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ABSTRACT

This study aims to investigate the physical and rheological properties of binder modified with nano-titanium dioxide and to evaluate the fatigue performance of modified asphalt mixture using Indirect Tensile Fatigue Test. Nano-titanium dioxide of 2%, 4%, 6%, 8% and 10% by weight of binder has been incorporated into unaged 80/100 binder. Through rotational viscosity test, added 10% TiO₂ shows the most reduction of mixing and compaction temperature with approximately 15°C, which indicates that the viscosity was improved. Nano-TiO₂ was also able to elevate the complex modulus of a binder a sides from increasing the temperature susceptibility. In terms of fatigue performance testing, addition of 10% additive does help in improving the fatigue properties as it shows greater result than the control binder. The resilient modulus of the modified mixture shows similar results to each other, which indicates it has similar property although it is compacted at lower temperature. In conclusion, modified nano-TiO₂ is great in improving the physical and rheological properties of the binder asides from improving the fatigue properties.

Key words : Titanium Dioxide, modified asphalt, viscosity, rheology, complex modulus.

1. INTRODUCTION

Fatigue is one of the most distressing mechanisms that take place in asphalt pavement. It is presented by a series of interconnected cracks that resemble the skin of an alligator. The crack usually initiates from the bottom of the asphalt layer which later propagate to the top as a result of high tensile stress associated to the repetitive traffic loading[1]-[3]. The initiation and propagation of cracking generally relates to the magnitude of dissipated energy produced by external loading. At the stage of crack initiation, water will trap in the cracks and lead to reduction of material strength when loaded repeatedly [4].

This strength reduction causes the initial crack to propagate and further degrade. Other than that, factors that could be contributing to the structural distress are the structural design of the pavement, asphalt content, consistency of the asphalt cement, asphalt concrete properties, poor compaction, binder aging and also the distribution of temperatures. This damage also seriously discussed since implementing warm mix asphalt (WMA). Incomplete drying of the aggregates due to lower temperature used during mixing may cause the trapped water in aggregate to result in moisture damage [4]. Although, several researches present a new approaches to mitigate this problem. The use of warm-mix asphalt technology requires the addition of an additive into the mix, and minor changes in production and construction procedures, in specific, temperature related, which could influence the short-term and long-term performance of the pavement [5]-[6]. Other than that, in a research done by Gandhi and Amirkhanian, they claimed that usage of nanomaterial in warm mix asphalt reduces the aging of binder significantly through the lower mixing and compaction temperature [7]. Improvement in low temperature fatigue cracking resistance is also observable as the viscosity of binder is greatly improved with the presence of additive. Lower aging properties can extend the life of the pavement as the oxidation process takes place later than conventional binder.

In addition, Shafabakhsh et al., in their research have evaluated about the effect of nano-titanium dioxide (TiO₂) on the rutting and fatigue behavior of asphalt mixture [8]. Based on the results obtained, TiO_2 is best used at the rate of 5% by weight of the binder. It shows its optimum potential at this percentage by decreasing the penetration value, increasing the viscosity, softening point temperature and ductility. It is also proven that the addition of nano-TiO₂ as bitumen modifier can improve the creep behavior of asphalt mixtures even under high temperatures and stresses. A Generation of tensile and vertical cracks by horizontal tensile stresses were found to be less visible which help in preventing propagation. Other than that, Hassan et al., in their finding stated that addition of nano-TiO₂ in WMA pavement do help in improving the air quality by mitigating the production of nitric oxide (NO) and nitrogen oxide (NO_x) when the pavement is exposed to UV light from the direct sunlight [9]. When used as a binder modifier, the photocatalytic compound is not effective in degrading NO_x in the air stream although there is increment in the percentage. The effectiveness is almost negligible. This could be due the fact that only a tiny amount of additive present on the surface. However, when nano-TiO₂ is used as a thin coating, distinct difference of percentage can be seen. This evidence shows that nano- TiO_2 is effective in improving air quality but the proper method of applying it is still unclear.

In another research, Shafabakhsh et al. also proven that addition of nano-TiO₂ in asphalt binder has improved the physical characteristic of the binder by decreasing the penetration value and increasing the softening point⁸. This is an indication of improvement in the hardness and stiffness of the modified bitumen. It is also said that the adhesive bonding strength of binder and aggregates is improved with the present of nano-TiO₂. In terms of rutting and fatigue, it is learned that lower sensitivity of modified asphalt concrete towards stress produce better resistance against rutting and lower viscosity at lower temperature decreases the cracking formation on the asphalt concrete.

In this study Titanium dioxide is a nanopowder that uses for reducing the mixing and compaction temperature of asphaltic concrete mixture, also increase fatigue resistance. Titanium dioxide is a white powder which is able to breakdown most of the organics in the medium that it encounters when exposed to sunlight. It is non-toxic and this naturally occurring oxide can also be mined from other minerals such as ilmenite or leucoxene $ores^{10}$.TiO₂ came in three mineral forms, rutile, anatase and brookite. Upon heating, both the anatase and brookite convert into rutile which is also the most stable form among the phases [10]-[11].

Research has shown that TiO_2 in anatase phase exhibits the highest photoactivity in environmental purification [12]. Nano titanium dioxide is made up of 80% anatase and 0% rutile, have a specific surface area of 50 and an average size of 21 nm. Nano TiO_2 has 500% higher surface area and 400% lower opacity compared to ordinary TiO_2 . Large specific surface area (SSA) which is area to volume ratio as well as small particle diameter under 100 nm are of characteristics of TiO_2 , making it useful as a nanoparticle [13]. TiO_2 has the potential to help reduce the emission of greenhouse gases which will benefits the environment¹⁰. Other than it is expected to improve the performance of asphalt binder in terms of rheology.

2. EXPERIMENTAL METHODS AND MATERIALS

Bitumen of penetration grade 80/100 was mixed with nano-TiO₂ of 2%, 4%, 6%, 8% and 10% by weight of binder. The mixing speed used for binder preparation is 3500 rpm, for duration of 30 minutes at 155°C. The samples were then labeled with BT0, BT2, BT4, BT6, BT8 and BT10 for each different quantity of additive added. Full description of the samples is shown in Table 1. Penetration and Softening point test were done to determine the physical properties of binder. Higher penetration value indicates softer consistency of the binder and lower value indicates that the stiffness of the binder is improved. To evaluate the fatigue parameter of asphalt binder, dynamic shear rheometer (DSR) test was conducted to obtain the complex shear (G*) and phase angle (δ) of the binder. The multiplication value of these parameters should not exceed 5000 kPa, else the binder is considered too

stiff and would not be suitable in the construction of pavement. An elastic asphalt binder is important in resisting fatigue cracking therefore minimum value of G*sin\delta is more favorable. For asphalt concrete performance, Indirect Tensile Fatigue Test (ITFT) is conducted to examine the fatigue resistance. In this testing, fatigue cracking is considered stress-controlled and strain-controlled. Cylindrical asphalt concrete is loaded repetitively with a load of 600 kPa until obvious cracking is shown. The total number of load applications before a fracture occurred is defined as the fatigue life. Energy dissipated during the loading could also be used to further define the fatigue failure. Fatigue cracking can be mitigated by conducting laboratory research to develop for better fatigue resistance in binder and also in asphalt concrete.

Composition	ID
Virgin Binder	BT0
Virgin Binder + 2% Nano-TiO ₂	BT2
Virgin Binder + 4% Nano-TiO ₂	BT4
Virgin Binder + 6% Nano-TiO ₂	BT6
Virgin Binder + 8% Nano-TiO ₂	BT8
Virgin Binder + 10% Nano-TiO ₂	BT10

3. EXPERIMENTAL RESULTS

3.1. Physical properties

Table 2 given the results of penetration and softening point test. It can be seen that the penetration value is gradually getting lower as more additive is added into the binder.

Table 2: Summary of physical testing results

Sample	Penetration at	Softening	PI	
	25°C	Point		
BT0	89.1	46.4	-0.71	
BT2	88.2	46.2	-0.80	
BT4	75.3	46.6	-1.07	
BT6	67.1	50.0	-0.5	
BT8	60.8	48.6	-1.1	
BT10	56.4	49.2	-2.85	

This shows that addition of additive does help in improving the hardness of the binder, which is a desired factor in modified binder property. Higher softening point indicates stiffer binder and lower temperature susceptibility at high temperature, which is more preferred in hot climates. Also shown in the table, BT2 shows lower softening point compared to the control binder, BTO. However, the value started to increase from BT4 until BT6. At BT8, the value decrease slightly before it rose up again at BT10. This indicates that proper amount of TiO₂ added can help in improving the stiffness of the binder. Through the results obtained from penetration and softening point test, the Penetration Index (PI) can calculated to determine the temperature susceptibility of the binder. The PI value normally ranges from approximately -3 to approximately +7. Negative value indicates that the binder is susceptible for high

temperature while positive value indicates low temperature susceptibility. All the modified binders have negative PI value which is a desired property in modified asphalt industry. All modified binders have lower PI value compared to the control binder except for BT6 which has a value slightly higher than BT0.

3.2. Viscosity

Asphalt is a typical viscoelastic material, which exhibits both viscous and elastic properties over the working temperature of pavement under normal traffic loads. Brookfield Viscometer was used in rotational viscosity test to measure the flowing resistance of asphalt materials. The Superpave[™] specification of AASHTO M 320 requires that the maximum viscosity of asphalt binder is no greater than 3Pa.s at 135 °C for the convenience of storage and pumping in construction period Conventional binder is known to have a higher mixing and compaction temperature compared to modified binder. In this study, as shown in Figure 1, it is found out that BT0 and BT2 has a mixing and compaction temperature that is very close to each other. However, BT8 and BT10 show exceptionally better temperature with a difference of approximately 15°C.

3.3. Dynamic Shear Rheometer

DSR is an apparatus to measure the complex modulus (G*) and phase angle (δ) to characterize the viscous and elastic behaviour of asphalt binders



Figure 1 : Viscosity of modified binders at different temperature

The G^* can be considered the sample's total resistance to deformation when applied repeatedly shear loading, while δ , is the lag between the applied shear stress and the resulting shear strain. Intuitively the higher the G^* value the stiffer the asphalt binder. Whereas, the lower the phase angle, the less viscous the material, which means the asphalt binder is able to recover to its original shape after being deformed by a load. Haake software was set to run 10 cycles of test repetitions, at a constant oscillating speed of 1.592 Hz and test temperature ranging from 46°C to 70°C. From Figure 2, it can be seen that addition of additive does help to improve the complex modulus of modified binder except for BT2, but it does not help in improving the phase angle of the modified binder. This proposed that addition of TiO_2 will not produce a promising result for unaged binder.



Figure 2: Isochronal graph of G* and δ against temperature for unaged binder

3.4. Fatigue Resistance of Modified Binder

Figure 3 shows results of complex modulus and phase angle of binder after long term aged. It can be seen that BT2 has the highest complex modulus, followed by BT8, BT6, BT4, BT10 and BT0. These 125°C compacted mixtures show greater G* values than the control binder, BT0, which is one of the preferable criteria. However, in terms of phase angle, BT0 has the most desirable value, followed by BT2, BT4, BT8, BT6 and BT10.



Figure 3: Isochronal graph of G* and δ against temperature for long term aged binder

By looking at the overall result, BT2 has the highest temperature susceptibility among the other binders. This result was followed by BT8, BT6, BT4, BT0 and BT10. The values of G*Sin\delta for each binder samples after PAV aging are illustrated in the Figure 4. According to SHRP, binder that is prone to fatigue cracking will have a G*sinδ value greater than 5000 kPa. From this chart, it can be seen that BT2, BT6 and BT8 are more susceptible to fatigue cracking at 16°C as its value exceeds the standard specification. At 19°C, BT2 is the only binder that are prone to cracking. Other modified binders have a value higher than the control binder, however, the values are still below the specification limit, and therefore, it is considered to be able to resist fatigue cracking.



Figure 4 : Value of fatigue cracking resistance of every modified binder

3.5. Fatigue analysis

In this study, the fatigue analysis is done through the operation of Universal Testing Machine (UTM). The samples of BTB0 was prepared at mixing and compaction or 165°C and 155°C respectively and other samples were prepared at mixing and compaction or 135°C and 125°C respectively. Indirect tensile fatigue test is a test conducted under repeated loading to determine the fatigue resistance of a mixture. The testing was done at 20°C to represent the condition where fatigue cracking is likely to happen. A loading of 600 kPa was implied on the cylindrical sample throughout the testing using UTM until it comes to a termination. The fatigue characteristic was then evaluated by the number of cycles count. From the graph of Figure 5, mixture compacted at 155°C have higher number of cycles when compared to the mixtures compacted at 125°C except for BTB10. The trend for mixtures compacted at 125°C is showing an increasing pattern but it starts to decline from BTB8 and rise back at BTB10. This could indicate that proper amount of TiO₂ addition will help to improve the reaction between the binder and the aggregates, which eventually increases the strength of the mixture as a whole.



Figure 5: Number of cycles against TiO₂ content

3.6. Indirect tensile resilient modulus

Indirect tensile (IDT) resilient modulus test is a non-destructive test and it is one of the important parameters in determining the mechanical properties of asphalt mixtures. Figure 6 shows the result of resilient modulus (MR) conducted at 25°C using three different repetition periods. From the chart, it shows that the trend is going down as the repetition period goes higher. However, the case is different for BTB0. The repetition period at 3000 ms seems to be slightly higher than that of 2000 ms. BTB4 displays the highest MR during the 1000 ms pulse repetition period while BTB6 has the lowest value, with a difference of approximately 650 MPa. For 2000 ms pulse repetition period, BTB2 generates the highest MR while BTB4 produces the least. For 3000 ms pulse repetition period, BTB6 shows the highest modulus and BTB10 shows the lowest. From the result, it can be clearly seen that mixtures compacted at 125°C show similar performance as mixture compacted at 155°C.



Figure 6: Resilient modulus against TiO₂ content

4.CONCLUSION

In this study, nano-TiO₂ of 2%, 4%, 6%, 8% and 10% were added to the base binder to investigate for the effectiveness of the additives in improving the properties of the binder. Based on the results presented, the following findings and conclusions can be deduced with respect to the applications:

• The addition of nano-TiO₂ has significantly improved the penetration and softening point of the binder. This

demonstrated the improvement of stiffness of the binder and also better temperature susceptibility.

- After short-term aging has been conducted on the binder through RTFO test, it is found that the mass loss of all the modified binder is less than 1%, which indicates less aging will happen during mixing and construction phase.
- The fatigue resistance of modified binder is greatly improved as shown by the value of G*sinδ after the process of long term aging. There is a huge improvement on the complex modulus and also the temperature susceptibility of the binder.
- Addition of TiO₂ has the potential in reducing the mixing and compaction temperature of the mixture. Other than BT2 and BT6, all the modified binder shows lower mixing and compaction temperature.
- In terms of fatigue cracking performance of mixture, addition of modified nano-TiO₂ does aid in enhancing the static strength and stiffness of the modified bituminous mixture. However, the amount of TiO₂ added does play a big role in controlling the effectiveness of mixture towards fatigue resistance.
- The resilient modulus of the modified mixture shows similar performance in compared to control mixture. This indicates that modified nano-TiO₂ has the potential to be used as additive in WMA.

In conclusion, the addition of modified nano-TiO₂ is great in improving the physical and rheological properties of the binder. However, in terms of fatigue performance, this modified mixture display satisfying results only at the addition of 10% additive. Besides that, the best percentage of modified nano-TiO₂ to be used is showing different results in binder performance and also mixture performance. For binder, the best percentage for workability would be 8% while for fatigue performance evaluation, the best percentage would be 10%.

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