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Application of nano silica particles to improve high-temperature rheological performance of tyre pyrolysis oil-modified bitumen

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Application of nano silica particles to improve high-temperature rheological performance of tyre pyrolysis oil-modified bitumen

Tyre pyrolysis oil (TPO) is considered a well-established alternative to conventional crumb rubber (CR) for bitumen modification due to its lower manufacturing temperature. However, TPO-modified bitumen still has certain drawbacks, such as poor high-temperature rheological properties. Nano silica (NS) has been widely applied due to its unique physical and chemical characteristics in improving the properties of materials, including bitumen. Therefore, this study aimed to evaluate the effects of NS particles on the high-temperature rheological properties of TPO-modified bitumen. Physical tests, short-term aging, temperature sweep test and multiple stress creep recovery (MSCR) test were conducted to evaluate the effects of NS on the high-temperature rheological performance. Results indicate that the inclusion of NS to TPO-modified bitumen displayed significant improvement in rutting resistance before and after aging. Meanwhile, MSCR results showed that the high-temperature performance grade of bitumen was upgraded from PG 64S in the case of base and TPO-modified bitumen to PG 64V at 10% TPO and 4% NS. Statistical analysis also showed that the effects of NS particles on the rutting performance of TPO-modified bitumen are statistically significant within the confidence interval of 95%. These findings present a remarkable enhancement in the rutting performance of TPO-modified bitumen.

Keywords: nano silica; tyre pyrolysis oil; rheological properties; rutting resistance; MSCR; rubber modified bitumen

1. Introduction

Bitumen is a complex viscoelastic material that is considered one of the main essential components of asphalt pavement (Ziari, Amini, Goli, & Mirzaeiyan, 2018). It is commonly used for pavement construction. Asphalt pavement contribute approximately 95% of constructed pavements worldwide. Pavement undergoes various types of distresses over time due to the increasing traffic loads and different environmental conditions (Walubita et al., 2012). Amongst these distresses, rutting and fatigue are the most common and considered two of the main parameters that affect the durability of asphalt pavement (Bernier, Zofka, & Yut, 2012).

The higher performance grade bitumen is mostly recommended to overcome the permanent deformation distress of the bitumen and asphalt mixtures with taking into consideration the workability and sensitivity of the asphalt mixtures for fatigue distresses by controlling the mix design to balance the rutting and fatigue performance of the asphalt mixtures (Walubita, Hoeffner, & Scullion, 2012). Millions of US dollars (USD) have been spent every year to maintain and repair pavement's failures (Shafabakhsh, Sadeghnejad, & Sajed, 2014). At present, researchers are working hard to determine methods for enhancing the properties of bitumen to resist rutting and fatigue, especially with the increase of axial loads, heavy traffic and change in environmental conditions during the last few decades (Ziari, Amini, Goli, & Mirzaeiyan, 2018).

To reduce pavement distresses and improve the physical, chemical and rheological properties of base bitumen, bitumen modification is required using various suitable materials. These enhancements in binder properties result in the extension of the service life of the asphalt pavements (Bala, Napiah, & Kamaruddin, 2018; Miller, Little, Bhasin, Gardner, & Herbert, 2012). Previously, polymer materials were commonly used to enhance the properties of base bitumen, such as thermoplastic plastomers and elastomers (Bala, Napiah, & Kamaruddin, 2018; Yusoff, Breem, Alattug, Hamim, & Ahmad, 2014). It was also reported that the addition of recycled asphalt materials (RAM) into soft bitumen could enhance the permanent deformation and low-temperature cracking resistances of RAM-modified bitumen and mixtures through the role of the RAM in increasing the stiffness of bitumen to prevent the deterioration in form of cracking distress at low-temperatures (Bai et al., 2020; Zhang, Simate, Hu, Souliman, & Walubita, 2017).

In addition, using fillers such as limestone in order to enhance the characteristics of bitumen and asphalt mixtures is another technique that has been commonly used during the last few decades (Kim, & Little, 2004). The effect of filler on the properties of asphalt mastic and mixtures is a function of the texture, particle size distribution, shape, surface area and the physical-chemical interaction between the filler and bitumen, therefore the effects will be different from one filler to another (Clopotel, Velasquez, & Bahia, 2012; Kim, & Little, 2004). As an example of traditional fillers used, limestone showed an enhancement in the rheological properties of asphalt mastic and permanent deformation of asphalt mixtures due to the improvement of the viscosity and stiffness of bitumen (Clopotel, Velasquez, & Bahia, 2012; Faheem, Wen, Stephenson, & Bahia, 2008).

Another study was conducted by comparing cement filler and nano silica as a partial replacement for the filler in asphalt mixtures, the results showed that the asphalt mixtures containing NS filler exhibited significant improvement in the resilient modulus compared to that containing cement filler. It was also reported that both mixtures showed satisfactory improvement in the tensile strength of asphalt mixtures (Hamedi, Sohrabi, & Sakanlou, 2019).

At present, bitumen is being mixed with nanomaterial additives to improve its durability and performance due to their excellent properties, such as high dispersion ability, large surface area, high chemical purity, excellent stability and strong absorption (Bala, Napiah, & Kamaruddin, 2018; Kong et al., 2012; Singh, Karade, Bhattacharyya, Yousuf, & Ahalawat, 2013). Moreover, using nanomaterials with polymers for modification of bitumen is cost-effective as it reduces the quantities of the polymers used, and they also enhances the compatibility of the polymers with bitumen (Bala, Napiah, Kamaruddin, & Danlami, 2017; Yusoff, Breem, Alattug, Hamim, & Ahmad, 2014).

Nano silica is commonly used by industries for producing cement and concrete mixtures and reinforcing elastomer rheological solutions (Bala, Napiah, Kamaruddin, & Danlami, 2017; Yusoff, Breem, Alattug, Hamim, & Ahmad, 2014). It is also applied in bitumen binders as an inorganic filler to improve the performance of bitumen and asphalt mixtures (Ellie H Fini, Hajikarimi, Rahi, & Moghadas Nejad, 2015; Karnati, Oldham, Fini, & Zhang, 2019; Nazari, Naderi, & Nejad, 2018; Taherkhani, Afroozi, & Javanmard, 2017). Nano silica particles have played a promising role in enhancing material characterisations, such as bitumen, due to their high surface area, strong adsorption, excellent stability, high chemical purity and good dispersal ability (Yao et al., 2012b).

Apart from the dimension and shape of NS particles, they are also desirable for bitumen binder applications due to their higher surface area of interaction in the bitumen matrix compared with conventional fillers. Such particles have a relatively highly chemically active surface and polarity (Ellie H Fini, Hajikarimi, Rahi, & Moghadas Nejad, 2015). New studies found that the addition of 2%–4% NS by the weight of base bitumen can reduce rutting by almost 50%. From previous studies, nanomaterial-modified asphalt mixtures showed increased rutting resistance and lower rut depth compared with control mixtures as the amount of nanomaterials in mixtures increased (Yao et al., 2012a; You et al., 2011; Yusoff, Breem, Alattug, Hamim, & Ahmad, 2014). The results indicate that NS will be a suitable choice to be used for enhancing the rutting resistance of bitumen. Therefore, it was used in the present study.

Recycled tyre rubber has become a popular waste material used in various applications, such as civil engineering for bitumen modification (Shu, & Huang, 2014). The wet process of preparing rubberised bitumen was utilised since the 1960s, and rubberised asphalt mixtures are used at different places worldwide to solve various quality problems such as rutting distresses (Hu et al., 2020; Presti, 2013). Approximately 300 million pieces of scrap tyres are produced in the United States every year (Shu, & Huang, 2014). However, most of these waste materials are disposed of in a manner that is unbeneficial to the environment. Only 5.5% of these materials are used in civil engineering applications and 40% for generating energy. Hence, the use of such waste in bitumen applications should be improved, given that they are useful for enhancing the properties of bitumen and asphalt mixtures (Shu, & Huang, 2014).

Waste tyre rubber has a good compatibility with base bitumen due to its high solubility in bitumen especially at the high shear process, making it applicable in asphalt paving industries, such as reclaimed asphalt pavement, warm mix asphalt and bio-asphalt technology (Shu, & Huang, 2014). The compatibility of waste tyre rubber, particularly with bitumen using the wet process, improves the properties and performance of bitumen and asphalt mixtures, such as rutting and fatigue resistance due to increasing of binder stiffness and film thickness and then improving its ductility, which enhances the durability of the asphalt mixture (Mashaan, Ali, Koting, & Karim, 2013; Shu, & Huang, 2014). The use of waste rubber in bitumen modification also reduces environmental pollution, maintenance cost and overall road construction cost (Mashaan, Ali, Koting, & Karim, 2013).

The three most common ways of recycling tyre rubber are pyrolysis, pulverisation and reclamation. Among them, the pulverisation method converts tyre rubber to ground tyre rubber as a powder and manifests the lowest cost amongst the aforementioned three methods. Tyre pyrolysis oil (TPO) has properties that can improve and enhance the performance of asphalt mixture better than crumb rubber (CR) (Wu, Wang, & Dong, 2016). The properties of TPO depend on the rubber sources, type of reactor, processing parameters and pyrolytic conditions and mechanism (Yousefi, Ait-Kadi, & Roy, 2000). The application of light pyrolysis to convert tyre rubber into a liquid elastomer will be an alternative for improving the high- and low-temperature properties of bitumen and asphalt mixtures by using higher percentages compared with those of the conventional rubberised technology (utilizing the rubber in the form of powder) (Wu, Wang, & Dong, 2016) .

Fini et al. (Elham H Fini, Hosseinnezhad, Oldham, & Sharma, 2016) studied the effects of TPO on the aging and rheological characteristics of bitumen. They found that TPO can enhance the aging resistance of bitumen due to the lower changes in modulus values after aging compared with base bitumen. However, the high-temperature properties of TPO-modified bitumen were slightly reduced as the TPO content increased.

The application of TPO as an alternative for CR to overcome the drawbacks of using CR in bitumen, such as poor mix workability and hot storage stability was investigated by (Presti, Izquierdo, & del Barco Carrión, 2018). They found that using TPO as an alternative for CR reduced the usual manufacturing temperature up to 30 °C. The low and intermediate-temperature rheological properties of TPO-bitumen were improved compared with those of base bitumen binders. However, the high-temperature rheological properties were unsatisfactory (Presti, Izquierdo, & del Barco Carrión, 2018). The authors also proposed further investigation of the aging properties of TPO–bitumen.

Therefore, the present study aims to investigate the effects of NS on the high-temperature rheological properties of TPO–modified bitumen in unaged and rolling thin film oven (RTFO)-aged conditions in order to enhance the rutting performance. This study also aims to explore the ability of NS particles to enhance the incorporation of TPO as a partial replacement for bitumen. TPO is used as an extender for bitumen at concentrations of 0%, 5%, 10% and 15% by the weight of base bitumen. Whereas, the NS was used at concentrations of 2%, 4% and 6% by the weight of base bitumen.

2. Materials and methods

2.1 Materials

2.1.1 Bitumen

The bitumen used in this study was obtained from the PETRONAS Refinery Melaka, Malaysia. The bitumen has a 60/70 penetration grade. The other specific characteristics of the base bitumen are presented in Table 1.

Table 1. Characteristics of the base bitumen

Test	Standard	Standard limit		Results
		Min.	Max.	
Penetration at 25 ºC, 0.1mm	ASTM D5	60	70	60
Softening Point, ºC	ASTM D36	49	56	49
Ductility at 25 ºC, cm	ASTM D113	100	-	+ 100
Specific gravity at 25 ºC	ASTM D70	-	-	1.03
Viscosity (Pa.s) at 60 ºC	ASTM 4402	-	-	0.863
Flash point, ºC	ASTM D92	230	-	283
Fire point, ºC	ASTM D92	-	-	> 310
Mass loss, RTFO, %	ASTM2872	-	1	0.12
Penetration Index (PI)	-	-	-	-1.04

2.1.2 Nano silica

The physical and chemical properties of the NS materials used in this study are presented in Table 2. Nano silica was supplied by Benua Sains Chemical Sdn Bhd Malaysia. Figure 1(a) shows the physical appearance of the NS used in this study. A high-shear mixer was used for mixing at a temperature of 160 °C with 4000 rpm for 2 h to ensure good dispersion of the NS particles in base bitumen matrix. The selected NS amounts of 2%, 4% and 6% by the weight of bitumen were used in this experimental study, this selection was based on the preliminary study and previous literature (Han et al., 2017).

Table 2. Characteristics of the nano silica particles used in this research

Property	Value
Appearance	High dispersive white
Hydrophobicity	Strong hydrophobicity
SiO2 content (%) (950 ºC, 2h)	99.8
Purity (%)	> 99.9
Loss of ignition (%)	≤ 6
Surface density (g/ml)	0.15
Average particle size	10-25
PH value	6.5 – 7.5
Specific surface area (m^2/g)	100 ± 25

2.1.3 Tyre pyrolysis oil (TPO)

The tyre pyrolysis oil used in this study is the sludge or liquid rubber produced from the pyrolysis process of conversion of scrape tyre to bio-gas in the bio-fuel manufactures. The TPO is technically produced from the vacuum pyrolysis of waste tyre scrap and during the vacuum pyrolysis, the thermochemical changes taking place occur, while the waste rubber particles are heated. After that the waste tyre scraps were broken down into its different component, the liquid part was removed and the pasty residue was collected separately. This was used in this study as tyre pyrolysis oil. TPO was obtained from Tyre Oil (M) Sdn. Bhd Manufacturer, Perak, Malaysia. The characteristics of TPO are shown in Table 3. While Figure 1(b) exhibits the TPO physical appearance. The concentrations of TPO used are 0%, 5%, 10% and 15% by the weight of base bitumen as an extender.

(a) (b)

Figure 1. (a) Nano silica (b) Tyre pyrolysis oil.

Table 3. Characteristics of the TPO used in this research

Property	Value
Appearance	Thick liquid with a dark colour
Viscosity at 60 ºC and 10 s^{-1} shear rate, Pa.s	11.9
Viscosity at 60 ºC and 100 s^{-1} shear rate, Pa.s	1.713
Carbon, %	78.77
Hydrogen, %	7.967
Nitrogen, %	1.105
Sulfur, %	0.0305
Others, %	12.13

2.2 Methods

2.2.1 Preparation of materials

A high-shear mixer was used for blending samples in this study. The mixing procedure has been identified on the basis of literature review and trial and error method. The trials were carried out in accordance with the method of Han et al. (Han et al., 2017) to fix the procedures for blending NS and TPO with base bitumen 60/70. Base bitumen was heated to 160 °C and certain amount of TPO was added gradually. Rubber was allowed to be absorbed in bitumen for 10 minutes. The NS powder was then added into the mixture, and the final blend was sheared at 4000 rpm for 2 hours at 160 °C.

On the basis of the above-mentioned procedure, 16 blends were prepared at 0%, 2%, 4% and 6% of NS and 0%, 5%, 10% and 15% of TPO by the weight of base bitumen. The composite materials were designed using response surface methodology (RSM) software which is well known for experimental design. The identifiers (IDs) of bitumen blends prepared in this study are presented in Table 4. Three replicates of each blend for physical properties tests were prepared and tested and the average results were reported. Similarly, at least two replicates for rheological properties tests of each blend for each of the aging conditions (before and after aging) were prepared and tested.

Table 4. IDs of bitumen blends used in this research

Blend types	IDs
Control bitumen	RMB 00
98% base bitumen mixed with 2% NS by weight	RMB 02
96% base bitumen mixed with 4% NS by weight	RMB 04
94% base bitumen mixed with 6% NS by weight	RMB 06
95% base bitumen mixed with 5% TPO by weight	RMB 50
93% base bitumen mixed with 5% TPO and 2% NS by weight	RMB 52
91% base bitumen mixed with 5% TPO and 4% NS by weight	RMB 54
89% base bitumen mixed with 5% TPO and 6% NS by weight	RMB 56
90% base bitumen mixed with 10% TPO by weight	RMB 100
88% base bitumen mixed with 10% TPO and 2% NS by weight	RMB 102
86% base bitumen mixed with 10% TPO and 4% NS by weight	RMB 104
84% base bitumen mixed with 10% TPO and 6% NS by weight	RMB 106
85% base bitumen mixed with 15% TPO by weight	RMB 150
83% base bitumen mixed with 15% TPO and 2% NS by weight	RMB 152
81% base bitumen mixed with 15% TPO and 4% NS by weight	RMB 154
79% base bitumen mixed with 15% TPO and 6% NS by weight	RMB 156

2.2.2 Physical properties tests

Penetration grade and softening point temperature are the most common empirical tests to evaluate the consistency of the bitumen (Bai et al., 2020; Hu et al., 2020). Penetration and softening point tests were carried out on base bitumen as a control specimen, TPO-modified bitumen, NS-modified bitumen and NS-TPO-modified bitumen. The penetration indexes (PIs) for all specimens were also calculated and analyzed on the basis of the data collected from the test. The penetration test was conducted in accordance with ASTM D5 (ASTM D5, 2013). The softening points of the specimens were measured in accordance with ASTM D36 (ASTM D36, 2014).

The changes in the consistency of the bitumen as a function of temperature can be defined as a temperature susceptibility. Changing the temperature susceptibility of the TPO-modified bitumen with NS was investigated by calculating the PI. The PI values can vary between -3 and 7, the lower the PI value of bitumen, the higher its temperature susceptibility. In another word, the bitumen with PI value of 7 is non-susceptible to temperature compared to the bitumen with PI value of -3 which is highly susceptible to temperature (Arabani, & Tahami, 2017). The PI was calculated using the softening point results and penetration at 25 °C. Equation 1 was used to calculate the PI.

$$PI = \frac{1952 - 500 * Log\ Pen - 20\ SP}{50 * Log\ Pen - SP - 120} \tag{1}$$

where *Pen* is the penetration at 25 °C and *SP* is the softening point in °C.

2.2.3 Rolling thin film oven (RTFO)

Aging of the bitumen is one of the serious issues that causes a pavement deterioration and reduction in its service life (A. M. Al-Sabaeei, Napiah, Sutanto, Alaloul, & Usman, 2020; Walubita et al., 2012). Therefore, all bitumen binders prepared in this study were aged using RTFO in accordance with ASTM D2872 (ASTM D, 2012) to simulate the loss of volatiles of hot mix asphalt during the production and construction in the field. The control, TPO-modified and NS-TPO composite binder samples were kept in an oven at a temperature of 163 °C for a time period of 85 min. The binder was exposed to continued air flow. The aged binders obtained from the RTFO were utilized for rheological property investigation tests.

2.2.4 Rheological properties tests

2.2.4.1 Dynamic shear rheometer (DSR) test

The DSR test was developed to characterise the viscoelastic properties of bitumen binders over a wide range of temperature (Jahromi, & Khodaii, 2009; Walubita et al., 2012; Walubita, Hoeffner, & Scullion, 2012). A Kinexus Malvern Instrument Rheometer was used for rheological analysis in accordance with AASHTO T 315(AASHTO T, 2012). A 25 mm plate and 1 mm gap geometry were used for testing. The effect of separate TPO and NS and NS–TPO composite on rheological parameters complex modulus (G*) and phase angle (δ) was obtained. The rutting resistance factor G*/sin δ at the standard frequency of 1.59 Hz (10 rad/s) was then calculated to evaluate the performance at high temperature. The testing temperatures were 40 °C, 46 °C, 52 °C, 58 °C, 64 °C, 70 °C and 76 °C to evaluate the high-temperature performance of the NS–TPO-modified bitumen. The DSR test was conducted on unaged and RTFO-aged samples to investigate the effects of short-term aging on the rheological properties of modified bitumen.

2.2.4.2 Multiple stress creep recovery (MSCR) test

The MSCR test was also carried out using DSR equipment in accordance with AASHTO T350 standard. The testing temperature used was 64 °C, the maximum work temperature of base bitumen was PG 64, which is equivalent to 60/70 penetration grade. All the binders were subjected to RTFO aging before the MSCR test. The diameter and thickness of the specimen were 25 and 1 mm, respectively. A 1 second shear creep load was applied on the specimen, followed by a 9 seconds recovery. The first 10 cycles under 0.1 kPa were used for conditioning of the sample followed by another 10 cycles of creep and recovery under same shear load. Another 10 cycles were applied on the same sample under the shear load of 3.2 kPa.

The nonrecoverable creep compliance (J_{nr}) is the rutting resistance index from the MSCR test, whereas percent recovery predicts the elastic properties of bitumen (Zhang, Simate, Hu, Souliman, & Walubita, 2017). Both factors were investigated to evaluate the effect of composite NS and TPO on the high-temperature performance of bitumen. J_{nr} and percent recovery were expressed using Equations 2 and 3 as follows:

$$J_{nr}= \frac{Nonrecoverable\ strain}{Stress\ level} \qquad (2)$$

$$Perecent\ recovery = \frac{Recovered\ strain}{Maximum\ strain} \tag{3}$$

2.2.5 Design of Experiments and Statistical Analysis

User-defined design (UDD) is one of the most common design methods used with RSM for evaluating the mathematical relationship between the independent and dependent (responses) variables. In this study, the UDD approach was applied with four levels by using Design-Expert software version 10.0.8. The independent variables considered in this study were TPO and NS content. The responses considered were G*/sinδ (kPa) at 64 °C (unaged), G*/sinδ (kPa) at 64 °C (aged), J_{nr} at 0.1 kPa (kPa^{-1}), J_{nr} at 3.2 kPa (kPa^{-1}), R% at 0.1 kPa (%) and R% at 3.2 kPa (%). The selection of the independent variables and their experimental range was based on an existing literature review and a preliminary study (Ellie H Fini, Hajikarimi, Rahi, & Moghadas Nejad, 2015; Elham H Fini, Hosseinnezhad, Oldham, & Sharma, 2016). Sixteen experiments were performed on each of the responses on the basis of the RSM design. At least two specimens' replicates were prepared for each experimental blend per test.

Analysis of variance (ANOVA) was conducted to evaluate the interactions between the independent parameters and the appropriateness of the model. ANOVA measures and confirms the suitability of the selected models and the significances of each variable factor. The fitness of the actual experimental data to the selected models was also assessed by using the coefficient of determination (R^2). Fisher's F test was also applied to ensure the probability (P value) within the typical 95% confidence level. The application of RSM for conducting ANOVA analysis and Fisher's F test used in this study is based on reported literature that applied RSM for the same purpose (A. Al-Sabaeei, Napiah, Sutanto, Alaloul, & Ghaleb, 2020; Del Barco Carrión, Subhy, Rodriguez, & Presti, 2020)

3. Results and discussion

3.1 Physical properties

3.1.1 Penetration

Bitumen penetration indicates the degree of hardness and consistency of bitumen. Figure 2 illustrates the penetration of the control, TPO-modified, NS-modified and NS-TPO composite bitumen specimens at 25 °C. It can be seen that the penetration values slowly decrease with the increase of NS dosages in the base bitumen. However, the penetration values decreased with the addition of TPO up to 5% and then increased. The composition of NS with TPO at different dosages rapidly decreased the penetration values, which implies that the NS–TPO composite can greatly improve the hardness and consistency of the bitumen binders. In contrast, the separate addition of TPO or NS slightly improved the bitumen hardness. It can be also noted that the lowest penetration value was found at 5 % TPO and 6 % NS which is about 70 % lower compared to control bitumen penetration value. This reduction in penetration values is considered the primary indicator of the improvement of the intermediate temperature resistance of the NS–TPO-modified bitumen.

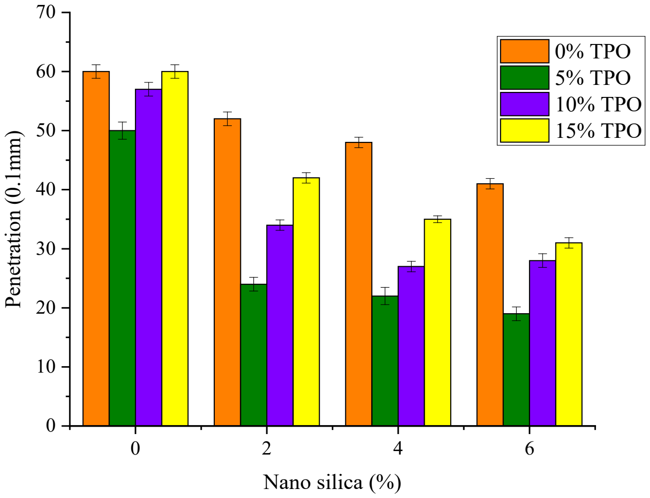

Figure 2. Penetration values of control and NS-TPO composite bitumen.

3.1.2 Softening point

Figure 3 presents the relationship between the softening point values and different NS-TPO composite bitumen groups. The plot shows that the softening point value increases with increasing NS content regardless of the presence or absence of TPO, particularly for binders contain 0% and 5% of TPO. However, the softening point values decrease with the addition of TPO into the bitumen without NS, this could be attributed to the reduction in the viscosity of bitumen with increasing the TPO content. The softening point values of the NS–TPO composite were higher than those of the TPO-modified specimens. Additionally, the softening point value improved with the addition of NS to 15% TPO–bitumen. Such value exceeded the softening point of base bitumen. This finding indicates that NS is a useful additive to improve the viscosity of TPO-modified bitumen and incorporation of high TPO content in conventional bitumen decreases the softening point temperature.

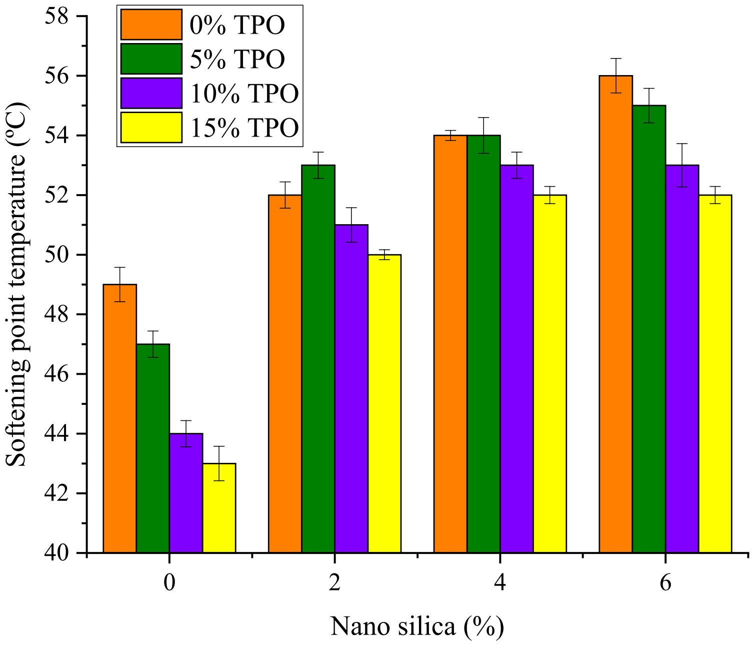

Figure 3. Softening point values of control and NS-TPO composite bitumen.

3.1.3 Penetration Index (PI)

PI is used to indicate the temperature susceptibility and flexibility of bitumen. As the PI increases, the flexibility of bitumen increases. PI can also represent the types of colloid bitumen. When the PI is greater than +2, the bitumen is considered a gel structure; when the PI is lower than -2, the bitumen is considered a sol structure; others can be considered gel–sol structures (Roberts, Kandhal, Brown, Lee, & Kennedy, 1991). Figure 4 shows the influences of NS, TPO and NS–TPO composite on the PI of base bitumen 60/70. It is apparent that the PI of the TPO-modified bitumen increases with the addition of TPO. This situation indicates that the TPO could improve the elasticity and temperature susceptibility of bitumen. The temperature susceptibility decreases with the increase of NS content in base bitumen. In the case of the NS–TPO composite, the PI value decreases as the TPO increases for the same amount of NS. This result indicates that the composite NS–TPO-modified bitumen most likely will performs well at high-temperature environments.

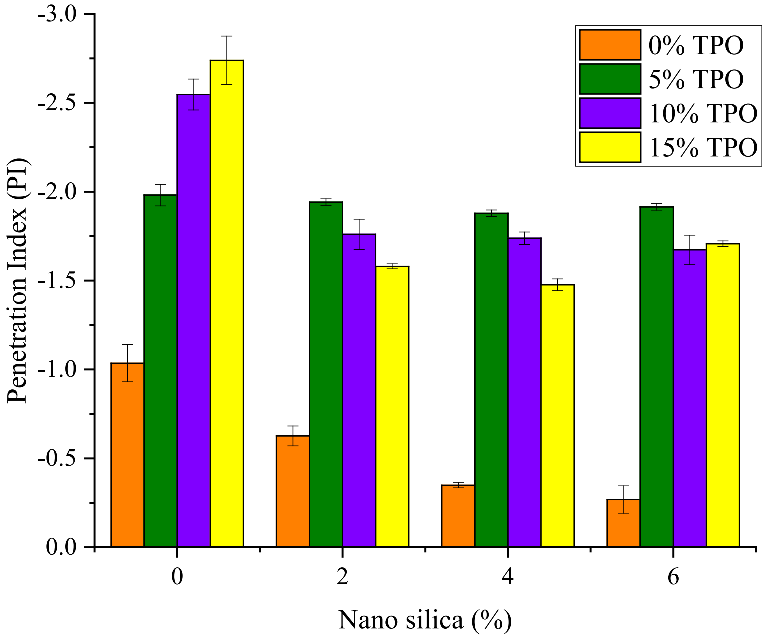

Figure 4. Penetration Index values of control and NS-TPO composite bitumen.

3.2 Rheological properties

3.2.1 Rutting resistance factor ($G^*/\sin\delta$) of unaged binders

The rutting resistance factor (G*/sinδ) has been identified as a good indicator to predict the rutting performance of the base and modified bitumen binders. The G*/sinδ of the unaged control, NS-modified, TPO-modified and NS–TPO composite bitumen binders were plotted in Figure 5 based on the complex modulus (G*) and phase angle (δ) at 10 rad/s. The effect of NS on the rutting parameter of the TPO-modified bitumen was analyzed. It was observed that the rutting resistance factors for control and all modified binder groups reduced with increasing test temperature from 40 °C to 76 °C. However, in contrast, various binders reveal different rutting resistance factors at specific testing temperatures. The base and TPO-modified bitumen showed lower rutting resistance at all test temperatures compared with the NS-modified and NS-TPO composite bitumen. This can be due to the high concentration of aromatic in TPO which leads to reduction in the viscosity of TPO-modified bitumen and also the high shear susceptibility of TPO can be another reason for the reduction of rutting resistance as the content of TPO increases (Elham H Fini, Hosseinnezhad, Oldham, & Sharma, 2016). However, the rutting resistance of the NS-TPO composite bitumen was significantly improved at 4% and 6% of NS. This can be attributed to the high surface area, high molecular weight and specific gravity of NS that leads to enhance the interaction of NS particles in the bitumen matrix which in turn improves the viscoelastic properties and rutting resistance of the bitumen (Ellie H Fini, Hajikarimi, Rahi, & Moghadas Nejad, 2015).

According to the performance grade specification, the minimum G*/sinδ for the base bitumen is 1 kPa. The failure temperatures of the base and modified binders were determined. The failure temperature of the control bitumen was observed at 64.2 °C. The higher failure temperature amongst the TPO-modified bitumen for RMB 50 was obtained at 69.92 °C. The NS-TPO composite bitumen yielded a significant improvement in terms of high-failure temperature amongst all binder groups in this study. The high-failure temperature was 77.12 °C for RMB 104. Such value improved by 12.92 °C and 7.2 °C compared with the high-failure temperature of the control and unmodified TPO-bitumen, respectively. Figure 6 shows the rutting parameters of all binders at a specified temperature (64 °C) to demonstrate the improvement due to the addition of NS to the TPO-modified bitumen. The above-mentioned results showed that the NS particles could enhance the high-temperature performance of TPO-modified bitumen at the unaged binder state. This situation could be attributed to the ability of NS in improving the TPO shear resistance to enhance the high-temperature rheological properties of TPO-modified bitumen and achieve compatibility of TPO in bitumen. These results are in agreement with those of Alhamali et al. (Alhamali et al., 2016).

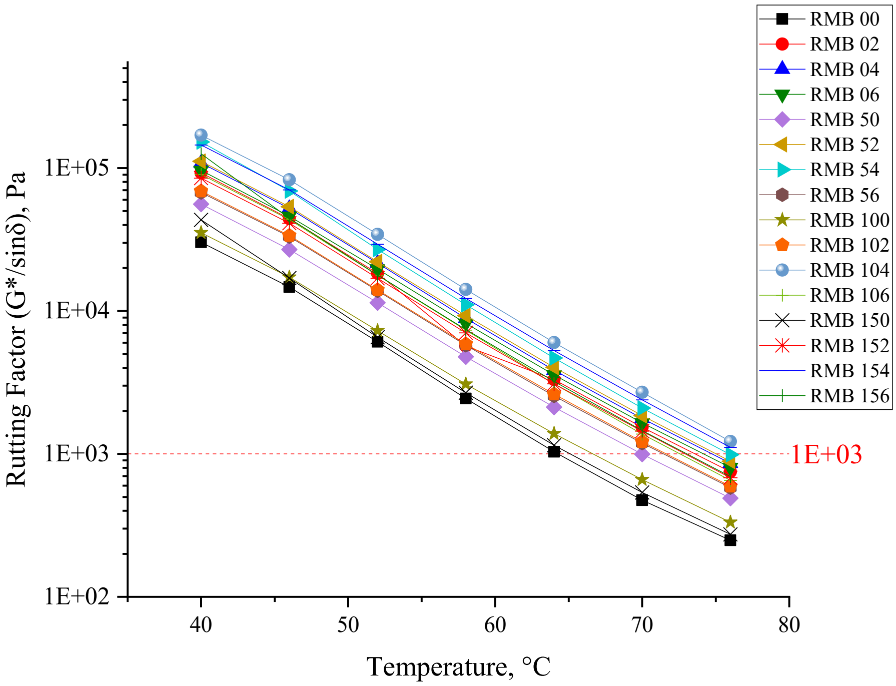

Figure 5. Rutting parameters of unaged control and NS-TPO composite bitumen.

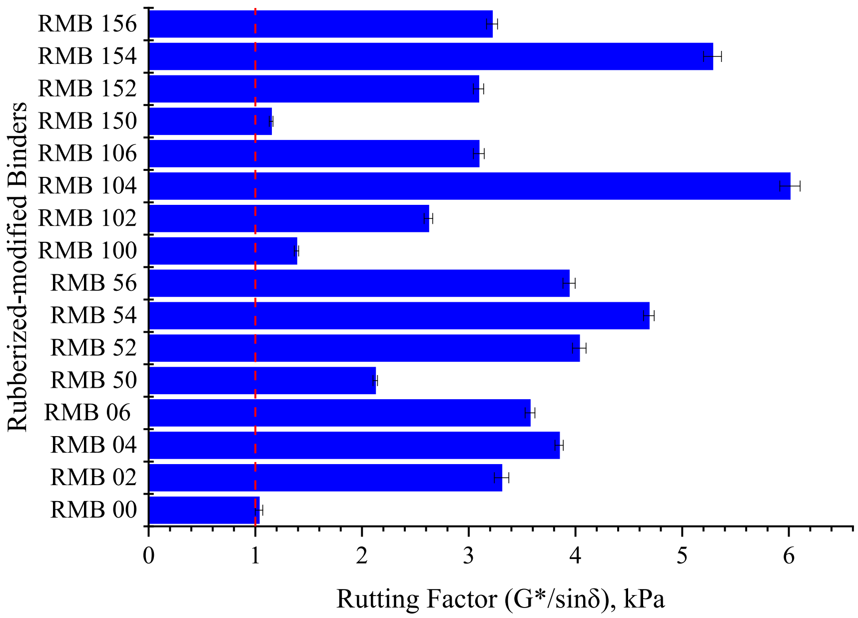

Figure 6. Rutting parameters of unaged control and NS-TPO composite bitumen at 64 °C.

3.2.2 Rutting resistance factor (G/sinδ) of aged binders*

Figures 7 and 8 show the rutting parameters of base bitumen, NS-modified bitumen, TPO-modified bitumen and NS–TPO composite bitumen after short-term aging. It can be seen that the rutting resistance factors of the control and modified bitumen binders decreased with the increase in temperature from 40 °C to 76 °C. The NS-modified and NS–TPO composite bitumen binders have higher rutting resistance at all testing temperatures compared with the base and TPO-modified bitumen. However, the rutting resistance of the NS–TPO composite bitumen was significantly enhanced at 4% of NS. This enhancement can be attributed to the NS particles reinforcement for the base and TPO-modified bitumen binder's structure by increasing the bond and enhancing the cohesion of bitumen. Another reason could be a results of the clumping in the NS particles or the filler settlement in the bitumen matrix at the high content of NS (Karnati, Oldham, Fini, & Zhang, 2019; Li, Xiao, Amirkhanian, You, & Huang, 2017).

The failure temperatures of the aged control and modified bitumen binders in this study were determined for comparison with the rutting parameter minimum of 2.2 kPa according to the Superpave specification. The failure temperature of the base bitumen was obtained at 66.79 °C. Nevertheless, the highest failure temperature amongst the TPO-modified bitumen for RMB 50 was at 70.25 °C. This finding is in good agreement with that before short-term aging as discussed above. The NS-TPO composite bitumen showed the highest failure temperature amongst all binders tested in this study. The failure temperature for RMB 104 was 76.32 °C, showing an improvement of 9.53 °C and 6.07 °C in the high-temperature performance grade compared with the base and TPO-modified bitumen binders without NS, respectively. Therefore, NS has a significant influence on the improvement of the high-temperature performance of RTFO-aged base and TPO-modified bitumen binders. These findings are in agreement with the results of the rutting resistance of unaged NS-modified bitumen as discussed above.

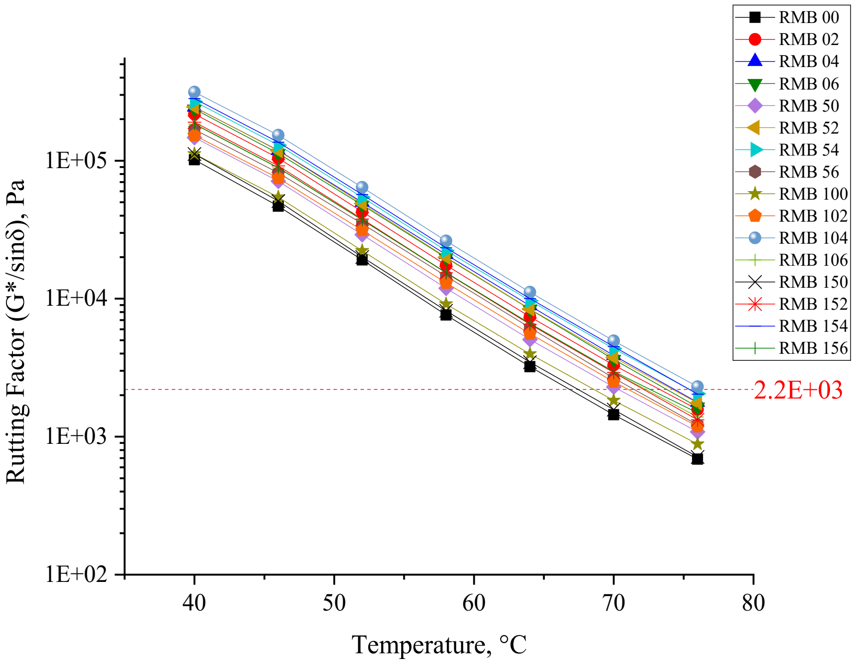

Figure 7. Rutting parameters of aged control and NS-TPO composite bitumen binders.

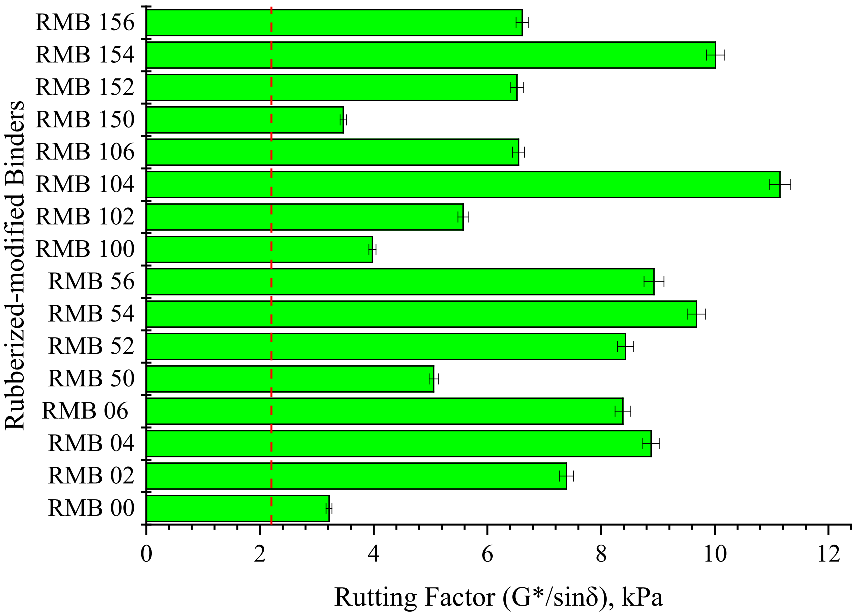

Figure 8. Rutting parameters of aged control and NS-TPO composite bitumen binders at 64 °C.

3.2.3 Multiple stress creep recovery (MSCR)

Figures 9 and 10 present the average of 10 creep (1 s) and recovery (9 s) cycles of the base bitumen, TPO-modified bitumen, NS-modified bitumen and NS–TPO composite bitumen binders at shear stress levels of 0.1 and 3.2 kPa, respectively. It is seen that the actual strain increased with the increase in loading time at the creep phase. All the TPO-modified bitumen binders have lower actual strain compared with the control bitumen at 0.1 and 3.2 kPa shear stresses. The actual strain was also increased with the increase in TPO content. However, the addition of NS particles to the TPO-modified bitumen caused a significant reduction in the creep strain, which can be attributed to the ability of NS particles to increase the creep recovery at the end of each cycle. These findings are in agreement with those reported in previous literature (Ghanoon, & Tanzadeh, 2019). The least amount of creep strain was observed for the bitumen modified with 10% TPO and 4% NS at 0.1 and 3.2 kPa shear stresses which represent the high creep resistance of NS-TPO composite bitumen at mentioned TPO and NS content. During the recovery phase, the actual strain quickly recovered at the beginning, but the recovery rate decreased with time for all binders tested in this study. The immediate recovery of the elastic strain and gradual recovery of the viscous strain reflect the viscoelastic properties of the rubber materials and bitumen binders. The actual strain of the binders at the 3.2 kPa creep level is higher than that at 0.1 kPa. Hence, the maximum strain increases with the increase in creep level.

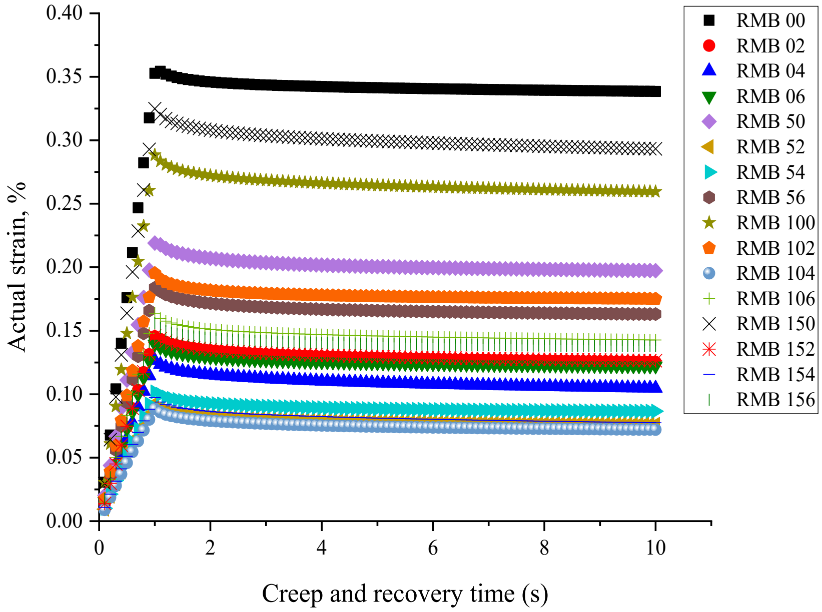

Figure 9. Creep and recovery curves of unmodified and NS-TPO composite bitumen binders at 0.1 kPa.

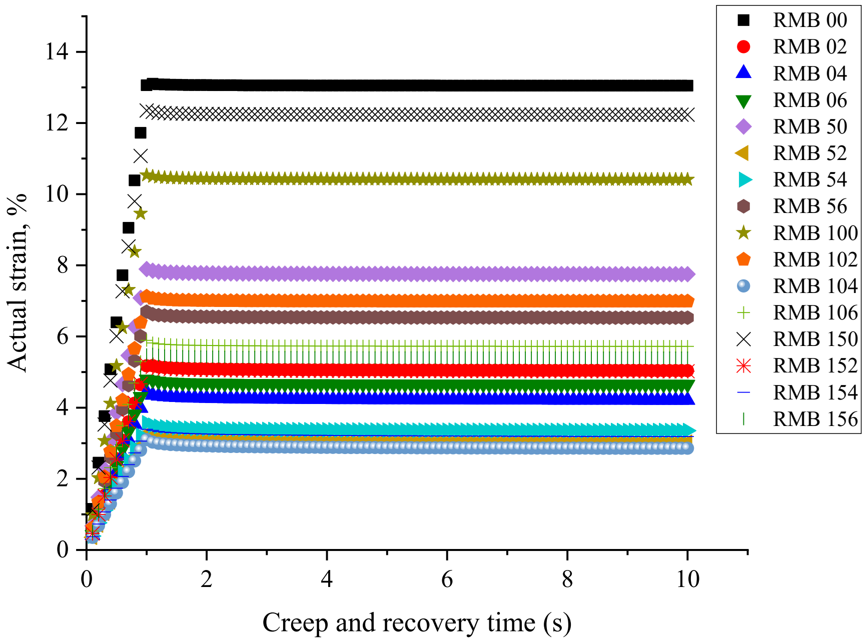

Figure 10. Creep and recovery curves unmodified and NS-TPO composite bitumen binders at 3.2 kPa.

Figures 11 and 12 present the effects of TPO, NS and NS–TPO composite on the J_{nr} of bitumen at 0.1 and 3.2 kPa shear stresses, respectively. In general, all modified bitumen binders had lower J_{nr} compared with the control bitumen at both stress levels. At 5% TPO, J_{nr} reduced and then began to increase with the increase of TPO content in bitumen. This finding can be attributed to the sensitivity of TPO to shear stress. The rutting resistance sensitivity of TPO was increased in similar trend to that found in the above-mentioned complex modulus and rutting parameter. These findings are in agreement with what was found in the TPO viscosity characteristics at different shear rates that have been evaluated in this study. Another study reported a similar trend of the sensitivity of TPO to shear stress with respect to the different TPO characteristics applied in that work (Elham H Fini, Hosseinnezhad, Oldham, & Sharma, 2016). The J_{nr} of the NS-modified bitumen was reduced with the increase in NS content. Such situation could be attributed to the high surface area and stability of the NS particles that have high chemical interaction in the bitumen matrix (Ellie H Fini, Hajikarimi, Rahi, & Moghadas Nejad, 2015).

However, the 4% NS showed the least J_{nr}, which is in agreement with the viscosity, complex modulus and rutting parameter results from this study; the slight reduction in J_{nr} beyond 4% is consistent with the findings in literature (Al-Omari, Taamneh, Khasawneh, & Al-Hosainat, 2018; Arshad, Samsudin, Masri, Karim, & Halim, 2017; Yao et al., 2012b) with respect to the different NS particle characterizations and mixing processes in bitumen. The slight reduction can be attributed to the new structure of bitumen formed during bitumen mixing due to the chemical reactions and physical dispersion of NS particles (Enieb, & Diab, 2017; Yao et al., 2012b). This situation will lead to the improvement of asphalt mixture workability and reduce the mixing and compaction temperatures toward lower energy consumption.

The NS-TPO composite bitumen showed lower J_{nr} compared with the TPO-modified bitumen that is unmodified with NS particles and control bitumen at the 0.1- and 3.2-kPa shear stress levels. These results are consistent with the rutting parameters found from the DSR test. Within the same TPO content, the J_{nr} value of the NS-TPO composite bitumen reduced with the increase of the NS particles due to the improvement in the stiffness of the TPO-modified bitumen as NS content increase. However, the 4% NS showed a low reduction in the TPO-modified bitumen. Most TPO-bitumen binders modified with NS particles showed $J_{nr} < 2.0$ kPa^{-1} compared with the TPO-modified bitumen and control binders that are > 2 kPa^{-1}. This notion indicates that the high-temperature performance grade was improved from the standard grade in the case of control bitumen to high traffic loading grade with the maximum replacement of the conventional bitumen of 15% TPO. The TPO-modified bitumen modified with 10% TPO and 4% NS showed a low J_{nr} value of 0.91 kPa^{-1}, thereby leading to the improvement of the performance grade of the control bitumen from standard to heavy traffic loading grade. RMB 154 also showed a J_{nr} of 1 kPa^{-1}, which is considered very heavy traffic loading grade according to AASHTO designation of J_{nr} at the stress level of 3.2 kPa (AASHTO M332, 2014). These findings could be an indicator of a significant effect of NS particles on the improvement of the rutting performance of the base and TPO-modified asphalt mixtures.

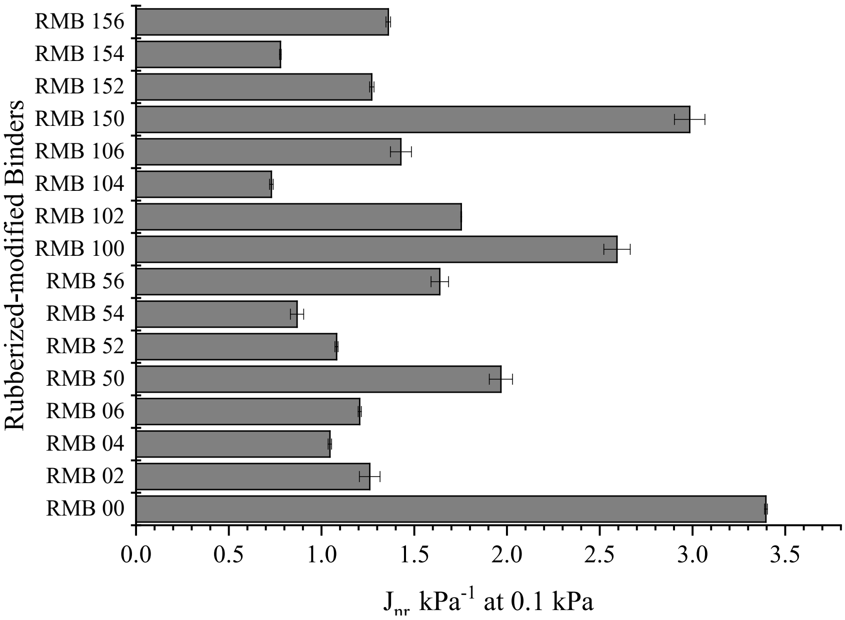

Figure 11. Nonrecoverable creep compliance (J_{nr}) at 0.1 kPa.

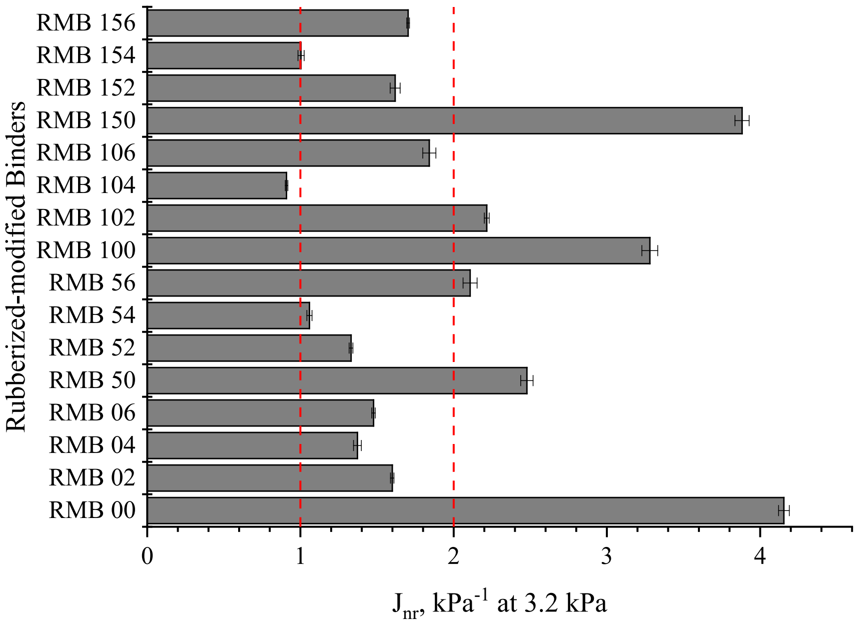

Figure12. Nonrecoverable creep compliance (J_{nr}) at 3.2 kPa.

The creep recovery percentage (R%) is an important parameter to assess the stiffness of bitumen against rutting because it is an indicator of the delay elasticity of bitumen. The higher the R%, the better the bitumen elastic performance (Mirsepahi, Tanzadeh, & Ghanoon, 2020). Figures 13 and 14 show the mean values of the recovery percentages of the control, TPO-modified, NS-modified and NS-TPO composite bitumen binders at 0.1 and 3.2 kPa shear stress, respectively. The modified binders with NS, TPO and NS–TPO present higher R% compared with the control bitumen. The R% of TPO-modified bitumen decreases as the TPO content increases. This occurrence could be a result of the low shear resistance of the TPO materials, which could be due to the depolymerization of rubber particles at the high temperature during the production of tyre pyrolysis oil, which means the polymer chains of rubber breakdown into small molecules (Shu, & Huang, 2014). An improved R% can be observed in the NS-TPO composite bitumen binders with the increase in NS content compared with that of the control and TPO-modified bitumen binders. This result indicates that the bitumen sensitivity to stress decreases with the increase in NS content up to 4%. The NS particles present a significant improvement in the percentage of the recovery of TPO-modified bitumen binders. High R% values were obtained for RMB 154 and 104 at 0.1 and 3.2 kPa shear stresses, respectively. These slight differences may indicate the sensitivity of TPO to the applied shear stress.

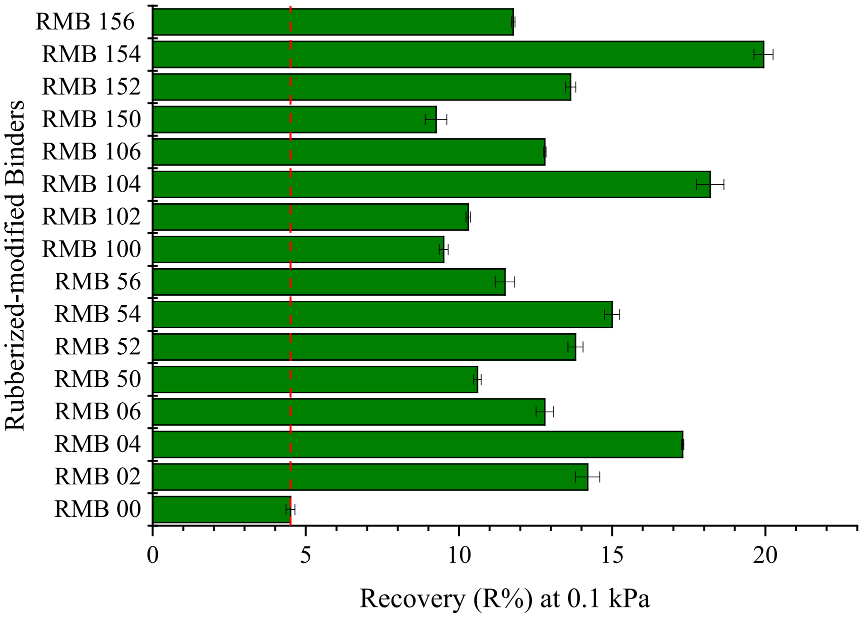

Figure 13. Percent recoverable strain (R%) at 0.1 kPa.

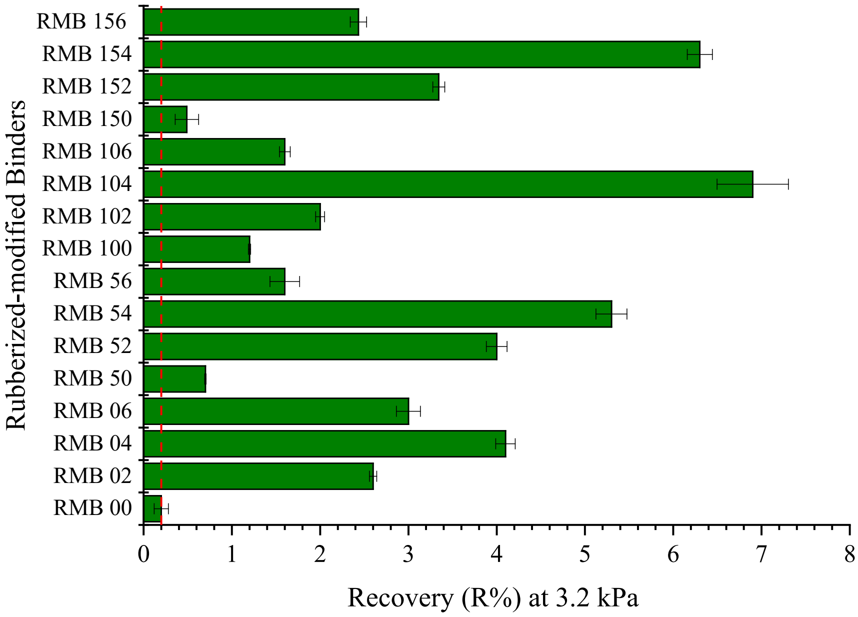

Figure 14. Percent recoverable strain (R%) at 3.2 kPa.

The sensitivity of the control and modified bitumen binders to the applied stress can be measured by J_{nr} diff. The J_{nr} diff values are the relative differences between the J_{nr} at 3.2 and 0.1 kPa shear stress. Table 5 shows the J_{nr} diff for all the control and modified binders. The J_{nr} diff values must be < 75% in accordance with the standard requirements (AASHTO M332, 2014; Mirsepahi, Tanzadeh, & Ghanoon, 2020). It can be seen that all binders have J_{nr} diff of < 75%. This finding indicates that all binders in this study met the standard requirements and are not sensitive to stress. A high $J_{nr,\ diff}$ value was obtained at RMB 150, which indicated the sensitivity of TPO to stress compared with those of the control and NS-modified binders. However, the sensitivity of the TPO-modified bitumen generally reduced with the increase in NS content in the blend and addition of NS particles.

Table 5. $J_{nr,\ diff}$ data for control and modified bitumen binders

Binder ID	$J_{nr\ @\ 0.1\ kPa}$ (kPa^{-1})	$J_{nr\ @\ 3.2\ kPa}$ (kPa^{-1})	$J_{nr,\ diff}$ (%)	Stress sensitivity (Meet specifications)
RMB 00	3.396	4.155	22.3	YES
RMB 02	1.26	1.599	27	YES
RMB 04	1.045	1.372	31.2	YES
RMB 06	1.206	1.477	22.4	YES
RMB 50	1.967	2.478	26	YES
RMB 52	1.081	1.33	23	YES
RMB 54	0.8681	1.059	22	YES
RMB 56	1.638	2.107	28.6	YES
RMB 100	2.593	3.28	26.5	YES
RMB 102	1.753	2.216	26.4	YES
RMB 104	0.7298	0.9091	24.6	YES
RMB 106	1.428	1.841	28.9	YES
RMB 150	2.985	3.882	30.07	YES
RMB 152	1.271	1.618	27.29	YES
RMB 154	0.7782	1.004	29	YES
RMB 156	1.36	1.703	25.24	YES

3.3 Statistical analysis results

Table 6 summaries the statistical analysis results of the rutting parameters before and after aging and the MSCR results of the 16 RMB binders tested in this study. The table shows that the P-values for all responses are < 0.05. This result indicates that the effects of NS particles and TPO on the high-temperature rheological properties of bitumen are statistically significant within the confidence interval of 95%. A correlation coefficient (R^2) was used to check the degree of correlation amongst the dependent and independent variables. All responses except R% at 0.1 kPa had R^2 values of > 0.8. This result indicates approximately 80% of changes in the high-temperature rheological properties of RMB binders in this study because of the effect of NS and/or TPO.

Table 6. Analysis of variance results summary

Parameter	Sum of squares	df	Mean square	F-value	P-value	R^2
G*/sinδ (kPa) @ 64 °C (Unaged)	3.69	3	1.23	18.06	< 0.0001	0.82
G*/sinδ (kPa) @ 64 °C (Aged)	1.77	3	0.59	21.15	< 0.0001	0.84
$J_{nr\ @\ 0.1\ kPa}$ (kPa^{-1})	2.61	3	0.87	20.89	< 0.0001	0.84
$J_{nr\ @\ 3.2\ kPa}$ (kPa^{-1})	2.60	3	0.87	19.29	< 0.0001	0.83
$R\%_{@\ 0.1\ kPa}$ (%)	3.32	3	1.11	9.74	0.0015	0.71
$R\%_{@\ 3.2\ kPa}$ (%)	5.01	5	1	9.93	0.0012	0.83

4. Conclusions

This study evaluated the conventional and high-temperature rheological properties of NS, TPO and composite NS–TPO-modified bitumen through empirical, DSR and MSCR tests. The following conclusions can be drawn on the basis of the test results and data analysis:

- The results of physical tests showed that the addition of NS particles to TPO-modified bitumen led to a decrease in the penetration values as well as temperature susceptibility and an increase of the softening point temperature, which indicates the possibility of utilizing TPO as a partial replacement for conventional bitumen modified with the NS.

- The incorporation of NS into the TPO-modified bitumen enhanced the high-temperature rheological characteristics in terms of complex shear modulus and rutting parameters before and after aging. This result indicates a significant improvement in the rutting resistance and compatibility of TPO with base bitumen. Such a situation could lead to the enhancement of the rutting performance of asphalt mixtures with NS–TPO-modified bitumen.
- The base and TPO-bitumen binders modified with NS particles showed a great reduction in the actual strain at 0.1 and 3.2 kPa shear stress compared with the control base bitumen. Such a result this indicates a significant improvement in the creep strain resistance of bitmen due to NS addition. This reduction in the creep strain can be attributed to the ability of NS particles to increase the creep recovery of the base and TPO-modified bitumen.
- The addition of NS particles to the base and TPO-modified bitumen revealed a potential decrease in J_{nr} and increase in R% up to 4% NS at 0.1 and 3.2 kPa shear stresses. The study found that 15% of the base bitumen can be substituted by TPO with the addition of 4% NS to enhance the high-temperature performance grade from PG 64S in the case of base bitumen to heavy traffic grade (PG64H). The best permanent deformation resistance of bitumen, where the performance grade reached the PG64V was found at 4% and 10% of NS and TPO respectively, which could be recommended as the optimum. This result indicates the positive effects of NS particles on the incorporation of TPO into base bitumen to enhance the high-temperature performance. It is also considered a remarkable contribution to environmental pollution reduction and the management of rubber waste.
- Statistical analysis presented a degree of correlation (R^2) > 0.8 and P-value < 0.05 for the G*/sinδ before and after aging, J_{nr} at 0.1 and 3.2 kPa and R% at 3.2

kPa, which indicates that the effects of NS on the rheological properties of the base and TPO-modified bitumen are statistically significant within the confidence interval of 95%.

- The proposed optimum combination of TPO and NS was evaluated in the laboratory level in terms of high-temperature performance-related properties for asphalt pavement applications in the tropical regions. It is expected that the developed composite binder will be cost-effective as a result of energy consumption reduction compared to bitumen modified with conventional crumb rubber.
- Further studies are recommended to investigate the performance of developed modified binders with NS/TPO composite at the level of asphalt mixtures, evaluate the environmental impacts and conduct life cycle cost analysis. It is also recommended that a combination of nano silica with traditional filler such as limestone be evaluated for the promising modified asphalt mixtures.

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Disclosure statement

The authors declare that they have no conflict of interest.

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