Linking the Fatigue Resistance of Nano-Modified Binders to Mixture Cracking

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ABSTRACT

This study examined the correlation between binder-level fatigue properties and mixture-level cracking resistance in asphalt binders modified with five Nanomaterials (NMs): Nano-Silica (NS), Nano-Alumina (NA), and Nano-Titanium dioxide (NT) at 2%, 4%, and 6% as well as Nano-Zinc oxide (NZ) and Carbon Nanotubes (CNTs) at 1%, 2%, and 3%. Modified binders were subjected to Rolling Thin-Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) aging and tested at 25 °C using the Linear Amplitude Sweep (LAS) test to determine fatigue life (N_f) and the fatigue parameter *G*.sin δ*. The corresponding asphalt mixtures were evaluated using the IDEAL-CT test. The results indicated strong correlations between binder and mixture performance for NT, NZ, and NA, with NS exhibiting high correlation at lower strain levels, while CNT-modified binders showed weak relationships due to dispersion issues. Overall Desirability (OD) analysis identified 6% NT, 4% NA, 2% NS, 1% CNT, and 1% NZ as the optimal formulations. These findings offered practical guidance for selecting suitable NM types and dosages to improve pavement durability and optimize fatigue performance in asphalt mix design.

Keywords-asphalt; fatigue; linear amplitude sweep test; ideal-CT; nanomaterials

I. INTRODUCTION

Asphalt mixture, a viscoelastic composite of asphalt binder, mineral fillers, and aggregates, is widely utilized in multilayer pavement systems, relying on proper interlayer bonding for effective load transfer [1]. However, early failures such as fatigue cracking, rutting, and thermal cracking remain prevalent, largely due to design, material, and construction deficiencies [2, 3]. Specifically, the rapid societal changes in Iraq have increased the need for vehicle usage, imposing heavier loads on pavements. Limited transport alternatives as well as high maintenance costs highlight the necessity for more efficient road construction [4-5]. The performance of asphalt pavement depends on durability and sustainability, which are influenced by binder cohesion, binder–aggregate adhesion, and environmental factors such as moisture, air, and temperature [6].

The incorporation of additives such as polymers, fibers, and Nanomaterials (NMs) in asphalt mixture has been used to address these challenges by improving resistance to moisture, fatigue, cracking, deformation, and aging [7-10]. Nanotechnology, with its broad applicability, modifies materials at the nanoscale, altering their surface structure and physicochemical behavior [11-12]. Nanoparticles, typically under 100 nm in size, exhibit high surface-area-to-volume ratios, leading to increased surface energy, reactivity, and bonding potential with asphalt binders. These unique properties make NMs promising modifiers for improving asphalt durability and overall performance [13].

Several studies have been conducted examining the effects of NMs in asphalt mixtures. Authors in [14], revealed that blending Nano-Silica (NS) into asphalt mixture improved stiffness, Marshall stability, and fatigue life, with optimal performance at 4%. Similarly, in [15] a concentration of 6% NS reduced moisture resistance. In [16], a uniform dispersion and enhancements in Performance Grading (PG) and rutting resistance were observed without compromising fatigue properties. Additionally, researchers in [17] examined the impact of Nano-Alumina (NA) at concentrations of 3-12%. The results showed enhanced high-temperature and fatigue performance, with 9% being optimal. In [18], the incorporation of 2-6% nano additives improved binder–aggregate adhesion and surface free energy, while time sweep tests in [19] confirmed increased fatigue life despite *G*sin δ* suggesting the opposite. Furthermore, Nano-Titanium (NT) at 3-7% improved fatigue life, creep, and adhesive bonding, with 5% being optimal [20]. In [21], 3% nano-TiO_2 delayed aging and improved high-temperature rheology, as well as in [22] it enhanced cohesive-adhesive cracking resistance. Nano zinc oxide (ZnO) improved fatigue life via adhesion and cohesion energy [23], while Carbon Nanotubes (CNTs) (0.1-2.25%) increased viscosity, strength, and fatigue performance while enhancing fracture energy and durability under intermediate conditions [24-25].

Despite the effectiveness of NMs in asphalt mixtures, continuous advancements are critical for optimizing their performance. This study investigated the relationship between the fatigue properties of asphalt binders modified with five NMs (SiO_2, Al_2O_3, nano-TiO2, nano-ZnO, and CNTs) and the cracking resistance of their corresponding asphalt mixtures. The evaluation was conducted through rheological characterization using the Linear Amplitude Sweep (LAS) test and mechanical performance assessment through the IDEAL-CT test, with all experimented performed at 25 °C.

II. MATERIALS AND METHODS

A. Materials

This study utilized a 40-50 penetration grade asphalt binder, obtained from Al-Doura Refinery in Baghdad, Iraq. Its physical and rheological properties are summarized in Table I. Aggregates were graded as Type D5 following ASTM D3515 [26]. Gradation details and limits are depicted in Figure 1.

TABLE I. BASE BINDER PROPERTIES AND SPECIFICATION LIMITS

Property	Result	Standards	Limit
Penetration (0.1 mm)	44	AASHTO T49 [27]	40-50
Ductility (cm)	+100	AASHTO T51 [28]	>100
Softening point (°C)	48.7	AASHTO T53 [29]	-
Rotational Viscosity (Pa·s)	0.745	AASHTO M320 [30]	3 (max)
G/sinδ* (kPa) (at 70 °C)	1.45		1 (min)
Mass loss (%)	0.254		1% (max)
G/sinδ* (kPa) (at 25 °C)	5019		5000 (max)

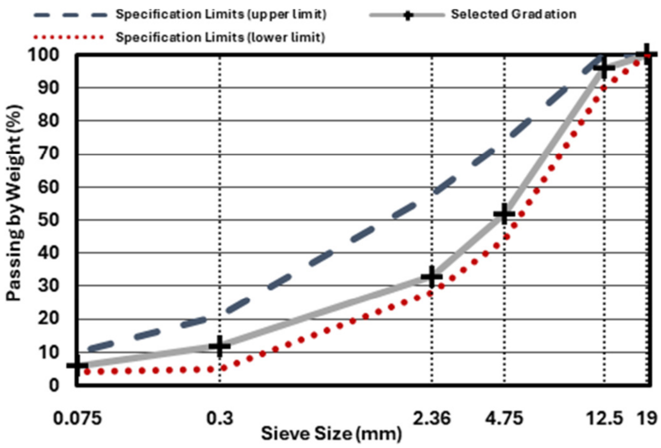

Fig. 1. Aggregate gradation curve and specification limit.

The incorporation of NMs into asphalt mixtures was based on a previous study which used high-shear mixing at 140 °C and 4000 rpm for 20 minutes, adding 2-8% NS, NT and NA at 4 g/min [31]. In the current research, five NMs were utilized: NS, NA, and NT oxide at concentrations of 2%, 4%, and 6%, as well as NZ oxide and CNT at 1%, 2%, and 3% by weight of asphalt binder. Figures 2 and 3 illustrate the powder morphology and SEM microstructure (at 15,000× magnification) of these NMs, respectively.

The NMs were added to 500 grams of asphalt binder and mixed using a high shear mixer initially at 400 rpm for 4 minutes at 140 °C, followed by a final mixing stage at 3,000 rpm for 20 minutes at 140-150 °C to ensure uniform dispersion.

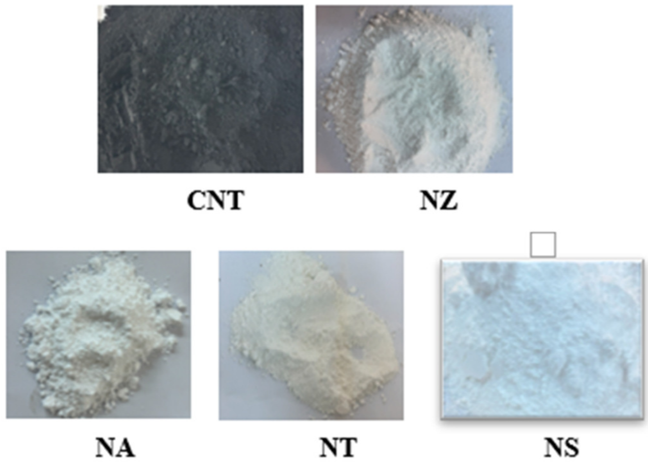

Fig. 2. The powder morphology of CNTs, NZ, NA, NT, and NS.

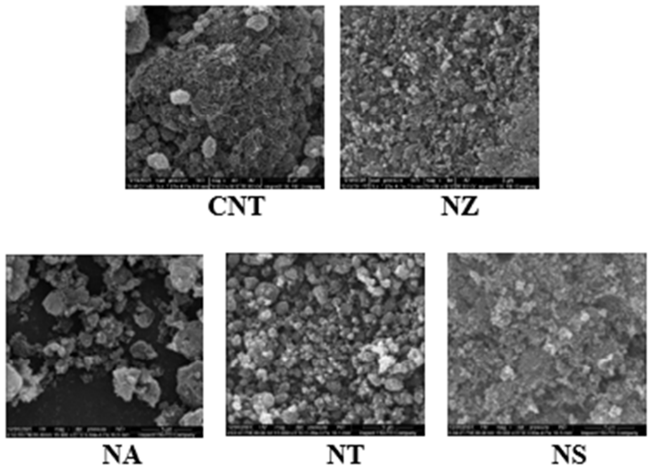

Fig. 3. SEM Images of NMs at 15,000× magnification.

B. Testing Method

A series of standardized tests were conducted to evaluate the rheological and fatigue performance of unmodified and NM-modified asphalt binders and mixtures. Specifically, rotational viscosity was measured at 135 °C following AASHTO T316 [32], as shown in Figure4, to assess binder workability. To simulate field aging, binders were subjected to Rolling Thin-Film Oven Test (RTFOT) (AASHTO T240) [33] and Pressure Aging Vessel (PAV) (AASHTO R28) [34].

Except these, the rheological fatigue parameter $G^* .sin(\delta)$ was evaluated at 25 °C using a performance threshold of 5000 kPa, in line with fatigue cracking criteria. The Linear Amplitude Sweep (LAS) test was performed according to

AASHTO T391 [35], incorporating both frequency and amplitude sweep phases on aged binders with 8 mm plates and a 2 mm gap, per AASHTO T315 [36]. Fatigue life was predicted using the Viscoelastic Continuum Damage (VECD) analysis based on test outputs.

For asphalt mixtures, the IDEAL-CT test was accomplished at 25 °C per ASTM D8225 [37] to assess cracking resistance. Cylindrical specimens (101.6 mm in diameter and 63 mm in height) were compacted using the Marshall method to achieve 4 ± 0.5% air voids. IDEAL-CT testing was performed three times, with mean values used for analysis. The optimum binder content (5.0%) was determined using the Marshall method per MS-2 guidelines for heavy traffic (> 10^6 ESALs). Finally, the CT Index was calculated based on fracture energy (G_f) and the post-peak slope at 75% load level, following the standard calculation method.

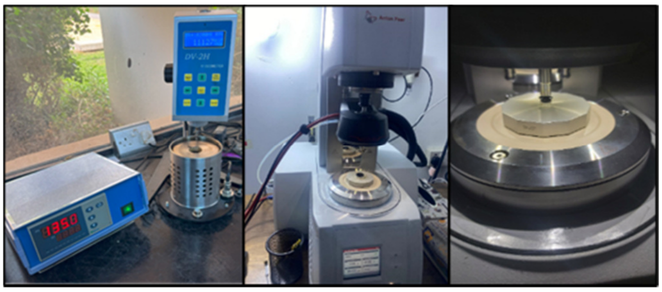

Fig. 4. Rotational viscometer and LAS test using Smart Pave 102e DSR.

III. RESULTS AND DISCUSSION

A. Correlation between IDEAL-CT Index and G.sin δ*

The fatigue parameter *G*.sin(δ)*, derived from Dynamic Shear Rheometer (DSR) testing, is commonly used to evaluate binder-level fatigue resistance. Lower values correspond to improved fatigue performance, indicating reduced stiffness and greater capacity to dissipate strain energy under repeated loading Figure 5 presents the correlation between *G*·sin(δ)* and CT Index values for all nano-modified binders at concentrations of 2%, 4%, and 6% for NS, NA, and NT and 1%, 2%, and 3% for NZ and CNT.

The NT-modified binders demonstrated a unique deviation from the expected inverse relationship. While their *G*.sin(δ)* values were not notably low, the CT index values, especially for 6% NT, were significantly high. This behavior may be attributed to a combination of factors, including superior nanoparticle dispersion, improved binder-aggregate adhesion, and NT's ability to enhance stress distribution and crack resistance at the mixture level. Although these factors not fully captured by binder-level rheological parameters alone, his complex behavior highlighted the limitations of relying solely on *G*·sin(δ)* to predict mixture-level fatigue performance, especially in nano-engineered systems. CNT and NS modified binders revealed weak or inverse correlations, suggesting that increased *G*.sin(δ)* values matched with reduced cracking tolerance. These observations supported the theoretical assumption that excessive binder stiffness may adversely affect fatigue performance at the mixture scale. Additionally, NZ and NA displayed intermediate behavior, with moderate nonlinear or negative trends, highlighting the complexity of the relationship between rheological and mechanical properties. These inconsistencies were likely due to differences in nanoparticle dispersion, compatibility with the asphalt matrix, and the influence of NM morphology on mixture behavior.

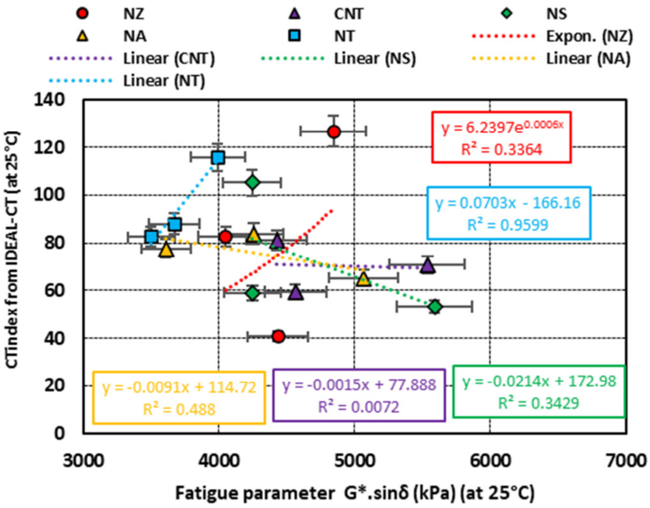

Fig. 5. CT index and *G*.sin(δ)* Correlation for Nano-Asphalt Binders.

B. Correlation between IDEAL-CT Index and LAS

The relationship between fatigue life (N_f at 2.5% strain) and cracking resistance (CT index) of all asphalt mixtures is presented in Figure 6.

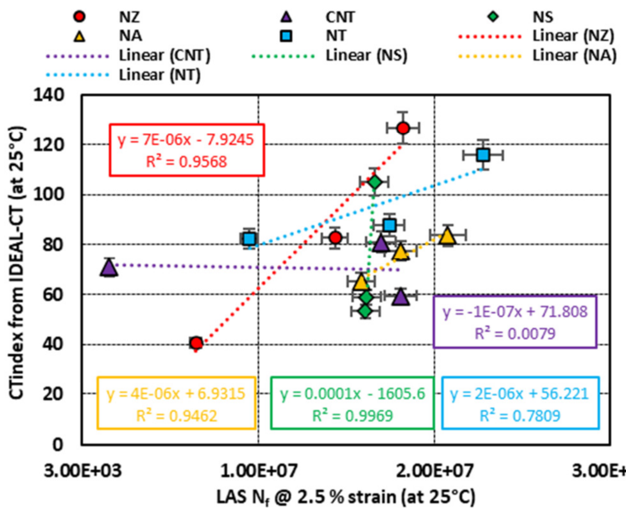

Fig. 6. CT index and N_f (2.5% strain) correlation for nano-asphalt binders.

A strong positive linear correlation was observed for NZ, NT, NA, and NS with R^2 values of 0.96, 0.78, 0.95, and 0.99, respectively. The highest CT index (126.7) and N_f ($1.82{\times}10^7$), were achieved at 1% NZ, highlighting enhanced mixture and binder fatigue behavior. Similarly, 6% NT and 6% NA demonstrated simultaneous increases in both CT index and N_f, indicating effective structural reinforcement. NS binders exhibited the strongest linearity, with CT index values rising proportionally with N_f, emphasizing silica's uniform effect on both fatigue and cracking tolerance. In contrast, the CNT group presented negligible correlation (R^2 = 0.0079), where N_f and CT index varied independently, probably due to poor dispersion or inconsistent interaction with the asphalt matrix. These findings suggested that, except for CNT, the fatigue behavior at the binder level aligned well with mixture performance, supporting the value of integrated fatigue evaluation in nano-modified asphalt binders.

Additionally, the correlation between N_f at 5% strain and CT index was evaluated across various nano-modified asphalt binders (Figure 7). Most materials exhibited a positive relationship between N_f and CT index, though with varying degrees of strength. Specifically, NZ displayed a strong linear correlation (R^2 = 0.8936), with CT index rising from 40.7 to 126.7 while N_f increased from 263,491 (3% NZ) to 701,396 (1% NZ), indicating that binder-level fatigue resistance was well reflected in mixture-level performance. NT also showed a substantial trend (R^2 = 0.7258), as 6% NT achieved the highest CT index (115.8) and N_f (907,908), suggesting consistent mechanical synergy. NA revealed a high correlation as well (R^2 = 0.9605), where CT index and N_f increased concurrently, particularly in 6% NA (CT index = 83.9, N_f = 835,059).

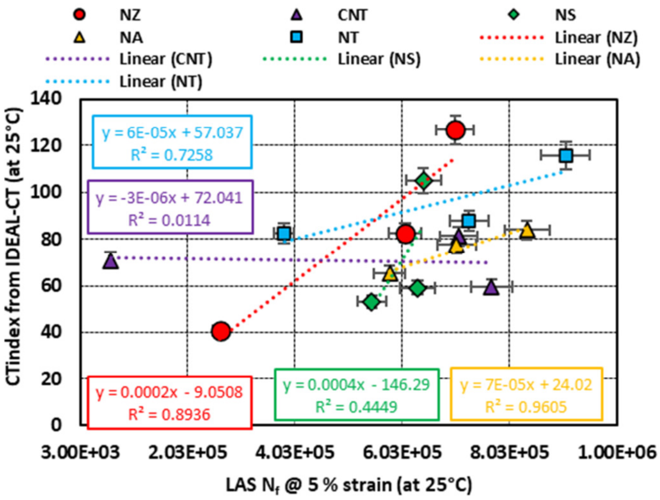

Fig. 7. CT index and N_f (5% strain) correlation for nano-asphalt binders.

On the other hand, NS modified binders exposed a strong correlation between N_f and CT Index at 2.5% strain (R^2 = 0.7809), indicating that binder fatigue behavior governed mixture cracking resistance under moderate strain conditions. However, this correlation weakened significantly at 5% strain (R^2 = 0.4449), likely due to damage localization, strain-sensitive fracture mechanisms, and potential nanoparticle agglomeration at higher dosages, further reducing dispersion quality and stress transfer efficiency. These factors diminished the predictive reliability of binder-level fatigue life on mixture-level performance under elevated deformation and complex loading states. CNT binders demonstrated virtually no correlation (R^2 = 0.0114), as N_f varied widely with little corresponding change in CT index, revealing potential issues with dispersion or interaction uniformity in the asphalt matrix.

These results confirmed that while LAS-derived N_f values could serve as a useful indicator of mixture cracking performance, the strength of this relationship was highly dependent on the NM type and its integration within the binder system.

Numerous studies have confirmed the positive impact of NMs on asphalt fatigue performance. NA significantly improved fatigue life in binders [38]. NS enhanced fatigue behavior due to inorganic network formation, while NT showed reduced performance when increased from 2% to 4% due to competing mechanisms [39]. In [40], CNT improved performance at 0.4%, 0.75%, and 2.25%, with better load transfer despite slight stiffness loss at higher contents. Additionally, NZ was effective at 1-4%, peaking at 2%, but declined at 5% due to agglomeration [41].

Figure 8 presents the SEM images of all asphalt binders used in this study, both neat and nano-modified, at 1,000× magnification. A clear morphological difference was observed between both types of binders. Nanoparticle addition generally altered surface texture, with increased roughness and particle dispersion evident at higher dosages.

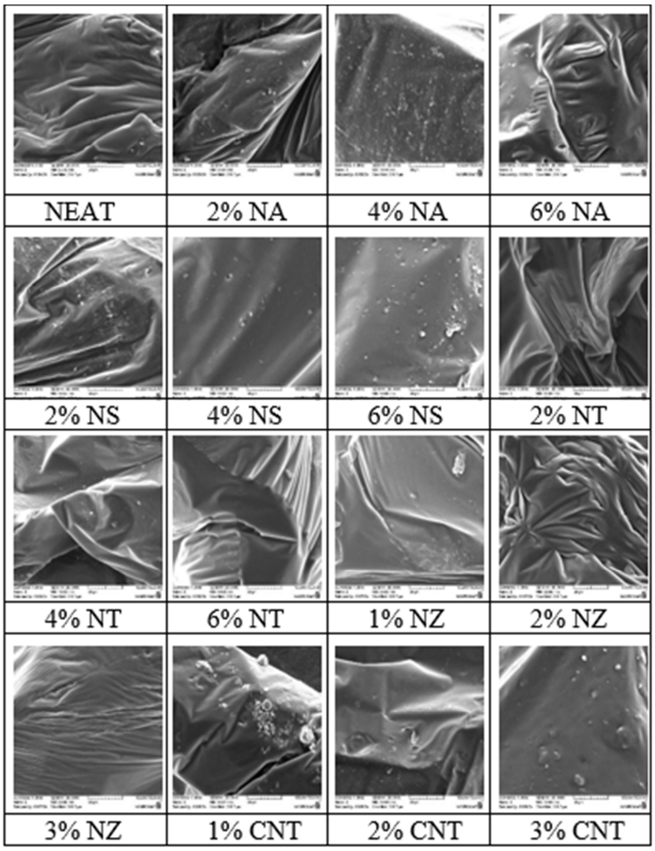

Fig. 8. SEM images of modified binders at 1,000 × magnification.

C. Overall Desirability Analysis

Overall Desirability (OD) is a multi-criteria decision-making tool used to identify the optimal NM dosage for asphalt binder modification [42]. The OD was calculated based on four key performance indicators: Rotational Viscosity (RV), fatigue parameter ($G^*.sin\,\delta$), fatigue life at 2.5% strain (N_f 2.5%), and CT index. Each parameter was normalized on a 0-1 scale, where 1 indicated the most desirable outcome. The normalization was conducted using either the "larger-the-better" (1) or "smaller-the-better" approach (2), depending on the performance criterion. Specifically, N_f 2.5% and CT index were treated as "larger-the-better," while RV and $G^*.sin\,\delta$ followed the "smaller-the-better" rule. The final OD value (3) was computed as the geometric mean of all normalized parameters to determine the most effective binder formulation.

$$x_i^*(k) = \frac{x_i(k) - \min x_i}{\max\, x_i - \min x_i} \quad (1)$$

$$x_i^*(k) = \frac{max\ x_i - x_i(k)}{max\ x_i - min\, x_i} \quad (2)$$

$$y_{OD}(k) = [x^*1(k)x^*1\,(k) \dots x^*m(k)]^{1/m} \quad (3)$$

where i refers to the index of the evaluation criterion (i = 1, 2, ..., m), and k denotes the sample or data point index (k = 1, 2, ..., n). The original (non-normalized) value of the i-th criterion for the k-th sample was represented by $x_i(k)$, while $x_i^*(k)$ is the corresponding normalized value. The maximum and minimum values of the i-th criterion across all samples are specified as $max(x_i)$ and $min(x_i)$, respectively. (Table II).

TABLE II. BINDER PROPERTIES, NORMALIZED VALUES, AND OD SCORES

Binder	RV (mPa·s)		$G^*.sin\,\delta$ (kPa)		N_f 2.5%		CT index		OD
	$x_1^{(0)}$	x_1^*	$x_2^{(0)}$	x_2^*	$x_3^{(0)}$	x_3^*	$x_4^{(0)}$	x_4^*	
NEAT	745	0.941	5,019	0.272	9,641,519	0.381	35	0.000	0.000
2% NS	1,129	0.392	4,243	0.643	16,601,257	0.709	105.1	0.764	0.608
4% NS	1,183	0.315	4,245	0.642	16,172,962	0.689	59	0.262	0.437
6% NS	1,368	0.050	5,589	0.000	16,087,517	0.685	53.3	0.200	0.000
2% NA	1,112	0.416	5,067	0.250	15,872,060	0.675	65.2	0.329	0.390
4% NA	1,214	0.270	3,606	0.948	18,127,718	0.781	77.6	0.465	0.552
6% NA	1,245	0.226	4,256	0.637	20,791,911	0.906	83.9	0.533	0.514
2% NT	707	0.996	3,669	0.918	17,469,764	0.750	87.9	0.577	0.793
4% NT	704	1.000	3,497	1.000	9,498,965	0.375	82.5	0.518	0.664
e	705	0.999	3,988	0.765	22,780,068	1.000	115.8	0.881	0.906
1% NZ	840	0.805	4,845	0.356	18,226,057	0.786	126.7	1.000	0.689
2% NZ	852	0.788	4,050	0.736	14,365,220	0.604	82.7	0.520	0.653
3% NZ	1,150	0.362	4,435	0.552	6,459,667	0.231	40.7	0.062	0.232
1% CNT	1,216	0.268	4,424	0.557	17,006,368	0.728	81.2	0.504	0.484
2% CNT	1,257	0.209	4,563	0.490	18,114,785	0.780	59.5	0.267	0.382
3% CNT	1,403	0.000	5,538	0.024	1,544,237	0.000	70.9	0.391	0.000

The highest OD values were observed at 6% NT (0.906), 4% NT (0.664), and 2% NT (0.793), indicating that NT significantly enhanced the balance between stiffness, fatigue resistance, and crack tolerance. Similarly, binders incorporating NZ (1% NZ and 2% NZ) achieved high OD scores (0.689 and 0.653, respectively), reflecting effective improvement in both rheological and mechanical behavior. Conversely, the neat binder, 6% NS, and 3% CNT recorded OD values of 0.000, emphasizing important deficiencies in overall performance. This may be attributed to the presence of extreme values in one or more parameters, such as excessive $G^*.sin\,\delta$, which negatively influence the composite OD score. Moderate OD values were reported for 4% NA, 2% NS, and 1% CNT, suggesting that the effectiveness of NA, NS, and CNT depends on dosage level and interaction quality within the asphalt matrix.

Overall, these findings underscored the value of OD analysis as a multi-criteria decision-making tool for assessing the integrated performance of nano-modified asphalt binders.

IV. CONCLUSIONS

This study investigated the relationship between fatigue properties of asphalt binders modified with five Nanomaterials (NMs) and the cracking resistance of their mixtures using Linear Amplitude Sweep (LAS) and IDEAL-CT tests, both conducted at 25 °C. From the analysis, the following conclusions were established:

- Nano-Titanium (NT): Exhibited the highest OD score (0.906) and strong correlation between LAS and CT results, indicating superior enhancement in fatigue life and cracking resistance, particularly at 6% dosage.
- Nano-Zinc (NZ): Demonstrated strong binder–mixture correlation and high CT index at 1% dosage, supporting its effectiveness in improving elasticity and cohesive behavior.
- Nano-Alumina (NA): Showed excellent alignment between fatigue life and CT index (R^2 = 0.96) and consistent performance at 6%, confirming its role in enhancing mechanical synergy.
- Nano-Silica (NS): Achieved strong CT-N_f correlation at 2.5% strain (R^2 = 0.99) but weaker performance at higher strains, indicating dosage- and strain-dependent efficiency.
- Carbon Nanotubes (CNTs): Presented minimal correlation between binder and mixture behavior, with OD values approaching zero, likely due to poor dispersion and interaction with the asphalt matrix.
- Based on the OD values, the optimal dosages for enhancing fatigue resistance were 6% NT, 4% NA, 2% NS, 1% CNT, and 1% NZ.

The significance of this research lied in its comprehensive evaluation approach, combining binder-level rheological testing with mixture-level mechanical assessments. Nano-

Titanium at a 6% dosage emerged as the optimal NM with the best fatigue and cracking resistance.

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