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EVALUATION OF NANOMODIFIED ASPHALT WITH $CaCO_3$: RHEOLOGICAL AND MIXTURE PERFORMANCE

Ahmed Mahir Mohammed[1,*], Alaa H. Abed[2]

[1]Civil Engineering Department, University of Baghdad, Baghdad, Iraq
[2]Civil Engineering Department, AL-Nahrin University, Baghdad, Iraq

*E-mail of corresponding author: ahmed.m.886@coeng.uobaghdad.edu.iq

Ahmed Mahir Mohammed 0009-0001-4585-323X, Alaa H. Abed 0000-0002-6569-4598

Resume

The impact of using three contents of 1, 3 and 5 % of nano CaCO3 on the composition of asphalt binder and the mechanical properties of Hot Mix Asphalt (HMA) wearing course was studied. Laboratory tests were performed to examine the physical and rheological characteristics of nanomodified asphalt binder. In addition, Superpave Indirect Tensile Test (IDT) was conducted to evaluate the mechanistic characteristics and moisture susceptibility of the HMA. The test findings pointed that the nano modified asphalt with 5% nano $CaCO_3$, decreased the temperature sensitivity due to asphalt stiffening and improved resistance to permanent deformation. Additionally, HMA mixture performance exhibits a substantial increase in moduli value at temperatures varying from 5 to 40 °C and increased flexibility at lower testing temperatures as compared to the neat HMA mixture.

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1 Introduction

Researchers and agencies have recently focused on a trend of growing attention to the development of longer pavement life that sustains heavy traffic loads along with environmental impact. Nanomaterial has recently been incorporated into the asphalt as the key solution to overcome and survive the pavement lifecycle against the common distress that occurs in high-temperature climates as permanent deformation, fatigue, and thermal cracking besides stripping and reveling action that easily extracts asphalt film from aggregate leading to early premature failure within the pavement. Researchers worldwide have extensively used various types of nanomaterials, including TiO_2.SiO_2, ZnO, Fe_2O_3, and $CaCO_3$ that has been investigated in this study. Due to the rapid progress in nanomaterial technology, nano $CaCO_3$ is currently being employed to prepare the modified asphalt as a dependable and cost-effective material. The Nano-$CaCO_3$ is identified as economical material for producing modified asphalt; it has a white powder consisting of particles that have an average size ranging from 10 to 100 nanometers is produced using two methods: (a) the mineral carbonation process, which involves using industrial wastes such as CaO and $CaCl_2$ as a source of calcium in a packed bed reactor [1], and (b) utilizing the CO_2 exhaled during the cement manufacturing process at a cement plant [2]. Thus, employing nano $CaCO_3$ as a modifier additive not only reduces CO_2 emissions but also proves to be a cost-efficient approach to improving the rheological properties of the asphalt binder. It is composed of roughly 98.5 % calcium carbonate. Combined with asphalt, it enhances the performance of HMA mixtures by establishing a stable system that improves temperature susceptibility, particularly at high temperatures.

A study conducted by Hao et al. [3] revealed that adding 6 % nano $CaCO_3$ to asphalt concrete improved dynamic and residual stability. This enhancement was observed in both high-temperature and water stability. A separate group of scientists that was investigating to assess the rheological, physical, and performance properties of hot mix asphalt (HMA) that was improved by the addition of nano $CaCO_3$ found that higher nano concentration results in reduced penetration, increased stiffness and viscosity, heightened susceptibility to permanent deformation, and enhanced anti-aging effects [4-7]. Zhang et al. [8] conducted a study demonstrating the positive impact of incorporating up to 5 % nano-$TiO_2/CaCO_3$ on the mechanical properties of bituminous materials. This inclusion resulted in a decrease in penetration, while simultaneously increasing the softening point and viscosity. As a result, the sensitivity of the bituminous materials was reduced.

Additionally, it improves the durability of the bituminous material against deformation and wear caused by repeated stress at moderate temperatures. Adding $TiO_2/CaCO_3$ increased the asphalt stiffness modulus while raising its viscosity. The unique characteristic of nano $CaCO_3$ is its capacity to improve the durability of modified asphalt binder at elevated temperatures by strengthening it through its dispersing properties. Xing et al. [9] established that reducing particle sizes leads to a higher density of particles, hence enhancing the yield stress of the modified asphalt via the Orowan mechanism. According to the literature, nano $CaCO_3$ has improved different elements of asphalt performance, including its sensitivity to temperature, susceptibility to moisture, resistance to cracking fatigue, aging, adhesion, and dispersion in asphalt binder. Adding nano $CaCO_3$ to HMA mixtures increased rutting while decreasing susceptibility to high temperatures. When 4 % $CaCO_3$ is added, increasing the viscosity of the asphalt binder improves the tensile and compressive strengths of the HMA, leading to a 55.8 % improvement in fatigue life [10]. Zhai et al. [11] have investigated the impact of different concentrations (3 %, 4 %, 5 %, 6 %, and 7 %) of nano $CaCO_3$ modified asphalt in SBR polymer.

The study aimed to assess the performance of the HMA mixture using various tests, including Wheel track, static creep, overlay, and beam bending tests. The results demonstrated a significant improvement in the ability to resist rutting by incorporating 5 % $CaCO_3$/SBR. This improvement can be attributed to the increased surface area of nanoparticles, which enhanced viscosity and adhesion. As a result, the micromechanical properties, including adhesion, dissipated energy, anti-cracking resistance, and flexural deformation, were greatly enhanced compared to using asphalt modified only with SBS.

In a recent study conducted by Yarahmadi et al. [12], it was found that utilizing Nano $CaCO_3$ may successfully decelerate the progression and spread of fatigue fractures in Stone Matrix Asphalt (SMA) mixes. The performance of the SMA combination was enhanced at elevated temperatures and stress levels. The indirect tensile strength (ITS) test findings demonstrated that the addition of nanotechnology-enhanced materials to the SMA mixes enhanced its ability to withstand moisture damage and perform well when exposed to water. Li et al. [13] examined how adding nano

***Table 1** Doura Asphalt physical test*

Property	Units	Result
Penetration at 25 oC, 100 gm, 5 sec	0.1 mm	46
Softening Point	oC	49
Specific gravity at 25 oC	----	1.03
Flash point	oC	288
Ductility	cm	114
Residue from thin-film oven test AASHTO T 179		
Retained penetration, % of original	0.1 mm	61
Ductility at 25 oC, 5 cm/min	cm	87

***Table 2** Doura Asphalt Rheological test*

Property	Result	Temp.	M320
Original Test on Binder			
Rotational Viscosity, Pa.s.	0.63	135 °C	Max. 3
	0.16	165 °C	--------
Dynamic Shear Rheometer, G*/sin δ, kPa	2.34	64 °C	Min. 1
	0.84	70 °C	
	Tests on RTFO Residue		
	1.68	70 oC	Min 2.2
Tests on PAV Residue			
Dynamic Shear Rheometer, G*.sin δ, kPa	4266	25 °C	
	3452	28 °C	Max. 5000
Creep Stiffness, S, MPa	241	-12 °C	Max. 300
Creep Slope, m value.	0.322	-12 °C	Min. 0.3

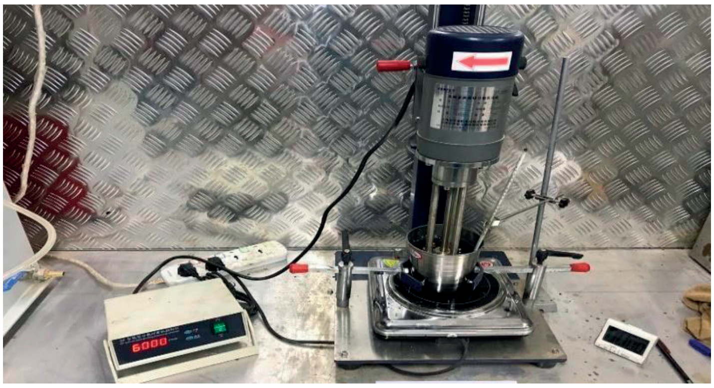

Figure 1 *Mixing of NCC with Asphalt*

$CaCO_3$/ SBR and ZnO/SBR composite modifiers affects the rutting resistance and viscosity of AK-70 asphalt binder to investigate various concentrations of nano CaCO3 (4 %, 5 %, 6 %), Nano ZnO (1 %, 3 %, 5 %), and SBR (3 %, 4 %, 5 %) evaluated using Dynamic Shear Rheometer (DSR) and Rationale viscosity (RV) tests. The results indicated that the modified asphalt with composites exhibited increased viscosity and improved resistance to rutting compared to the neat asphalt. Vasilievici et al. [14] investigated the impact of a 5 % Polystyrene ratio on the stiffness of the asphalt binder D50/70, including nano $CaCO_3$, utilizing dynamic mechanical analysis. Polystyrene was shown to increase the stiffness of the nano $CaCO_3$ asphalt binder.

This research hypothesis shows the impact of using three contents of 1, 3 and 5 % of nano $CaCO_3$ types of nanomaterials on the composition of asphalt binder and the mechanical properties of HMA wearing. Laboratory tests including physical asphalt test and RV, DSR, Bending Beam Rheometer (BBR), Rolling Thin Film Oven (RTFO), and Pressure Aging Vessel (PAV), were performed to examine the physical and rheological characteristics of the binders. Additionally, Superpave IDT was conducted to evaluate the mechanistic characteristics and moisture susceptibility of the HMA.

2 Material selection

2.1 Asphalt

The 40-50 penetration grade of asphalt yielding to PG (64-16) from Doura petroleum refinery was used in this study, physical and rheological properties of which are described in Tables 1 and 2.

2.2 Nano $CaCO_3$

The study employed nano $CaCO_3$ (NCC) as an asphalt modifier. The NCC is a white substance powder that cannot be dissolved and has an average particle size of 15 to 40 nanometers with a purity level of approximately 97.5 %; it was imported from Sky-Spring Company, United States.

2.3 Blending of nanomodified asphalt

A dry mixing technique was developed in this study, involving high-speed stirring to distribute the nanomaterials evenly throughout the asphalt binder matrix. The NCC was introduced into a sole asphalt PG (64-16) at different concentrations, specifically 1, 3, and 5 % by weight of the neat asphalt. The dry mixing approach employs a high-speed shear mixer (HSM) to disperse the nanomaterials evenly. Next, the nanomaterial is introduced, and the HSMs exhibit a unique tip design that sets them apart from traditional mixers.

Figure 1 illustrates the shear mixing action that occurs over a specific duration. The initial batch of asphalt material, weighing 500 ± 2 g, was heated to a temperature of 160 °C. It was then manually stirred for 20 minutes. The nano additive was added gradually, at a rate of 2 to 4 g per minute. This process continued until all the nanoparticles were thoroughly dispersed within the binder. The specific conditions used a speed of 6000 rpm and 45 minutes as a blending time. The mixing uniformity is sufficient to prevent asphalt aging. After completion, the asphalt was poured into metal cans and allowed to cool at room temperature for further testing.

3 Testing protocols

3.1 Physical and rheological asphalt test

Both neat and nanomodified asphalt at varying percentages were introduced into physical routine tests, including penetration test ASTM D5, softening point ASTM D36, ductility ASTM D113, storage stability ASTM D7173. Finally, asphalt temperature sensitivity is measured using the penetration Index (PI) [15]:

$$PI = \frac{1952 - 500\log_{10}P25 - 20SP}{50\log_{10}P25 - SP - 120} \quad (1)$$

Asphalt viscosity was determined using RV according to AASHTO TP 48 at 135 °C. On the other hand, the DSR was utilized at 10 rad/sec frequency, employ ing 8 mm and 25 mm diameter and the gaps were 1 and 2 mm, respectively. The failure temperatures of asphalt were determined by observing the point at which G*/sinδ went below 1 kPa. The aging process involved using the Rolling Thin Film Oven (RTFO) for the short-term aging and the Pressure Aging Vessel (PAV) for the long-term aging was employed to assess the high-temperature rating for modified asphalt and their ability to withstand both high and moderate-temperature environments and repeated loading circumstances. Finally, BBR was utilized to determine low-temperature characteristics of neat and modified asphalt based on AASTHO T313.

4 Discussion and analysis

4.1 The physical response

Figure 2 indicated that with increasing the nano content from 1 to 5 %, the penetration value decreases by about 7, 16 and 20 % as compared to neat asphalt, which can be attributed to the dispersion of the NCC inside the asphalt, which leads to increased stiffness and resistance to penetration. This behavior is an indication that the NCC has acquired the high-temperature viscoelastic characteristics as a result of nanoparticle's higher surface area, which might lead to the asphalt's reduced sensitivity towards temperature [6].

Similar trends are also noted in Figure 3; hence, nanomodified asphalt had reduced ductility value by 6, 13, and 21 % as nano content increased from 1 to 5 %, respectively, compared to the neat asphalt.

On the other hand, the modification of the neat asphalt presents sensitivity towards the temperature variation; for this, the softening point suffers a change in falling to temperature of 4 °C at 1 % NCC content, reaching 8 °C at 5 % NCC, as shown in Figure 4.

The drop in penetration value and the increase in softening point imply an increase in specimen stiffness and a decrease in temperature susceptibility, resulting in improved resistance to rutting at high temperatures.

The penetration test results are consistent with research conducted by Hao et al. [3]. The rise in softening point is a positive indication as bitumen with a higher softening point tends to be more resistant to permanent deformation [16]. The PI values gets higher with the increment of modifier content, which indicates the enhancement of modified bitumen against high-temperature susceptibility. Figure 5 flows that the PI value increased to - 0.82, - 0.59, and - 0.28 for nano content range 1 to 5 %. Nanomodified asphalts with NCC having high PI values are more resistant to low-temperature cracking and permanent deformation.

The final comment adheres to the storage stability of nanomodified asphalt since it relates to the agglomeration issue and is presented in Figure 6; the

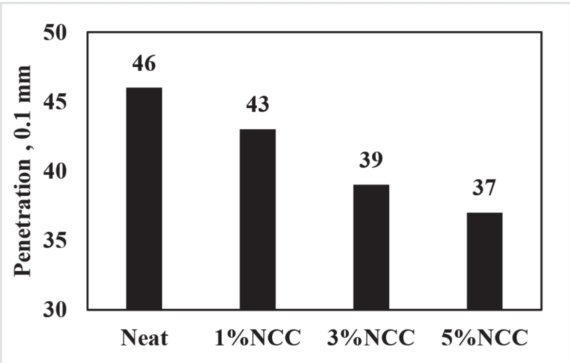

Figure 2 *Penetration value of neat and NCC modified asphalt*

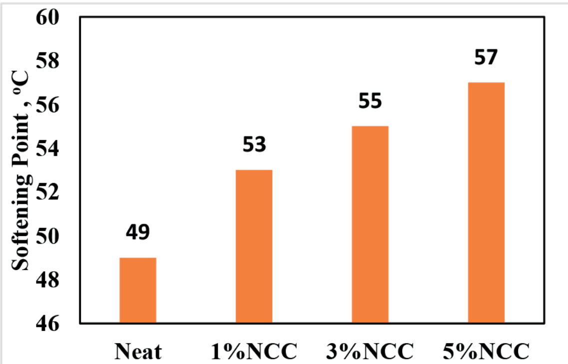

Figure 4 *Softening point value of neat and NCC modified asphalt*

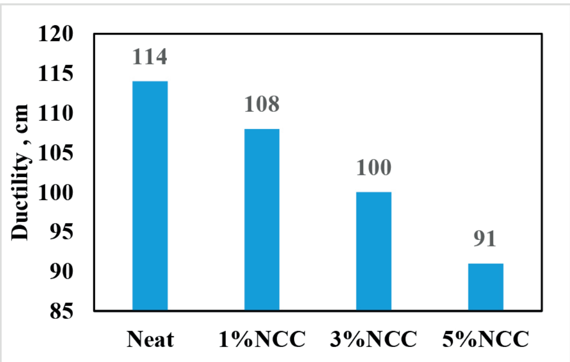

Figure 3 *Ductility value of neat and NCC modified asphalt*

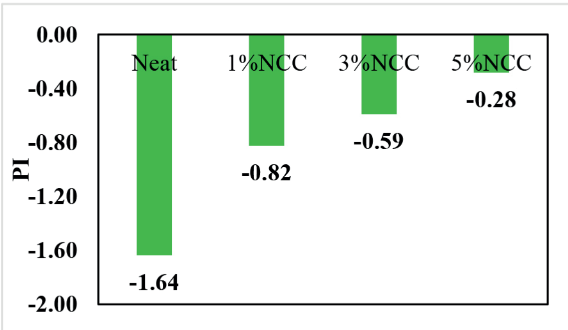

Figure 5 *PI value of neat and NCC modified asphalt*

used HSM with 6000 rpm has significantly approved that with the increasing content, the difference between softening reading will not reach beyond the critical value of 2.5 °C, thus, ensuring a good dispersion for the NCC modified blends.

The result obtained from physical testing indicated an improvement related to the modification of the asphalt binder by stiffening the asphalt within the penetration grade; the use of 5 % Nano content had the greatest impact on the properties of the asphalt with NCC. While the penetration index and softening point rise with the amount of nanomaterial, asphalt ductility is decreased due to the material's decreased temperature sensitivity, improving resistance to low-temperature cracking and permanent deformation.

4.2 The rheological response

A DSR was used to find the Superpave PG system's upper critical temperature (T_{crit}). The neat and nanomodified bitumen samples, both unaged and RTFOT-aged, were exposed to DSR oscillatory shear at 10 rad/s (1.59 Hz), corresponding to field traffic traveling at around 90 km/h. Initial temperature values were established at 64 °C for unaged samples and 70 °C for samples undergoing RTFOT aging, with a six-degree increment.

Using the G*/sinδ values, the highest critical temperatures for each sample were calculated and employed in the PG system. The DSR standards' rutting, and fatigue requirements are displayed in Table 3, Figure

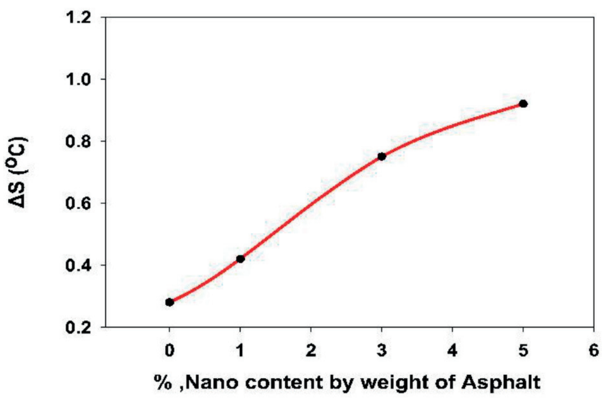

Figure 6 *Storage Stability of neat and NCC modified asphalt*

Table 3 *Criteria required for PG - DSR limit*

Item	Property	Limits	Concern
Original	G*/sinδ	≤ 1.1 kPa	
RTFO	G*/sinδ	2.2 kPa ≤	Rutting
PAV	G*/sinδ	5000 kPa≥	Fatigue

Table 4 *Rutting and upper critical temperature for neat and nanomodified asphalt*

Binder	Temp. °C	G*/sinδ, kPa		T_{crit}
		Original	RTFO	
Neat	64	2.341	3.48	64
	70	0.842	1.68	
	76	0.452	0.589	
1% NCC	64	2.51	3.81	64
	70	1.10	1.84	
	76	0.53	0.77	
3% NCC	64	3.07	4.25	70
	70	1.97	2.85	
	76	0.61	1.30	
5% NCC	64	5.42	5.28	70
	70	3.37	3.49	
	76	1.06	1.89	

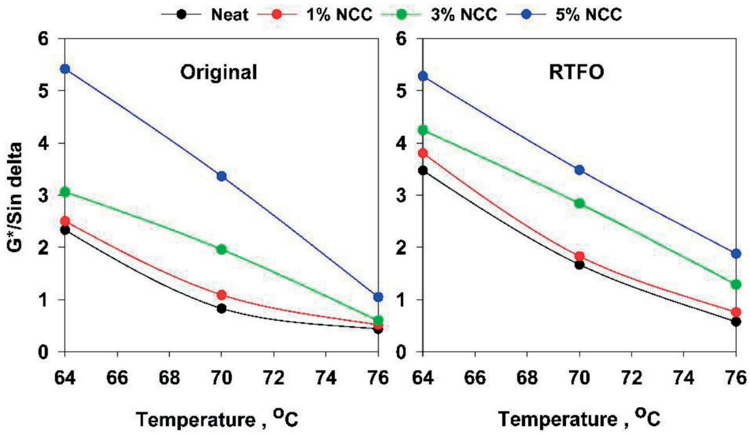

Figure 7 *Result of DSR-rutting parameter G*/sinδ for neat and NCC modified asphalt*

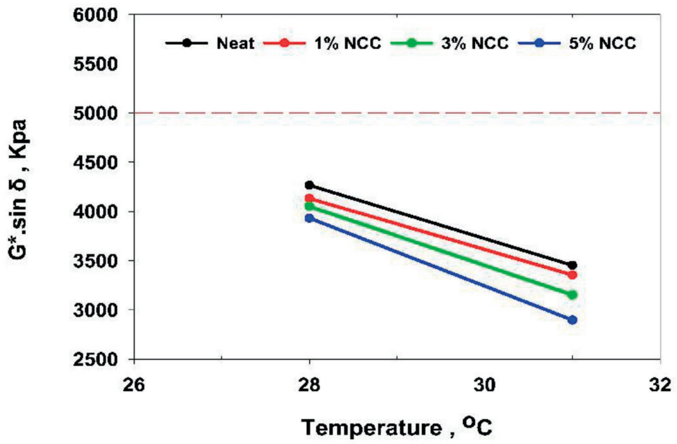

Figure 8 *Result of DSR-fatigue parameter G*.sinδ for neat and NCC modified asphalt*

7, while Table 4 lists the upper critical temperatures for each sample. The upper critical temperature (T_{crit}) serves as a means of evaluating the rutting performance of a specific bitumen sample. A higher G*/sinδ number indicates greater resistance of a sample to permanent deformation.

The results indicate that adding the NCC at concentrations of 3 % and 5 % has positively affected by shifted PG by one grade to a temperature of 70 °C. Modifications in the upper PG grading are linked to the enhancement of high-temperature performance caused by the augmentation of the materials' hardness or stiffness. As a result, they become more resilient to external forces and demonstrate superior resistance to rutting. At a temperature of 70 °C, the RTFO nanomodified asphalt demonstrates improved resistance to rutting. This is achieved by raising the value of G*/sin δ, which reaches 2.85 and 3.49 kPa when the nano range is between 3 and 5 %.

In comparison, neat asphalt at the same temperature only reaches 1.68 kPa. It is worth noting that the G*/sinδ values for un-aged and RTFO-aged samples were constant at the same temperature. Therefore, it can be inferred that the NCC modified RTFO aged samples do not undergo rapid hardening or oxidation, which is often due to aging. Meanwhile, the optimum content of NCC at 5 % relatively moves asphalt specification when tested at PG +6 °C, i.e., 64 °C, indicating less susceptibility to fatigue damage than other Nano types. It is shown that for all Nano types, the 1 % has the lowest stiffness value, which could be attributed to the low amount that did not disperse uniformly in the asphalt. It can be concluded that, 5 % NCC nano modified asphalt is almost satisfying and improves the rutting of asphalt. The improvement in these characteristics suggests that nanomodified asphalt is more resistant to permanent deformation, and rutting at high temperatures will perform better in hot regions where the pavement permanently deforms. An increase in this characteristic also suggests that bitumen will perform better when it is manufactured and used (short-term aging stage). The fatigue factor G*.sin δ was examined at moderate temperatures (25 and 28 °C) to assess the durability against fatigue cracking. The maximum allowable value for G*.sinδ, as per the long-term aging (PAV) criterion, is 5000 kPa. Consequently, a lower value of G*.sinδ is considered a favorable attribute regarding resistance to fatigue cracking. Figure 8 presents the fatigue factor values obtained following the PAV process. The NCC-modified asphalt exhibited a somewhat lower G*.sinδ due to the increased loss in moduli. It is evident that increasing the nano content from 1 to 5 % results in

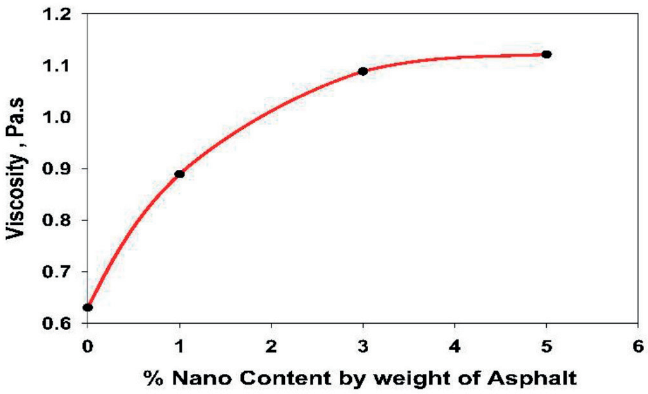

Figure 9 *Viscosity values for neat and NCC modified asphalt*

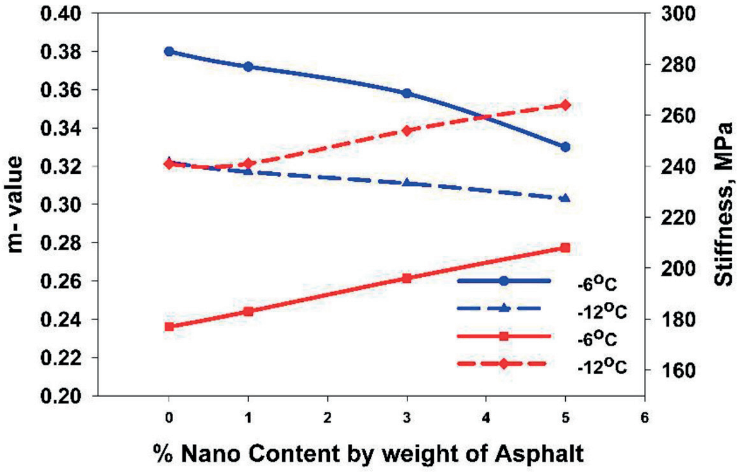

Figure 10 *BBR results stiffness and m-value for neat and NCC modified asphalt*

a drop in the value of G*.sinδ. This decrease occurs at an average rate of approximately 4, 7, and 12 % when tested at temperatures of 25 and 28 °C. This suggests a potential antioxidant effect of nano NCC following PAV [17-18]. Nanoparticles exhibit significant performance potential due to their size and high specific area, and their use is expanding to enhance the asphalt binder's performance characteristics. The effectiveness of asphalt binders modified with metal oxide nanoparticles against the buildup of microcracks and fatigue, as well as its relationship to oxidative aging, is one of the topics that has hardly ever been the focus of prior studies.

According to Figure 9, increasing the NCC content from 1 to 5 % leads to an enhancement in asphalt viscosity. This results in the growth of the binder film thickness and the coating of aggregates in the heated mixture. A viscosity value of 1.121 Pa s was observed using a 5 % concentration of NCC. Consequently, the viscosity of all the nanomaterials increased as the Nano content increased at a temperature of 135 °C. Specifically, a 5 % increase in NCC resulted in a 43 % increase in asphalt viscosity.

The asphalt needs to retain a low stiffness level to mitigate the risk of low-temperature cracking in colder pavement conditions. By raising the Nano content to 5 % at temperatures of -6 and 12 °C, the m-value would be marginally reduced for any content below 8 % compared to pure asphalt. This reduction indicates a decreased capacity for stress relaxation. Figure 10 demonstrates that the inclusion of NCC particles leads to a decrease in the m-value when compared to pure asphalt at both temperatures. The stiffness of the NCC-modified asphalt has exhibited a modest alteration at temperatures of -6 and -12 °C compared to clean asphalt. This alteration is shown in Figure 10 and characterized by an increase in creep stiffness of around 3.6 % and 10 % when the NCC content is raised from 1 % to 5 %. However, adding the NCC to the asphalt has not significantly enhanced its performance at low temperatures and had a negligible impact on low-temperature cracking. This finding aligns with previous studies conducted by [10] and [19].

The physical and rheological properties of nanomodified asphalt with varying levels of NCC content indicate an enhancement in asphalt performance compared to regular asphalt. However, these enhancements are quite small, especially when it comes to a low-temperature examination, which includes BBR and fatigue factor. Consequently, after analyzing the test findings, a certain nano content was selected for each kind to study the HMA mixture. The data indicates that there is a 3 to 5 % rise in modified asphalt. Nevertheless, the incorporation of 5 % Nano content exhibited the

Table 5 *Physical properties of coarse aggregate*

Property	Result		Limits
	5-12 mm	5-9 mm	
Bulk Specific gravity, ASTM C127	2.627	2.618	--------
Apparent Specific gravity, ASTM C127	2.674	2.674	
Absorption, %, ASTM C127	0.66	0.797	
Fractured Face, %, ASTM D 5821	93	95	Min. 90
Consensus Properties			
Coarse Aggregate Angularity, %, ASTM D 5821	97	98	Min. (95/90)
Flat, elongated particles, %, ASTM D 4791	1.2	0.8	Max. 10
Source Properties			
Abrasion %, ASTM C131	21	15	Max .30
Soundness, %, ASTM C88	3.71	2.81	Max .12

Table 6 *Physical properties of fine aggregate*

Property	Result		Limit
	Crusher Sand	River Sand	
Bulk Specific gravity, ASTM C127	2.576	2.545	--------
Apparent Specific gravity, ASTM C127	2.635	2.656	
absorption, %, ASTM C127	0.854	1.647	
Consensus Properties			
Angularity, %, ASTM D1252	60	49	Min. 45
Sand Equivalent, %, ASTM D2419	78	53	Max. 45
Source Properties			
Deleterious. materials, %, ASTM C142	0.58	0.92	Max .10

most significant impact on the characteristics of the asphalt. Therefore, rising the Nano content in asphalt to 5 % may meet the requirement specification, and the asphalt can resist rutting. Ultimately, adding 5 % nano content improves the properties of asphalt binders and will be further explored as the optimal nano content to improve the IDT and moisture damage, as presented in the further manuscript sections.

5 HMA

5.1 Mixing design

An aggregate maximum size of 12.5 mm, crushed quartz sourced from north of Baghdad. The Al-Nibaie quarry serves as the origin of this aggregate for their physical, source, and consensus properties were listed in Tables 5 and 6. Aggregates were prepared to establish a Job Mix Formula (JMF) for the combined aggregate blended with different blending ratios for each raw material, as graphically shown in Figure 11.

Duplicate specimens that have 150 mm in diameter and 115 mm in height were prepared at target design asphalt content of 4.5 % by weight of the mixture and varying percentages of 0.5 % above and below target asphalt as well as 1.0 %. The final volumetric properties and optimum asphalt content are listed in Table 7 for the neat and NCC modified mixture.

5.2 Specimen fabrication and test procedure

In this study, the effect of different percentages was identified to evaluate the Resilient Modulus, Creep, and tensile strength of HMA mixtures at a temperature of 5, 15, 25 and 40 °C with the aid of DTM 50. A cylinder of dimensions 150 mm diameter and 165 mm height was initially designed for air void content (4 %) and fabricated using Superpave Gyratory Compactor (SGC).

The specimen had at least 6 mm sawed off both sides, and four samples were created by sawing specimens to 150 mms diameter by 50 mm thickness, as presented in Figures 12 and 13. The Superpave IDT tests include resilient modulus (Mr), creep (D_t), and tensile-strength tests (TS), which adhere to the test

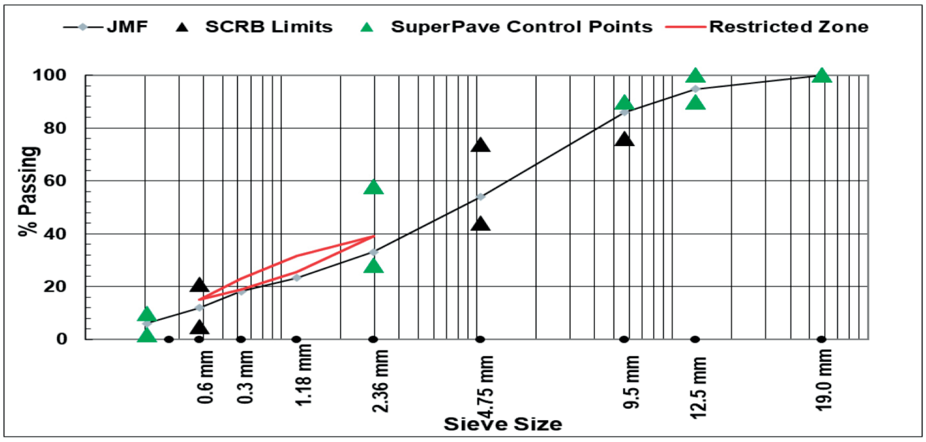

Figure 11 *HMA mix design gradation*

Table 7 *volumetric properties of HMA*

Property	Neat Asphalt	5% NCC modified Asphalt	Requirement
OAC, %	5.0	5.2	4.0-6.0
VA, %	4.0		4.0
VFA, %	74.4	72.92	65-75
VMA, %	15.23	14.73	Min 14.0
DP, %	1.03	1.04	0.8-1.6
% G_{mm} at $N_{initial}$	87.11	86.61	≥ 89%
% G_{mm} at N_{design}	96.13	96.23	96%

OAC = Optimum Asphalt Content, VA= Air Voids, VFA = Void Filled Asphalt, VMA = Voids in Mineral Aggregate, DP= Dust to Binder Ratio

Figure 12 *IDT preparation and cutting*

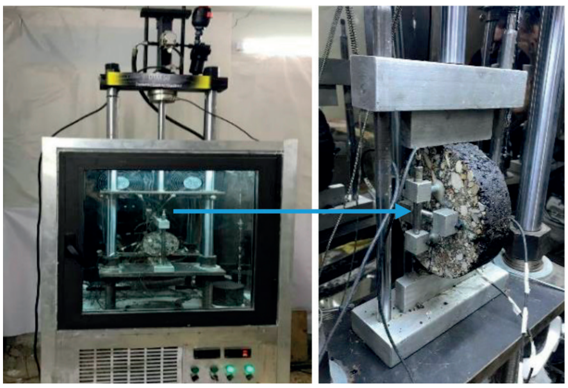

Figure 13 *Configuration of IDT using DTM 50*

procedures established by Roque and Buttlar 1992 ,1994 [20-22] to assess the cracking resistance.

The cylindrical samples underwent a test, where a repeated peak load was applied, causing horizontal deformations within the 0.038 to 0.089 mm range. Each load cycle has a 0.1 s load application interval followed by 0.9 s of rest. The computer continuously recorded the load and deformation. The specific methodologies for estimating the Mr can be located in the works of Roque and Buttlar [20-22] using:

$$M_r = \frac{P \times GL}{\Delta H \times t \times D \times C_{CMPL}} \quad (2)$$

where GL is the gauge length equal to ¼ diameter, P is the maximum load applied, t and D are the thickness and diameter, respectively, ΔH is the Horizontal Deformation and C_{CMPL} is a non-dimensional factor. The creep compliance was adopted on the same specimen with a static load of 100 s, and the recorded stress and strain were applied to find creep compliance as follows:

$$D_j = \frac{\Delta H \times t \times D \times C_{compliance}}{P \times GL}. \quad (3)$$

where D_j (GPa) is the creep compliance at time t, $C_{compliance}$ is the correction factor. Finally, the destructive test was conducted to find the *TS* at a static rate of 50.8 mm/min within DTM 50 following Equation:

$$TS = \frac{2P(C_{SX})}{\pi . t . D}. \quad (4)$$

Hence, C_{SX} is a factor corresponding to the stress correction and P is the maximum recorded load. To study the role of NCC as anti-stripping to prevent the negative impact of moisture damage, the AASHTO T283 procedure was followed. Specimens of a diameter of 101 mm and a thickness of 63 mm are made using OAC % for Marshall testing. Compaction levels range from 43 to 54 blows per each face for neat and nanomodified asphalt to achieve target air voids of 7 ± 0.5 %. The samples were divided into two groups, each consisting of three samples. One group was unconditioned, while the other group was conditioned. The control groups was kept in a water bath at 25 °C for 2 hours before testing. ITS was measured by applying a compressive force to the specimen, using a Marshall testing apparatus at a 50.8 mm/min rate until the specimen fractured. The second set of conditioned samples is placed in a vacuum chamber for the ITS testing. The specimens underwent the vacuum pressure treatment to get the targeted saturation level, which should fall within the 70 to 80 % range. Then the samples that have undergone vacuum saturation were stored in a freezer at -18 °C for at least 16 hours. Afterwards, the samples were taken from the freezer and placed in a water bath for 24 hours at 60 °C. after which they were transported to the water bath maintained at 25 °C and left there for 2 hours. The ITS test is performed on the conditioned samples mentioned above, and the maximum load is documented. The ITS of the samples is calculated by determining the maximum loads for both conditioned and unconditioned samples using:

$$TSR\,\% = \frac{2000 \times P}{\pi \times t \times D}, \quad (5)$$

where: *ITS* value for each specimen is in kPa, P is the peak load at failure, and t and D are thickness and diameter in mm, respectively.

$$TSR\,\% = \frac{Condition\ specimen}{Un - condition\ speimen} \times 100. \quad (6)$$

A total of 24 samples were employed in this study for both neat and nanomodified asphalt. The moisture susceptibility of a combination, as defined by AASHTO M 320-2002, was regarded as satisfactory if its TSR value was equal to or higher than 80 %.

5.3 IDT testing result

The results of the Resilient modulus test between the nanomodified HMA mixture and the neat asphalt mixes are compared in this section. Three duplicates were conducted at different temperatures. Table 8 displays the peak load, Poisson ratio, maximum horizontal deformation, and moduli value for both mixtures. Nanomodified asphalt enhances the moduli values, exhibiting a consistent upward trend. The addition of nanoparticles in asphalt, specifically NCC, results in a higher modulus value compared to the neat asphalt. The NCC has been found to increase the modulus Mr value at temperatures of 15, 25, and 40 °C by 11.2 %, 16.6 %, and 8.0 % respectively. At 5 °C, there is a slight effect of 4 %.

The NCC has a positive impact on these Mr value. This is because the nanoparticles have a large specific surface area, which enhances viscosity, adhesion, and strength, ultimately improving the modulus value at higher and intermediate temperatures. Figure 14 illustrates the disparity in Mr values between the neat and 5 % NCC modified asphalt at different temperatures.

The inclusion of 5 % NCC has had a significant effect on the moduli value, particularly at temperatures of 25 and 40 °C. This is because nanomaterials possess distinct features that increase viscosity and result in stiffer HMA mixes. These factors likely contribute to the observed improvement in Mr characteristics. To summarize, the aforementioned improvements led to increased Mr values, which are displayed in Table 8.

This suggests that adding 5 % nanomodified asphalt to the mixtures improved their ability to withstand stress and strain, while also reducing the non-linear behavior of the HMA mixtures. The decrease in non-linearity further reinforces the resilience of flexible pavements against permanent deformation.

The creep compliance quantifies the correlation

***Table 8** Mr value, load, Poisson, and horizontal deformation of neat and nanomodified*

Temp. °C	Item	Peak load, N	Deformation, (µm)	*Poisson*, V	Mr, MPa
5	Neat	2257	19.08	0.260	15223
	5% NCC	2143	18.87	0.259	15862
15	Neat	1875	27.89	0.297	9670
	5% NCC	2019	26.16	0.292	10889
25	Neat	1461	36.58	0.361	5362
	5% NCC	1548	34.58	0.348	6430
40	Neat	555	75.00	0.405	1199
	5% NCC	578	73.08	0.389	1303

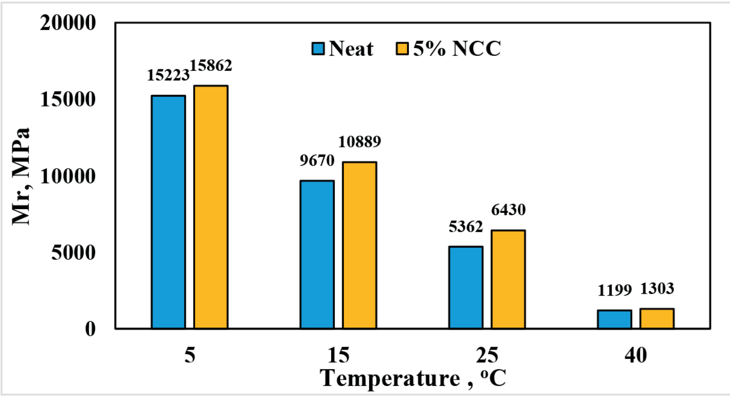

***Figure 14** Effect of NCC nanomodified asphalt on Mr at varying temperatures*

***Table 9** IDT Creep test parameter*

Type	Temp, (°C)	D_1 (1/GPa)	m-value	D(t), (1/GPa)
Neat	5	0.0823	0.475	0.587
	15	0.286	0.546	3.63
	25	0.448	0.625	7.96
	40	0.6304	0.704	16.57
5% NCC	5	0.079	0.465	0.551
	15	0.353	0.515	3.46
	25	0.436	0.617	7.76
	40	0.598	0.680	14.32

between the tension and the strain that occurs over time. Assessing the extent of damage that has occurred in HMA is of the utmost importance. The experimental research conducted in [23] suggests that the creep strain rate is primarily governed by the two crucial parameters: the m-value and the D_1 value. There is a clear difference between these two concepts. The m-value controls the pace at which the creep strain occurs in the long-term section of the creep compliance curve, whereas the D_1 value mainly affects the early portion. The m-value is more significant in terms of the rate at which irrecoverable strain accumulates. Conversely, a drop in the m-value indicates a decrease in the amount of accumulated damage. The utilization of the power law was employed to demonstrate the creep compliance fitting for both neat and NCC nanomodified asphalt. Enhancing the creep performance of HMA mixture is achieved through the utilization of nanomaterial to modify the asphalt binder.

The addition of nanoparticles to asphalt resulted in increased flexibility at lower testing temperatures of 5 and 15 °C, as well as altered creep values at intermediate and higher temperatures of 25 and 40 °C. These changes are anticipated to improve the resistance of HMA mixtures to rutting and thermal cracking. Furthermore, they provide a dependable validation of the results obtained in previous research studies carried out by [24-25]. Figure 15 illustrates a comparison of the data presented in Table 9. The creep parameters, specifically the m-value and D1, of nanomodified asphalt mixes are lower than those of mixtures containing the neat binder. The addition of NCC resulted in a modest drop in the D_1 and m-value compared to the plain mixture. This can be due to the reduced deformation of

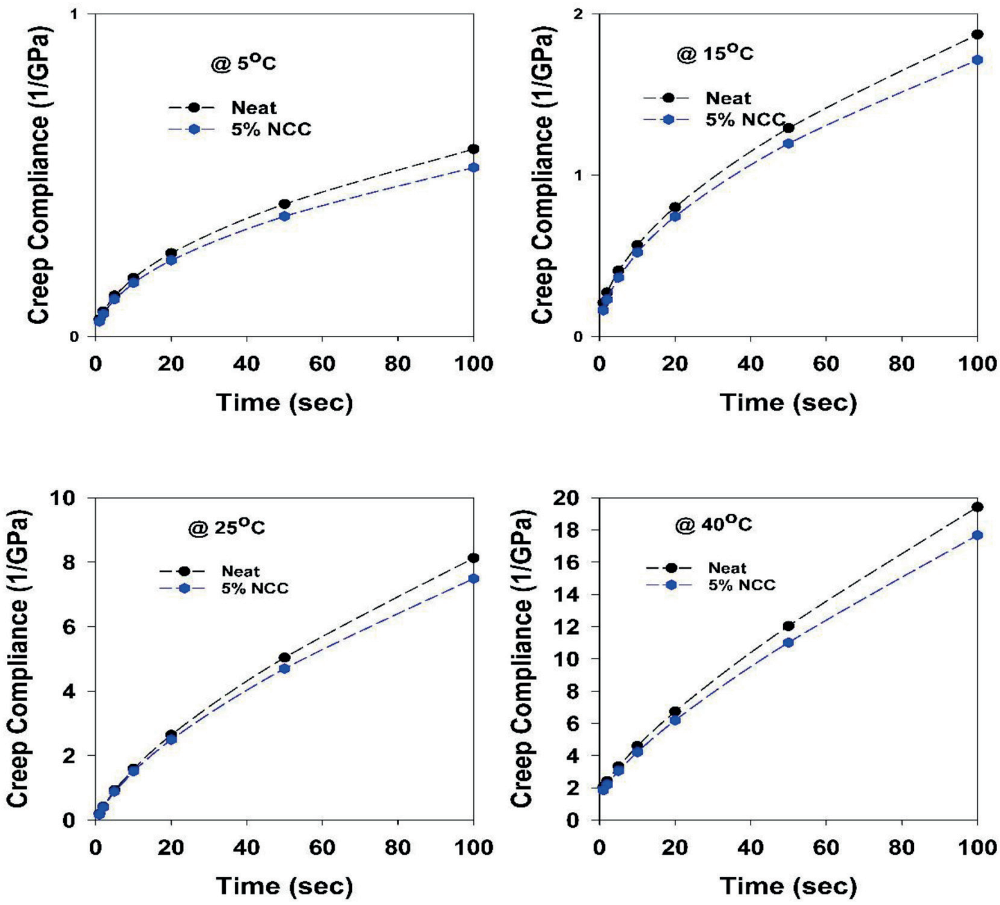

Figure 15 *Effect of NCC nanomodified asphalt on creep at varying temperatures*

the nano modified mixes, which is corroborated by the stiffening effect of NCC.

The tensile stress exhibits the anticipated trends, indicating that the strength value diminishes as the temperature increases. At lower temperatures of 5 and 15 °C, the ratings for all the mixtures show an increase in TS value for NS and NT modified mixtures. However, there appears to be little variation in NCC, which corresponds to neat asphalt. Conversely, the thermal susceptibility (TS) value of nanomodified mixes is greater than that of neat HMA mixtures at temperatures ranging from 25 to 40 °C. This suggests that the addition of 5 % nanomodified asphalt positively affects the strength of HMA at higher temperatures.

The NCC-modified HMA mixture had a greater TS value compared to the neat HMA mixture at lower temperatures of 5 and 15 °C. The behavior is expected to vary as a result of the temperature sensitivity of the NCC mixture. This sensitivity causes the viscosity of the asphalt binder to rise, which in turn enhances the tensile strengths of HMA by 12 % at 25 °C and 22 % at 40 °C. Nevertheless, FS exhibits a consistent upward tendency of no more than 9 % across all the temperatures for the plain mixture. Based on the previous investigation, the use of nanomodified asphalt binder has the potential to improve the tensile strength and failure strain as compared to neat asphalt.

This improvement is particularly significant at temperatures of 25 and 40 °C, with even greater enhancement observed at lower temperatures. As a result, this would lead to an increase in the fracture energy value, enabling HMA mixtures to absorb a greater amount of energy. The improved energy dissipation capacity of HMA mixtures will boost their resistance to fatigue and fracture failure, hence leading to a prolonged lifespan of asphalt pavements. Conversely, the changed mixture showed significant improvement at lower temperatures, while its influence was less pronounced at mid to higher temperatures compared to the neat asphalt. Figure 16 and Table 10 present a comparison of the TS and FS values for both neat and nanomodified asphalt.

5.4 Moisture damage

Pavements that are consistently exposed to water might lead to premature moisture damage. Moisture damage, indicated by the reduction in the bonding strength between the aggregate and asphalt binder when water is present, is a significant issue that affects asphalt pavements. Figure 17 demonstrates that the ITS

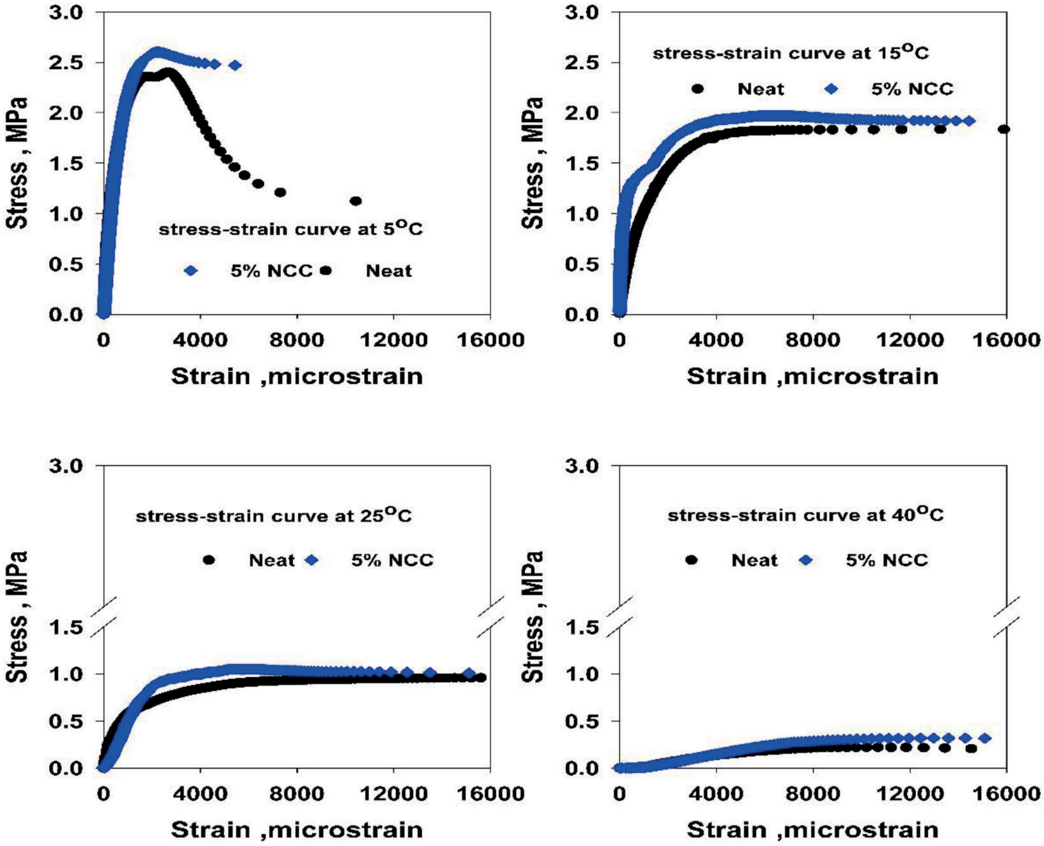

Figure 16 *Effect of NCC nanomodified asphalt on Tensile Strength at varying temperatures*

Table 10 *TS and FS value for neat and nanomodified asphalt at varying temperature*

Neat				
Temp, °C	5	15	25	40
TS, (MPa)	2.35	1.79	0.92	0.21
FS (10^3 micro)	1.66	4.51	7.06	10.78
5% NCC				
Temp, °C	5	15	25	40
TS, (MPa)	2.47	1.84	1.04	0.26
FS (10^3 micro)	1.84	4.92	7.54	11.16

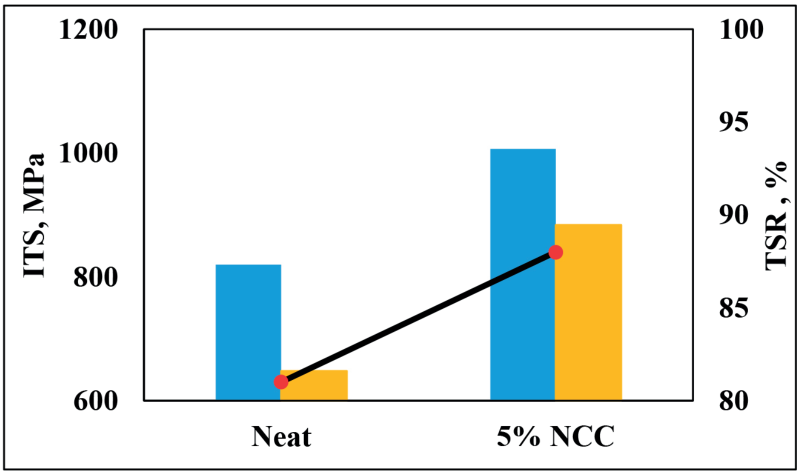

Figure 17 *TSR and ITS result for neat and NCC nanomodified asphalt*

and TSR values of HMA for condition and un-condition samples. The predictability of this result arises from the fact that water reduces the cohesive force between the aggregate and asphalt binder. As a result, conditioned samples show decreased resistance when subjected to loading. Therefore, all of the testing results met the parameters specified by AASHTO T283 for a minimum of 80 % TSR.

The influence of nanomaterial on the ITS and TSR of the HMA combination is more significant compared to neat asphalt. As anticipated, the NCC modified mixes exhibited improved ITS values compared to neat asphalt. The ITS values of conditioned and unconditioned samples have been modified by 5 % using the NCC. Unconditioned and conditioned mixtures led to a 19 % and 27 % enhancement in ITS values, respectively, when compared to asphalt. This phenomenon occurs as a result of the beneficial impact of NCC particles, which increases the stiffness of the asphalt. Consequently, the HMA mixture samples exhibit improved resistance to damage caused by moisture.

As a result, the TSR values experienced a substantial increase to 88 % as compared to the original asphalt. Moreover, this enhancement can be attributed to the fact that NCC modified asphalt has formed a strong connection with the aggregate due to the presence of extremely minimal dispersion forces. Due to the presence of a "polar surface," most aggregates have an electrical charge that largely attracts the lower active polarity of asphalt binder, which is composed mainly of high molecular weight hydrocarbons. Therefore, the NCC mitigates the moisture-induced harm in HMA by improving the bond between asphalt and aggregate and creating a robust nano network structure, as stated in reference [6].

It is important to mention that earlier research [3, 4, 23, 25] have reached a consensus on NCC contribution to moisture damage. Overall, the incorporation of nanomodified into asphalt has resulted in increased tensile stress resistance, greater adhesion within the asphalt matrix, enhanced resistance to water intrusions into HMA mixes and improved TSR value. The greater effect was demonstrated by NCC, which resulted in a TSR increase of up to 88 %, compared to neat asphalt with 81 %. Demonstrating the function of these nanoparticles as additives that prevent or reduce damage caused by moisture to some level, based on the specific surface area of these nanomaterials and the increased stiffness they provide to asphalt binder.

6 Conclusion

The conclusions drawn from this study are as follows:

1. The incorporation of 5 % NCC content had the most significant impact on the characteristics of the asphalt. This led to a decrease in its sensitivity to temperature due to the stiffening of the asphalt within the specified penetration grade. Additionally, it improved the resistance to permanent deformation, as indicated by higher values of softening point and penetration index. Furthermore, it enhanced the ability of the asphalt to withstand low-temperature cracking by reducing its ductility.
2. Asphalt viscosity increases by 43 %, hence improving the performance of the asphalt binders at high temperatures.
3. The DSR test findings showed that the rutting factor value in the RTFO test was observed while using 5 % NCC as an additive. This resulted in a better $G^*/\sin\delta$ value of 3.49 kPa compared to neat asphalt, which maintained a value of 1.68 kPa.
4. The BBR testing result indicated that the NCC had minor effect at low temperatures.
5. Nanomodified asphalt mixture exhibits a substantial increase in moduli value compared to the neat mixture, with enhancements of approximately 4 %, 11.2 %, 16.6 %, and 8.0 % for NCC at temperatures ranging from 5 to 40 °C, furthermore affected the creep, tensile stain and strength at moderate to high temperatures.
6. Nanomodified asphalt mixtures with 5 % NCC present the higher TSR value up to 88 % leading to prevent and reduce damage caused by moisture.

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Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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