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Rutting Performance of Nano-Silica-Modified C320 Bitumen

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Abstract: Nanomaterials exhibit novel properties and profound attributes as an additive in asphalt binder modification. However, the application of nano-silica in asphalt binders and mixture modification is still limited, and further research is required. Along these lines, in this work, nano-silica with a content from 2% to 8% and an increment of 2% was utilized in modifying the bitumen binder type C320, which is considered the most conventional type of bitumen used in Western Australia road asphalt mixtures. Various tests were performed to assess their properties, including complex shear modulus, penetration, softening point, and multiple stress creep recovery (MSCR) test. The extracted results revealed an increase in the strength and stiffness properties by lowering the penetration, improving the softening point, and increasing the complex shear modulus of all the nano-silica-modified bitumen samples. Interestingly, much of the content of nano-silica leads to higher rutting resistance. However, the rutting resistance was affected by the size of the nano-silica coated with the silane coupling agent. The ideal sample of nano-silica-modified C320 was determined as NS-15 nm (NS-A), which can improve the rutting resistance by about 7.1 kPa. In the current study, the results of the penetration and softening point using 6–8% of NS-A resulted in a relatively significant improvement of up to 45% in comparison with the non-modified binders. Nevertheless, the rutting resistance of the modified asphalt mixtures needs to be further investigated in the future to elaborate on the impact of nano-silica as modified binders on the mechanical properties of Australian asphalt mixtures.

Keywords: nano-silica; C320 bitumen; rutting; penetration; DSR; MSCR

Citation: Mashaan, N.S. Rutting Performance of Nano-Silica-Modified C320 Bitumen. *Eng* **2022**, *3*, 635–645. https://doi.org/10.3390/eng3040043

Academic Editors: Leszek Adam Dobrzański, Jingwei Zhao, Christof Sommitsch, Sabu Thomas, Lech Bolesław Dobrzański, Emilia Wołowiec-Korecka, Borut Kosec, Jorge Roberto Vargas Garcia and Gilmar Ferreira Batalha

Received: 9 October 2022
Accepted: 8 December 2022
Published: 9 December 2022

1. Introduction

Road pavements start experiencing functional deterioration once they are open to heavy traffic. One way to increase the service life of road surfaces is using certain additives such as polymers to modify and improve the properties of the asphalt mixtures [1,2]. Polymer-modified binder mixtures have been widely used in various civil engineering and construction projects. The addition of polymers within these mixtures improves their stiffness and significantly enhances their robustness against temperature fluctuations. This modification, in turn, improves the mixtures' resistance to pavement cracking. In addition, the incorporation of polymers into the binder results in a significant increase in its cohesiveness and adhesiveness, allowing it to effectively bind the mixture of components together [2]. However, the employment of some types of polymers for bitumen modification has certain disadvantages in terms of storage stability, bitumen–polymer phase separation, and elevated costs [3]. Therefore, the scientific community aspires to find an alternative additive that could improve the bitumen binder's properties. From this perspective, nanomaterials are currently employed to adjust the characteristics of bitumen binder and asphalt mixtures. The superior surface area and effective particles network created in the modified bitumen increase their stiffness and improve the asphalt mixtures' resistance to permanent deformation [4,5].

Nano-silica has several applications in the medical and engineering sectors. As far as the building material and concrete industry is concerned, nano-silica shows an essential part in the adhesion and cohesion of concrete. In road and pavement materials, nano-silica exhibits promising potential for improving asphalt mixtures' mechanical and engineering properties. It can also be used in bitumen and asphalt modification owing to its several advantages, including low-cost production and practical characteristics [4]. Nano-silica is a novel material that has many desirable characteristics such as a high surface area, enhanced absorption, high allocation, good stability, and high purity; moreover, it is cost-effective by nature [4]. In Section 2, the application of nano-silica in bitumen modification and the performance properties will be thoroughly illustrated and discussed.

To the best of the author's knowledge, nano-silica has not been used to modify the structural properties of bitumen C320, which is the most common bitumen used to design wearing course materials in Australia. Under this direction, the goal of this work is to investigate the rheological–physical properties and rutting resistance of nano-silica within the C320 bitumen and establish the ideal type of nano-silica to be used as an additive in the future nano-silica modified asphalt mixtures.

2. Nano-Silica in Bitumen Modification

Nanomaterials including nano-silica display unique characteristics in comparison with conventional material configurations due to their small size and high surface-to-volume ratio. Consequently, nano-silica holds crucial features, which can be employed in asphalt pavement as an additive [6,7]. The outstanding characteristics of nanomaterials such as their bulky surface area and self-assembly properties, which are quite different from the majority of the commonly used materials in construction, render them an idealistic materials as additives for asphalt-pavement applications. In addition, nanomaterials possess tremendous abilities, including self-cleaning and self-healing abilities [4,6,7]. These fundamental properties of nanomaterials successfully meet the requirements and standards of modern highway pavement. Therefore, nanoparticles have been already used to enhance the efficiency of asphalt [7].

Xiao and his associates' incorporated nanoparticles within asphalt to examine their rheological properties. In the last decade, numerous types of nanoparticles have been utilized to enhance asphalt's properties [8–10].

Several reports in the literature [11–17] have demonstrated that nanomaterials can significantly enhance the cohesion of asphalt mixture and establish an enhanced linking between the nano-silica and asphalt, thus preventing the growth of cracks. As a result, the fatigue life would possibly be increased, and the rutting failure would substantially decline [11–15]. Various studies have been also conducted by using different contents and sizes of nano-silica and different mixing conditions, as illustrated in Tables 1–3.

Table 1. Implementation of Nano-Silica in Asphalt.

Reference	Asphalt	Nano-Silica (%)
[11]	60/80	1.86 and 1.98
[13]	PG-76	2 and 4
[14]	PG 58–34	4 and 6
[15]	60/70	2, 4 and 6
[18]	AH-70	3, 5 and 7

Table 2. Specification of the Nano-silica Properties Based on Previous Studies.

Reference	Nano Silica Purity (%)	Size/Dia. (nm)	Surface Area (m^2/g)	Density g/cm^3
[4]	SiO_2 > 99	10	600	2.4
[5]	SiO_2 > 99	11–13	200	2.4
[19]	SiO_2 + carbon nanotube > 95	10–12	Not Given	2.64
[20]	SiO_2 > 99	Not Given	195	Not Given

Table 3. Mixing Aspects and Mixing Conditions of Producing Nano-Silica.

Authors	% Nano-Silica	Time	Temperature	Speed
[4]	2, 4, 6, and 8	2 h	135 °C	4000 rpm
[5]	1, 3, and 5	1 h	160 °C	3000 rpm
[19]	1, 3, and 5	2 h	160 °C	4000 rpm

As can be seen from Tables 1 and 3, the highest nano-silica content was up to the value of 7%, which can significantly improve the rutting resistance. The applied ideal mixing conditions were 2 h at 160 °C by using the high shear mixer of 4000 rpm, as illustrated in Table 3.

On top of that, the rutting and fatigue resistance of nano-silica has been systematically investigated [13–17], and the published results have indicated the remarkable ability of nano-silica to enhance the bitumen's rheology and the mixtures' mechanical properties. According to the literature [13–15], the unique features of nano-silica 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. In addition, various works focus on the physical-rheological properties of nano-carbon modified asphalt binder [4,9,10], while less attention has been given to the modification of the asphalt using nano-silica. Yao et al. [14] suggested that both chemical reactions and physical dispersion may occur during the blending of nano-silica and bitumen, which could lead to the production of a new network structure. Therefore, further studies are required to shed light on these effects.

The performance properties, in terms of improved stiffness and less sustainability to moisture damage, have been previously investigated [13,15,18]. In addition, the use of nano-silica with virgin polymer in bitumen modification, in terms of improving the physical and rheological properties, have been also examined. From the reported outcomes, it can be argued that the reaction between nano-silica and bitumen is not a trivial issue. Thus, further research is required to examine the underlying origins of the above-mentioned interactions.

3. Experimental Program

3.1. Materials

3.1.1. Bitumen

As far as binder selection is concerned, C320 bitumen was employed and supplied by SAMI Bitumen Technologies, which is in Perth city, Western Australia. This type of bitumen binder was selected following the specification and the recommendation of Main Road Australia and the Australian Asphalt Pavement Association, which both recommended the use of a C320 binder in wearing course materials design. Table 4 demonstrates the bitumen's physical properties [21].

Table 4. Bitumen's Properties.

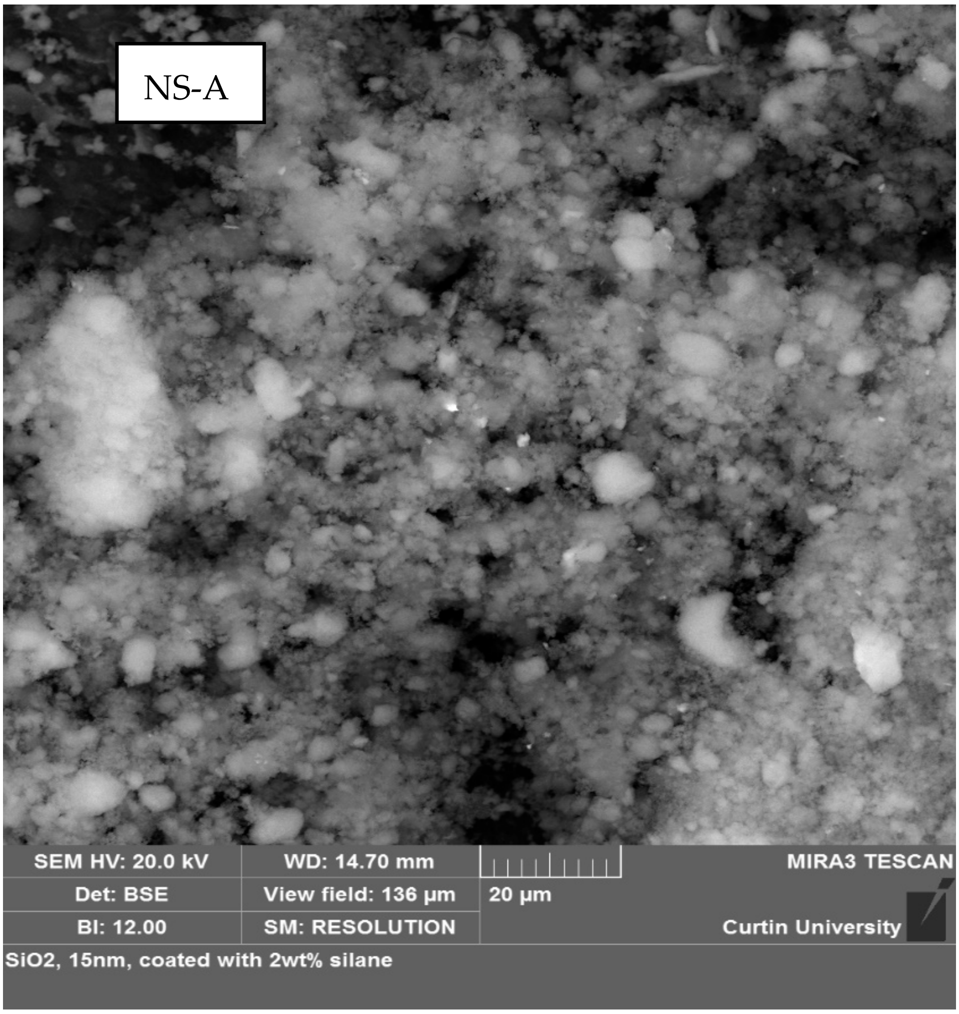
Properties	Value	Australian Standards
Penetration at 25 °C	42 mm	A2341.12
Brookfield Viscosity at 135 °C	0.502 Pa.s	AS 2341.2
Flash point	248 °C	AS 2341.14

3.1.2. Nano-Silica

In this work, three different sizes of nano-silica were used, as can be ascertained from the provided scanning electron microscopy (SEM) image in Figure 1. Generally, three types of nano-silica have been used in this work, two of which were coated with a silane coupling agent. The latter was used to improve the compatibility between the nano-silica and bitumen binder and to modify the silica's surface. Table 5 illustrates the properties of the three different types of nano-silica. NS-A stands for SiO_2, with a thickness value of 15 nm, coated with 2 wt% KH550-silane coupling agent. NS-B represents the nano-silica SiO_2, 99+%, with a thickness of 20–30 nm (no coating agent). NS-C denotes the nano-silica SiO_2, with a thickness of 30 mm coated with 3–4 wt% KH550-silane coupling agent.

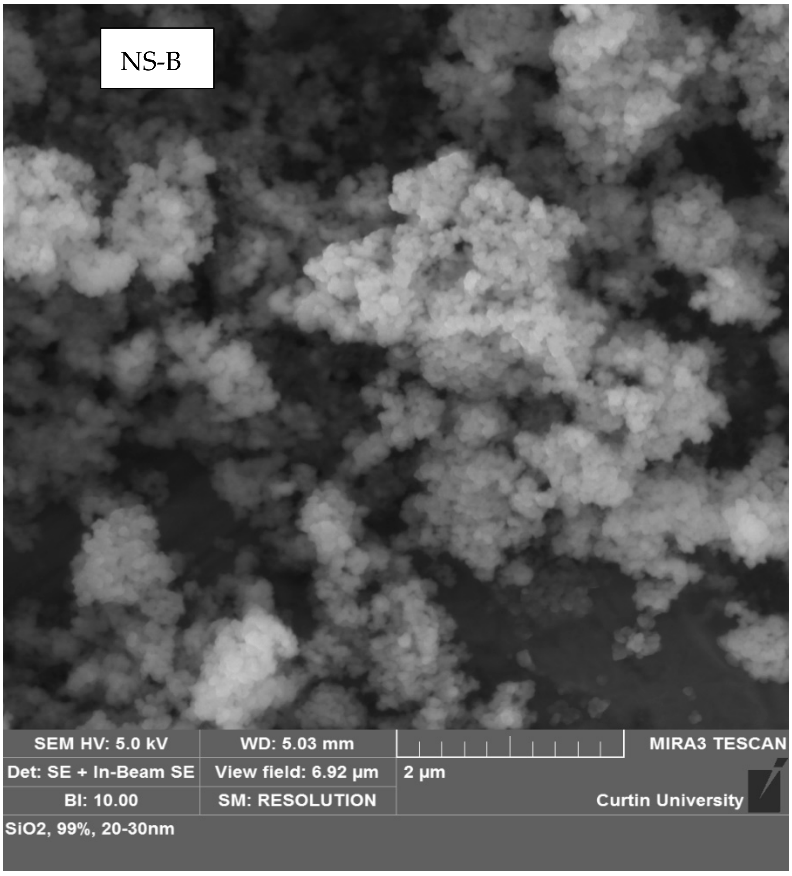
Table 5. Properties of Different Size of Nano-Silica.

Nano Name	Sample Name	Area	Size	Colour	True Density	Purity
SiO_2, 15 nm, coated with 2 wt% KH550-silane coupling agent.	NS-A	600 m^2/g	15 nm	White powder	2.4 g/cm^3	97.3%
SiO_2, 99+%, 20–30 nm (no coating agent)	NS-B	180–600 m^2/g	20–30 mm	White powder	2.4 g/cm^3	99%
SiO_2, 30 mm coated with 3–4 wt% KH550-silane coupling agent.	NS-C	130–600 m^2/g	30 mm	White powder	2.4 g/cm^3	96.3%

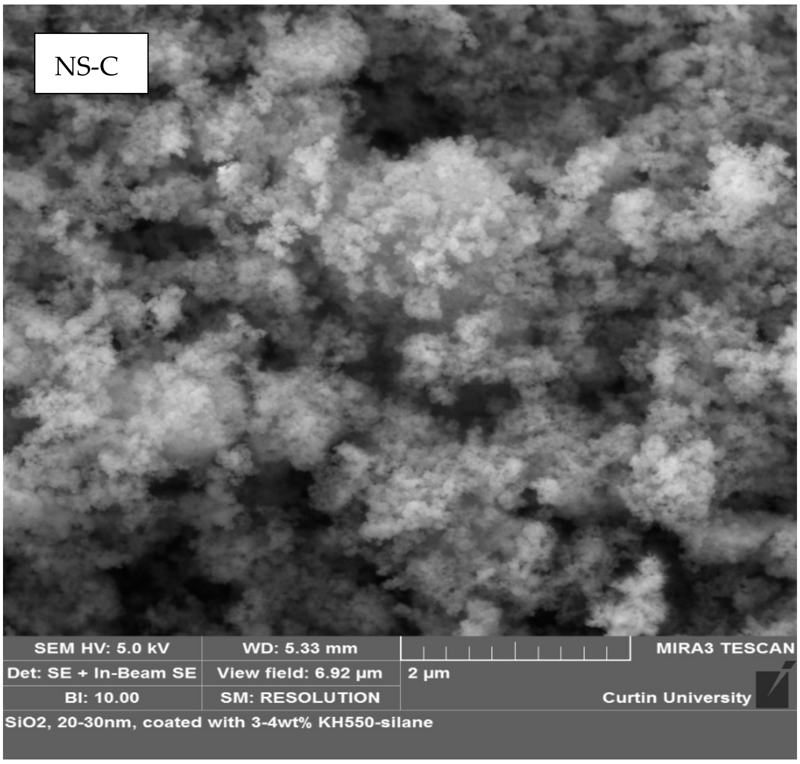


NS-A

Figure 1. *Cont.*



NS-B



NS-C

Figure 1. SEM images of the different types of nano-silica.

3.2. Sample Preparations

The high-shear mixer type Silverson L5M-A was used to create nano-silica-modified bitumen structures with a variety of contents: 0%, 2%, 4%, 6%, and 8%, by weight of bitumen for each sample of NS-A, NS-B, and NS-C, which refer to the sorts of nano-silica used, as is illustrated in Table 5. The mixing conditions included a high shear speed of 4000 rpm, a mixing time of 2 h, and a processing temperature of 170 ± 0.5 °C. The mixing process started at 700 rpm for the first 10 min, and then the speed was increased to 4000 rpm. The prepared samples of the modified bitumen were stored in a 1 L container, which were used for testing the physical, viscoelastic, and rutting properties.

3.3. Testing Methods

3.3.1. Penetration Test

The penetration test is considered to be a well-established method to determine the physical properties of the bitumen binder. The Australian Standard AS 2341.12 has been followed during this method. The penetration test conditions were 25 °C, 5 s and the enforcement of a 100 g load for measuring the vertical penetration distance of standard needle in the bitumen samples. The major outcome of the test was to determine the strength of bitumen and the capacity to resist rutting deformation.

3.3.2. Softening Point Test

The softening point test, which is also known as the ring and ball test, was used to determine the softening and stiffness levels of the bitumen. This approach can be used as an indicator of the bitumen's susceptibility to high temperature, whereas valuable insights regarding the lower temperature susceptibility and improved rutting resistance can be derived. The test was conducted according to the Australian Standard AS 2341.18 at 25 °C.

3.3.3. Dynamic Shear Rheometer (DSR)

This test was conducted according to the AASHTO 315 standards. This test aims to determine the impact of the complex shear stress and liner viscous on the elastic properties of bitumen through oscillatory shear. More specifically, the primary objective of this test procedure was to determine the stiffness, elasticity, and rutting resistance.

3.3.4. Multiple Stress Creep Recovery (MSCR) Test

The MSCR test was performed following the AASHTO T350-14 specification at 64 °C by using the DSR technique. The creep recovery and stiffness performance of the bitumen binder were determined, as well as the rutting resistance. The samples were tested in creep and recovery states under the application of stress loads of 0.1 and 3.2 kPa. The main two tested parameters of Jnr and %R represented the non-recoverable creep and recovery percentage, respectively.

4. Results and Discussion

4.1. Physical Properties

Figure 2 displays the penetration results of nano-silica at 25 °C. It is interesting to notice that the addition of different sizes of nano-silica indicates a considerable improvement in the penetration results for all of the contents of nano-silica. However, NS-A exhibits better results at 6–8%, which could be attributed to the impact of the small size of the nano-silica (15 nm) and the use of KH550-silane as the coupling agent. The use of KH550-silane can significantly improve the compatibility and interaction between the bitumen and nano-silica [7,17].

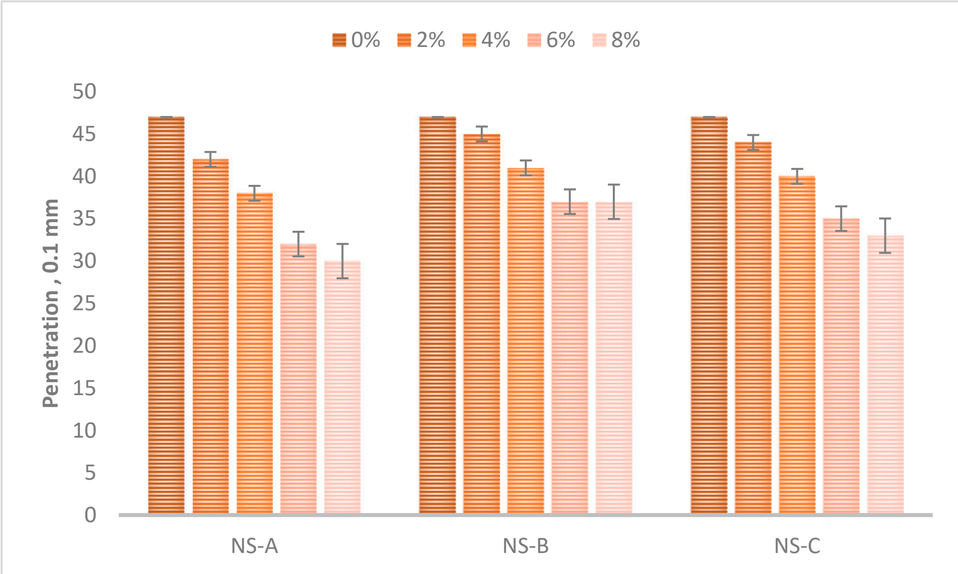


Figure 2. Penetration results.

In addition, a similar trend was observed, in terms of the softening point, as is shown in Figure 3. The optimum physical properties could be achieved by using NS-A, which is nano-silica with a size of 15 nm coated with the coupling agent. However, all nano-silica modified samples have higher softening points than the non-modified C320 bitumen. The finding is in line with the previously reported findings in the literature [12,15]. However, in the current study by using the 6–8% of NS-A, a relatively high improvement in the penetration and softening properties up to 45% was achieved compared to the non-modified binders.

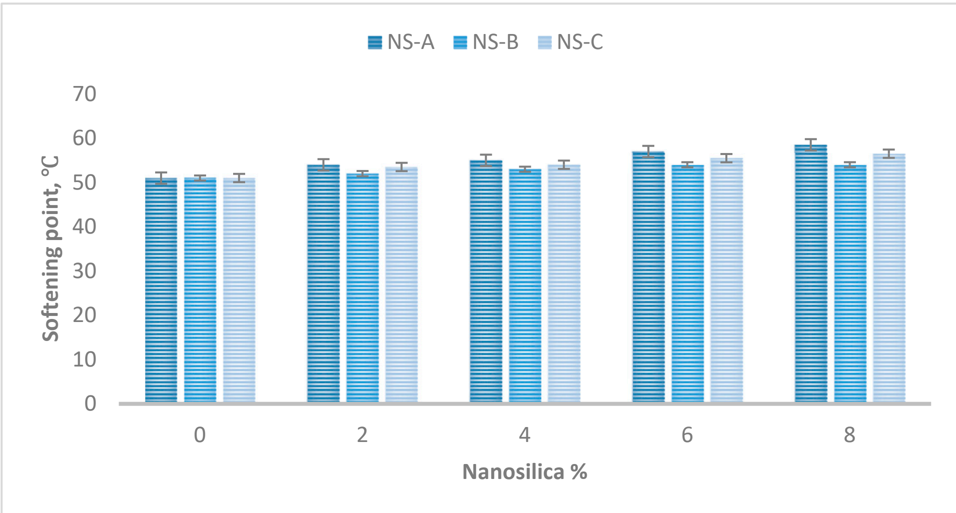


Figure 3. Softening point results.

4.2. Rheological Properties of Nano-Silica Modified C320 Bitumen

Tables 6–8 illustrate the stiffness properties, in terms of the complex shear modulus for all the different types of nano-silica, by using various temperatures: 50 °C, 58 °C, 60 °C, 64 °C, 70 °C, and 76 °C. Tables 6–8 illustrate the influence of nano-silica percentage, size, and types on the complex shear modulus of the modified bitumen. The results show that the different sizes and different contents of nano-silica would significantly improve the stiffness properties by increasing the complex shear modulus, resulting in better stability and high resistance to deformation. Thus, nano-silica modified C320 bitumen results in an enhancement in rutting resistance. Modifying and advancing the properties of the bitumen and asphalt mixture by using certain additives, such as nano-silica, is one way of boosting the service life of road surfaces [4,19].

Table 6. Complex Shear Modulus (kPa) of NS-A.

	50 °C	**58 °C**	**60 °C**	**64 °C**	**70 °C**	**76 °C**
0%	14.2	3.87	3.2	2.76	0.8	0.4
4%	15.89	7.6	5.4	2.9	1.9	0.42
6%	16.66	5.9	4.3	2.5	2.1	0.5
8%	18.6	7.1	4.8	4.2	2.78	0.5

Table 7. Complex Shear Modulus (kPa)of NS-B.

	50 °C	**58 °C**	**60 °C**	**64 °C**	**70 °C**	**76 °C**
0%	14.2	3.87	3.2	2.76	0.8	0.4
4%	14.89	5.6	4.4	1.9	1.9	0.42
6%	16.66	5.9	4.3	1.5	2.1	0.5
8%	18.6	9.1	5.8	1.2	0.78	0.15

Table 8. Complex Shear Modulus (kPa) of NS-C.

	50 °C	**58 °C**	**60 °C**	**64 °C**	**70 °C**	**76 °C**
0%	14.2	3.87	3.2	1.76	0.8	0.4
4%	16.89	5.6	4.4	2.9	1.9	0.42
6%	16.66	4.9	3.3	2.5	2.1	0.5
8%	19.6	6.1	4.8	3.2	2.78	0.5

4.3. DSR Rutting of Nano-Silica Modified C320 Bitumen

The rutting factor ($G^*/\sin\delta$) has been used as a rutting indicator, and the extracted results of the various samples modified with different nano-silica sizes are shown in Figure 4. At 64 °C, the 8% NS-A and NS-C samples had a higher rutting resistance than the unmodified bitumen. More specifically, the rutting factors of the NS-A and NS-C samples were 7.1 kPa and 5.88 kPa, respectively, whereas the rutting factor of NS-B was only 2.45 kPa. Although both NS-B and NS-C samples had the same size of 20–30 nm, the implementation of a 2% coating of silane coupling on NS-C resulted in an increase in the rutting resistance in comparison to NS at a thickness value of 20–30 nm without incorporating a coupling agent coating.

Nanomaterial and nano-silica could substantially improve the adhesion and cohesion of the bitumen binder and create a bridging impact between the bitumen and nanoparticle, evading the growth of deformation [15,17,20]. Therefore, in turn, the life service of road pavement may possibly be extended, and the rutting failure may significantly decline.

4.4. MSCR Rutting of Nano-Silica-Modified C320 Bitumen

Figure 5 illustrates the recovery percentage of nano-silica-modified bitumen C320 and non-modified bitumen with three different nano-silica sizes under the application of high- and low-stress levels after rolling thin film oven test RTFOT ageing. This study is considered to be of great importance for evaluating rutting deformation. From the acquired results shown in Figure 5, the incorporation of nano-silica as a modifier yields an increase in the elastic properties and the recovery performance of the modified bitumen at elevated temperatures. All of the nano-silica-modified bitumen structures possess a higher percentage of recovery in comparison to the conventional non-modified bitumen.

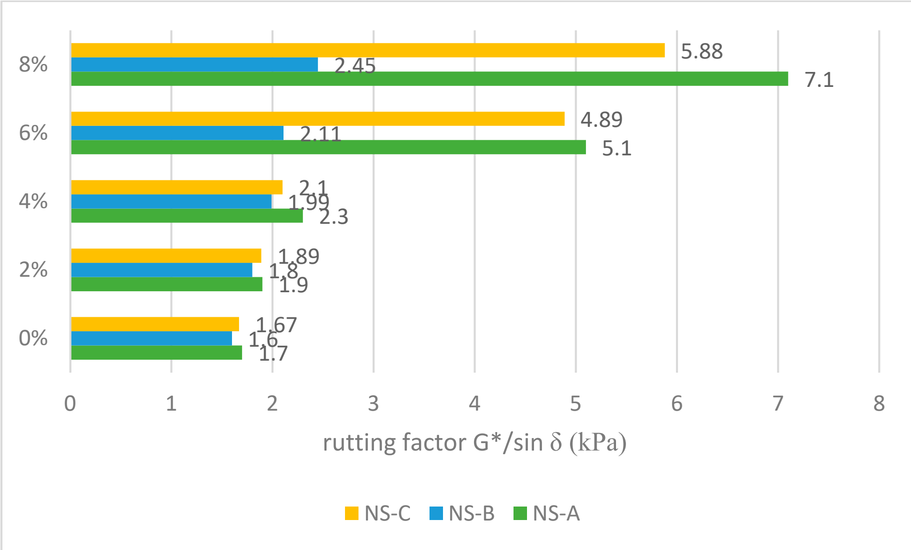


Figure 4. Rutting factor of nano-silica-modified bitumen.

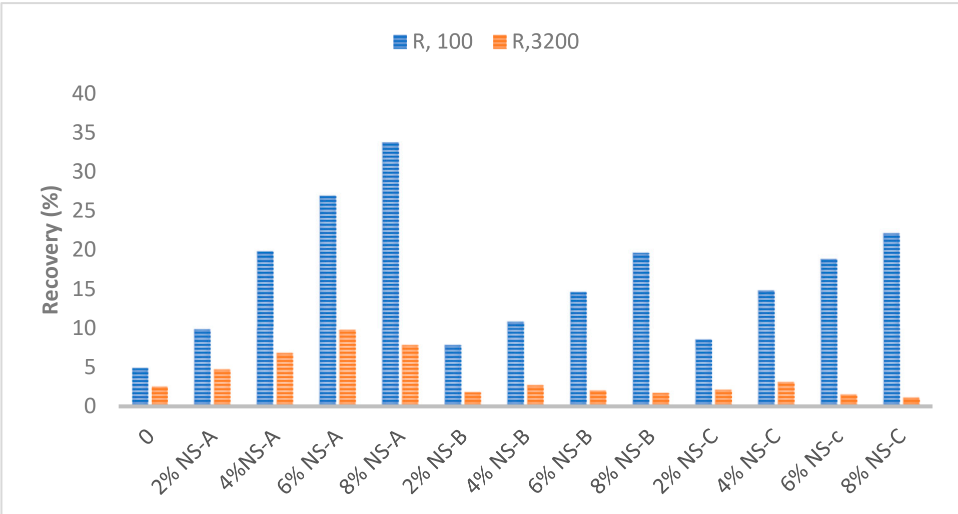


Figure 5. Recovery percentage of nano-silica-modified bitumen.

Based on the results of these experiments, adding the nano-silica significantly increased the rutting resistance of the C320-bitumen-modified mixture. Moreover, using nano-silica as an additive to bitumen samples results confirmed that the addition of the nano-silica improves the rutting resistance, workability, stiffness, and efficiency of the modified bitumen [4,12–16].

Figure 6 depicts the non-recovered strain for nano-silica-modified bitumen C320 and non-modified bitumen with three different nano-silica sizes under the implementation of high- and low-stress levels. Interestingly, most of the modified samples exhibit a decrease in Jnr at high-stress levels. Since non-recovered strain is considered a sensitivity indicator against rutting deformation, it is clear that—in comparison to the non-modified bitumen—all of the nano-silica modified bitumen samples have a lower susceptibility for the accumulation of non-recoverable strain under creep loading and recovery [22,23].

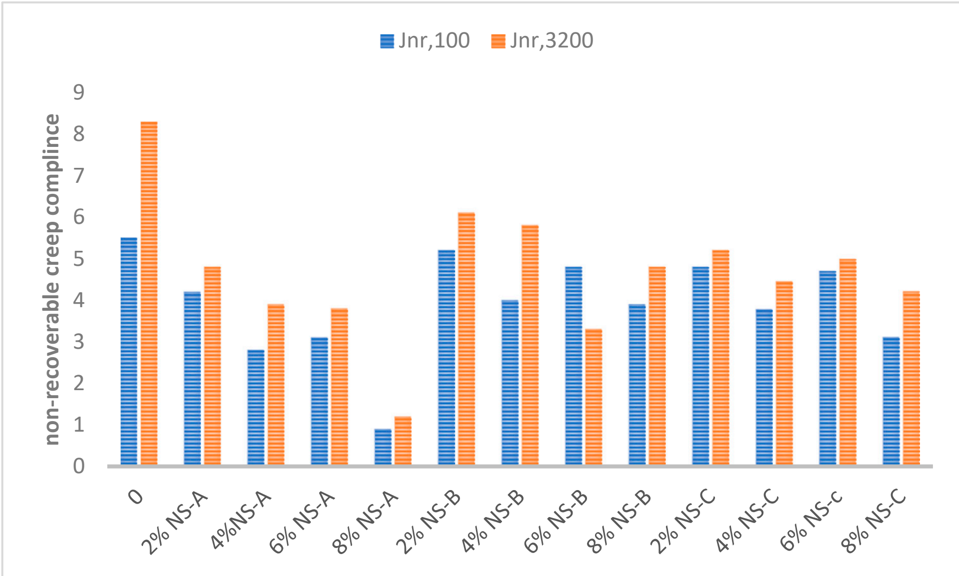


Figure 6. Non-recovered strain for nano-silica-modified bitumen.

5. Conclusions

The findings of the study are as below:

1. The results show an improvement in the resistance to temperature sustainability in terms of the better penetration and the softening points results of the modified samples. Thus, the modified bitumen can significantly improve the stiffness of the modified bitumen and enhance the asphalt mixtures' resistance to rutting deformation.
2. From the results, an increase in the stiffness properties was observed by lowering the penetration. Moreover, the mechanical strength was enhanced, in terms of the complex shear modulus of all nano-silica-modified bitumen samples. The incorporation of relatively high percentages of nano-silica (6% and 8%) led to increased rutting resistance. Nevertheless, the trend followed by the rutting factor was not consistent as the rutting resistance was also affected by the size of the nano-silica coated with the silane coupling agent.
3. The non-recovered strain for the nano-silica-modified bitumen C320 and the non-modified bitumen at three different nano-silica sizes for high- and low-stress levels was found. Most modified samples exhibited a decrease in Jnr under the application of high-stress loads. Therefore, the utilization of NS-A leads to a reduction in the levels of rutting deformation.
4. Based on the results of this work, the implementation of NS of 15 nm coated with the silane coupling agent was selected for further research in the next work on producing the hybrid polymer–nano–silica-modified C320 bitumen. Numerous tests, including rutting, durability, and fatigue, on both binders and asphalt mixtures, will be conducted.

Funding: Research Training Program (RTP 2018).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: The authors would like to acknowledge the contribution of the Australian Government Research Training Program (RTP 2018) in supporting this research project.

Conflicts of Interest: The author declares no conflict of interest.

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