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Performance evaluation of nanosilica-modified asphalt binder

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Abstract

Continuous efforts are being made to enhance the performance of the pavements for which various modifiers and additives are being utilized. Lately, emphasis has been given to the use of sustainable materials to be used in pavement construction. The paper explores the use of nanosilica, which can be manufactured from industrial and agricultural wastes, as an asphalt modifier and evaluates its effect on high-temperature properties of VG-10 binder. The paper investigates the rutting potential of nanosilica-modified binders by using different rheological approaches. Nanosilica was used in three concentrations (0.5%, 1% and 3%). It was found that adding nanosilica to asphalt binder improves its rutting resistance. Results of all the rheological approaches showed that resistance to permanent deformation increases with the addition of nanosilica. Nanosilica-modified binders have high resistance to oxidative ageing. Nanosilica-modified binders exhibited good storage stability at high temperatures.

Keywords Rutting resistance \cdot Superpave rutting parameter \cdot Shenoy's parameter \cdot Complex modulus \cdot Phase angle \cdot ZSV \cdot LSV \cdot MSCR \cdot Nanosilica

Introduction

Rutting has been identified as major distress in asphalt pavements and is considered as one of the major design parameters considered in the design of flexible pavements. Rutting mainly results due to densification and/or shear deformation in the asphalt layers. However, the overall permanent deformation from the accumulation of the permanent strain in different layers of the pavement contributes significantly to rutting.

The permanent strain accumulation in the pavement layers is dependent upon the component materials and the pavement design. Properties of the binder play a significant part in resisting the permanent deformation. The intrinsic capability of the asphalt binder to resist the permanent deformation affects the overall rutting resistance of the asphalt mixes. Asphalt being a viscoelastic material is

 Faheem Sadiq Bhat faheem_35phd17@nitsri.net
 Mohammad Shafi Mir shafi@nitsri.net highly dependent on time and temperature and deforms with time due to load application. The ability of the binder to resist this deformation is highly desirable to obtain a mix with high rut resistance [1]. Asphalt on its own is incapable of resisting the excessive loads, stresses, temperature variations and oxidative processes. Therefore, different types of modifiers are used to improve various properties of the asphalt. Several categories of asphalt modifiers are used, which include polymers, rubbers, sulphur, fibres and various chemical agents [2–17].

Recently, the impetus has been on the use of nanomaterials in various construction fields. Nanomaterials due to their high strain resistance, high functional density and high specific surface area may provide solutions to various problems associated with the pavements. Various types of nanomaterials are being used in asphalt modification; nanoclay, carbon nanotubes, aluminium trioxide and nanosilica are few of them [18–27].

Earlier studies have shown that adding nanosilica to the asphalt binder improves its rutting resistance. Most of the studies to evaluate rutting resistance have concentrated on the use of Superpave rutting parameter $G^*/\sin\delta$ [28–38]. The studies have used different concentrations of nanosilica ranging from 1 to 6%; the authors selected lower and higher concentrations based on the study carried out by

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Leiva-Villacorta and Vargas-Nordcbeck [39]. Various studies have pointed out the inability of $G^*/\sin\delta$ to capture the delayed elastic response of the modified binders and poor correlation with the rutting evaluation of mixes; this has raised concerns over the effectiveness of this parameter to rank the rutting resistance of various binders [40–45]. Few investigations have evaluated the rutting resistance of nanosilica-modified binders using other rheological approaches like Shenoy's parameter, zero-shear viscosity (ZSV), low shear viscosity (LSV) and multiple stress creep and recovery test (MSCR). All these approaches have been developed to overcome the constraints of the Superpave rutting parameter. Various studies have shown that parameters like ZSV, LSV and MSCR correlate very well with the rutting performance of mixes [43, 44, 46–60].

Objectives and scope of the study

The main objective of the investigation was to examine the rutting potential of nanosilica-modified binders and investigate its effect on the oxidative ageing and storage stability. The sub-objectives are as follows.

- Estimate the effect of adding nanosilica on Superpave rutting parameter $G^*/\sin\delta$.
- Examine the outcome of the addition of nanosilica on the zero-shear viscosity and low shear viscosity parameters of the asphalt binder.
- Explore the effect of nanosilica on the Shenoy's rutting parameter.
- Evaluate the outcome of the addition of nanosilica on the cyclic loading and unloading conditions using MSCR approach.
- Develop correlations between different rutting parameters.
- Examine the effect of nanosilica on oxidative ageing of the binders.
- Assess the storage stability of the nanosilica-modified binders.

Experimental program and procedures

Material characterization

A soft grade binder of 80/100 penetration grade was used in the study as the base binder. This binder shows good compatibility with diverse kinds of modifiers like polymers and crumb rubber. The physical properties of the base binder are enumerated in Table 1.

Nanosilica is utilized as a modifier in the current study; nanosilica was supplied by Platonic Nanotech private limited. The base binder is modified with varying concentrations of nanosilica, and its effect on rutting potential is investigated. Different properties of nanosilica as specified by the supplier are listed in Table 2.

Modification of binder and sample preparation

The base binder was heated to a temperature of 150 ± 5 °C; this temperature is mostly used for preparation of VG-10 binder mixes. The nanosilica was added to the base binder in three different concentrations of 0.5%, 1% and 3% by weight of the base binder. The highest concentration of SiO₂ was limited to 3% as per literature [39]. Nanosilica has a very high tendency for agglomeration; to prevent the agglomeration, nanosilica was added in small quantities over a time period of 15 min in the binder. The mixing was carried out using a high-speed mixer. Mixing temperature was maintained at 150 ± 5 °C by using a thermostat. The 2-h mixing time was selected after conducting trials with samples

 Table 2
 Physical properties of nanosilica

Specification	Value
Purity	99.5%
Average particle size	30–50 nm
Specific surface area	200–250 m ² /g
Bulk density	0.10 g/cm^3
Melting point	1600 °C

 Table 1
 Physical properties of binder

Characteristic value	Test method	Unit	Value	Specification limit (mini- mum)
Penetration at 25°	IS: 1203	0.1 mm	92	80
Softening point R&B	IS: 1205	°C	52	40
Ductility	IS: 1208	mm	>100	75
Dynamic viscosity at 60 °C	IS: 1206 (Part II)	Poise	1072	800-1200
Kinematic viscosity at 135 °C	IS: 1206 (Part III)	cSt	286	250
Flash point	IS: 1209	°C	298	220

prepared at different mixing temperatures and mixing times, and results showed that the modified binder showed the best performance after mixing for 2 h. The rotational speed was 3500 rpm, and the mixing was carried for a duration of 2 h. The base asphalt was subjected to the same set of conditions to remove the effect of ageing. The base asphalt and modified asphalt were made to undergo short-term ageing by conditioning in a thin-film oven (TFO) as per (ASTM D1754-2014) [61].

Tests conducted on binder

Rheological measurements

The primary objective of the study was to assess the rutting performance of the nanosilica-modified binders by using diverse rheological methods as listed in the objectives section. Five different rheological approaches were used in the present study. Tests were performed on dynamic shear rheometer (DSR) having parallel plate geometry with 25 mm diameter and 1-mm gap.

Figure 1a, b shows the values of $G^*/\sin\delta$ for unaged and short-term aged binders. $G^*/\sin\delta$ for unaged and TFO-aged binders, at diverse temperatures (46, 52, 58, 64, 70 and 76 °C), was evaluated as per ASTM-D7175-15 [62]. As per SHRP recommendations, the unaged samples are tested at 12% strain rate and for aged samples, the strain rate is 10%. Angular frequency of 10 rad/s is used for both conditions. The value of $G^*/\sin\delta$ is limited to 1 kPa for unaged binders and 2.2 kPa for aged binders. Complex modulus G^* and phase angle δ are obtained from the test.

Figure 2 shows the Shenoy's parameter at three different temperatures. The data obtained from $G^*/\sin\delta$ evaluation are used to obtain the Shenoy's rutting parameter. Shenoy's parameter is found to be highly sensitive to phase angle.

Zero-shear viscosity is a theoretical concept and is defined as the viscosity measured when shear rate is approaching zero. It is the viscosity measured under a defined stress when the shear rate is almost zero [63]. ZSV is an indicator of stiffness of the binder and permanent deformation under long-term loading. At high temperatures, conventional asphalt binders behave as Newtonian fluids and their behaviour is independent of shear rate. In comparison, modified binders behave as non-Newtonian and have strong dependence on shear rates. However, at low shear rates, the behaviour becomes less complex and binders behave like Newtonian fluids. This viscosity is called zero-shear viscosity. Zero-shear viscosity can be evaluated from creep tests or frequency sweep tests. In the present study, ZSV was evaluated from frequency sweep data. Frequency sweep was conducted in the range of 0.1-100 rad/s. In the present study, ZSV was evaluated by using the cross-model.



Fig. 1 $G^*/\sin\delta$ of a unaged binder and b TFO-aged binder



Fig. 2 Shenoy's parameter

Table 3 Fitted model parameters of ZSV for different concentrations of nanosilica

Nanosilica content	ZSV (Pa s)	k	m
0	496.334	0.02349	0.1498
0.5	1675.25	0.01548	0.1617
1	2007.029	0.00801	0.1804
3	3394.602	0.00821	0.1751

Table 4Low shear viscosity at 64 °C

SiO ₂ content (%)	Low shear viscosity at frequency of		
	0.1 rad/s	0.01 rad/s	0.001 rad/s
0	119.76	121.56	123.36
0.5	484.43	484.89	484.91
1	740.86	741.29	741.64
3	2731.7	2732.65	2732.67

$$\eta^* = \eta_{\infty} + \frac{\eta_0 - \eta_{\infty}}{\left(1 + k\omega\right)^m} \tag{1}$$

where η^* denotes the complex viscosity, η_0 denotes the zeroshear viscosity, η_{∞} denotes the infinite shear viscosity, ω is the angular frequency (rad/s), and k and m are the model constants. The ZSV and cross-model parameters are presented in Table 3.

Some modified binders show unrealistically high zeroshear viscosities at low frequencies, and therefore, De Visscher [64] proposed the concept of low shear viscosity. Low shear viscosity has been found to adequately characterize rutting potential of the unmodified and modified binders [46, 65, 66]. Low shear viscosity is calculated at frequency of 0.1, 0.01 and 0.001 rad/s using the cross-model. LSV values and cross-model parameters are presented in Table 4.

MSCR test was carried out on all the TFO-aged nanosilica samples at a temperature of 64 °C according to ASTM-D7405 [67]. The average non-recoverable creep compliance (J_{nr}) and the average percentage recovery were calculated for the 1-s creep and 9-s recovery time. The test was performed at stress levels of 0.1 kPa and 3.2 kPa. There was no time lag when the stress was increased from 0.1 to 3.2 kPa. Nonrecoverable creep compliance is the ratio of non-recoverable shear strain and shear stress. The two estimated parameters are presented in Eqs. 2 and 3, respectively:

$$J_{\rm nr} = \frac{\varepsilon_{\rm nr}}{\sigma} \tag{2}$$

where J_{nr} denotes the non-recoverable creep compliance (1/kPa), ε_{nr} denotes the non-recoverable strain at the end of rest period, and σ denotes the stress (kPa) applied during the loading time.

$$\varepsilon_{\rm r} = \frac{\varepsilon_1 - \varepsilon_{10}}{\varepsilon_1} * 100. \tag{3}$$

where ε_r is percentage recovery, ε_1 is the strain value at the end of creep portion, and ε_{10} is the strain value at the end of recovery phase.

For better performance, the asphalt binder is required to have lower value for J_{nr} and a high value of recovery.

Effect on ageing

Asphalt binders experience oxidation process during the mixture production and placement stage and also for the entire service life. Oxidative ageing results from oxidation of unsaturated bonds present mainly in the aromatics and resin fractions of the binder. For longer pavement life, it is intended that the binders used should have a higher resistance to oxidative ageing. Typically, the ageing resistivity potential is measured by empirical means of softening point test, penetration test and viscosity tests. Various kinds of polymers used in the binder modification suffer from a major drawback of phase separation when stored at high storage temperatures [4]. The success of using a modifier also depends on its ability to be stored at high storage temperature without undergoing phase separation.

The ageing resistance of the nanosilica-modified binder was estimated by using the ageing index parameter, which uses the Superpave rutting parameter. A higher value for the ageing index indicates a higher degree of vulnerability towards ageing. In the present study, the ageing index was evaluated for a temperature of 60 °C at the frequency of 10 rad/s.

Ageing index =
$$\frac{\left(\frac{G^*}{\sin \delta_{\text{aged}}}\right)}{\left(\frac{G^*}{\sin \delta_{\text{unaged}}}\right)}$$
. (4)

Storage stability

Storage stability tests help to determine the stability of a binder to resist phase separation at high temperatures. In this study, storage stability test of the nanosilica-modified binders has been determined as per (ASTM D7173-14) [68]. The modified binder was poured in an aluminium tube. The tube is put in an oven maintained 165 ± 5 °C for 48 h. After this conditioning period, the tube was put in a freezer maintained at -10 °C for a duration of 4 h. The tube was then cut into 3 sections of approximately equal length; the softening point difference between borders of top and bottom portion is found out. The blend is considered to be stable when the difference between softening points of top and bottom sections is less than 2.5 °C.

Results and discussion

Superpave rutting parameter

Figure 1a, b shows the effect of the addition of nanosilica on the Superpave rutting parameter $G^*/\sin\delta$. As evident from the figures, the addition of nanosilica improves the rutting potential of the asphalt binder for both unaged and aged conditions. The improvement is more pronounced with the increase in the nanosilica content. For example, with the addition of 0.5%, 1% and 3% nanosilica, the $G^*/$ $\sin\delta$ value of the base binder increases from 1.37 to 5.60, 11.95 and 21.54 kPa, respectively, at 64 °C. This improvement in the rutting resistance is due to the fact that the addition of nanosilica increases the complex modulus and decreases the phase angle of the binder.

Rutting resistance using Shenoy's parameter

Figure 2 demonstrates Shenoy's parameter $(G^*/(1/(1-(\sin\delta \tan\delta))))$ for base binder and nanosilica-modified binder at different temperatures. Shenoy's parameter is based on G^* and δ values, which are also used in the Superpave rutting parameter calculations. Shenoy's parameter has damping $\tan\delta$ which is more sensitive to change in the phase angle. As can be observed, Shenoy's parameter and Superpave parameter show similar trend, the addition of nanosilica improves the rutting resistance of the binder, and improvement is more substantial at higher concentrations. It was observed that the values of Shenoy's parameter are higher as compared to the Superpave parameter.

Zero-shear viscosity

Zero-shear viscosity η_0 and the related model parameters (*k* and *m*) of the cross-model were calculated using multiple nonlinear regression analysis. Solver function of excel was used to fit the desired equation on the curve of complex viscosity. Figure 3 illustrates the effect of the addition of nanosilica on the ZSV of the binder evaluated at temperature of 64 °C. The model constants are tabulated in Table 3. The addition of nanosilica improved the ZSV of the binder significantly. As can be seen from Fig. 3, the addition of 3% nanosilica improves the ZSV of base binder from 496.334 to 3394.602 Pa s at 64 °C. This increase in the ZSV shows rutting resistivity will improve with the increase in nanosilica content.



Fig. 3 Zero-shear viscosity

Low shear viscosity

Low shear viscosity was calculated at the frequencies of 0.1, 0.01 and 0.001 rad/s. The frequency of 0.0001 rad/s was not chosen as it has been shown by various studies that LSV calculated at this frequency overestimates rutting potential. Table 4 gives the values of low shear viscosity at different frequencies. The higher concentration of nanosilica shows a significant increase in the low shear viscosity, indicating a higher rutting resistance. The LSV values did not show any significant change with the decrease in frequency.

Multiple stress creep and recovery test

Figure 4a shows the change in non-recoverable compliance J_{nr} with the incorporation of nanosilica in asphalt binder. It can be seen that the J_{nr} decreases with the addition of nanosilica. For example, at 3.2 kPa, 0%, 0.5%, 1% and 3% of nanosilica showed J_{nr} values of 7.9585, 2.9416, 0.4104 and 0.385, respectively. The decrease in the J_{nr} value with the addition of nanosilica may be attributed to the increased stiffness of the modified asphalt binder.

Figure 4b shows variation in recovery values of the binder as attained from the MSCR test. It can be seen that the addition of nanosilica increases the recovery value. For example, at the stress level of 3.2 kPa, the base binder shows zero recovery and with the addition of 0.5%, 1% and 3% of nanosilica, the recovery increased to 1.21%, 10.53% and 15.915%, respectively. Similar type of improvement was observed at 0.1 kPa stress level.

The decrease in non-recoverable creep compliance and increase in the recovery values show that modified nanosilica has higher resistance against rutting.



(a) Effect of nanosilica on non-recoverable creep compliance



Fig. 4 a Effect of nanosilica on a non-recoverable creep compliance and \mathbf{b} recovery

Elastic modification index

Complex shear modulus (G^*) comprises two components storage modulus (G') and loss modulus (G''). The inphase component of complex modulus is called as storage modulus (G'); storage modulus is the real part of the complex modulus. Loss modulus (G'') is the out-of-phase component of the complex modulus. To access the elastic modification of the nanosilica-modified binders, concept of elastic modulus is used [69]. Elastic modification index is defined as:

$$E_{\rm mi} = \frac{\text{(Elastic modulus of nanosilica modified binder)}}{\text{Elastic modulus of control binder}}$$
(5)

The elastic modification index $(E_{\rm mi})$ for different concentrations of nanosilica was calculated using Eq. (5) and has been plotted against temperature as shown in Fig. 5. $E_{\rm mi}$ has been calculated in the temperature range



Fig. 5 Elastic modification index

of 46–64 °C. It can be seen from figure that there is a continuous increase in elastic modification index with the increase in nanosilica content. The improvement is visible in the entire temperature domain. Improvement in elastic modification index is an indicator of improved elastic nature of the modified binder and enhanced the recovery aspect of the binder. This type of response of the binder is an indicator of improved rutting resistance of the modified binder.

Cole-Cole diagram

A Cole–Cole diagram is a plot between storage modulus (G') and loss modulus (G'') of an asphalt binder. Cole–Cole plot helps in representing the viscoelastic properties of asphalt binder without incorporating temperature or frequency as one of the axes [70]. Figure 6 represents a Cole–Cole diagram for control and nanosilica-modified asphalt binder. As



Fig. 6 Cole–Cole diagram

Table 5 Ageing index of nanosilica-modified binder

Binder type	Ageing index = $\frac{\left(\frac{G^*}{\sin \delta_{\text{aged}}}\right)}{\left(\frac{G^*}{\sin \delta_{\text{unged}}}\right)}$
Base + 0% SiO ₂	1.86
Base + 0.5% SiO ₂	1.40
Base + 1% SiO ₂	1.19
Base + 3% SiO ₂	1.074

 Table 6
 Softening point values of top and bottom sections from storage stability test

Binder type	(Softening point °C) _{top}	(Softening point °C) _{bottom}	(Soften- ing point °C) _{difference}
Base + 0% SiO ₂	50.2	50	0.2
Base + 0.5% SiO ₂	53.3	52.7	0.6
Base + 1% SiO ₂	56.6	55.8	0.8
Base + 3% SiO_2	60.1	59	1.1

can be seen from the figure, the slope of curves of binders shows a shift towards the storage modulus axis. As the concentration of nanosilica increases, the shift becomes more prominent. Asphalt binder modified with 3% nanosilica shows the highest shift towards the storage modulus axis. This is an indication of improvement in elastic nature of the binder, which will enhance the rutting resistance of the binder.

Ageing resistivity

Table 5 shows the ageing resistivity of base binder and nanosilica-modified binder, as is evident the addition of nanosilica decreases the ageing index of the binder, which is an indication that the addition of nanosilica decreases the oxidation process in the binder and therefore improves the ageing resistance of the modified binder.

Storage stability

Table 6 shows the variation in softening point of the base binder and the nanosilica-modified binders. As can be seen from the table, with the addition of nanosilica, the difference in the softening point of the top and bottom sections increases; however, it is below 2.5 °C for all the concentrations of nanosilica.

Relationship between different rutting parameters

Figure 7a–d shows relationship of $G^*/\sin\delta$ with $J_{\rm nr}$, $G^*/\sin\delta$ and ZSV, $G^*/\sin\delta$ and $(G^*/(1/(1 - (\sin\delta \tan \delta)))$ and $G^*/\sin\delta$

and LSV, respectively. The relationships have been established for TFO-aged binders at a temperature of 64 °C. Most suitable trend line has been selected for each correlation. It is evident from the figure that increase in $G^*/\sin\delta$ resulted in the decrease in J_{nr} . This is due to the stiffening of the modified asphalt binder which makes it more rut resistant. ZSV, LSV and $(G^*/(1/(1 - (\sin\delta\tan\delta))))$ are found to increase with the increase in $G^*/\sin\delta$ values. These correlations can be helpful in predicting the rutting parameters based on $G^*/$ $\sin\delta$. However, these correlations are based on limited data and further investigations are needed to be carried out in order to fully establish them.

Nanosilica as a sustainable alternative for binder modification

Use of polymers for asphalt modification is being practiced throughout the world. Polymer-modified asphalts, however, are costly and suffer from drawbacks like phase separation and high-temperature storage stability problems. These drawbacks of the polymer-modified binders can be overcome by using sustainable modifiers like nanosilica. Various authors have reported the production of nanosilica from waste products like silica fume and rice husk [29, 71–73]. Use of nanosilica in asphalt modification can reduce the cost of the construction and at the same time help to produce a sustainable product that will have all the attributes of the polymers and will be environment friendly.

Conclusions

Different rheological approaches have been used in the study to investigate the permanent deformation characteristics of the nanosilica-modified binder. From the Superpave rutting parameter and Shenoy's parameter, it can be concluded that the addition of nanosilica makes the asphalt binder more elastic in nature, thus making it more rut resistant. ZSV and LSV parameters calculated from frequency sweep data showed that the addition of nanosilica has a positive impact on the rutting resistance of the binder. The recovery of the asphalt binder increased with the addition of nanosilica, and the non-recoverable creep compliance decreased which is an indication that nanosilica-modified binder has better resistance against rutting. The trend of improvement in rutting resistance versus the amount of modifier added is identical for all the rheological approaches. An attempt has been made to correlate various rutting parameters, and these parameters showed good correlations, which could be useful in predicting the rutting parameters. Results showed that addition of nanosilica to the asphalt binder improves its resistance against oxidative ageing. Nanosilica-modified binders were found to be stable when stored at high temperatures.



Fig. 7 a Plot of $J_{nr_0.1}$ kPa versus $G^*/\sin\delta$, b plot of ZSV versus $G^*/\sin\delta$, c plot of $(G^*/(1/(1 - (\sin\delta\tan\delta))))$ versus $G^*/\sin\delta$ and d plot of LSV versus $G^*/\sin\delta$

Nanosilica is a sustainable product that can be manufactured from waste materials, and therefore, nanosilica-modified binder will have a positive impact on the environment.

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