

# Experimental Assessment and Prediction of Physical Properties of Nanosilica-Modified Asphalt Cement

Sheelan A. Ahmed<sup>1†</sup>, Hirsh M. Majid<sup>2</sup>

<sup>1</sup>Department of Civil Engineering, Faculty of Engineering, Koya University, Koya 44023, Kurdistan Region – F.R. Iraq

<sup>2</sup>Department of Civil Engineering, College of Engineering, University of Sulaimani, Kirkuk Road, Sulaymaniyah, Kurdistan Region – F.R. Iraq

**Abstract**—Modifying the asphalt binder is one technique for improving asphalt pavement performance. When nanoparticles are utilized to modify asphalt binders, they exhibit unique and significant properties. Without a doubt, silica is the most commonly added element as nanoparticles to asphalts. The main objective of this study was to investigate the effect of mixing speed used to produce nanosilica (NS)-modified-asphalt cement on conventional physical properties of asphalt cement and prepare a model to predict the physical properties of NS-modified asphalt cement. For this purpose, four different nanosilica contents (1.5%, 3%, 4.5%, and 6% of the asphalt cement weight) were utilized. Each content was mixed at a temperature of 160°C using a high shear mixer set to four different speeds (1,000 rpm, 2,000 rpm, 3,000 rpm, and 4,000 rpm) for 30 min. Conventional asphalt cement tests, including penetration, softening point, rotational viscosity, and ductility tests, are conducted to evaluate the nanosilica-modified asphalt cement properties. The results showed that higher NS contents improve the physical properties of modified asphalt cement, but the improvement is more noticeable when mixing at a faster speed, as this lowers penetration and rotational viscosity and raises the softening point. By utilizing the Minitab statistical program, the physical properties prediction models of the NS-modified asphalt showed a direct correlation between input data (NS content, NS average size, mixing temperature, mixing rotational speed, mixing time, and neat asphalt properties) and predictive models, demonstrated by a high  $R^2$  value and acceptable standard deviation values.

**Index Terms**—Ductility, Nanosilica-modified asphalt, Penetration, Rotational viscosity, Softening point

## I. INTRODUCTION

Silica is the term usually used to depict silicon dioxide, it is an inorganic substance mostly made from silica precursors,

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<sup>†</sup>Corresponding author's e-mail: sheelan.abdulwahid@koyauniversity.org

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such as silica fume or rice husk ash, and has undergone chemical processing (Liebau, 2012). Nanosilica (NS) represents one of the nanomaterials that can improve asphalt binder mechanical characteristics. All across the world, colloidal silica, silica fume, and silica gels are made with silica as a key component (Yang and Tighe, 2013). Silica nanocomposites have been attracting scientific interest. The advantages of these nanomaterials reside in the low cost of production and the high-performance features (Leiva-Villacorta and Vargas-Nordebeck, 2019). Nano-silica has demonstrated that, including nanomaterials, enhances the physical and chemical characteristics of the asphalt binder, resulting in nano-modified asphalt with higher performance. It has good self-healing and adhesive characteristics (Li, et al., 2017). Whereas the exfoliated structure of the NS, which functions as a barrier to stop oxygen from entering the binder matrix and light components of the binder from evaporating, was found to be responsible for the aging resistance, other study results have indicated the remarkable ability of NS to enhance the bitumen's rheology and mixture mechanical properties (Al-Sabaei, et al., 2021). The unique features of NS have considerably contributed to the tremendous enhancement of the modified bitumen properties. These features include extraordinary chemical purity, excellent dispersal skill, adsorption, and outstanding stability (Yusoff, et al., 2014).

### A. Effect of Physical Properties of Nano silica

Modified asphalt binders containing varying amounts of silica powder are used to investigate the effect of particle size on asphalt binder physical properties (penetration grade, softening point temperature, penetration index, flash point, and ductility) (Dai, et al., 2023). Sadeghnejad and Shafabakhsh (2017) introduced nano-SiO<sub>2</sub> at low concentrations (0.3–1.2%) into standard 60/70 bitumen and measured its properties. The results showed that adding 1.2% nano SiO<sub>2</sub> could decrease penetration while increasing the softening point. Moreover, Mirabdolazimi, Kargari, and Pakenari, (2021) modified two types of bitumen (60/70 and 85/100 penetration grade) using NS varying (0.2–0.9%). They found that for both types of asphalt, the optimal amount of NS is 0.7%. This percentage of NS content increased the softening

point, flash point, and penetration grade. Furthermore, Saltan, Terzi, and Karahancer (2017) utilized 0.1–0.5% NS to modify standard 60/70 bitumen. They observed that adding NS decreases penetration while increasing the softening point and rotational viscometer (Zghair et al., 2019a; Zghair et al., 2019b; Allothman et al., 2022). Previous studies investigated the modified asphalt binder (60/70) by adding 2%, 4%, and 6% of NS with an average size of 11 nm, likewise (Rasheed, Joni and Al-Rubaee, 2022; Rasheed, Joni and Al-Rubaee, 2023) utilized the same contents of NS and binder penetration grade, with an average size of 15 nm, whereas (Al-Sabaei, Safaeldeen and Napiah, 2023; Al-Sabaei, et al., 2022) they added the NS with an average size of 17.5 nm, while (Qasim et al., 2022; Sadeghpour Galooyak, et al., 2015) utilized the average particles size 15 nm and 25 nm, respectively. They concluded that the optimum amount of NS to modify the bitumen was 6%, and that complied with the results of Badgujar (2018), who employed a different asphalt binder grade of (40/50) with the same NS contents as well as the average size of 17.5 nm. However, other investigations (Mashaan, 2022; Aboelmagd, et al., 2021; Qasim et al., 2022; Albayati, et al., 2024) extended the content of NS up to 8%. They concluded that the best performance for asphalt cement was identified at 4% content of NS, where NS shows a dense arrangement of particles, indicating a large surface area which is critical for integrating with the asphalt binder. Other range of contents studied by (Saltan, Terzi and Karahancer, 2018; Taherkhani and Afrozi, 2016; Taherkhani and Afrozi, 2017), they modified the asphalt binder (60/70) by adding 1%, 3%, and 5% NS with an average size of 12 nm and 15 nm. Whereas, some other researchers (Ezzat, 2016; Ahmed Hussein, Hussein and Jalal Khoshnaw, 2021; Mohammed and Abed, 2023; Motamedi, Shafabakhsh and Azadi, 2020) utilized the NS contents of 3%, 5%, and 7%. However, all these studies agreed that the penetration value for asphalt cement decreased with increasing NS content, while the softening point increased, indicating that its temperature sensitivity decreased with increasing NS content. This decrement in the temperature sensitivity of asphalt cement leads to an improvement in its resistance to persistent deformation and low-temperature cracking.

#### B. Effect of Asphalt-NS Mixing Properties

Yusoff, et al. (2014) modified the asphalt cement with NS by heating it to 160°C and blending it for 60 min with a shear mixer at 1,500 rpm to achieve homogenous composition. Similarly (Ezzat, 2016; Ahmed Hussein, Hussein and Jalal Khoshnaw, 2021), they used the same speed and time for mixing, but by heating the asphalt cement to a temperature of 145°C. Whereas, a study by (Enieb and Diab, 2017; Rasheed, Joni and Al-Rubaee, 2022; Rasheed, Joni and Al-Rubaee, 2023; Aboelmagd, et al., 2021), they heated the asphalt cement to a temperature of 160°C and continued mixing the asphalt with NS for 60 min utilizing a rotational speed of the mixer of 2,000 rpm. Likewise, the researchers (Taherkhani and Afrozi, 2016; Taherkhani and Afrozi, 2017; Al Naser, et al., 2022) used the same mixing

time and temperature but with a mixing speed of 3,000 rpm. In another investigation, Bhat and Mir (2021) blended NS at a mixing speed of 3,500 and for 120 min with asphalt cement after heating it at 150°C. On the other hand, Zghair et al., 2019b employed two different mixing times 30 and 60 min with a mixer speed of 2,000 rpm for mixing NS with the asphalt cement heated to a temperature of 140°C. Another research by Zghair et al., 2019a utilized the same mixing times and temperature but with three different speeds of mixer (2,000, 4,000, and 6,000) rpm, and observed that a high shear mixing set at (4,000–6,000) rpm for mixing time reached 30 min at 140°C, indicating that the mixing temperature was more useful especially to produce a good dispersion of NS powder in asphalt binder. Besides, some researchers (Badgujar, 2018; Al-Sabaei, Safaeldeen and Napiah, 2023; Al-Sabaei, et al., 2022; Saltan, Terzi and Karahancer, 2018) investigated the blending of NS with the asphalt binder, the mixing carried out with the use of a high-shear mixer at a speed of 4,000 rpm, a temperature of 160°C, and 120 min interaction time, while Mashaan (2022) employed an interaction time of 60 min. A summary of previous studies utilizing nanosilica to modify asphalt cement is shown in Table I, which will be employed to prepare a statistical model to predict the physical properties of NS-modified asphalt cement.

## II. EXPERIMENTAL PROGRAM

#### A. Materials Characterization

##### NS powder

The NS utilized was produced by Handan Yueci New Materials Company in China and consists of a white solid powder with good dispersibility, wear resistance, and hardening coating. The physical properties of NS powder are summarized in Table II.

##### B. Asphalt Cement

One type of asphalt cement with a penetration grade of (40–50) was employed in this study, brought from the Phonex oil refinery in Sulaymaniyah city, northern Iraq. To qualify the asphalt cement, it must meet the parameters outlined in the Iraqi specifications. The penetration, ductility, rotational viscosity, softening point, flash point, and fire point tests were conducted to verify these parameters. The physical properties of the asphalt samples are given in Table III.

##### C. Sample Preparation

The modified asphalt cement was produced by heating the asphalt binder to 160°C to ensure that the asphalt cement was fully melted for proper blending of the modifier. The nanosilic powder was directly added to asphalt with four contents (1.5%, 3%, 4.5%, and 6%) by weight of asphalt cement. The mixture was prepared by blending the NS manually for 10 min up to the asphalt cement coating all particles of nanosilica. After that, transfer the sample to an oil bath heated to 160°C to ensure a constant temperature during the mechanical mixing by a high shear mixer for 30 min, with four different speeds of 1,000, 2,000, 3,000,

TABLE I  
SUMMARY OF PREVIOUS STUDIES UTILIZING NANOSILICA TO MODIFY ASPHALT CEMENT

References	Asphalt grade	NS properties		Mixing characteristics			Conventional tests			
		Size (nm)	Content (%)	Temp. (°C)	Speed (rpm)	Time (min)	Penetration	Softening point	Ductility	Viscosity
Hussein, Hussein and Khoshnaw, 2021	40–50	20–30	3,5,7	145	1,500	60	✓	✓	✓	✓
Sadeghnejad and Shafabakhsh, 2017	60–70	80	0.3,0.6, 0.9,1.2	150	4,000	60	✓	✓	✓	✓
Taherkhani and Afrozzi, 2017	60–70	11–13	1,3,5	160	3,000	60	✓	✓	✓	✓
Zghair, Joni and Hassan, 2019a	60–70	11–12	2,4,6	140	2,000	30	✓	✓	✓	✓
					4,000	60				
Zghair, Jony and Hassan 2019b	60–70	11–12	2,4,6	140	2,000	30	✓	✓	✓	✓
					60					
Rasheed, Joni and Al-Rubaee, 2022	60–70	15	2,4,6	160	2,000	60	✓	✓	✓	✓
Zafari, et al., 2014	60–70	5	2,4,6	180	4,000	120	✓	✓	✓	✓
Rasheed, Joni and Al-Rubaee, 2023	60–70	15	2,4,6	160	2,000	60	✓	✓	✓	✓
Mashaan, 2022	40–50	20–30	2,4,6,8	170	4,000	120	✓	✓		
Albayati, et al., 2024	40–50	25–35	2,4,6,8	140	4,000	20+	✓	✓		✓
Mirabdolazimi, Kargari and Pakenari, 2021	60–70	20–30	0.2,0.4, 0.7,0.9	150	4000	30	✓	✓	✓	✓
		85–100								
Taherkhani and Afrozzi, 2016	60–70	11–13	1,3,5	160	3,000	60	✓	✓	✓	✓
Aboelmagd, et al., 2021	75	20–100	2,4,6,8,20,30,40,50	160	2,000	60	✓	✓		✓
Mohammed and Abed, 2023	40–50	15–20	1,3,5,7	140	2,500	60				
Abdel-Wahed, Abdel-Raheem and Moussa, 2022	60–70	30–50	1,2,3,4	145	4,000	60	✓	✓		✓
Enieb and Diab, 2017	60–70	NA	2,4,6	160	2,000	60	✓	✓		✓
Sadeghpour Galooyak, et al., 2015	60–70	20–30	2,4,6	160	3,000	30	✓	✓	✓	
Motamed, Shafabakhsh and Azadi, 2020	85–100	20–30	3,5,7	160	4,000	60	✓	✓		✓
Guo, et al., 2016	81.3	≤100	0.5,1,1.5, 2,3,4	160	4,000	60	✓	✓		✓
Al-Sabaei, Safaeldeen and Napiah, 2023	60–70	10–25	2,4,6	160	4,000	120	✓	✓		
Qassim, et al., 2022	60–70	15	2,4,6,8	163	2,000	60	✓	✓	✓	✓
Alothman, Gökçeşu and Ali, 2022	60–70	11	2,4,6	160	4,000	120	✓	✓	✓	
Al-Sabaei, et al., 2022	60–70	10–25	2,4,6	160	4,000	120	✓	✓	✓	
Bhat and Mir, 2021	VG–10	30–50	0.5,1,3	150	3,500	120	✓	✓	✓	✓

TABLE II  
PHYSICAL PROPERTIES OF NANOSILICA POWDER

Properties	Unit
Chemical composition	SiO <sub>2</sub>
Appearance	White Powder
Particle size	30 nm
Purity	99.5%
specific surface area	220±30 m <sup>2</sup> /g

TABLE III  
PHYSICAL PROPERTIES OF ASPHALT CEMENT

Properties	Unit	ASTM	Test results
Penetration at (25°C, 100 g, 5 s)	0.1 mm	D5	41.66
Rotational viscosity @135°C	Pa.S	D4402	448
Softening point (Ring and Ball)	°C	D36	51.16
Ductility (25°C, 5 cm/min)	cm	D113	132
Flash point	°C	D92	>232
Fire point	°C	D92	>250

and 4,000 rpm, to obtain a homogeneous material. Fig. 1 illustrates the stages of the mixing process.

The asphalt cement samples modified with 6% NS were chosen, which was the maximum amount in this study, to assess the dispersion of the NS inside the asphalt cement.

These samples were mixed for 30 min at four different speeds (1,000 rpm, 2,000 rpm, 3,000 rpm, and 4,000 rpm). Scanning electron microscopy (SEM) was employed for this purpose; the results indicated that NS dispersion improved significantly with increasing speed, as shown in Fig. 2.

In addition, Fourier-transform infrared spectroscopy spectroscopy was utilized to identify interactions between NS particles and asphalt molecules, as well as to determine whether the modification process resulted in chemical alterations in the binder. The findings, as illustrated in Fig. 3, showed that mixing speed does not add new functional groups to the asphalt binder; however, higher rotational speed raises the peak absorbance intensity, especially in the O–H and C=O regions, indicating improved modifier dispersion and higher oxidation at higher speed, where the curves appear more stable. NS modifies asphalt cement physically rather than chemically.

#### D. Testing Methodology

To achieve the objectives of this study, NS was added to the asphalt cement at four different contents (1.5%, 3%, 4.5%, and 6.0%). The research methodology was divided and performed into two stages: First, preparation of NS-modified asphalt cement by employing a high shear mixer with four



Fig. 1. Mixing procedure to produce nanosilica-modified asphalt cement.

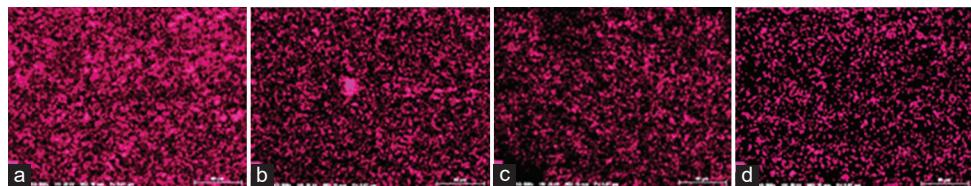


Fig. 2. SEM images of 6% nanosilica-modified asphalt cement using rotational speed: (a) 1,000 rpm. (b) 2,000 rpm. (c) 3,000 rpm. (d) 4,000 rpm.

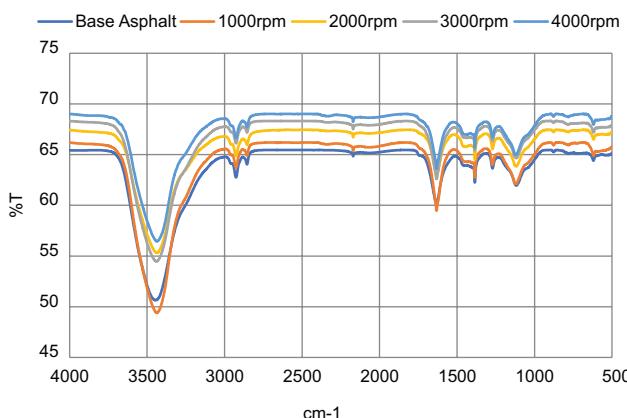


Fig. 3. Fourier-transform infrared spectroscopy spectrum of 6% nanosilica-modified asphalt cement mixing with different rotational speeds.

rotational speeds (1,000 rpm, 2,000 rpm, 3,000 rpm, and 4,000 rpm) for 30 min, and second, evaluation of asphalt cement properties with different NS contents and different mixing speeds. Using the Minitab statistical program, the results of the present study and data from previous studies will be used as input to develop models predicting the physical properties of NS-modified asphalt cement, including penetration (MP), softening point (MSP), ductility (MD), and rotational viscosity (MRV). These input data will include the physical properties of neat asphalt (penetration [NP], softening point [NSP], ductility [ND], and rotational viscosity [NRV]) as well as the NS contents (PC), average particle size (PS), mixing rotation speed (RS), mixing time (TI), and mixing temperature (TE). To evaluate the model reliability, statistical assessment parameters were utilized, such as the coefficient of determination ( $R^2$ ), standard deviation (Std.), root mean square error, mean absolute error, and scatter index. The research methodology of the present study is outlined step by step in the form of a flowchart in Fig. 4.

### III. RESULTS AND DISCUSSION

The effect of varied NS concentrations and mixing speeds on the conventional physical properties of asphalt cement samples was investigated in this study.

#### A. Penetration Test

Fig. 5 illustrates the correlation between the penetration values, mixing speed, and NS content. As the NS concentration increases, the penetration values decrease; however, as the rotation speed rises, the difference becomes more pronounced. This sharp drop was brought on by a higher NS concentration and faster mixing speed, which improved nanoparticle dispersion and might have stiffened the base asphalt cement.

$$MP = -1.414 (PC) - 0.01922 (PC)(PS) + 0.005548 (NP)(TE) + 0.000612 (TE)(RS) + 0.000023 (NP)(RS) - 0.001557 (RS * TI) \quad \text{Eq (1)}$$

The training data analysis produced an  $R^2$  value of 99.33 and a SD of 4.2162, according to the Minitab statistical program. The  $R^2$  and SD of the testing data, which make up 29.8% of the total data, are 98.82 and 5.1978, respectively. The differences between the actual and anticipated penetration levels of NS-modified asphalt cement as a result of Eq. (1) are shown in Fig. 6.

#### B. Softening Point Test

Fig. 7 shows that adding varying amounts of NS to asphalt cement increased its softening point at mixing speeds of 1,000 and 2,000 rpm, but the effect diminishes at higher speeds, where these variations are less pronounced between the NS contents. This effect can be explained that the distribution of NS particles for all contents becomes homogeneous at high speeds, meaning that the slight variations in softening point values are exclusively caused by an increase in NS content. On the other hand, at low speeds, the variations were caused

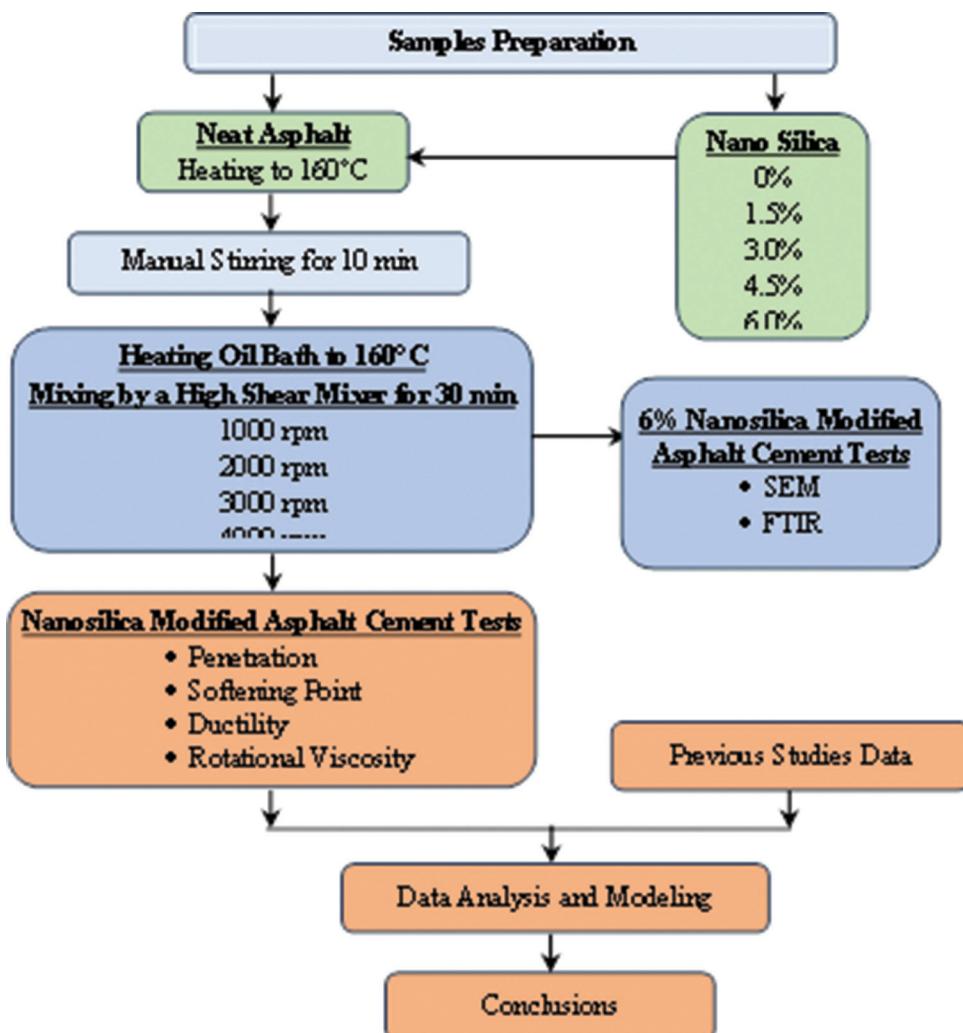


Fig. 4. The flow chart of the study.

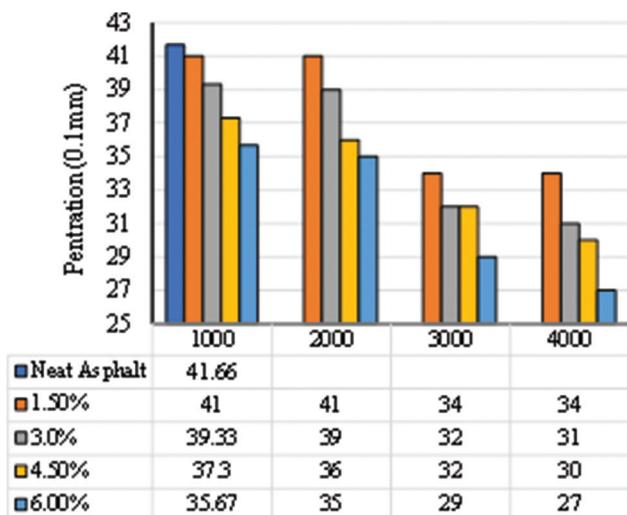


Fig. 5. Effect of nanosilica content and mixing speed on penetration of asphalt cement.

by both an increase in NS content and inadequate mixing to effectively disperse the NS particles.

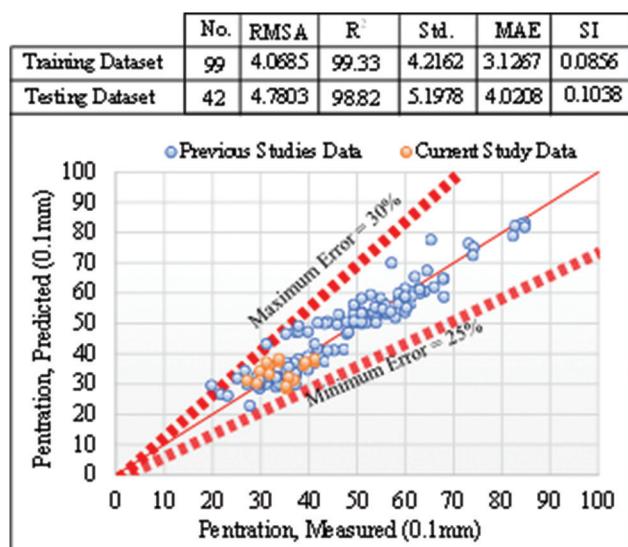


Fig. 6. Comparison of measured and predicted penetration values of the nanosilica-modified asphalt cement.

$$MSP = 2.377 (PS) + 0.38099 (TE) + 0.01814 (RS) - 1.302 (TI) - 0.01569 (PS) (TE) - 0.000375 (TE)(TI) + 0.01267 (RS)$$

(NSP)+0.01314(RS)(TI)

Eq. (2)

The training data analysis yielded a SD of 3.81707 and an  $R^2$  of 99.52. The testing data, making up 30% of the overall data, has a SD of 4.35146 and an  $R^2$  of 99.38. The differences between the observed and predicted softening point values of NS-modified asphalt cement as a result of Eq. (2) are shown in Fig. 8.

### C. Ductility Test

Ductility is used to show the adhesion and cohesion of asphalt cement. Fig. 9 illustrates that at 25°C, the ductility of

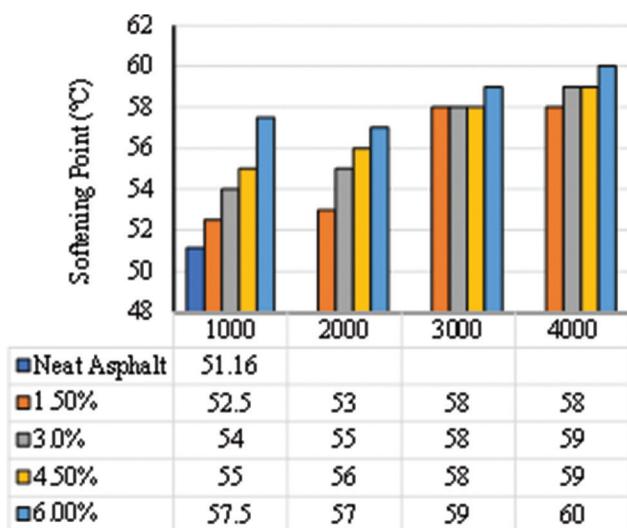


Fig. 7. Effect of nanosilica content and mixing speed on softening point of asphalt cement.

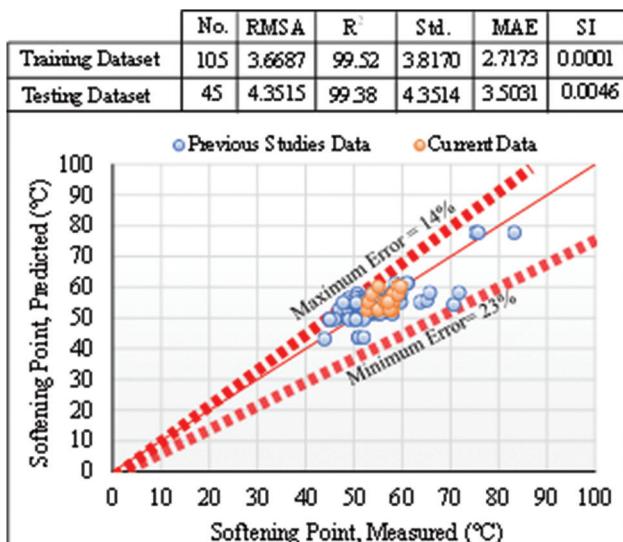


Fig. 8. Comparison of measured and predicted softening point values of the nanosilica-modified asphalt cement.

asphalt cement decreases as the amount of NS increases and the mixing speed increases. At a mixing speed of 4,000 rpm, ductility decreased significantly with increasing NS content. This reduction can be attributed to the absorption of the volatiles by NS, resulting in an increase in the stiffness of the NS-modified asphalt cement (Taherkhani and Afroz, 2016). Thus, adding NS can negatively affect cracking in cold climates, especially at high concentrations of NS.

$$MD = 0.05240 (RS) - 0.000088 (PS)(RS) - 0.1235 (PC)(TI) + 0.007325 (ND)(TE) - 0.000229 (TE)(RS) - 0.000270(ND)(RS) + 0.000003 (RS)2 \quad \text{Eq. (3)}$$

An  $R^2$  of 98.87 and a SD of 10.1007 were found in the training data analysis results.  $R^2$  is 98.80, and the SD is 10.4810 for the testing data, which represents 30.5% of the entire data. The differences between the observed and anticipated ductility values of NS-modified asphalt cement as a result of Eq. (3) are illustrated in Fig. 10.

### D. Rotational Viscosity Test

Rotational viscosity at the test temperature (135°C) of the asphalt cement increased with an increase in the NS content with faster mixing speed, as shown in Fig. 11. This can be explained by the hardening of the asphalt cement due to the addition of NS powder and its well-dispersed in the asphalt cement. This strengthens the bond and restricts the flow of asphalt, eventually increasing rotational viscosity. High-viscosity asphalt binder combined with NS can create a thicker film around the aggregate, thereby increasing cohesive strength. Thus, pavement durability may be enhanced (Qasim, Al-Sahaf and Al-Jameel, 2022).

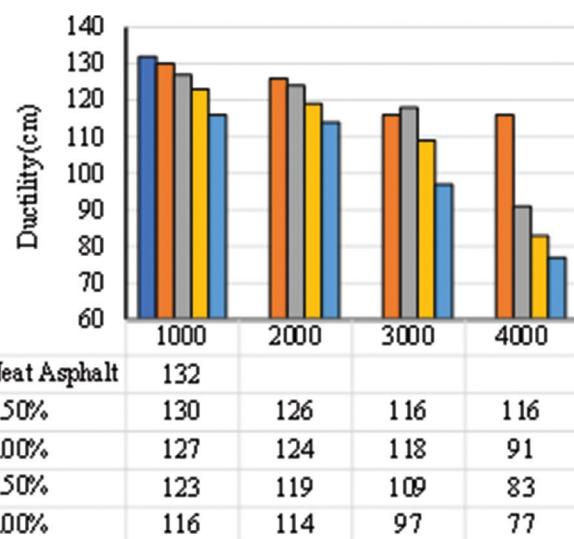


Fig. 9. Effect of nanosilica content and mixing speed on ductility of asphalt cement.

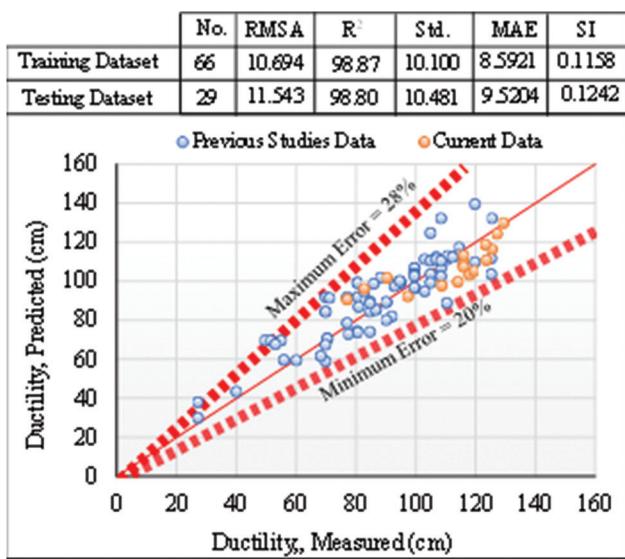


Fig. 10. Comparison of measured and predicted ductility values of the nanosilica-modified asphalt cement.

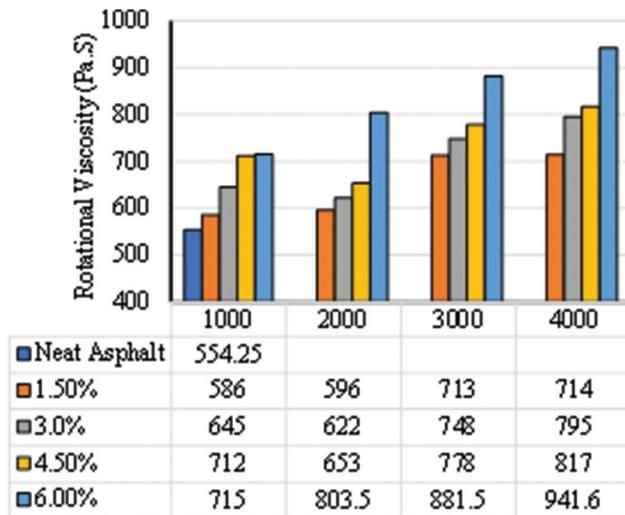


Fig. 11. Effect of nanosilica content and mixing speed on rotational viscosity of asphalt cement.

$$MRV = -6.13(PS) + 58.65(PC) + 15.59(TE) - 0.1734(RS) + 0.002894(TI)(PS) + 0.000267(TE)(TI) - 0.07649(TE)^2 \text{ Eq. (4)}$$

A SD of 106.383 and an R<sup>2</sup> of 97.87 were observed in the training data analysis outcomes. The testing data, which comprise up to 30.3% of the overall data, have a SD of 106.145 and an R<sup>2</sup> of 98.11. The variation between the calculated and observed penetration values of NS-modified asphalt cement, as determined by Eq. (4), is shown in Fig. 12.

#### IV. CONCLUSION

In this study, the evaluation of NS-modified asphalt cement was presented in terms of conventional physical properties of

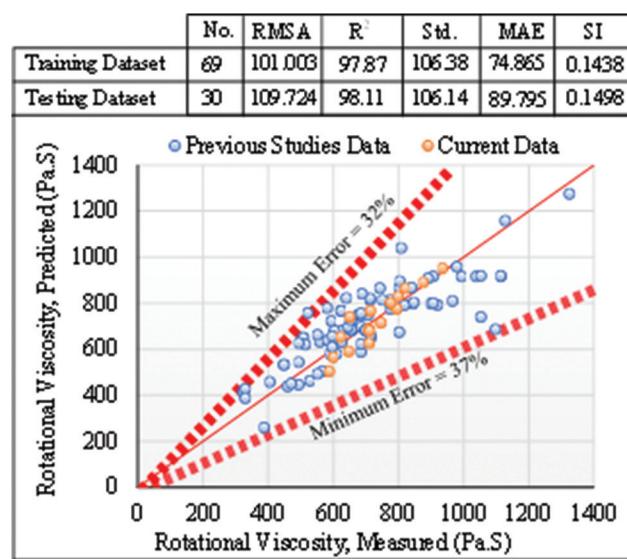


Fig. 12. Comparison of measured and predicted softening point values of the nanosilica-modified asphalt cement.

asphalt cement and was performed by adding NS and mixing at different speeds. The test results obtained from the testing program yield the following outcomes:

- Penetration test results indicated that adding 6% NS to asphalt cement at 1,000 rpm and 4,000 rpm resulted in a 14% and 35% drop in penetration results, respectively.
- The softening point of asphalt cement increased from 2% to 12% when 1.5% and 6% NS concentrations were added at a mixing speed of 1,000 rpm. In contrast, the softening point increased slightly from 13% to 17% when a mixing speed of 4,000 rpm was employed.
- All NS contents utilized to modify asphalt cement with mixing speeds up to 3,000 rpm demonstrated significantly good adhesion performance and acceptable ductility values.
- Utilizing a higher mixing speed led to a well-dispersed NS in the asphalt cement. This resulted in a significant increase in rotational viscosity, indicating an improvement in the stiffness and hardness of the modified asphalt cement.
- The models were generated to predict the physical properties of the NS-modified asphalt cement. These predictive models for penetration, softening point, ductility, and rotational viscosity demonstrated a direct correlation with input data (NS content, NS average size, mixing temperature, mixing rotational speed, mixing time, and neat asphalt properties), as evidenced by acceptable values of statistical assessment parameters.

Finally, concluded that the NS content enhanced the physical properties of asphalt, but this enhancement becomes more evident when using a higher mixing speed. This can be attributed to the better dispersion of NS, which produces stiffness and consistency of the modified asphalt cement.

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