The Effect of Nano Materials on the Rheological Properties of Asphalt Binder

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ABSTRACT

This study investigates the effects of nano-silica, nano-chitosan, and nano-clay as asphalt binder modifiers, focusing on their physical and rheological properties. The nanomaterials were incorporated at concentrations of 2.5%, 5.0%, 7.5%, and 10% by weight. This research was driven by the growing adoption of nanomaterials, many of which are known for their toxicity and environmental impact. The results demonstrated a significant improvement in both physical and rheological properties, particularly in rutting resistance. Nano-silica demonstrated the greatest enhancement, leading to a reduction in permeability values, an increase in softening point, viscosity, and overall resistance to permanent deformation. Nano-chitosan exhibited a similar trend but with slightly lower performance values than nano-silica. Nano-clay provided the least improvement, though it still contributed to increased asphalt binder stiffness and reduced temperature sensitivity. Overall, the findings confirm that nano-silica, nano-chitosan, and nano-clay can improve asphalt binder properties, extending pavement lifespan and performance under increasing traffic demands. Further studies are recommended to explore the long-term aging resistance, fatigue behavior, and field applications of these nanomaterial-modified asphalt binders.

Keywords-nano materials; chitosan; nano silica; nano clay; physical properties; rheological properties

I. INTRODUCTION

Asphalt, a hydrocarbon-based material, is the primary component of hot asphalt mixtures. Its properties have been a key focus of research due to their significant impact on pavement performance and durability. Factors such as climate conditions and the increasing weight of traffic load influence the longevity and effectiveness of asphalt pavements. One of asphalt's most valuable characteristics is its ability to resist permanent deformation at high temperatures and low frequencies due to its stiffness, while also demonstrating fatigue resistance and elasticity at lower temperatures and higher frequencies [1].

Traditionally, asphalt roads have been classified based on consistency, determined through experimental methods such as penetration and viscosity tests. However, with rising traffic volumes and unpredictable weather conditions, there has been growing interest in enhancing asphalt performance and refining experimental techniques for more precise evaluations. In response, the Strategic Highways Research Program (SHRP) was developed in 1993 [2]. SHRP facilitated asphalt modification using polymeric materials and introduced advanced testing methodologies. This initiative paved the way for incorporating new additives, including nanomaterials and polymeric nanocomposites, to improve the performance of asphalt pavements [3]. Research on asphalt concrete modified with nanomaterials has already led to practical applications in the paving industry.

Despite concerns about the potential health risks associated with nanomaterials, their benefits in asphalt modification have encouraged widespread adoption. Since the materials used in asphalt mixtures play a crucial role in pavement efficiency, researchers continue to explore emerging technologies to enhance asphalt performance. This study aims to evaluate the effects of different nanomaterials, including nano-silica, nano-chitosan, and nano-clay, on the physical and rheological properties of asphalt binders.

II. LITERATURE REVIEW

The addition of nanoparticles as modification additives is known to reduce the phase angle and increase the complex shear modulus, significantly enhancing the viscoelastic properties of asphalt binders. Among various nanoparticles, nanoclay is a preferred choice for asphalt modification due to its availability and cost-effectiveness.

Several research has demonstrated that incorporating nanoclay into asphalt binders improves both flexibility and stiffness compared to unmodified binders [4-6]. In [1], authors found that adding 4% nanoclay increased their shear modulus by 125%, while a 2% nanoclay-modified binder exhibited a 66% increase. Additionally, another form of nanoclay improve the shear modulus by 196% at a 4% concentration, whereas a 2% concentration elevated the modulus by 184%. In asphalt binder specifications, the rutting factor ($G^*/sin\delta$) serves as a key indicator of resistance to deformation under high temperatures. A higher $G^*/sin\delta$ value indicates better rutting resistance. Research in [7] demonstrated that incorporating Cellulose Nanofibers (CNF) into asphalt binders resulted in a 47% increase in $G^*/sin\delta$, significantly improving rutting resistance. Another study [8] suggested that appropriate amounts of nanoclay and nanosilica could further enhance the rutting factor. To evaluate the rutting resistance of nano-modified asphalt, researchers in [9] utilized the dissipated energy indicator. Their findings revealed that adding 4% Non-Modified Nanoclay (NMN) increased dissipated energy by an average of 50%, while a 2% NMN-modified binder showed a 12% improvement. The results indicated that energy loss per loading cycle increased in NMN-modified asphalt, making it less susceptible to rutting during the Rolling Thin Film Oven (RTFO) aging process. Additionally, researchers in [10, 11] demonstrated that nanomaterials such as nano-SiO_2, nanoclays, and nanofibers contributed to improved aging resistance in asphalt binders. The study in [12] investigated the effect of Layered Double Hydroxides (LDHs) and found that oxidation and water exposure were the primary causes of asphalt binder degradation. LDH-modified asphalt binders exhibited greater stability at high temperatures and better resistance to thermal aging in the absence of oxygen compared to unmodified binders. In [13], researchers employed the aging index to assess oxidative aging sensitivity, revealing that the inclusion of carbon nanotubes (CNTs) enhanced asphalt's aging resistance. Another study [14] examined the rheological properties of Montmorillonite (MMT)/SBS-modified asphalt after exposure to ultraviolet (UV) aging. Furthermore, studies in [15-17] investigated the structural impact of nanomaterials on asphalt mixtures and their performance over the service period.

III. MATERIALS USED

This study utilized three types of nanomaterials at concentrations ranging from 0% to 10%, increasing in 2.5% intervals, to investigate their influence on the physical and rheological properties of the asphalt binder. The selected nanomaterials are as follows:

A. Nano-Silica

Nano-silica enhances asphalt performance material due to its chemical stability, resistance to acids and alkalis, well-developed pore structure, high surface activity, low oil absorption heat resistance, strong mechanical properties, excellent electrical insulation, and UV resistance. Table I presents the physical characteristics of the nano-silica used in this study.

TABLE I. PHYSICAL PROPERTIES OF NANO-SILICA

Physical characteristics	**Results**
Purity	99.8%
Odor	Odorless
The point of melting	1610-1728 ˚C
Boiling Point Range	2230
Density, 20 °C	2.17-2.66
Water	Insoluble
Appearance	White powder

B. Nano-chitosan

Nano-chitosan is a structurally stable polysaccharide polymer widely used in various fields, such as medicine. It serves as a suitable material for producing nanoparticles, nanocoating, and nanocarriers. Chitosan is derived from chitin, a biopolymer found in fungi, crustaceans (such as shrimp and crabs), and insects, making it the second most abundant biopolymer after cellulose. Table II presents the test results for the nano-chitosan used in this study.

TABLE II. PHYSICAL PROPERTIES OF NANO-CHITOSAN

Test	**Specification limits**	**Test Results**
D.A.C, %	≤ 95	95.7
Viscosity	≤ 100	25
Insoluble (%)	≤ 1	0.10
Ash (%)	≤ 1	0.10
Moisture (%)	≤ 1	9.45
Particles size	≤ 80nm	Pass
Heavy metal (ppm)	≤ 10	<1.0
Arsenic (ppm)	≤ 0.5	0.02
Density(g/ml)	0.28	0.03

C. Asphalt Binder

The asphalt binder used in this study was sourced from the Doura refinery with a penetration grade of 40-50. Physical and rheological tests were conducted following ASTM standards, as shown in Table III.

TABLE III. PHYSICAL AND RHEOLOGICAL PROPERTIES OF 40-50 ASPHALT BINDER

Test	**Unaged binder**	**Aged binder (10 rad/sec, 1.59Hz)**	**Standard Method**
Penetration (0.1mm), 100g, 5s, 25 °C	42	31.6	ASTM D5
Softening Point (Ring and Ball), °C	49	52.5	ASTM D36
Flash Point, °C	315	-	AASHTO T-48
Rotational Viscometer, Viscosity (Pa·s), 135°C	0.602	-	ASTM D-4402
Rotational Viscometer, Viscosity (Pa·s), 165°C	0.198	-	ASTM D-4402
Mass Loss, RTFO, %	-	0.3 %	AASHTO TP-5

D. Nano-clay

The nano-clay used in this study is an organic Montmorillonite product sourced from the United States (product code: 682624). Four different percentages of nano-clay were tested. Table IV provides the physical characteristics of the nano-clay.

TABLE IV. PHYSICAL PROPERTIES OF NANO-CLAY

Physical characteristic	Result
Organic Montmorillonite Treatment	Quaternary ammonium compounds, bis (hydrogenated tallow alkyl) dimethyl, chlorides.
Cation Exchange Capacity (C.E.C.)	(35 – 45)% wt. dimethyl dialkyl (C14-C18) amine matrix
Appearance	White powder
Mositure Content (%)	3.0

IV. THE PHYSICAL CHARACTERISTICS OF ASPHALT BINDER MODIFIED BY NANOMATERIALS

This study evaluates the relative performance of asphalt binders modified with nanomaterials by analyzing their conventional rheological and viscoelastic properties. A fundamental rheological bond test using Dynamic Shear Rheometer (DSR) was conducted, and pavement performance was predicted using the Superpave rutting parameter ($G^*/\sin \delta$). To simulate the aging process of both pure and nanomaterial-modified asphalt binders, the standard RTFO test procedure (AASHTO T-240) was applied. The test conditions included an aging duration of approximately 85 minutes at a temperature of 163 °C with an airflow rate of 4000 m³/min.

A. *Physical Characteristics of Nano-Modified Asphalt Binders*

The flow characteristics of asphalt binders were analyzed to assess how different types and concentrations of nanomaterials influence binder performance and mixture behavior. The study examined how variations in nanomaterial type and modification ratios impact the rheological and mechanical properties of asphalt binders. The following sections discuss the effects of different nanomaterial additives on the modified asphalt binder properties.

1) *Nano-Silica*

The physical properties of the modified binder were evaluated using a series of tests conducted both before and after short-term aging. These tests included the penetration test at 25 °C (ASTM D5), the softening point test (ASTM D36), and the Brookfield viscometer test (ASTM D4402). The penetration test assesses bitumen consistency or hardness, while the softening point test determines the temperature at which bitumen transitions from a solid to a more fluid state, indicating its viscosity. Unlike crystalline materials, bituminous substances do not have a distinct melting point; instead, they exhibit a gradual and continuous change in consistency as the temperature rises. Figure 1 illustrates the variation in penetration and softening point values between the base binder and the nano-silica-modified binder, both before and after RTFO aging. At room temperature, the hardness and toughness of the nano-silica-modified asphalt binder improve, as evidenced by a decrease in penetration values with increasing nano-silica content. Similarly, the softening point of the binder rises as the nano-silica ratio increases, indicating a higher degree of hardness and reduced temperature sensitivity. This phenomenon occurs because nano-silica absorbs light volatiles (oily matter) from asphalt and converts them into resinous materials within the asphaltene fraction [18, 19].

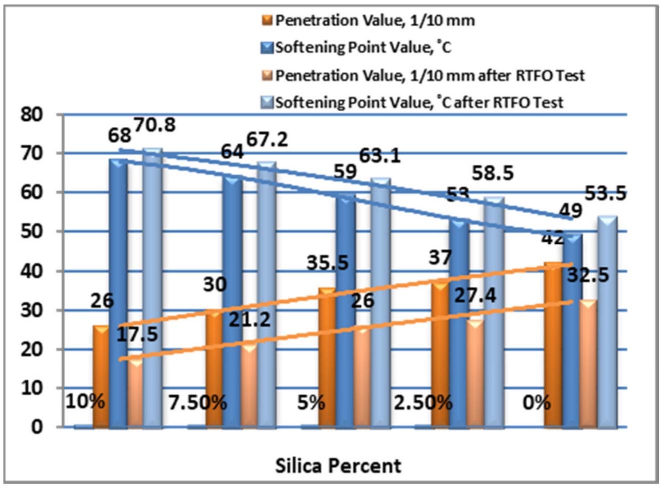

Fig. 1. Pre- and post-rolling thin film oven testing of asphalts modified with nano-silica.

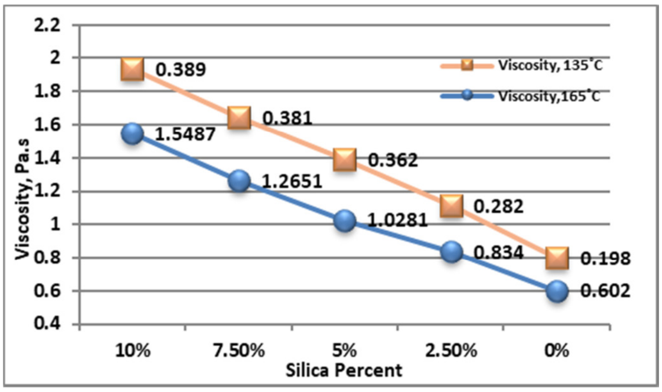

Fig. 2. Viscosity values of modified asphalt with nano-silica.

Additionally, nano-silica-modified asphalt binders exhibit greater hardness compared to unmodified asphalt cement binders, as nano-silica particles themselves possess higher hardness. Figure 2 further demonstrates a clear increase in viscosity as the nano-silica concentration increases under the same test conditions. This behavior is consistent with the permeability trends observed earlier. The increase in test temperature also significantly affects inverse viscosity values, a common characteristic of asphalt binders, even when modified with performance-enhancing additives.

2) *Nano-Chitosan*

Figure 3 depicts the penetration and softening point values of asphalt modified with nano-chitosan, both before and after RTFO aging, along with their respective ranges. During the preparation process, which involves 30 minutes of mixing, a notable decrease in penetration values was observed, along with an increase in softening point values. This behavior can be attributed to the polymeric nature of chitosan, a biodegradable polymer derived from chitin, a major structural component found in crustacean exoskeletons and fungal cell walls. Additionally, Figure 4 clearly demonstrates an increase in viscosity values at both 135 °C and 165 °C, which can be attributed to the interaction of nano-chitosan's polymeric structure with the asphalt binder. The polymer enhances the viscosity of asphalt by increasing its structural cohesion and reducing temperature susceptibility.

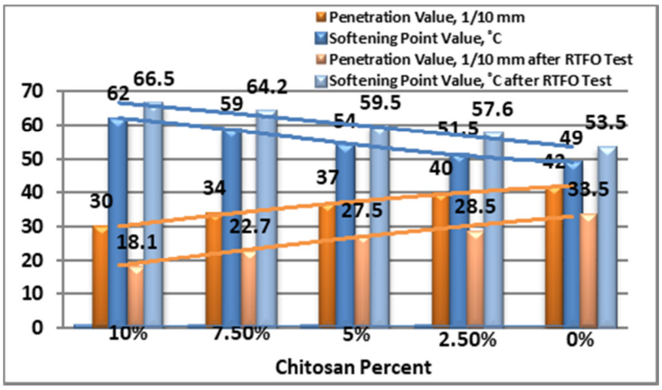

Fig. 3. Pre- and post-rolling thin film oven testing of asphalts modified with nano-chitosan.

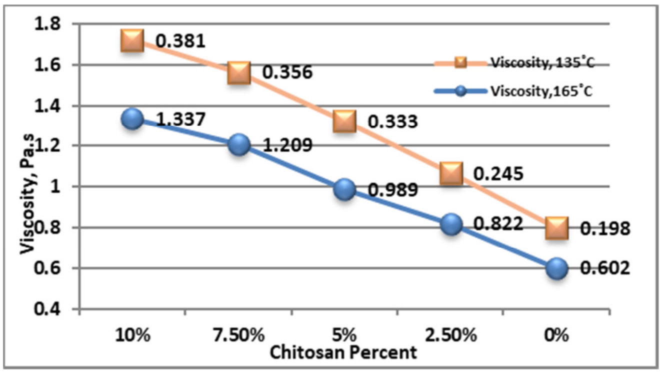

Fig. 4. Viscosity values of asphalt modified with nano-chitosan.

3) *Nano-Clay*

Similarly, an increase in nano-clay content resulted in lower penetration values and higher softening point values. This behavior is primarily due to the interaction between nano-clay and the oily components of asphalt, where the soft materials in nano-clay absorb or reduce the presence of light volatile compounds, making the asphalt more viscous [20]. This effect is demonstrated in Figures 5 and 6. Since nano-clay consists of compact silicate layers and contains traces of metal oxides and organic materials, it improves the viscosity of asphalt. However, it is important to consider the long-term aging effects of such materials.

To compare the effects of the three nanomaterials under the same testing conditions, the physical test results showed that as the percentage of nanomaterials increased, the penetration values of the modified binder decreased, while the softening point values increased. This trend indicates an improvement in cohesive strength, as the added nanomaterials help increase the asphalt's structural integrity, giving it a more solid and stable appearance compared to the unmodified binder. Since nanomaterials provide additional structural support to the asphalt binder, they effectively raise the softening point temperature, reducing the risk of permanent deformation (rutting) or splitting under high temperatures [21].

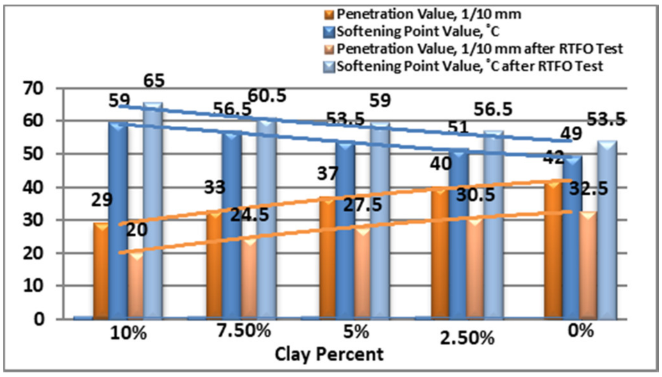

Fig. 5. Pre- and post-rolling thin film oven testing of asphalts modified with nano-clay.

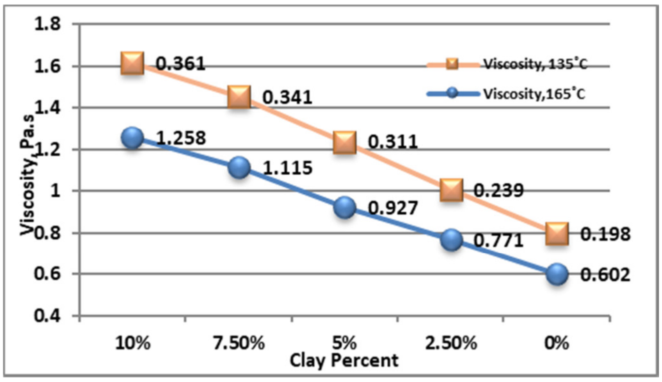

Fig. 6. Viscosity values of modified asphalt with nano-clay.

The comparative analysis highlights that nano-silica had the most significant impact in reducing permeability values. Nano-chitosan ranked second in effectiveness while nano-clay had the least impact on permeability reduction. For the softening point values, nanoclay had the smallest increase, even at higher additive percentages, nano-chitosan has a moderate effect, and nano-silica resulted in the highest increase. This behavior can be attributed to the structural characteristics of the nanomaterials and their varying degrees of influence on the asphalt binder. The viscosity test remains a key indicator of an asphalt binder's temperature sensitivity and its response to the presence of modifying agents.

V. THE RHEOLOGICAL CHARACTERISTICS OF ASPHALT BINDER MODIFIED BY NANOMATERIALS

In this test, the complex shear modulus (G^*) and phase angle (δ) were measured for both unmodified and nano-modified asphalt binders using DSR. The modified binders contained nano-silica, nano-chitosan, and nano-clay, and the temperature range for testing was 40 °C to 82 °C.

The rutting resistance of the asphalt binder, represented by $G^*/\sin\delta$, follows the standards set by the Superpave performance grading system. Increasing G^* or decreasing $sin\delta$ results in greater resistance to permanent deformation (rutting), as a stiffer yet flexible binder provides better structural integrity. For an asphalt binder to effectively resist permanent deformation at its design temperature, it must meet the minimum Superpave requirement of $\frac{G^*}{sin\delta} \geq 1\ kPa$. This value serves as the failure criterion in this test. Figures 7-9 illustrate the failure temperatures of asphalt binders modified with different nanomaterials. The results highlight the impact of nano-additives on the rheological performance and thermal stability of the modified asphalt binder. The RTFO-aged asphalt binder, modified with nano-silica, nano-chitosan, and nano-clay at four different concentrations, underwent a temperature sweep test. The results are illustrated in Figures 10-12. To ensure the asphalt binder can adequately resist permanent deformation, a minimum value of 2.2 kPa for $G^*/sin\delta$ has been suggested [22]. This threshold was set as the failure criterion for the test.

Figures 10-12 demonstrate that, regardless of aging condition (unaged or RTFO-aged), the complex shear modulus (G^*) of nanomaterial-modified asphalt is higher than that of pure asphalt, particularly at both low and high temperatures. This indicates that asphalt modified with nanomaterials exhibits reduced temperature sensitivity, meaning it remains more flexible at low temperatures while becoming stiffer at high temperatures. For nano-clay modified asphalt, the results were similar but showed lower rigidity compared to silica and chitosan modified binders. This could be due to differences in the modulus of elasticity (G') and deformation resistance (G^*) among the nanomaterial types. Specifically, the nano-clay-modified asphalt binder in this study did not achieve the same elastic modulus or stiffness as asphalt modified with silica and chitosan. In evaluating calcination as a stress-controlled cyclic loading event, SHRP researchers analyzed its effect on rutting resistance. Table V ranks the asphalt binders, including the nanomaterial-modified binders, at a high temperature of 58 °C, where calcination is a crucial factor. The higher the $G^*/sin\delta$ values, the better the rutting resistance, indicating that the material behaves more like a resilient elastic solid.

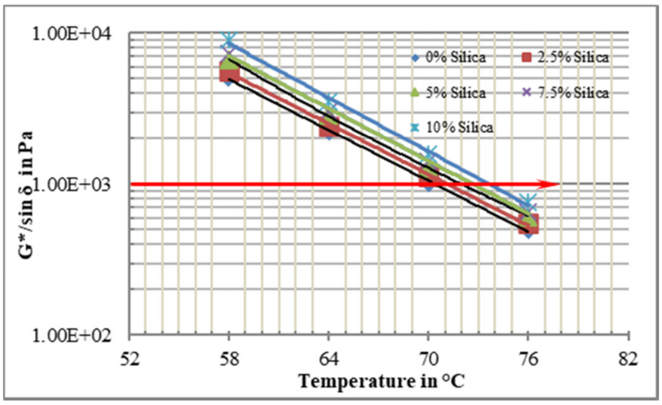

Fig. 7. Temperature sweep test of silica-modified binder.

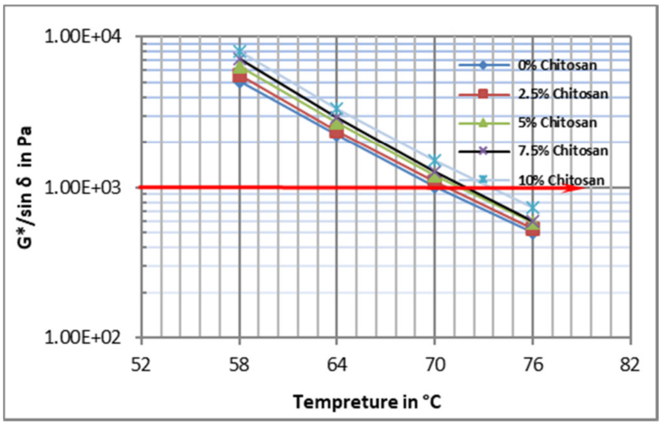

Fig. 8. Temperature sweep test of chitosan-modified binder.

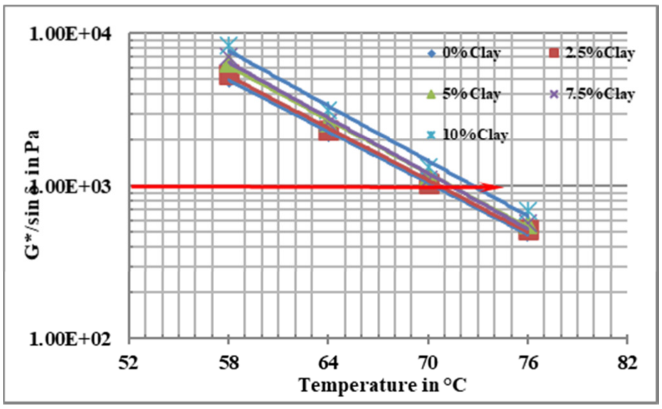

Fig. 9. Temperature sweep test of clay-modified binder.

The nanomaterial modified asphalt binder consistently exhibited an increase in G* and a decrease in sinδ, regardless of the type of nanomaterial used. The phase angle of the modified asphalt binder sample was higher compared to unmodified asphalt. This behavior is attributed to the enhanced internal bonding provided by nanomaterials, which reduces deformation and improves the asphalt binder's resistance to permanent deformation.

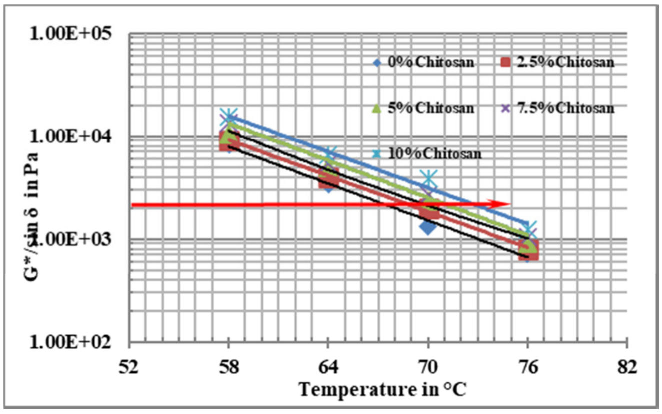

Fig. 10. Temperature sweep test of RTFO-aged nano-silica modified binder.

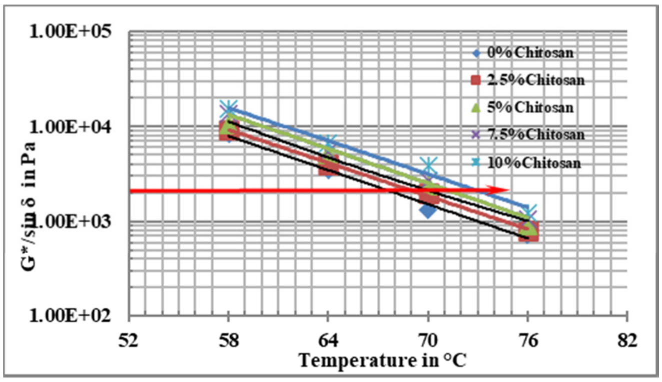

Fig. 11. Temperature sweep test of RTFO-aged nano-chitosan modified binder.

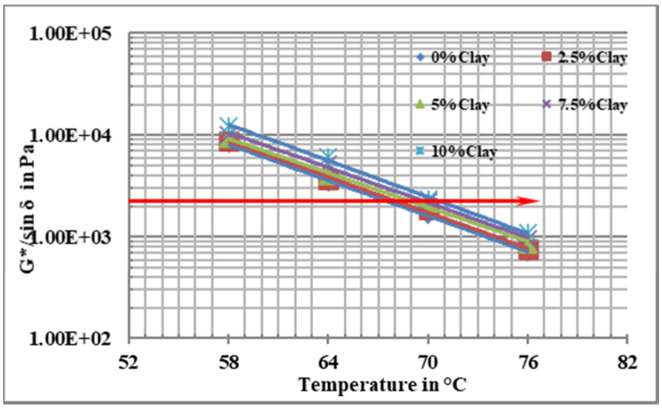

Fig. 12. Temperature sweep test of RTFO-aged nano-clay modified binder.

TABLE V. RUTTING PARAMETER OF NANO MATERIALS MODIFIED BINDER AT 58 °C (REFERENCE TEMPERATURE)

Blend type	**f Parameter rutting ($G^*/ sin\delta$) in kPa**	**The Parameter of rutting ($G^*/ sin\delta$) Of RTFO-aged binder in kPa**
M B-N0	5110.89	8210.898
MB-NS2.5	5669.44	10469.44
MB-NS5.0	6650.81	12450.81
MB-NS7.5	7344.4	15044.40
MB-NS10	8969.28	16999.28
MB-NCH2.5	5561.8	9169.44
MB-NCH5.0	6451.6	10450.81
MB-NCH7.5	7114.4	13544.40
MB-NCH10	8089.5	15299.28
MB-NC2.5	5449.1	8849.1
MB-NC5.0	6401.4	9201.4
MB-NC7.5	7026.2	10026.2
MB-NC10	8345.28	12345.28

The results demonstrated a clear increase in the complex modulus of elasticity (G^*) as the nanomaterial concentration increased. Nano-silica demonstrated the highest increase in G^*, followed by nano-chitosan, and nano-clay. This trend aligns with the findings from penetration, softening point, and viscosity tests, which also indicated that nano-silica provided the greatest hardening effect at an optimal concentration.

As nanomaterial content increases, the asphalt mixture becomes stiffer due to a reduction in free asphalt, leading to enhanced structural integrity. This suggests that rutting resistance will improve proportionally with an increase in the calcination coefficient ($G^*/ sin\delta$), further reinforcing the durability and longevity of nanomaterial modified asphalt.

VI. CONCLUSION

This study investigated the effects of nanomaterials (nano-silica, nano-chitosan, and nano-clay) on the physical and rheological properties of asphalt binder. The research focused on evaluating how different nanomaterials, added in varying proportions (0% to 10% in 2.5% increments), influence the binder's consistency, softening point, viscosity, and resistance to permanent deformation (rutting). Standard laboratory tests were conducted, including penetration, softening point, viscosity measurements, and dynamic shear rheometer (DSR) tests, both before and after aging using the Rolling Thin Film Oven (RTFO) procedure. The key findings of this study are detailed below:

- Physically, the incorporation of nano-silica, nano-chitosan and nano-clay resulted in an increase in both softening point and viscosity values, while penetration values decreased with higher additive ratios.
- Among the three nanomaterials, nano-silica presented the most significant enhancement, followed by nano-chitosan and then nano-clay, due to their unique chemical compositions and interaction with the asphalt matrix.
- The Dynamic Shear Rheometer (DSR) test demonstrated that modified binders had higher complex modulus (G^*) values and lower phase angle ($sin\delta$), leading to improved rutting resistance.
- The Superpave performance grading (PG) system indicated that the PG classification of the modified binders increased, confirming that nanomaterials contribute to extending the temperature range in which the binder can perform effectively.
- Nano-silica consistently showed the best performance, making the asphalt stiffer and more resistant to permanent deformation, while nano-chitosan ranked second, followed by nano-clay.
- The study also examined the effect of short-term aging (RTFO) on the modified binders, showing that nanomaterials reduce the aging susceptibility of asphalt by enhancing its structural stability.
- The nano-clay modified binder exhibited the least increase in viscosity post-aging, suggesting that it may have a different impact on aging resistance compared to nano-silica and nano-chitocsan.

VII. FUTURE WORK

Future work recommendation includes conducting laboratory tests to study the effect of nanomaterials, especially chitosan, on asphalt mixtures of hot, warm and cold types, to verify the possibility of using the additive or not.

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