust was used as a filler to prepare an asphalt concrete mixture for this study. It was bought from an Iraqi lime plant in Karbala. The gradation of the aggregate and filler was chosen following the **(SCRB/R9, 2003)** requirement. **Fig. 2** illustrates the aggregate gradation that was utilized.

Table 2. Physical Properties of Fine and Coarse aggregate

Property	ASTM Designation Method	Coarse Aggregate	Fine Aggregate
Bulk Specific Gravity	C-127 & C-128	2.58	2.63
Water Absorption %	C-127 & C-128	0.57	0.731
Los Angeles Abrasion %	C-131	15.26	-----

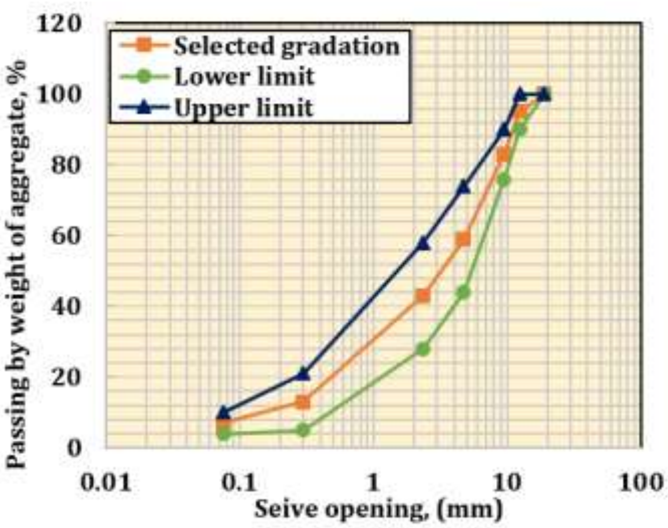

Figure 2. Design aggregate gradation

Figure 3. Digital image of Nano silica

2.3 Nano Silica (NS)

A high-dispersion powdered form of hydrophobic Nano silica (NS) particles supplied by US Research Nanomaterials, Inc. was used. Nano silica has been employed as an additive to enhance binder characteristics, which improves bituminous mix performance. NS particles used in this work are presented in **Fig. 3**, while the characteristics of NS are presented in **Tables 3** and **4**.

Table 3. Analysis of Nano silica

SiO_2	Ca	Ti	Fe	Na
99.5%	70 ppm	120 ppm	20 ppm	30 ppm

Table 4. Properties of Nano silica

Particle Size	True Density	Surface Area	Bulk Density	Purity
20-30 nm	2.4 g/cm^3	180-600 m^2/g	< 0.10 g/cm^3	+99%

2.4 Incorporating of Nano silica (NS) with Asphalt

Asphalt cement was modified with NS at three concentrations (2%, 4%, and 6%) by weight of the binder, respectively. The bitumen modification with NS was conducted using a high-shear mixer to accomplish the mixing process. After heating the Asphalt to 163°C, NS was gradually added to achieve the desired level while the mixer rotated at 500 rpm to achieve uniform dispersion of NS particles. After 10 minutes, the mixing speed was raised to 3000 rpm for an hour **(Alhamali et al., 2016)**.

2.5 Microstructural Analysis

Field Emission Scanning Electron Microscopy (FE-SEM) was used to ensure nano silica dispersion in the asphalt binder. The surface morphology and microstructural modifications of NS-modified binder were investigated using FE-SEM images of asphalt binder. A Zeiss Field Emission Scanning Electron Microscope (FE-SEM) at the Oxford Instrument in Tehran, Iran, was used to describe the distribution and morphology of NS in asphalt binder and asphalt mixture. After mixing NS with Asphalt at the three concentrations (2%, 4%, and 6%), a slice from each variety was tested.

2.6 Marshall Test

This test aimed to determine the OAC, air voids, density, stability, and flow of Asphalt concrete mixtures. Using five contents of Asphalt (4%, 4.5%, 5%, 5.5%, and 6% by weight of the total mixture), three specimens were made for each of the five contents to determine the optimum asphalt content for the HMA. Similar specimens were prepared using Asphalt modified with 2%, 4%, and 6% NS. The same procedure was applied to maintain the OAC for the modified mixtures. The Marshall parameters were calculated using the Marshall test with Marshall specimens measuring 63.5 mm in height and 101.6 mm in diameter. **Fig. 4** shows the procedure of the Marshall test.

(a) Preparing mixture (b) Group of Marshall specimens (c) Marshall test

Figure 4. Marshall test procedure.

2.7 Tensile Strength Ratio Test (TSR)

The **ASTM D-4867** was utilized to investigate the moisture sensitivity of asphalt specimens. The trial method was employed to prepare four Marshall specimens without any additive that complies with the job-mix formula, using (45,55,65,75) blows successively to achieve the number of blows that give 7±1% air voids. 54 blows were applied to achieve this percentage, as illustrated in **Fig. 5**.

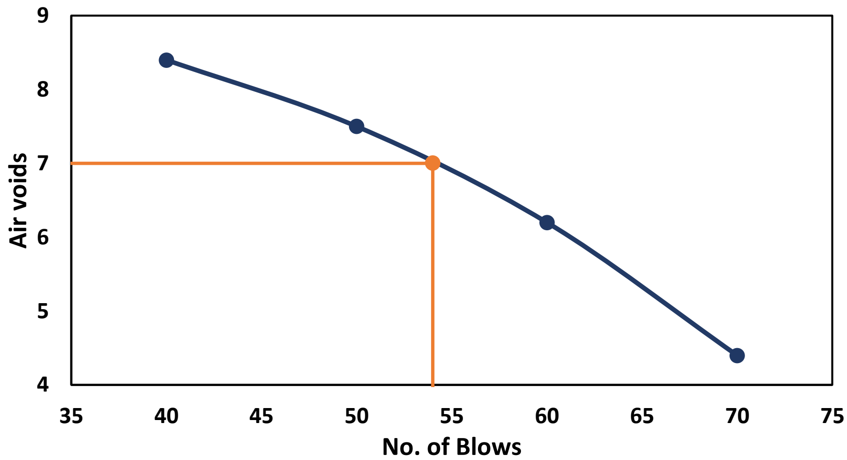

Figure 5. Relationship between air voids percent and No. of blows.

The **AASHTO R 30-2019** standard procedure simulated the mixtures' aging. The asphalt mixture underwent short-term (ST) aging, which simulates the aging that occurs when the Asphalt is exposed to heat and air during the construction and paving stages. Following mixing, the asphalt mixture was placed in a forced-draft oven for 4 hours and 5 minutes at a compaction temperature of 135 °C, stirring hourly to maintain uniform conditioning. Next, the mixture was removed from the oven and compacted before testing. Long-term (LT) aging replicates the aging throughout the Asphalt's service life. For LT specimens, the loose samples first underwent a short-term aging procedure at 135°C for 4 hours after mixing. Then, the specimens were compacted and placed in a laboratory oven at 85 °C for five days (120 h) to simulate the long-term aging process. After preparing Marshall specimens for three aging conditions (unaged specimens, short-term aged (ST) specimens, and long-term (LT) aged specimens), the Marshall specimens were classified into four groups of two sets. One of the groups was without any additive, and the remaining were modified with 2, 4, and 6% of NS.

The first set (three specimens) was selected as unconditioned specimens, immersed for 30 minutes in a water bath at 25 °C, and tested in an indirect tension test. The second set of specimens from each group was immersed in distilled water at 25 °C under a vacuum to remove air and then subjected to one cycle of freezing and thawing (16 h at -18 °C and then 24 h at 60 °C). After that, they were taken out and placed in a water bath at 25 °C for an hour and performed the same test as the first set (conditioned specimens). For the ITS computations, the highest force required to break the specimen was recorded after loading the specimens until failure at a 50.8 mm/min load at 25 °C. The following equation was utilized to evaluate the ITS value:

$$\text{ITS} = \frac{2000\,Pmax.}{\pi\, t\, D} \qquad (1)$$

where ITS is the indirect tensile strength (kPa), *Pmax.* is the maximum value of the applied vertical load (N), *t* and *D* are the sample's thickness and diameter (mm). The ratio of the ITS of wet set of specimens (*con.ITS*) to the ITS of dry set of specimens (*un con.ITS*), is used to measure the indirect tensile strength ratio (TSR), according to Eq. (2).

$$\text{TSR}\ \% = \frac{ITS_{con}}{ITS_{uncon}} * 100 \qquad (2)$$

The procedure of this test is shown in **Fig. 6.**

3. RESULTS and DISCUSSION

3.1 Microstructural Analysis

FE-SEM images of NS-modified Asphalt assisted in estimating NS dispersion within the Asphalt and understanding the microstructure of modified Asphalt. As seen in **Fig. 7** to **Fig. 12** the asphalt mixture with NS particles had a considerably different microstructure from the control binder. The FE-SEM images of the NS and NS-modified asphalt binder show that the nanoparticles were dispersed homogeneously throughout the binder. This may be related to NS particles' huge surface area and good dispersion potential. However, some agglomerations can be seen in the mixture. The agglomerations are caused by the

(a) Indirect tensile strength specimens

(b) Set of specimens in the oven for LT aging

(c) Freezing cycle

(d) Sample under Test

Figure 6. The steps of the **TSR** test

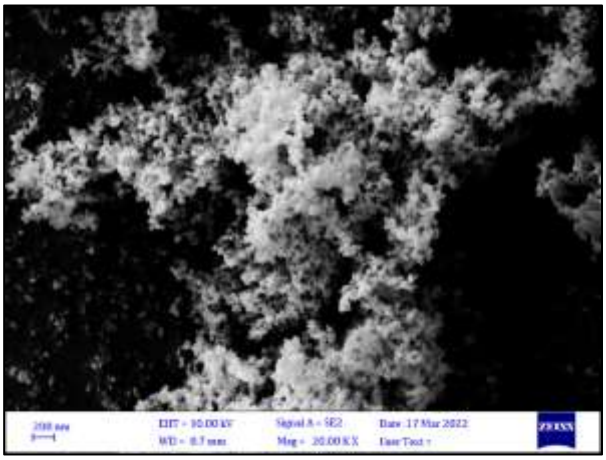

Figure. 7 FE-SEM image of nano silica

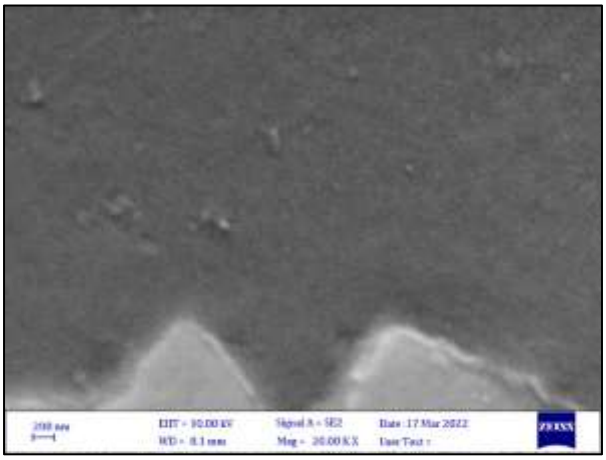

Figure. 8 FE-SEM image of control binder

Ostwald ripening phenomenon **(An et al., 2012).** Ostwald ripening causes small crystals or solution particles to dissolve and redeposit onto large crystals or solution particles. Establishing a dense framework structure in the asphalt mixture modified with NS may also lead to improved bonding characteristics between aggregates and binders, as shown in **Fig. 12.**

Figure. 9 FE-SEM image of 2% NS-modified binder

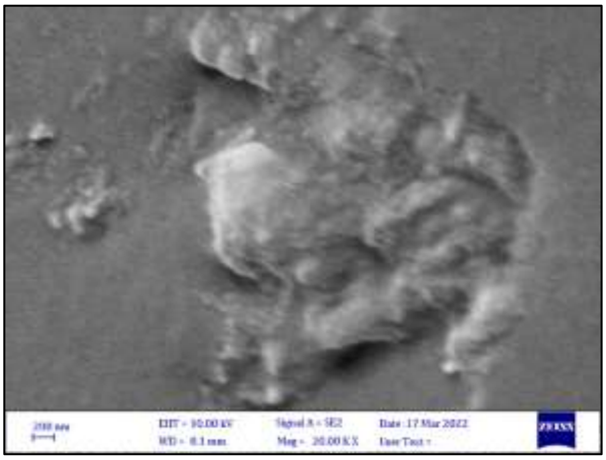

Figure. 10 FE-SEM image of 4% NS-modified binder

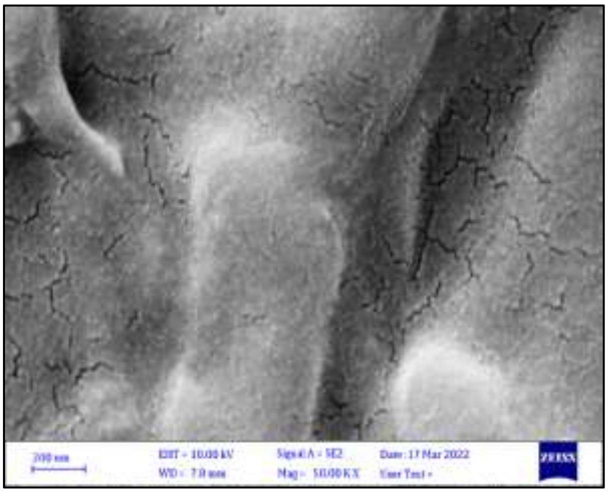

Figure. 11 FE-SEM image of 6% NS-modified binder

Figure. 12 FE-SEM image of 4% NS-modified mixture

3.2 Marshall Test Results

The optimum asphalt content increased by adding 2%, 4%, and 6% of NS from 4.92% in an unmodified binder to 5.21%, 5.3%, and 5.5%, respectively. The Marshall stability results show that adding 2, 4, and 6% NS increased the Marshall stability of the mixture by 11.47, 26.09, and 39.9, respectively, as shown in **Fig. 13**. This increase in stability is attributable to an increase in binder stiffness caused by the increased specific surface area of NS, which increases the surface chemistry of nano silica and asphalt absorption. **Fig. 14** shows that the addition of NS decreased the Marshall flow. The absorption of volatiles by NS and the increase in stiffness caused by NS modifications lead to a decrease in flow. The bulk density increased as the percentages of NS increased up to 6%. The mixture became denser due to the high surface area of NS added to the Asphalt. **Fig. 15** shows the effect of NS on the bulk density of the asphalt mixture.

As illustrated in **Fig. 16**, the air voids reported low values compared to the conventional mixture. This is owing to the modified binder's higher viscosity due to the addition of NS. Increases in viscosity may increase the binder film that coats the aggregates, reducing the percentage of air voids. The VMA% decreased as a result of the addition of 2%, 4%, and 6% of NS, as shown in **Fig.17**. The amount of voids filled with bitumen was increased due to the high surface area of the NS, Which improves Asphalt's ability to fill these voids, resulting in a more durable asphalt mixture. The VMA% was reduced when NS was added to asphalt cement, as illustrated in **Fig. 18**. The reduction in VMA% can be related to an increase in bulk-specific gravity.

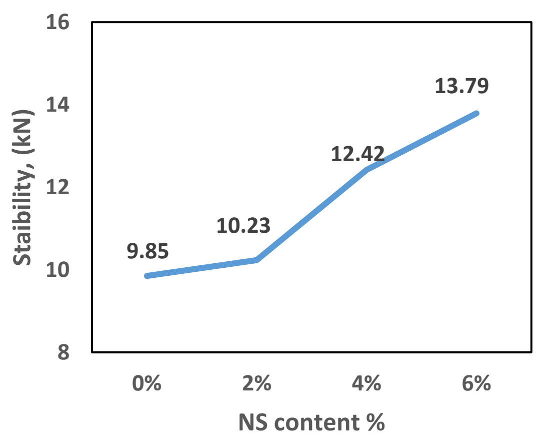

Figure 13. Effect of NS on Marshall stability.

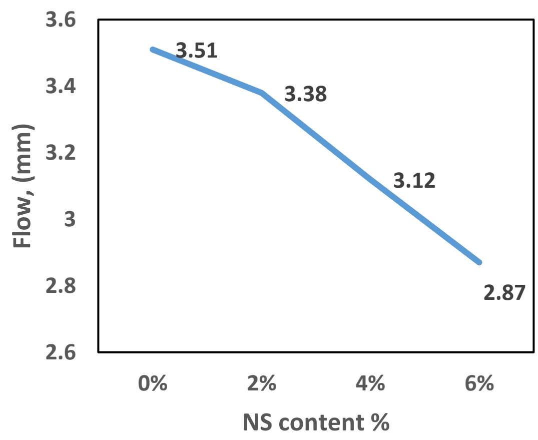

Figure 14. Effect of NS on Marshall flow.

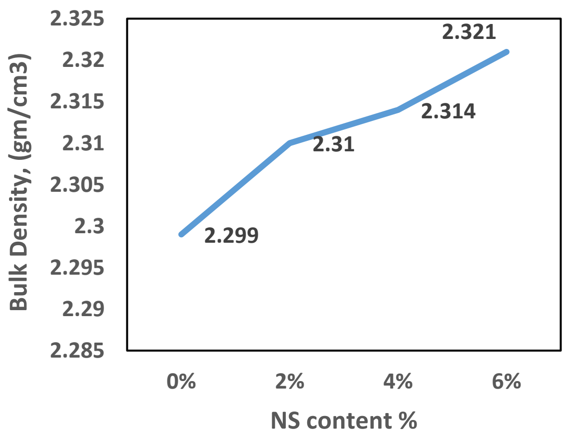

Figure 15. Effect of NS on bulk density.

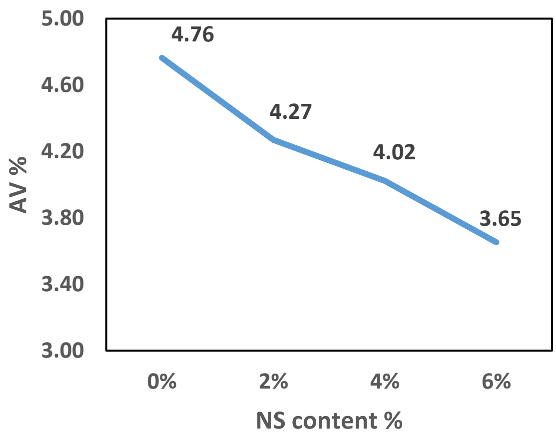

Figure 16. Effect of NS on air voids.

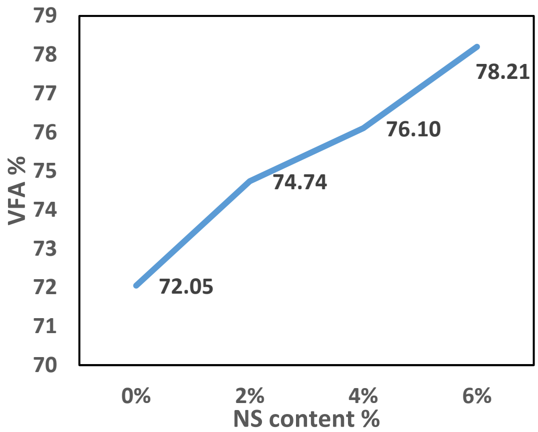

Figure 17. Effect of NS on VFA.

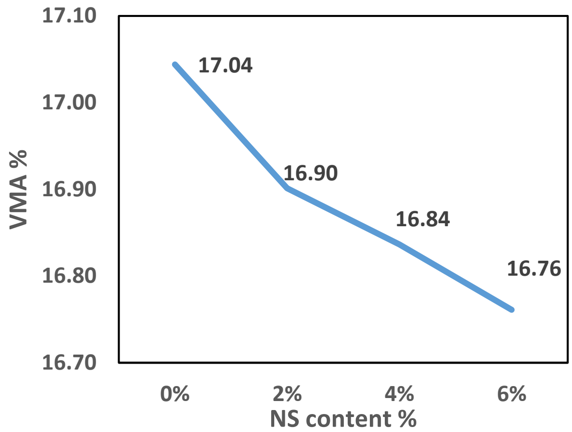

Figure 18. Effect of NS on VMA.

3.3 Indirect Tensile Strength Test Results

The indirect tensile strength values of modified mixtures at unaged conditions are higher than the ones for the conventional mixture. The dry (ITS) increased by 7.14%, 14.94%, and 24.68% with additions of 2%, 4%, and 6 % NS, respectively, as compared to the control mixture, while the wet (ITS) increased by 11.2%, 22.4%, and 40.8% with additions of 2%, 4%, and 6% of NS, respectively, as shown in **Fig. 14**. As the mixtures aged, the **ITS** values increased due to binder stiffening. The NS-modified mixture exhibits higher values when subjected to ST aging, just like the unaged condition. The dry (ITS) increased by 7.01, 16.56, and 23.57%, adding 2, 4, and 6% of NS, respectively. The wet (ITS) increased by 10.77, 26.15, and 31.54% with adding 2, 4, and 6% of NS, as shown in **Fig. 15**. When the mixtures underwent the LT aging process, the NS-modified mixtures again had higher ITS values than the control mixture. The results of the dry (ITS) for modified mixtures with 2%, 4%, and 6 % NS were NS higher than conventional mixtures by 8.13%, 21.25%, and 12.50%, respectively, while the wet (ITS) increased by 16.28%, 37.21%, and 20.93% with the addition of 2%, 4%, and 6% of NS, respectively, as shown in **Fig. 16**.

For an unaged condition, due to the increase in ITS in wet conditions, the TSR increased by 3.79, 6.49, and 12.93% with the addition of 2, 4, and 6% NS, respectively, compared with the conventional mixtures. TSR values for ST and LT aged mixtures follow the same trend as unaged mixture TSR values, with the modified mixtures having higher TSR values than the conventional mixture. The TSR increased by 3.52, 8.23, and 6.45% for ST aging and by 7.54, 13.16, and 7.49% for LT aging with 2, 4, and 6% of NS, respectively, compared with the control mixture as shown in **Fig. 17**. Aging and NS modification increased the binder's stiffness, allowing the mixture to withstand higher tensile stresses before failure. The interaction of a large number of NS particles with the binder causes the stiffness to increase; the particles absorbed the solvents from the binder, which resulted in the binder stiffening. Oxidation and volatile loss increase mixture hardness. The resistance to permanent deformation and load-bearing capacity is significantly improved by increasing the mixture hardness, particularly in ST aging. The increase in binder viscosity due to the high NS concentration and the progressive oxidation during aging may be related to the decrease in **ITS** value when using 6% of NS at LT aging. The results are consistent with previous studies **(Taherkhani and Tajdini, 2019; Ganesh and Prajwal, 2020).**

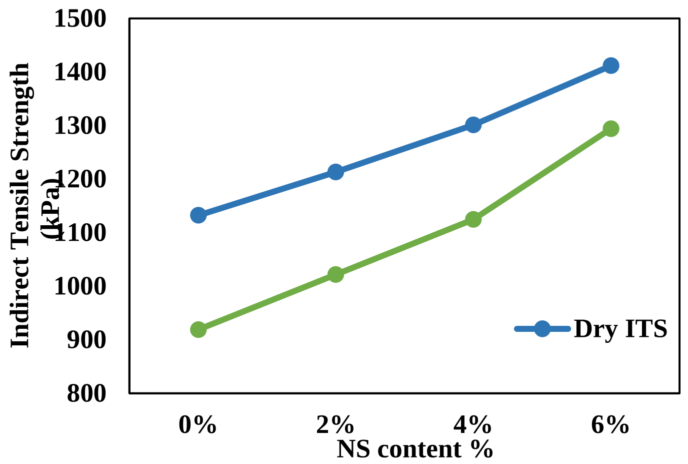

Figure 14. Effect of NS on dry and wet ITS for unaged condition

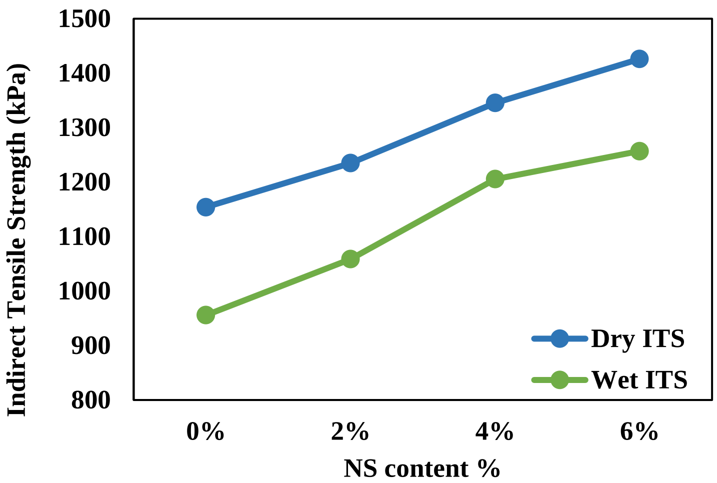

Figure 15. Effect of NS on dry and wet ITS for ST aging condition

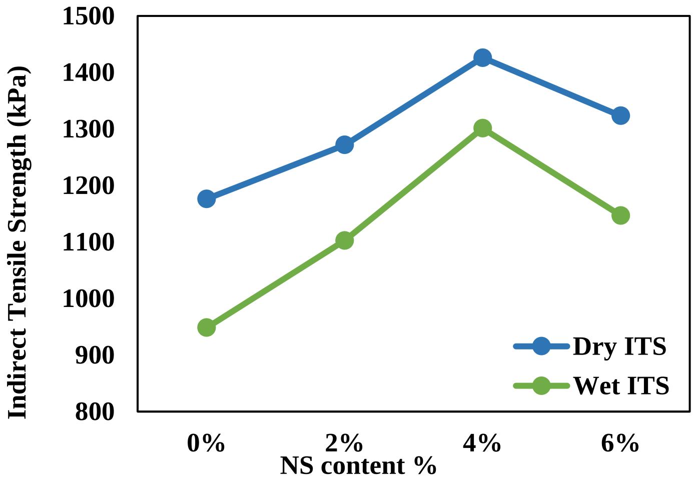

Figure 16. Effect of NS on dry and wet **ITS** for LT aging condition

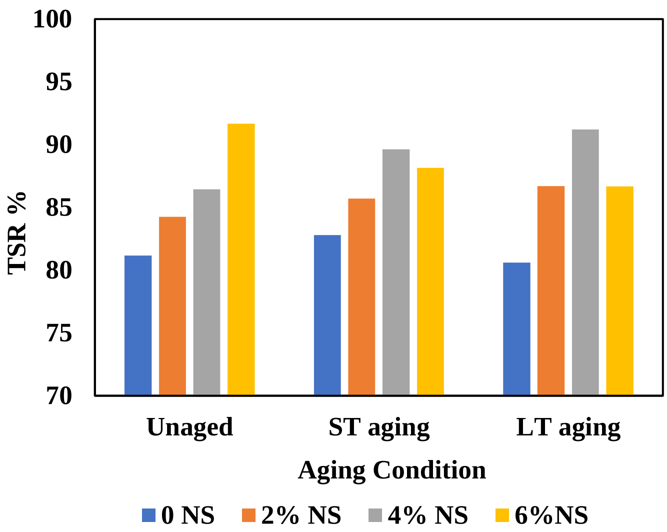

Figure 17. Effect of NS on **TSR** for different aging conditions

4. CONCLUSIONS

The performance of asphalt mixtures modified with nano silica is investigated in this work. The following conclusions were reached:

1. The addition of NS enhanced the asphalt mixture's Marshall properties. The highest Marshall stability was recorded when 6% of NS was added; it was 13.79 kN compared to 9.85 kN for the control mixture. The addition of NS reduced Marshall flow, with 4% of NS causing the highest reduction.
2. The FE-SEM images showed that the NS particles were distributed uniformly in the binder matrix and that the adhesion properties between the NS-modified asphalt binder and aggregates improved.
3. Modified bitumen's indirect tensile strength values are higher than those of unmodified bitumen. Regardless of the aging process used, aging significantly increases the indirect tensile strength values due to binder hardening. The **ITS** test results revealed that aged samples have a higher **ITS** than unaged samples and that 4% NS has the highest value for long-term aging. This suggests that increasing the NS content reduces vulnerability to oxidative aging significantly, especially during long-term aging.
4. Based on **TSR** values, NS seems to delay the aging-related hardening process. As a result, the strength improves without affecting consistency or adhesion properties, making it less susceptible to moisture.

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