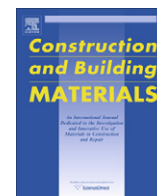




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Nanoclay-modified asphalt materials: Preparation and characterization

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ABSTRACT

The objective of this study is to review existing literature in the area of nano-modification of asphalt and proceed to apply nano-materials to asphalt to improve the performance. This study integrates literature review, preparation, and characterization of nano-modified asphalt materials. In the experimental testing montmorillonite, nanoclay at 2% and 4% by weight of asphalt was blended in asphalt binder at a high temperature to exfoliate the nanoclay within the asphalt. The asphalt binder was then characterized using the Superpave™ rotational viscosity, dynamic shear modulus, and direct tension test. The rotational viscosity results indicate that the addition of the two types of nanoclay, Nanoclay A and Nanoclay B, increased the rotational viscosity by an average of 41% and 112%, respectively, across test temperatures 80, 100, 130, 135, 150 and 175 °C. It was found that the dynamic shear complex modulus (G^*) value increases significantly across a range of testing temperatures (from 13 to 70 °C) and loading frequencies (0.01–25 Hz). With 2% Nanoclay A reinforcement in the asphalt binder, the complex shear moduli generally increased by 66% while the 4% Nanoclay A reinforcement in the asphalt binder generally increased the shear complex moduli by 125%. The 2% and 4% Nanoclay B increased the shear complex moduli by 184% and 196%, respectively. In terms of direct tension strength, the use of Nanoclay A and Nanoclay B reduced the strain failure rate of the original binder while the secant or direct tension moduli showed increase with the addition of the nanoclays. In furtherance of this research, nanoclay-modified asphalt is being tested at percentages higher than 4% to underscore the fact that nanoclays may have the potential to reduce rutting and cracking.

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1. Background and Introduction

1.1. Asphalt materials

In the United States, transportation infrastructure investments account for 7% of the Gross Domestic Product (GDP) according to the National Asphalt Pavement Association [1]. Over 550 million tons of hot-mix asphalt (HMA) is produced annually for construction projects. Increasing traffic loads and traffic volume, combined with the rising cost of asphalt, have led to an urgent need to improve the durability, safety and efficiency of asphalt pavements through asphalt modification.

Asphalt mixtures are composed of very irregular aggregates bound together with hydrocarbon-based asphalt, with a low volume fraction of voids dispersed within the matrix. Polymer-modified asphalts have increasingly been used over the last decade

to minimize low-temperature cracking and high-temperature rutting while improving the fatigue cracking resistance of asphalt concrete [2,3]. Polymer modifiers fall into one of two major categories: (1) elastomeric (rubber/polymer) modifiers including styrene/butadiene polymers, natural rubber, and crumb rubbers (both virgin and recycled) [4–19] and (2) plastomeric modifiers such as polypropylene and polyethylene [20].

1.2. Nanotechnology and the current development in pavement materials

Nanotechnology is the creation of new materials, devices, and systems at the molecular level as phenomena associated with atomic and molecular interactions strongly influence macroscopic material properties [21]. Even though engineers are interested in material properties at the macro and meso scales, the nano and micro scales provide fundamental insight for the development of science and technology. Fig. 1 illustrates the evolution of length scales of an asphalt concrete material (in macro scale), to meso, micro [22], nano, and to quantum scales. Although improvements

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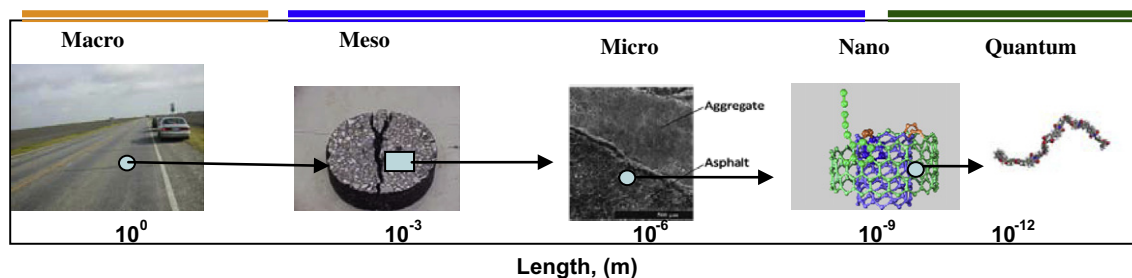


Fig. 1. Illustration of the evolution of different asphalt dimensions.

in asphalt performance have been achieved through polymer modification, it will be interesting to explore what nanotechnology offers in improving asphalt pavement performance.

In August 2006, the *NSF Workshop on Nanomodification of Cementitious Materials* was held to develop a “Roadmap for Research” for Portland cement concrete and asphalt concrete using nanotechnology. Part I [23] states that nano-science and nanotechnology may lead to progress in asphalt pavement technology. Researchers [24] are planning to form an ad hoc consortium to study and develop tools to model asphalt’s physio-chemical properties that effectively establishes the field of “asphalt nano-material science. Greenfield [25] has studied the molecular simulation of asphalt-like materials. Birgisson [26] envisions that nano studies are needed to develop safe and sustainable pavement infrastructure, stating that the future life of pavements could reach two to three times their current life. Other researchers have also initiated investigations into the use of nanotechnology in Portland cement concrete [27–34].

Ideal asphalt [35] should possess both: (1) high relative stiffness at high service temperatures (summer) to reduce rutting and shoving and; (2) increased adhesion between asphalt and aggregate in the presence of moisture to reduce stripping. The project team conducted preliminary tests, blending small percentages of nanoclay-composites into virgin asphalt with the hope of producing a binder that is less susceptible to high-temperature rutting and low-temperature cracking. The motivation is to significantly reduce the temperature sensitivity of the binder at service temperatures while maintaining workability at construction temperatures. Nano-modified asphalt may potentially improve the rutting, crack and fatigue resistance of asphalt mixtures.

2. Literature review

2.1. Asphalt chemical components and modification efforts

Most asphalt molecules, primarily consisting of carbon and hydrogen, also contain one or more of the elements sulfur, nitrogen, and/or oxygen [35]. Within asphalt, heteroatoms typically replace carbon atoms in the asphalt molecule; the interaction of heteroatoms and the hydrocarbons leads to the unique chemical and physical properties of various asphalt mixtures [36].

Asphalt molecules are classified in two major fractions based on solubility. These fractions are the asphaltenes and maltenes [36]. Both the asphaltenes and maltenes constitute the non-volatile, high-molecular-weight fractions of petroleum but the maltenes constitute the fraction of asphalt which is soluble in *n*-alkane solvents such as pentane and heptane. The three basic types of molecules in asphalt are aliphatics, cyclic, and aromatics.

Research conducted in the Netherlands suggests nanoclay modifications improves some characteristics of asphalt binders and asphalt mixtures, but more research is required before it can be applied on a large scale [37]. The major reason for additional research is that while the nanoclay increased the rutting

resistance little is known about how it mitigates the fatigue problem of asphalt. In China, similar research has been conducted on nano calcium carbonate (nano- CaCO_3) modified asphalt [38,39]; it was found that the nano- CaCO_3 can enhance asphalt’s rutting-resistance as well as improve its low-temperature toughness. It was found that the mixture of nano- CaCO_3 and asphalt forms a uniform and steady system which improves temperature susceptibility of asphalt at high temperatures. However, the mechanism of asphalt material behavior with this modification is not well understood.

In China, Yu has studied the effect of a nanoclay (montmorillonite) on properties of styrene–butadiene–styrene (SBS) copolymer modified asphalt [40] by melt blending with different contents of sodium montmorillonite (Na-MMT) and organophilic montmorillonite (OMMT). It was found that the addition of Na-MMT and OMMT increases the viscosity of SBS-modified asphalt. In addition, the nanoclay/SBS-modified asphalt gained a higher complex modulus and lower phase angle, implying stiffer and more elastic asphalt. Therefore, the nanoclay-modified asphalt was determined to have good rutting resistance compared to the original asphalt or the SBS-modified asphalt. It was found that MMT-modified asphalt may form an intercalated structure, whereas the OMMT-modified asphalt may form an exfoliated structure based on the X-ray diffraction (XRD) results [41,42].

2.2. Nanoclay, montmorillonite, and organic modification of nanoclay

Layered silicates (nanoclay) are widely used in the modification of polymer matrices to realize significant improvement in mechanical, thermal, and barrier properties. One of the most frequently used layered silicates is montmorillonite (MMT), which has a 2:1 layered structure with two silica tetrahedron sandwiching an alumina octahedron. Roy et al. [45,46] enhanced the compressive and shear strength of thermoplastic polymers using only a small weight percent of nanoclay reinforcement.

When the polymer penetrates between the adjacent layers of the nanoclay, the gallery spacing is increased and the resulting morphology is an intercalated structure [43]. An exfoliated morphology occurs when the clay platelets are extensively delaminated and completely separated due to thorough polymer penetration by various dispersion techniques. In combination with nanoclay, couplings agents, such as silane, are used to disperse the nanoclay in a stable manner [MMT surface modification]. Also, silane coupling agents have been observed to enhance the bonding of nanoclay to the polymer matrix.

3. Laboratory testing and results

3.1. Laboratory program design

The research team used the information gathered from the literature review and evaluations conducted to shape the direction of the laboratory program. three types of asphalt binder were used:

(1) Superpave™ grade original binder 64–28 from Michigan Department of Transportation (MDOT) US -127 Project; (2) The US-127 asphalt binder modified with 2% and 4% of a first nanoclay (Nanoclay A) by weight of asphalt binder; (3) The US-127 asphalt binder modified with 2% and 4% of a second nanoclay (Nanoclay B) by weight of asphalt binder. Using the Superpave™ Binder Test Specification Standards, the conglomerate tests conducted on the asphalt binder, Nanoclay A and Nanoclay B were:

- Rotational viscosity test (*standard method for viscosity determinations of unfilled asphalts using the thermosel apparatus, ASTM 4402*).
- Dynamic shear rheometer test (*Rheological Properties of Asphalt Binder Using Dynamic Shear Rheometer AASHTO TP5*).
- Rolling thin film test (*Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt, AASHTO T240*).
- Direct tensile test (*Method for Determining the Fracture Properties of Asphalt Binder in Direct Tension, AASHTO TP3*).

3.2. Preparation of the nanoclay particles

3.2.1. Fabrication of asphalt nanocomposite

The addition of a silane coupling agent was first carried out using the procedure outlined by Qian et al. [44]. The surfactant-modified nanoclay was dispersed in organic solvent (isopropanol) and the desired amount of silane coupling agent was added with continuous stirring in a water bath until the solvent was completely evaporated. The products were then dried in a vacuum at approximately 80 °C, wetted, and filtered using a 280 mesh. The surfactant-modified nanoclay in conjunction with the coupling agent was then dispersed in the asphalt. The asphalt nanocomposite was fabricated using a high-shear mixer. The asphalt was first heated to 160 °C to a fluid state and the surfactant-modified nanoclay, A or B, was added to the system and mixed at 2500 rpm for 3 h to disperse the intercalated MMT nanoclay. The detailed parameters such as temperature, speed, and mixing duration were adjusted to control the quality of the modified asphalt. Fig. 2 illustrates the chemical structure of Nanoclay A. Nanoclays A and B are similar in terms of the structure.

3.2.2. Morphological characterization

The morphology in the final nanoclay-modified asphalt product was determined using the X-ray diffraction (XRD) characterization techniques. XRD was used to provide a quantitative analysis of the interlayer gallery spacing and it provided a way to determine the extent of dispersion of the nanoclays in the binder. XRD was performed individually on the original binder and the Nanoclay A before the prepared nanoclay-modified binder was also tested. The former provided a useful guide to the interpretation of the combined XRD peaks revealed from the test on the modified binder. The tests were conducted only on Nanoclay A-modification because the motivation was to verify the efficiency of the rate of nanoclay dispersion initiated by the blending process. A higher rate of dispersion is required since nanoclays have a tendency to adhere to each other due to their inherent electrostatic charge.

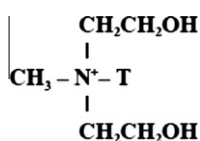


Fig. 2. Nanoclay A structure.

3.3. Experimental test program

3.3.1. Superpave™ characterization of nanocomposite-modified asphalt binder

The rotational viscometer (RV) test was used to evaluate the difference in viscous behavior between the baseline asphalt binder and the nanocomposite-modified asphalt binder. The temperatures over which the RV test was conducted were 80, 100, 130, 135, 150, and 175 °C.

The dynamic shear rheometer (DSR) was applied to characterize the mechanical properties of the modified and baseline asphalts prepared under temperatures ranging from 13 °C to 70 °C and loading frequencies from 0.01 Hz to 25 Hz. The DSR measures the viscous and elastic behavior of the binder as represented by the complex shear modulus (G^*) and phase angle (δ), both used to evaluate performance parameters of rutting-resistance and fatigue cracking [36].

Tensile strength tests were performed on the neat binder and nanoclay-composite modified asphalt specimens. The three samples tested had percentages of 0%, 2.0% and 4% by weight of Nanoclays A and B. A modified version of the Superpave™ direct tensile tester (DTT) was applied to characterize the tensile strength of the modified and non-modified binders. The DTT measures the low temperature ultimate tensile strain/stress of asphalt. A dog-bone shaped specimen is loaded in tension at a constant strain-rate until it fails. The failure strain in the specimen is the change in length (ΔL) divided by the effective gauge length (L_e). On six samples per test, the DTT was conducted to measure the mean maximum failure strain of the modified and baseline asphalt specimens at different loading rates at –18 °C.

4. Experimental results and analysis

4.1. X-ray Diffraction

The XRD indicates the nanoclay particles were uniformly dispersed within the asphalt binder matrix. This confirms that the blending technique used is effective in providing exfoliation of the nanoclay within the asphalt matrix. Figs. 3 and 4 indicate the typical XRD peaks associated with Nanoclay A and US 127 asphalt binder. Fig. 5 shows the XRD peaks for the nanoclay-modified US 127 asphalt binder. It is evident that the peak expected for the nanoclay (5° and 1900 intensity) is not prominent in Fig. 5 compared to Fig. 3 suggesting the nanoclay has been well dispersed and uniformly distributed in the mix.

4.2. Rotational viscosity

From the rotational viscosity plots shown in Fig. 6, it is evident that with the addition of 2% of Nanoclay A, the viscosity increased

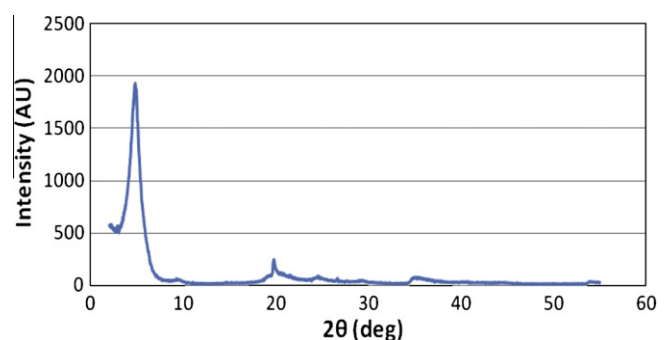


Fig. 3. XRD plot for Nanoclay A.

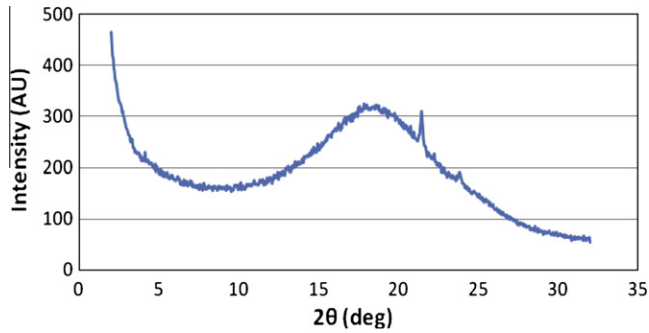


Fig. 4. XRD plot for US 127 asphalt binder.

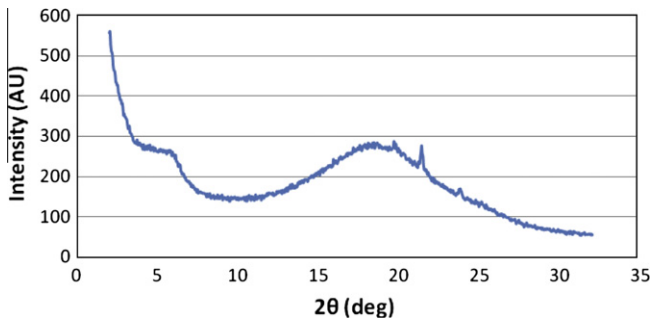


Fig. 5. XRD plot for 4% Nanoclay A-modified US 127 asphalt.

by an average of 41% while the Nanoclay B modified asphalt binder increased by 112% for the test temperatures ranging from 80, 100, 130, 135, 150 and 175 °C. The RTFO-aged neat binders viscosity also increased by 102%.

4.3. Dynamic shear rheometer (DSR)

Fig. 7 shows the DSR plot for the complex shear moduli difference between the original US-127 PG 64-28, 2% Nanoclay A-modified and the 4% Nanoclay A-modified US-127 PG 64-28 asphalt binder. With 2% Nanoclay A reinforcement in the asphalt binder, the complex shear modulus increased by approximately 66% while the 4% Nanoclay A reinforcement in the asphalt binder increased the shear complex modulus by 125%. Fig. 8 shows the DSR plot for the complex shear moduli difference between the original US-127 PG 64-28, 2% Nanoclay B-modified and the 4% Nanoclay

B-modified US-127 PG 64-28 asphalt binders. With 2% Nanoclay B reinforcement in the asphalt binder, the complex shear modulus increased by 184% while the 4% Nanoclay B reinforcement in the asphalt binder generally increased the shear complex modulus by 196%. An interesting feature in the shear complex moduli results of the DSR test is their convergence as the frequency increases from 0.01 Hz to 100 Hz. It is believed that as the frequency increases to 100 Hz (or traffic loading time decreases), the nanoclay molecules play less role in bearing the shearing load compared to low frequency or increased traffic loading where the action of the nanoclay molecules starts to become prominent. Therefore, it could be stated that at low frequency, both the action of the asphalt binder and nanoclay are significant, and at high frequency, the asphalt binder tends to become more significant than the nanoclay.

4.4. Direct tension test

4.4.1. Failure strain percent (%)

Fig. 9 shows the average strain at failure for the five different samples with an error bar of one standard deviation. The results show that the strain at failure decreases by 78% with the addition of 2% of Nanoclay A. Four percent of Nanoclay A modification of the US-127 PG 68-24 increases the strain at failure 64% above that of the 2% Nanoclay-modified asphalt binder. With the Nanoclay B modification, the strain at failure initially decreases by 47% with the addition of 2% of Nanoclay B. The strain at failure then increases slightly by 5.5% when the Nanoclay modification is increased from 2% to 4%. Between the 2% addition of Nanoclay and Nanoclay B modification of the asphalt binder, the strain at failure of the Nanoclay B modified asphalt increases over the Nanoclay B modified asphalt binder by 140%, which can be considered as highly significant. At 4%, the Nanoclay B increases the failure strain by 55% over the Nanoclay A modified asphalt specimen. Based on these results, it is proved that the use of Nanoclay B strengthens the tensile strength properties of the asphalt binder better than the Nanoclay A modifier since a lower failure strain% increases the potential for low-temperature cracking. The low-temperature cracking potential of the original binder was better than both Nanoclays A and B at both 2% and 4%; however, further research on nanoclay modification at percentages higher than 4% are being pursued to determine if this problem may be mitigated.

4.4.2. Secant moduli under direct tensile test

The secant modulus of the DTT results for the five different samples can be seen in Fig. 10 with error bars of one standard

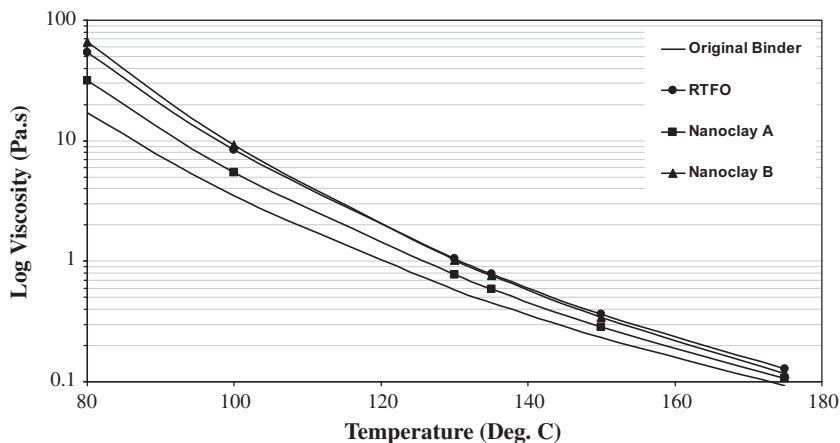


Fig. 6. Rotational viscosity plots for original binder, RTFO binder, Nanoclay A and Nanoclay B modified asphalt binder.

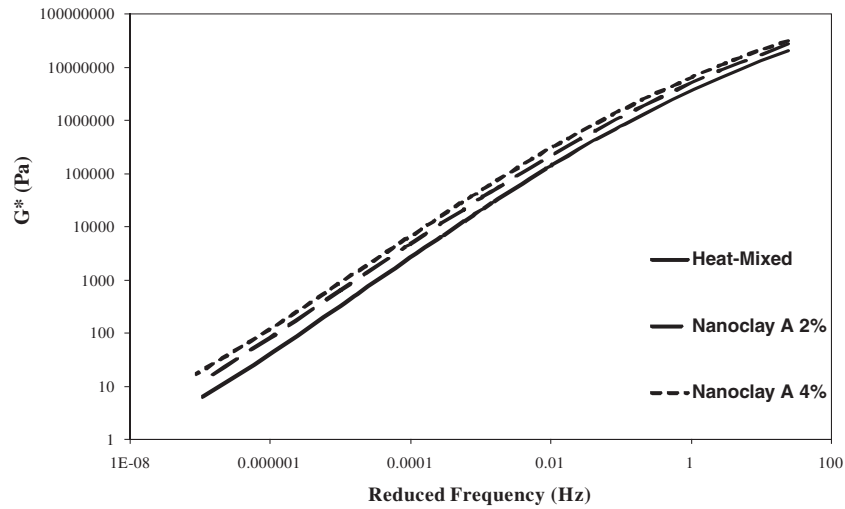


Fig. 7. DSR shear complex moduli (G^*) plot on original asphalt, 2% Nanoclay A modified asphalt, and 4% Nanoclay A modified asphalt.

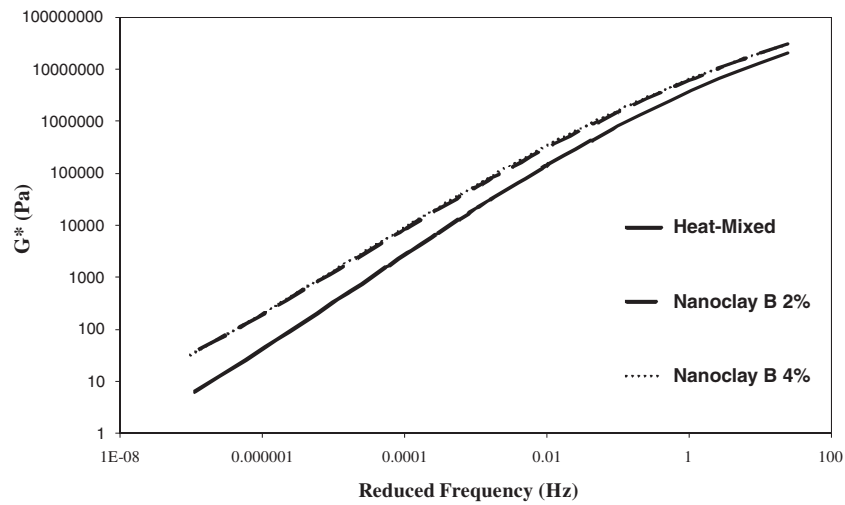


Fig. 8. DSR shear complex moduli (G^*) plot on original, 2% Nanoclay B modified and 4% Nanoclay B modified asphalt binder.

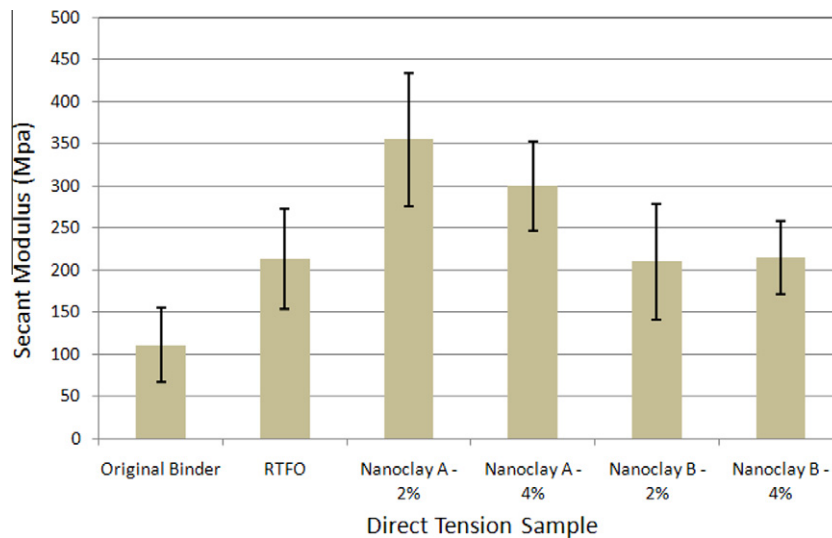


Fig. 9. Direct tension strain at failure% results for original, RTFO, Nanoclay A (2% and 4%) and Nanoclay B (2% and 4%).

deviation. The secant modulus is defined as the failure stress divided by the failure strain and provides an approximate value for the modulus of the stress/strain curve. The original binder sample

has the lowest secant modulus value when compared to the modified asphalts. The Nanoclay A modified specimen – 2% and 4% – had moduli greater than the original binder but the further

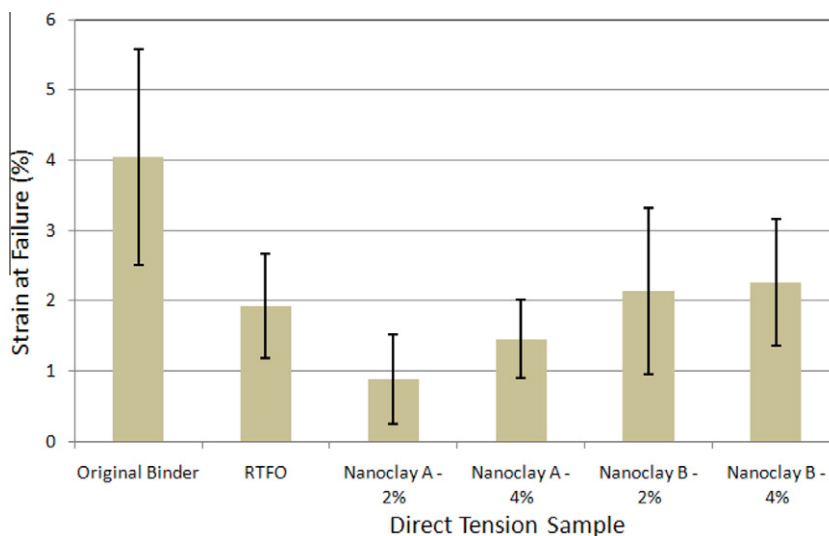


Fig. 10. Secant moduli results for original, RTFO, Nanoclay A (2% and 4%) and Nanoclay B (2% and 4%).

addition of the modifier from the 2% to 4% caused a reduction in the secant moduli. With the Nanoclay B modified specimen, there was insignificant difference between 2% and 4%.

4.4.3. Toughness under direct tensile test

The area under the stress and strain curve for the direct tensile was used to determine the toughness of each of the tested materials. Fig. 11 shows the trend that both the addition of Nanoclays A and B increase the toughness of the asphalt binder relative to the original and RTFO binder. This indicates that both the original and RTFO-aged binder do not have the toughness that the Nanoclay particles add to the asphalt. The trend can be explained by the stiffening behavior of the nanoclay as they form bond chains within the binder. Furthermore, the toughness of 4% Nanoclay A was higher than that of 2% Nanoclay A and this was expected as the percent of nanoclay corresponds to the stiffening effect. Similarly, 4% Nanoclay B had higher toughness than 2% Nanoclay B based on the same explanation. It was also seen that the error bars for the all nanoclays were higher than those for the original binder. This could be due to the distribution of nanoclays within the asphalt binder may have induced some different pockets of variable strength properties with the direct tension dog bone sample. This created some sense of variability within the test samples.

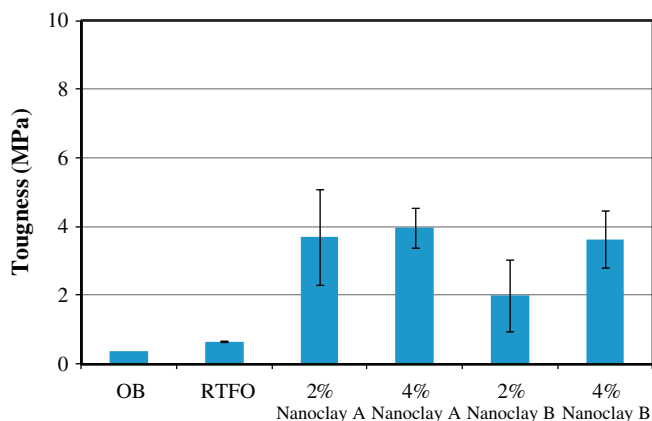


Fig. 11. Toughness results for original binder, RTFO short term aged binder, Nanoclay A (2% and 4%) and Nanoclay B (2% and 4%) modified binders.

This indicates that the shear mixing of the nanoclays into the asphalt binders may need further improvement.

5. Summary of findings to date

Using a shear mechanical blending device at 2500 rpm, it is possible to uniformly mix asphalt binder with nanoclay particles. The preliminary results from this research indicate that nano-modified asphalt binders hold promise for developing increased viscosities and complex shear moduli (G^*) with as little as 2–4% nano-material by weight of asphalt. Variances in percent nano-material result in minimal changes in G^* and viscosity; however, changes do appear to increase with an increase in nano-material composition. The tensile strength results show that the addition of either Nanoclays A or B (2% and 4%) caused a reduction in the failure strain%. Furthermore, the secant or direct tension moduli showed significant increase with the addition of the nanoclays.

6. Conclusions

This research has shown to a large extent that nanoclays can be effectively used as a modifier to improve the mechanical properties of asphalt binders. Two different nanoclay materials were used in the research – Nanoclays A and B. Both nanoclays used in this study improved the G^* and viscosity. Nanoclay B binder had more pronounced improvements in G^* and viscosity than Nanoclay A. The direct tension test indicates that Nanoclay A asphalt has better low-temperature cracking resistance than the Nanoclay B asphalt. Finally, it is noteworthy that the blending procedure is crucial in achieving well distributed nanoclay asphalt. Furthermore, the research team plans to conduct investigations on using nanoclays at different concentrations besides 2% and 4% to provide extensive test results to guide the use of nanoclays in asphalt binder. The ongoing research will focus on the low-temperature cracking performance and fatigue resistance of the nanoclay-modified binder as well as the asphalt concrete mixtures made with nanoclay-modified asphalt.

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