

Research Article

The Influence of Nano Titanium as Bitumen Modifier in Stone Mastic Asphalt

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Nanomaterials are emerging as one of the methods to improve the pavement industry. Due to that, this study explores the influence of nano TiO₂ modified binder and mixture with 60/70 grade bitumen. The first stage discovers the physical properties of bitumen by undergoing penetration and softening point tests. Then, the nano TiO₂ modified binder is evaluated in terms of its morphological and chemical properties. To verify the modified binder design, the mechanical performance of stone mastic asphalt (SMA) is assessed in terms of volumetric properties. From the results, 5% nano TiO₂ modified binder shows significant improvement in terms of penetration and softening point. The results also show that the 3% nano TiO₂ modified binder contributes the highest improvement in terms of its chemical properties and the morphological properties. Based on the morphological properties' evaluation, FTIR and XRD observed a sharp peak of nano TiO₂ while the bitumen containing nano TiO₂ is well distributed when SEM-EDX was applied. In addition, the volumetric properties of SMA also significantly improve with the addition of nano TiO₂. It can be said that the addition of nano TiO₂ is able to enhance the overall properties of bitumen.

1. Introduction

Stone mastic asphalt has widely been used in countries such as Unites States, Europe, and Asian countries like Malaysia [1]. The need to use SMA is due to the increasing traffic users over the years which make SMA a suitable choice since it is known to withstand high loads [2]. SMA originated from Germany only consists of coarse aggregates and bitumen as a filler in which the mix is held by the strong elastic properties of the binder [3]. However, with modernisation and new technology introductions, the SMA has been incorporated with many materials in such ways that the materials can improve the SMA properties. A modification to the mixture is one of the methods introduced. Some suggested changes include adding materials as an additive, rejuvenators, or even stabilizers. The focus would be on the aftereffect of the

mechanical effect that the materials have on the SMA, but binder modification has not been further researched in depth [4].

Nanomaterials are chemical compounds or products that are processed and used on a very small scale. The size of nanomaterials is between 1 to 100 nanometres (nm) [5]. They previously have been used in the healthcare industry, beauty products, and biology-related field, and it is priorly used in construction also before embarking to the pavement industry [6]. Nanomaterials have many types of applications depending on their respective type and size. Some of the commonly used nano materials that are always encountered are nano clay, nano silica, nano-sized metals such as copper, zinc, and aluminium oxide [7]. Aging is a concern of asphalt mixtures, and there are two types of it which are thermal oxidative aging and photo oxidative aging [8]. This is where

the surface modified nanomaterials such as nano silica, nano zinc oxide, and nano TiO_2 act as they could improve the antiaging behaviour of asphalt [9].

Nano clay is naturally occurring minerals, and its purity could affect the final nanocomposite properties. Due to their low cost of production, it is commonly used as it has potential to improve mechanical and thermal binder properties [10]. Nano silica properties such as its good stability, affordable cost, high surface area, chemical purity, and good dispersing ability have made it one of the most used materials over the years in improving the mechanical performance of asphalt such as aging, fatigue, and cracking [11].

Among the mentioned nanomaterials, nano TiO_2 was chosen due to its properties such as higher reactivity with bitumen due to its small size [12]. It possesses photocatalytic properties which could help in eliminating hazardous gases like nitrogen monoxide [13]. This promotes the environmentally friendly area of the asphalt pavement. Nano TiO_2 also improve the asphalt aging properties [14]. Furthermore, nano TiO_2 could reduce binder drain down properties by increasing the adhesion between aggregates and bitumen when added into the mix as the nano particles fill the voids that is present and make the structure more compact [15].

This study is aimed to investigate the binder modification by incorporating it with nano titanium and its effect to the SMA. Different percentages of nano TiO_2 starting from 1 to 5% will be mixed with the bitumen and will further be accessed. Among the test that will be done on the samples would be the traditional bitumen test which are penetration test and softening point. Then, the sample would be further analysed in terms of morphology and chemical aspects by Fourier transform method analysis (FTIR), scanning electron microscope (SEM-EDX), and X-ray diffraction analysis (XRD). The volumetric properties of SMA are also investigated after the nano TiO_2 modified bitumen is included in the SMA mix. The test results show that 3% nano TiO_2 modified bitumen has improved the bitumen in terms of its physical properties, rheology, morphology, and chemical characteristics. The volumetric properties of SMA also experience positive enhancement due to the binder modification.

2. Materials and Methods

2.1. Nano Titanium. The sizes of nano TiO_2 are between 10 to 15 nanometres and in white powder form supplied by the local supplier. Table 1 shows the properties of nano TiO_2 used for experimental works. Based on Table 1, the usage of nano TiO_2 is suitable for bitumen modification due to its high melting and boiling point as well as its structure.

2.2. Bitumen. The bitumen currently utilised has a 60/70 penetration grade. Table 2 shows the properties of the asphalt binder used in the experimental works.

2.2.1. Preparation of Nano TiO_2 Modified Binder. A mechanical mixer was used to prepare the nano TiO_2 modified binder at 1500 rpm and 160°C for 60 minutes. A

TABLE 1: Properties of nano TiO_2 .

Properties	Descriptions
Colour	White
Purity	99.9%
Primary particle size	20 nm
Structure	Anatase, Rutile, Brookite
Melting point	1843°C
Boiling point	2972°C
Relative density at 25°C	4.26 g/cm^3

predetermined amount of bitumen was obtained and put in the oven for approximately 1.5 to 2 hours until it melted. About 500 g of bitumen was then weighted before mixing with 1 to 5% nano TiO_2 . The mixture was mixed for 30 minutes while adding the nano TiO_2 gradually and further 30 minutes of additional mixing to ensure its homogeneity.

2.3. Experimental Methods

2.3.1. Stone Mastic Asphalt (SMA) Sample Preparation. SMA samples were prepared according to the Malaysian standard specification JKR/SPJ/S4, by incorporating 6.16% optimum binder content [16] and compacted using a Marshall compactor with 50 blows on each face. Figure 1 represents the aggregate grading used to fabricate asphalt mixture specimens. Sieve analysis was performed to obtain the required aggregated mass based on the targeted specification. The SMA aggregate grading is shown in Figure 1.

2.3.2. Morphological Tests for the Bitumen

(1) Fourier Transform Infrared (FTIR) Spectroscopy. Fourier Transform infrared (FTIR) spectroscopy is an analytical technique used to identify organic, polymeric, and, in some cases, inorganic materials. The technique uses infrared light to scan test samples and observe chemical properties. The qualitative analysis identifies characteristic absorption peaks for the functional group of interest, such as polymer components, carbon, and sulphur oxidation products [17]. In this study, the attenuated total reflectance Fourier-transform infrared (ATR-FTIR) spectroscopy was used to evaluate the modified binder. The sample was prepared by cooling off the bitumen-nano TiO_2 mixture before being compacted into a small sphere and placed on a small microscopic slide.

(2) X-Ray Diffraction (XRD). X-ray diffraction or XRD is a nondestructive test method used to analyse the structure of crystalline materials. XRD analysis identifies crystalline phases present in a material, revealing chemical composition information. X-rays are directed toward samples by applying the voltage, and reflected X-rays are gathered by the detector [18]. The phase identification of bitumen was analysed using XRD at $[\theta]$ theta. Samples were prepared by cooling the bitumen mixture and made into small solid spheres. XRD graph peaks were produced after the testing was finished.

TABLE 2: Properties of 60/70 grade bitumen.

Properties	Descriptions	Material used
Specific gravity @25°C	1.01/1.06 kg/cm ³	1.03
Penetration @25°C	60–70 mm	65
Softening point @25°C	49–56°C	53
Ductility @25°C	100 cm	100

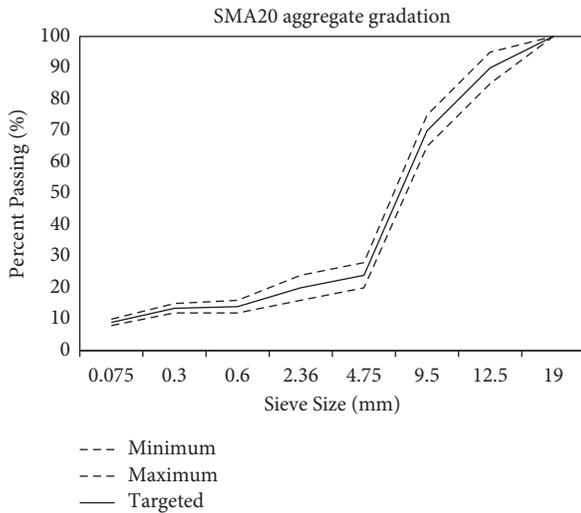


FIGURE 1: The SMA aggregate grading.

2.3.3. Scanning Electron Microscopy with Energy Dispersive X-ray Analysis (SEM-EDX). Scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) provides detailed high-resolution images of the sample by reprojecting a focused electron beam across the surface and detecting secondary or backscattered electron signal. Sample preparation involves cooling the bitumen until it hardens before scrapped into a small portion and observed under the microscope at desired magnifications. Further analysis using EDX was performed to identify the elements present in bitumen samples.

2.3.4. Physical Tests for Bitumen

(1) **Softening Point.** Bitumen was melted and poured into a pair of rings placed on a plate covered with Vaseline. After the specimen cooled, the ring was suspended in distilled water at $5 \pm 2^\circ\text{C}$ for 15 minutes. Steel balls were also placed on the surface of the bitumen in the ring. Then, the bath liquid was stirred and heated at a rate of $5 \pm 2^\circ\text{C}$ per minute. A thermometer was placed in the middle of the ring holder, levelled with the bottom of the ring. When the ball passed and dropped into the base plate, the temperature was recorded.

(2) **Penetration Test.** Specimens were prepared in a sample container and placed in a water bath at room temperature for 1 to 1.5 hours before the test. Then, the penetrometer dial reading was set to zero. The penetration needle was cleaned

and fixed into the holder and guide. For a normal test, the precisely dimensioned needle was loaded to 100 ± 0.05 g. Next, the needle was slowly lowered until the tip makes contact with its image on the sample's surface at right angles. The needle holder was released to penetrate the bitumen for 5 ± 0.1 s at a constant temperature of $25 \pm 0.1^\circ\text{C}$. The penetration was measured in millimetre (mm).

2.3.5. Volumetric Properties of SMA. This test was conducted on eight samples with two samples each for 0%, 2%, 3%, and 4%. Before testing, the samples were conditioned by placing them in a water bath at a temperature of 60°C for 30 minutes. Then, the samples were placed on a Marshall stability machine and subjected to a force sufficient to break the sample to determine the Marshall Stability. Volumetric properties obtained such as density, stability, flow, and stiffness were recorded.

The following tests were conducted according to the flowchart provided Figure 2.

3. Results and Discussion

3.1. Fourier Transform Infrared Spectroscopy. Figure 3(a) shows the IR spectrum of the original bitumen experiencing a N-H stretching at 3395.75 cm^{-1} . This peak is observed at the $3100\text{--}3500\text{ cm}^{-1}$ region which it could be seen that it appears as a wide and broad-shaped spectrum with medium strength. The spectrum then becomes longer and shorter as more nano TiO_2 is added. The two peaks at 2923.75 cm^{-1} and 2852.09 cm^{-1} which later appears are related to the C-H symmetric stretching (-CH₂) and the C-H asymmetric vibrations (-CH₃ and -CH₂) due to the presence of hydrocarbon chain segments that usually exist in the bitumen. Figure 3(b) shows some of the functional groups for the bitumen.

Next, from Figure 3(c), it shows that all of the nano TiO_2 modified bitumen have almost the same peaks, but at 3% nano TiO_2 modified bitumen, there is a new absorption bond that is observed. That is when nano TiO_2 is included the original region of $3100\text{--}3500\text{ cm}^{-1}$ which then makes the N-H bonds does not appear anymore. It is instead replaced with the two peaks at the $2851\text{--}3100\text{ cm}^{-1}$ region which are 2923.14 cm^{-1} and 2851.96 cm^{-1} . These peaks appear as there are interactions between the C-H and O-H bonds of the nano titanium with the bitumen mix; these bonds are stretching with medium strength. The results are consistent with a study by Lin et al.

Going into the 1700 cm^{-1} region, a remarkable peak at 1700.49 cm^{-1} is observed at the carboxylic region indicating that oxidation happens in this region and carbonyl group (C=O) is present. Next, the two peaks observed at 1462.37 cm^{-1} and 1376.67 cm^{-1} are assigned to the O-H bond which experiences bending with medium strength. In which this 1376.67 cm^{-1} peak is a response to the phenol group. Lastly, the 721.20 cm^{-1} peak is due to the presence of C double bond (C=C) in the nano TiO_2 modified bitumen sample.

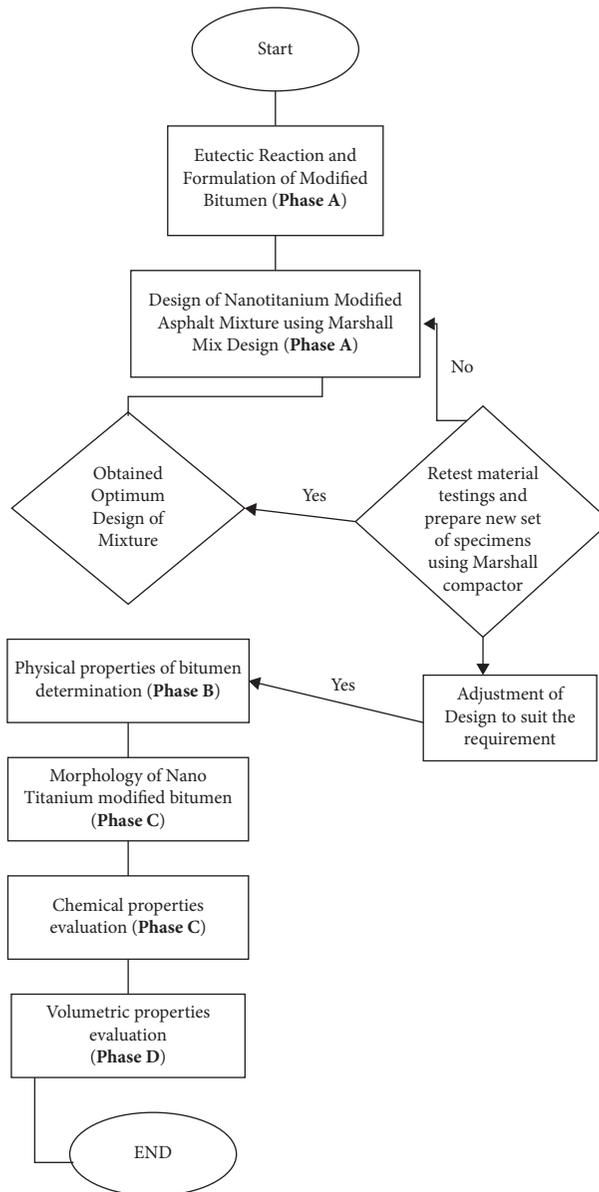


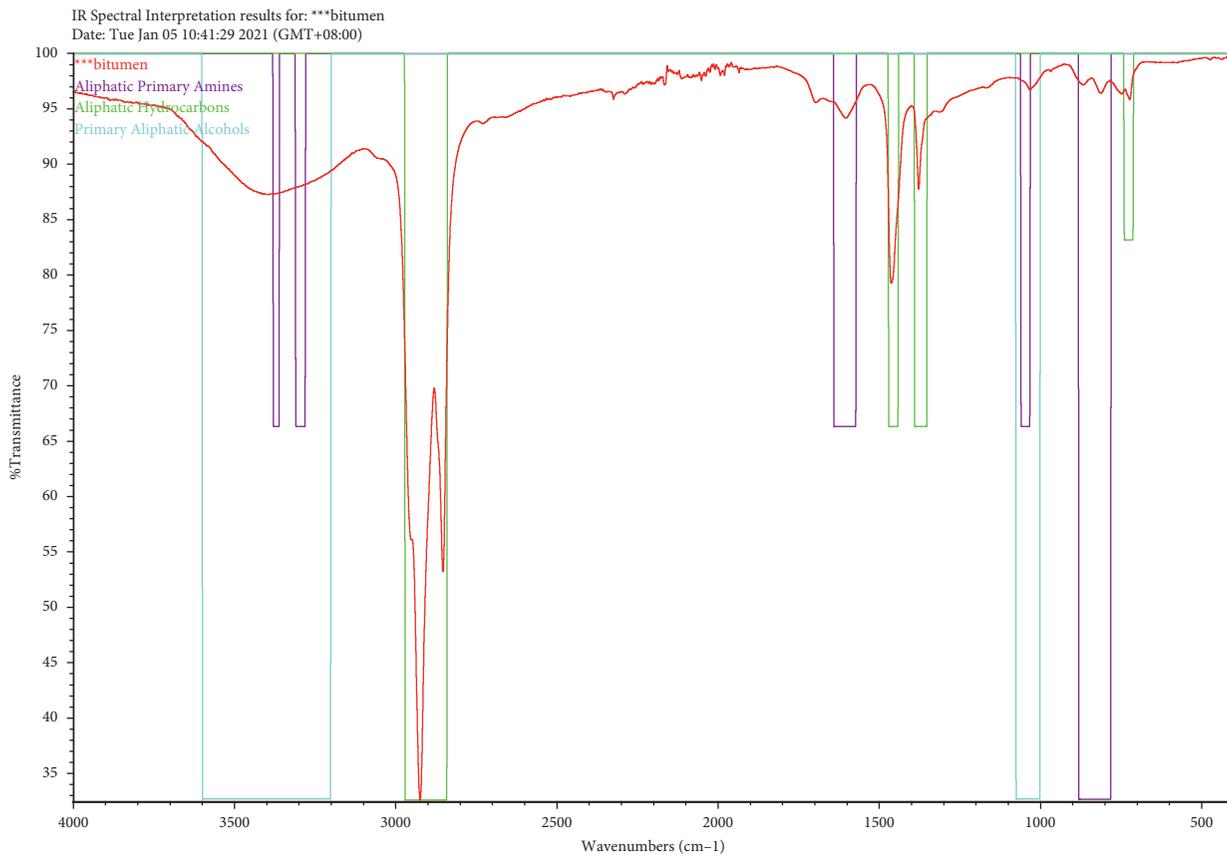
FIGURE 2: Flowchart of the research approach.

3.2. *X-Ray Diffraction (XRD)*. Figures 4 and 5 depict the result of XRD on the unmodified and modified bitumen with nano TiO_2 sample. A close check on the nano TiO_2 powder is also done revealing that the nano titanium is of crystallised structure since sharp peaks are produced along the 25–75-degree peaks. The peak for the virgin bitumen is observed between 20–25 degree which has the peak 23.96 and 26.27 degrees as these peaks are due to the aliphatic chains and layers of condensed saturated rings since it is plain bitumen and is completely amorphous.

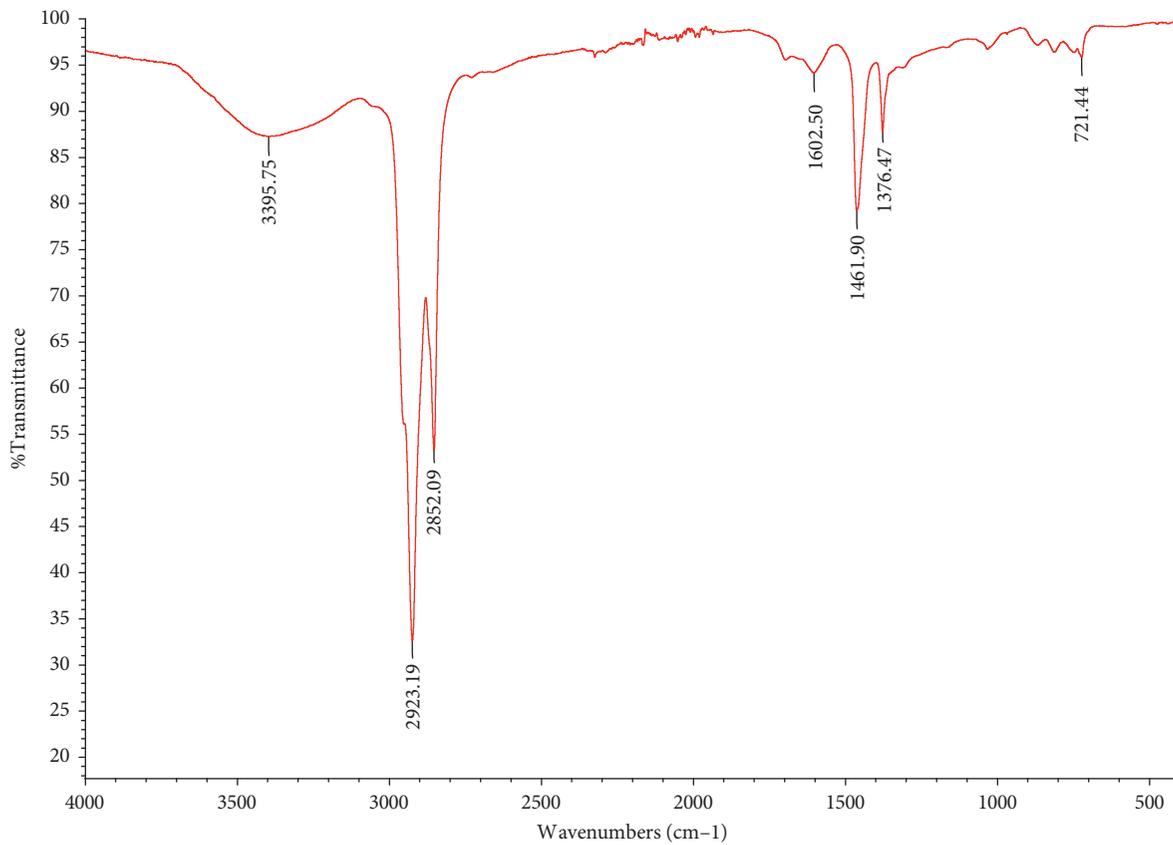
After the addition of nano TiO_2 in the bitumen, all of the samples show that the peak intensity rises, and this indicates that the nano TiO_2 particles have successfully incorporated themselves into the bitumen since the starting peak is higher than the starting peak for the original bitumen. The result trend is consistent with a study by Razavi and Kavussi [18].

The highest starts from 2%, followed by 3%, 5%, 4%, and 1%. But only 2% and 3% show that the peak intensity continues to develop up until 60 and 63 degrees, respectively. 3% is chosen as the bitumen continues to develop furthest with the influence of nano TiO_2 . This shows that a little amount of nano TiO_2 such as 2% is able to contribute to the inner structure improvement of virgin bitumen.

3.3. *Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis (SEM-EDX)*. Figure 6 shows the morphology of the bitumen both before and after being added with nano titanium powder. Again, SEM analysis is performed to see the condition of the nano titanium when mixed with bitumen. For the unmodified bitumen, the black stripe is the bitumen in the liquid state, and since the sample is semisolid,



(a)



(b)

FIGURE 3: Continued.

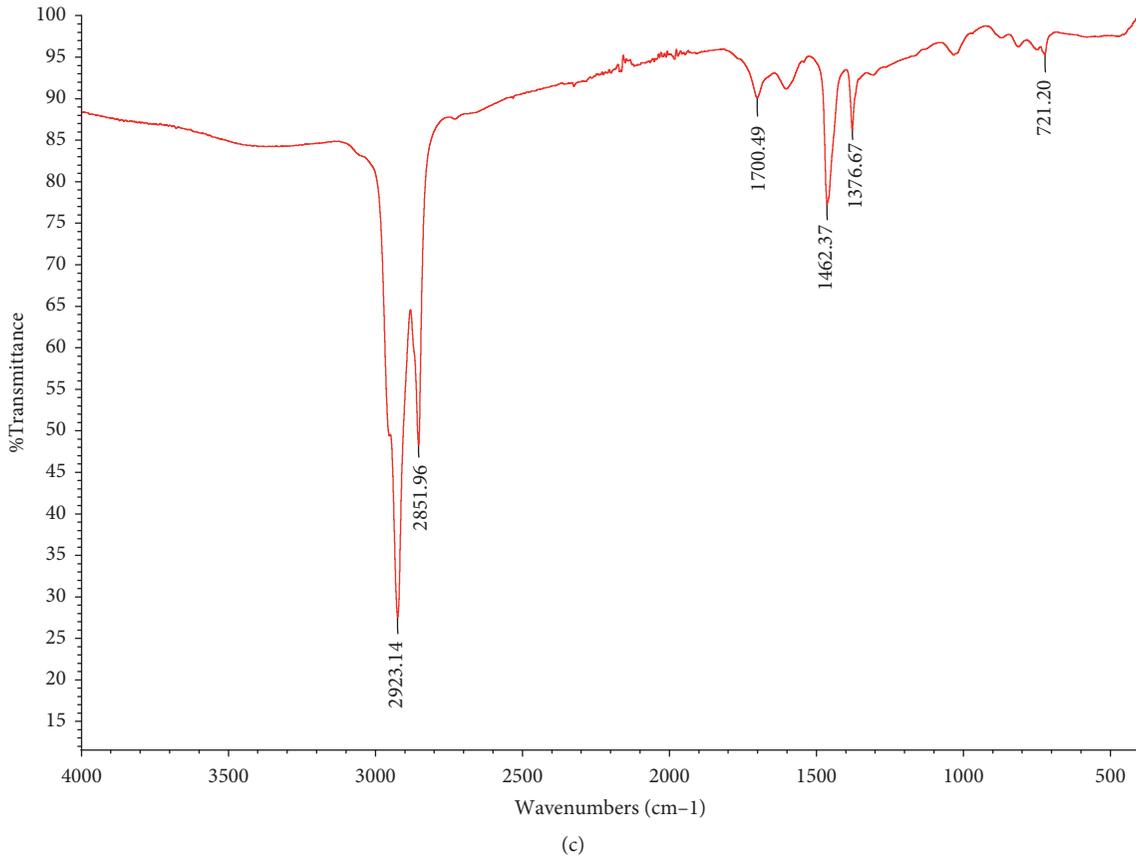


FIGURE 3: (a) Transmittance peaks of unmodified bitumen. (b) Transmittance (%) vs. wavenumber (cm⁻¹) spectral interpretation of unmodified bitumen. (c) Transmittance peaks of 3% nano TiO₂ modified bitumen.

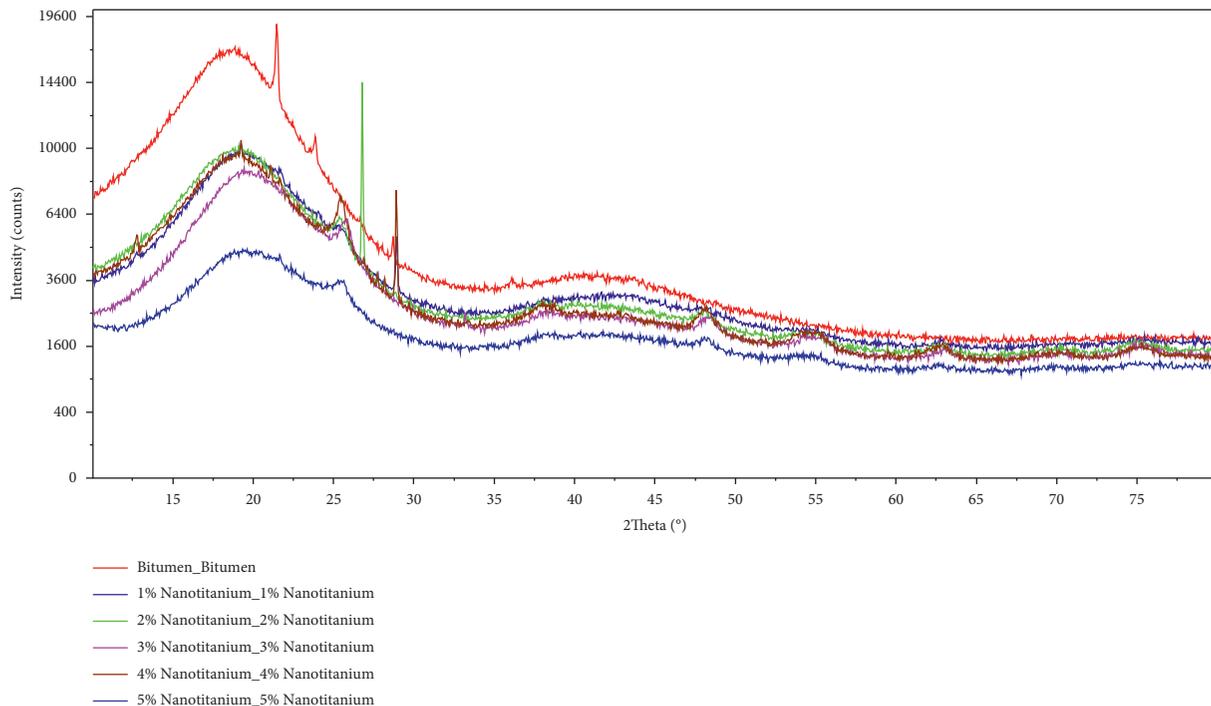


FIGURE 4: XRD for nano TiO₂ modified binder.

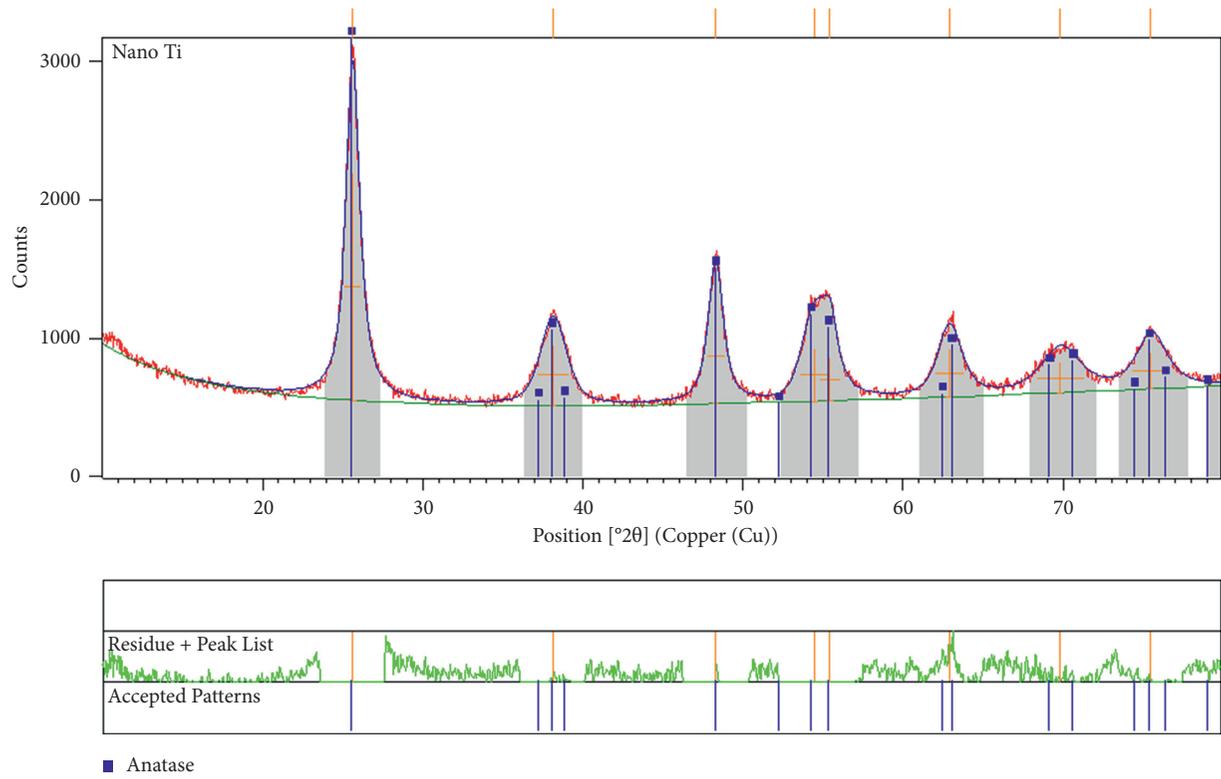


FIGURE 5: XRD peaks of Nano TiO₂ powder.

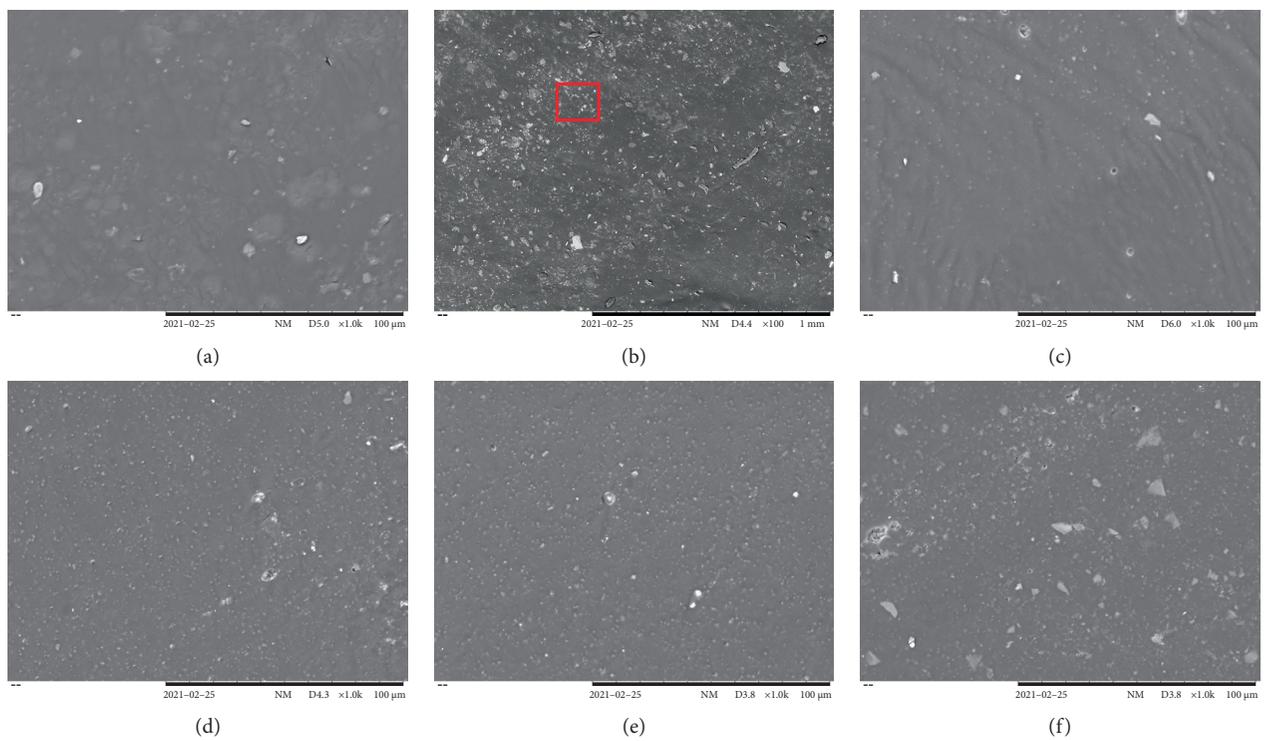


FIGURE 6: SEM images of modified and unmodified bitumen samples.

TABLE 3: Unmodified and modified bitumen elements.

Nano titanium (%)	Carbon		Oxygen		Sulphur		Titanium	
	Weight (%)	Atomic (%)						
0	76.36	84.83	12.32	10.28	5.68	2.36		
1	96.49	98.69	—	—	3.25	1.24	0.26	0.07
2	91.70	96.94			6.56	2.60	1.75	0.46
3	96.11	98.99			3.50	1.50	3.89	1.00
4	86.06	91.5	8.00	6.39	3.62	1.44	3.88	1.05
5	84.94	91.88	6.22	5.05	4.96	2.01	3.88	1.05

there seems to be a melted bitumen state and dry bitumen from the image.

The images generated from Figure 6 show the 0%, 1%, 2%, 3%, 4%, and 5% addition of nano TiO₂. The images generated show that some nano TiO₂ powder is still not blended well into the bitumen and the white spots are the powders that has not been able to mix with the bitumen. This can be further verified with reference to the previous Table 1 (properties of nano TiO₂). With the increasing amount of nano TiO₂, the bitumen has difficulty in blending well with the nano TiO₂ which then produces insoluble white lumps that is observed with increasing nano TiO₂ powder. Meanwhile, 3% nano TiO₂ modified bitumen can mix thoroughly and have lesser white streaks or clumps. However, the dispersion is still acceptable, and the improvement is verified based on the obtained results.

Next, EDX analysis is also carried out on the samples; from the elements observed from Table 3 and Figures 7(a)–7(f), it is most likely that the white flakes or clumps observed are from the aluminium or calcium residue composed in the bitumen. The visibility is due to the amount of those elements that still exists inside the nano TiO₂ modified sample. The elements from table confirm that the most abundant elements are carbon and oxygen. Other elements that are obtained include aluminium, sulphur, and calcium. The results are consistent with a few recent studies performed by Snehal et al. [11], Arshad et al. [19], and Babagoli [15].

The unmodified bitumen shows all the elements mentioned before, but when nano TiO₂ is added with various content, the chemical composition changes according to the amount of nano TiO₂ added. Hence, the optimum nano TiO₂ content in terms of SEM-EDX analysis is at 3% nano TiO₂ modified bitumen followed by 5%, 4%, 2%, and 1%, as shown in Table 3. Only two elements are present: carbon and oxygen, which mainly made up the sample alongside nano TiO₂ showing that the nano TiO₂ is mostly blended with the bitumen. Overall, 3% specimen has the most influence on the chemical composition changes and enhancement of virgin bitumen.

3.4. Softening Point. As shown in Figure 8, the softening point is the highest at 0% nano TiO₂ at 44.4°C compared to 5% nano TiO₂ which only record at 41.8°C. Further addition of nano TiO₂ makes the softening points drop lower than the initial reading recorded. The high softening point at 0% implies that the resistance of the bitumen to the heat is increased and shows that there is a low probability that the

bitumen will soften under high temperatures which is a resemblance of the hot weather. However, 4°C is not a large difference, and usually in summer seasons, the temperature on pavement surface is larger than 50°C, so with this bitumen, the risk of rutting exists always. This makes the bitumen less vulnerable when temperature changes. Rutting is one of the deformations happening related to temperature, and with this increase in softening point, the rutting would be reduced. The readings recorded are having the same trend as previous research by Crucho et al. [20] in which the increasing content of nano material added results in lower softening point.

3.5. Penetration Test. The results in Figure 9 show that bitumen penetration varies with different nano TiO₂ percentages. The penetration for nano TiO₂ mixture between 1 to 5% are 73 dmm, 61.5 dmm, 60.5 dmm, 55.5 dmm, 54.5 dmm, and 54 dmm, respectively. The consistency of bitumen is harder at 3% to 5% nano TiO₂ compared with 0% nano TiO₂ sample. This result is consistent with the earlier study on nano silica that observed a decrease in penetration tests [20]. These results are also reflected in the nano TiO₂ usage in the binder, which is similar to the utilisation of nanomaterials. This is caused by the increased viscosity of bitumen, as evident from the penetration value at increasing nano titanium percentages. Essentially, nano TiO₂ addition caused bitumen to be more consistent and harder. Thus, improving rutting resistance produces much stiffer bitumen which affects the resistance to fatigue cracking.

3.6. Volumetric Properties

3.6.1. Stability. Figure 10 shows the density and stability of the modified binder against nano TiO₂ percentages between 0 to 4%. The stability of the mixture is mostly influenced by the cohesion and internal friction of the matrix, which supports the coarse aggregates [21]. Figure 10 shows a comparable trend for density and stability, especially for the 3% nano TiO₂ addition. For this particular sample, the density is 2.238 g/cm³, while the maximum stability is 5945 N compared to 5879 N for 0% nano TiO₂ addition. It is found that increasing nano TiO₂ amount also increases the stability and density. However, nano TiO₂ addition beyond 3% does not increase both parameters. This indicates that the usage of 3% nano TiO₂ in SMA mixture is more effective in enhancing density and stability. These contribute to less

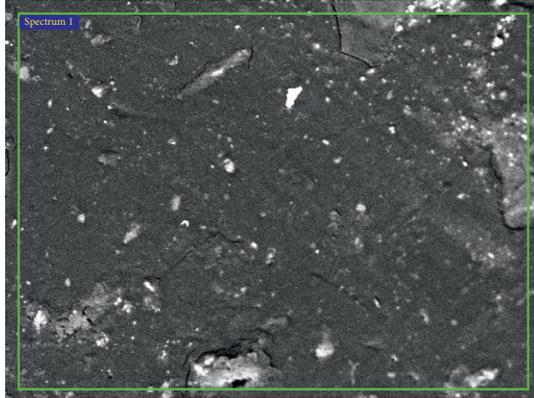
Spectrum details

Project: New project

Spectrum name: Spectrum 1

Electron Image

Image Width: 175.7 μm



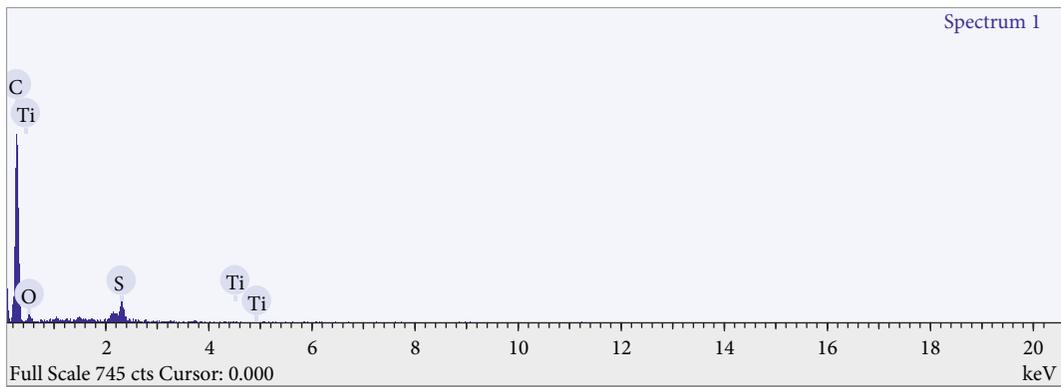
80 μm

Acquisition conditions

Acquisition time (s) : 29.9

Process time : 5

Accelerating voltage (kV) : 15.0



Quantification Settings

Quantification method : All elements (normalised)

Coating element : None

Summary results

Element	Weight %	Weight % σ	Atomic %
Carbon	96.490	0.469	98.689
Oxygen	0.000	0.000	0.000
Sulfur	3.246	0.395	1.244
Titanium	0.264	0.261	0.068

(b)

FIGURE 7: Continued.

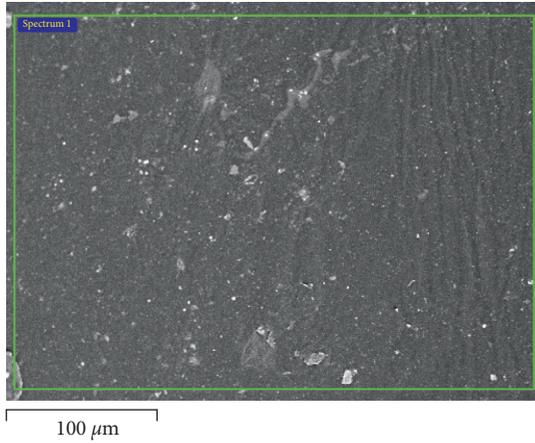
Spectrum details

Project: New project

Spectrum name: Spectrum 1

Electron Image

Image Width: 351.4 μm

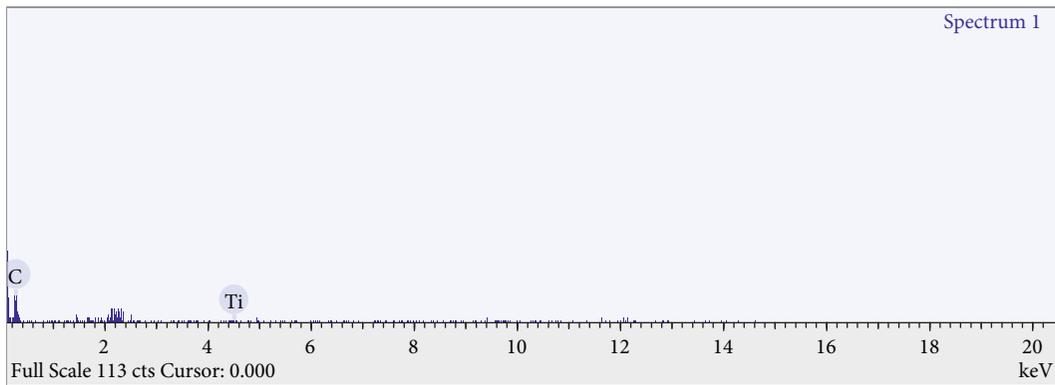


Acquisition conditions

Acquisition time (s) : 30.0

Process time : 5

Accelerating voltage (kV) : 15.0



Quantification Settings

Quantification method : All elements (normalised)

Coating element : None

Summary results

Element	Weight %	Weight % σ	Atomic %
Carbon	96.107	4.005	98.994
Titanium	3.893	4.005	1.006

(d)

FIGURE 7: Continued.

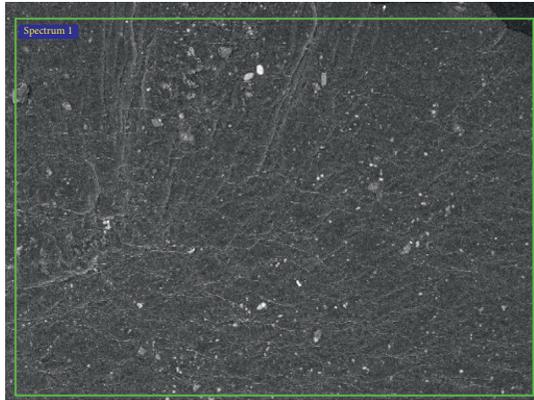
Spectrum details

Project: New project

Spectrum name: Spectrum 1

Electron Image

Image Width: 351.4 μm

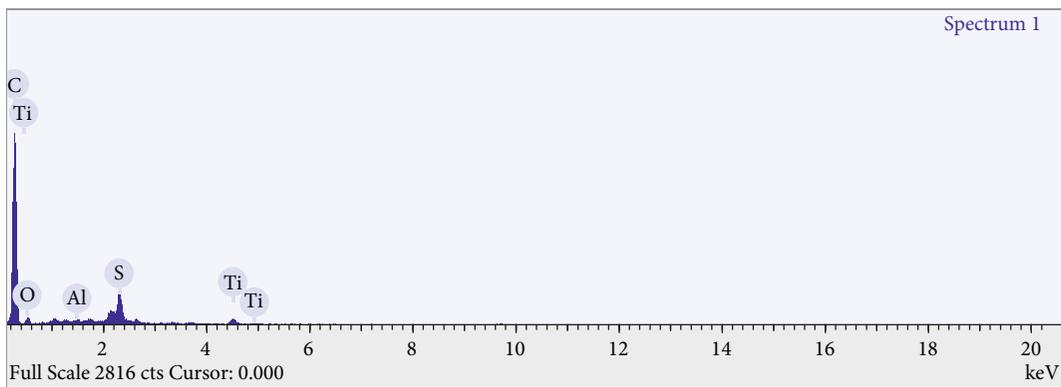


Acquisition conditions

Acquisition time (s) : 30.0

Process time : 5

Accelerating voltage (kV) : 15.0



Quantification Settings

Quantification method : All elements (normalised)

Coating element : None

Summary results

Element	Weight %	Weight % σ	Atomic %
Carbon	86.055	1.011	91.499
Oxygen	8.001	1.039	6.387
Aluminum	0.250	0.073	0.118
Sulfur	3.624	0.192	1.444
Titanium	2.070	0.183	0.552

(e)

FIGURE 7: Continued.

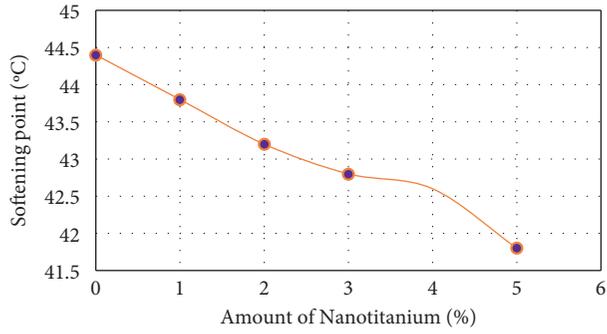


FIGURE 8: The softening point of nano TiO₂ modified bitumen.

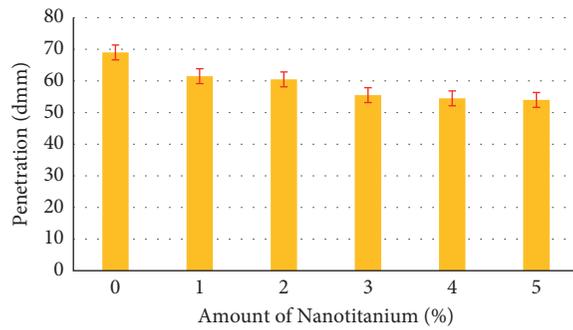


FIGURE 9: Penetration values of nano TiO₂ modified binder.

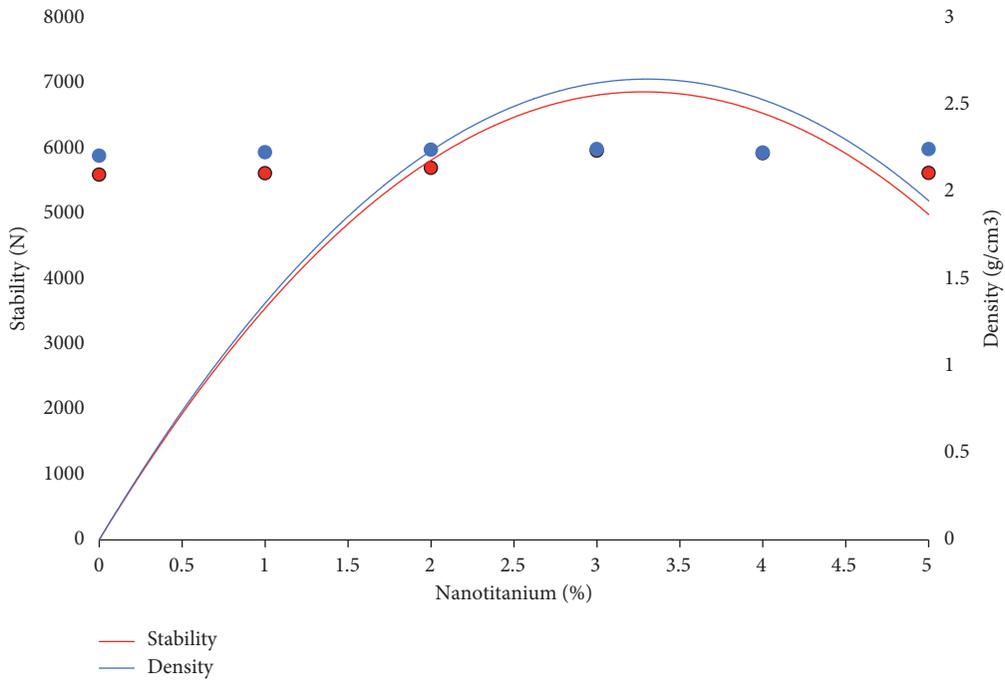


FIGURE 10: Stability and density against nano TiO₂ percentages.

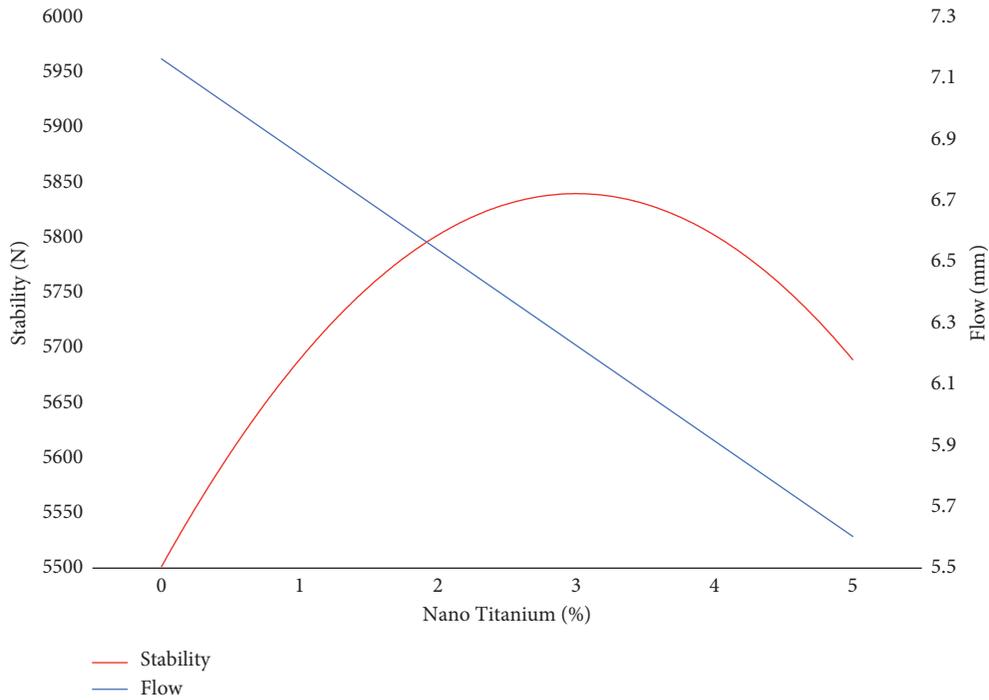


FIGURE 11: Stability and flow against nano TiO₂ percentages.

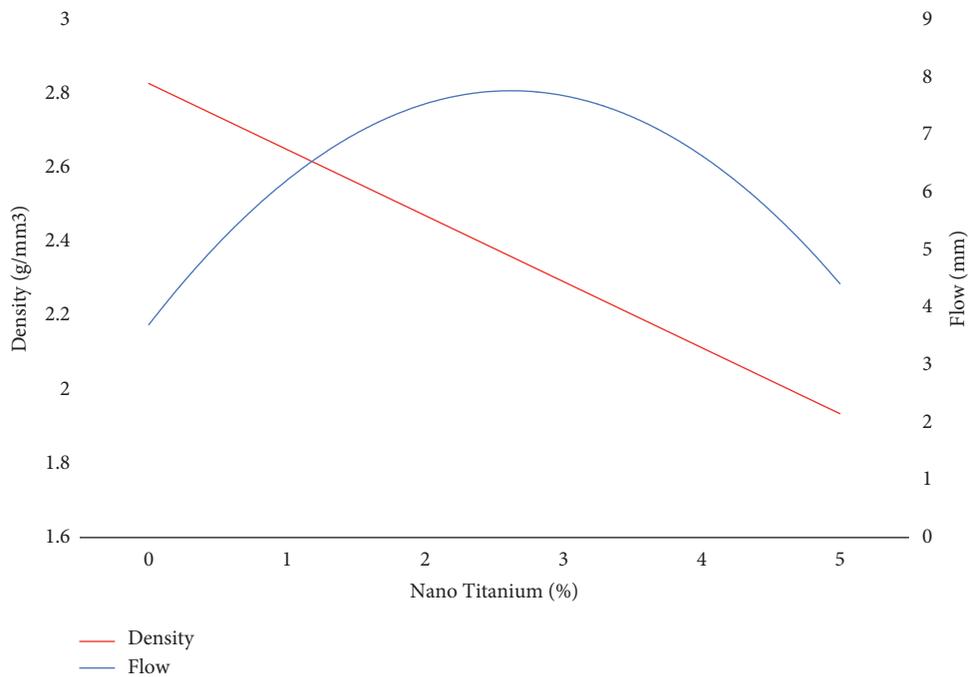


FIGURE 12: Density and flow against nano TiO₂ percentage.

with the inclusion of nano TiO₂. The flow at 3% nano TiO₂ complements both the density and stiffness. 0% nano TiO₂ produces a relatively higher flow value (7 mm) than the requirement by the Public Work Department (between 3 to 5 mm). The addition of nano TiO₂ reduces the flow, indicating that nano TiO₂ could influence the flow properties to a more accustomed value.

3.6.3. *Stiffness.* Figures 14 and 15 exhibits the stiffness of SMA mixtures against various nano TiO₂ percentages. The highest stiffness value is obtained by the 3% nano TiO₂ addition (965.67 N/mm), compared to 833.3 N/mm for 0% nano TiO₂ which is a 14.8% improvement. This shows that nano TiO₂ addition improves the stiffness characteristic of asphalt mixture.

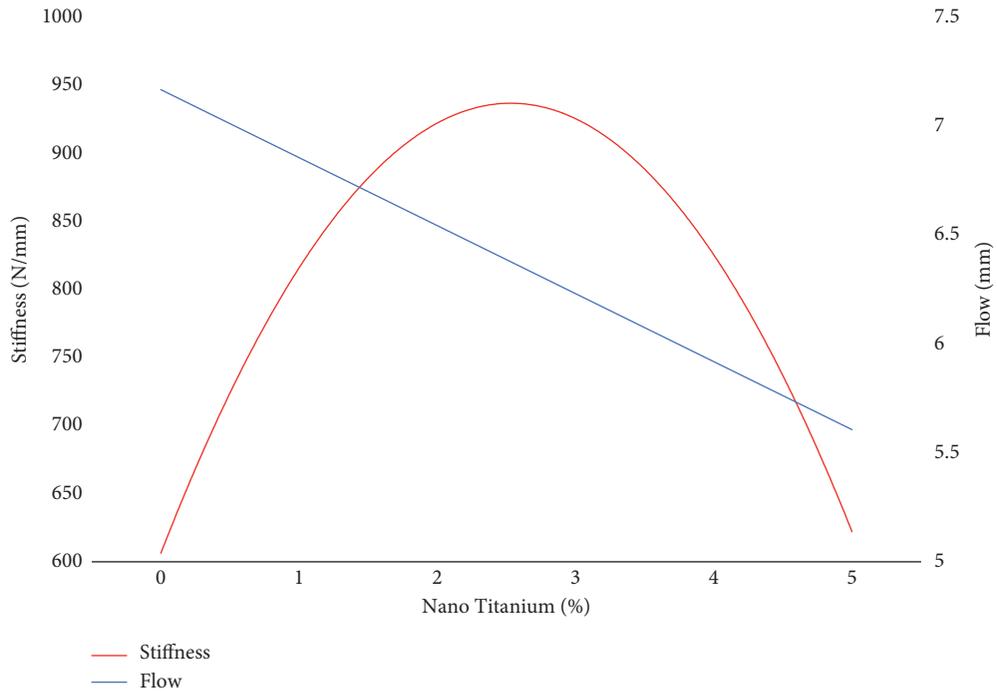


FIGURE 13: Stiffness and flow against nano TiO₂ percentages.

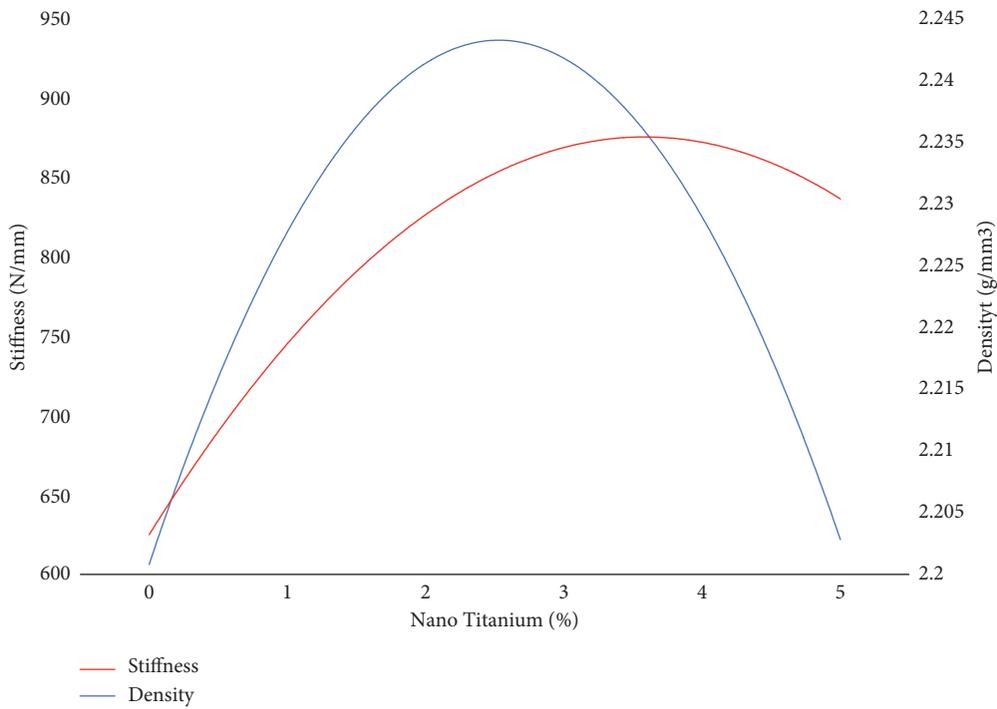


FIGURE 14: Stiffness and density against nano TiO₂ percentages.

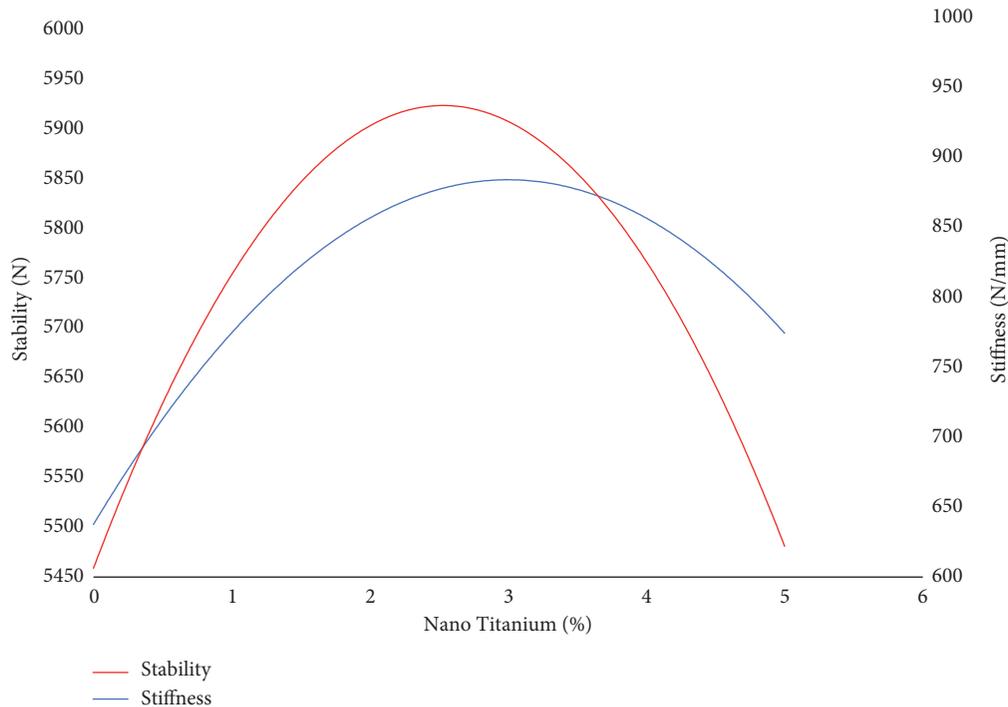


FIGURE 15: Stability and stiffness against nano TiO₂ percentages.

4. Conclusion

The nano TiO₂ modified binder significantly improves the microstructural, chemical, and physical properties of bitumen, as well as enhancing the mechanical properties of the SMA mixture. The details are as follows:

- From FTIR results, the change in chemical bonds happens when the nano TiO₂ is added into the bitumen showing that the nano TiO₂ successfully blends into the bitumen.
- XRD analysis peaks are higher and further compared to the virgin bitumen which has lower peak starting. This proved the existence of nano TiO₂ in the virgin bitumen.
- SEM-EDX results show that bitumen mixture particles are well dispersed depending on the nano TiO₂ percentages. The 3% addition of nano TiO₂ reveals that it is able to merge the nano TiO₂ particles fully into the bitumen.
- The penetration and softening point show a consistent trend similar to previous research studies as discussed earlier showing that 3% to 5% nano modification contributes the optimum value.
- Improvements of the physical, morphological, and chemical characteristics also enhance the mechanical performance of SMA as evident from volumetric properties.

The density, stability, flow, and stiffness properties of the 3% nano TiO₂ addition mixture are better than the 0% nano TiO₂ due to the addition of the modified nano TiO₂ binder. Therefore, it can be concluded that overall, 3% nano TiO₂

addition enhances the mechanical properties of SMA. Finally, the improvement of nano TiO₂ modified binder directly enhances the quality of SMA performance in terms of its volumetric properties [22].

Data Availability

All the data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest with other entities or researchers.

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