Influence of Clay Nanoparticles on the Physical and Rheological Characteristics of Short Term Aged Asphalt Binder

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Key words: Asphalt, montmorillonite, nanoclay, aging, physical properties, rheological properties.

This research paper presents laboratory investigation on the physical and rheological properties of asphalt binder modified with Organic Montmorillonite Nanoclay (OMMT). Two different concentrations (1% and 7% by weight of base asphalt) of OMMT was selected to blend with 80/100 penetration grade asphalt binder. The base as well as the OMMT modified asphalt binders was subjected to short term aging process by means of Rolling Thin Film Oven test (RTFO) in order to investigate the influence of the addition of OMMT nano clay in the asphalt binder properties after aging. Bituminous binder properties were investigated by both physical and rheological methods. In general, the physical test results demonstrated prominent increment in softening point; viscosity and decrement in penetration for both concentration of OMMT modified binder as compared to non-modified binder. The results of the experiments indicated that the addition of nano particles was helpful in increasing the complex modulus values and in improving rutting resistance of the RTFO binder. The phase angle of the binders generally decreased with an increase in nano content and RTFO aging procedure. Also, the results indicated that modified binders show better rheological properties compared to standard bitumen. The addition of OMMT to base asphalt binder has led to noticeable improvements in aging resistance this may be due to the homogeneous dispersion of nano particles consisting of layer silicate in the asphalt medium. Thus, nano clay is foreseen as a novel kind of resistance to aging and permanent deformation potential for bitumen.

INTRODUCTION

The focus on the efficiently of pavement structure is the most important issue that attract the interesting of the road engineers as the paved road reflect one of the progress features of the countries. It is well known that the pavement using the conventional asphalt binder safer from many distress during its construction and service life. The fatigue, rutting, thermal cracking and moisture induced damage are the most familiar distress that threatened the pavement structure (Lottman, 1978). In order to improve the asphalt properties, many of additives were adopted. Polymer, rubber, fiber and filler are the most known modifiers that used to enhance and reinforce the asphalt properties (Shad and Sang-Soo, 2003, Takaloulet al., 1987, Serfass and Samanos, 1996, Little and Epps, 2001) but, the thinking towards new materials which can collect new promising properties such as the lower price and the environmental friendly in addition to improving the binder and mixture properties, still occupied the researcher’s thinking. Nanotechnology using nano material now a day, is the most interesting technology that adopted by mostly the industries and academicians and showed unexpected reactive by the community. Montmorillonite nanoclay is one of the nano materials, which the silica is the dominant constituent and that used successfully by many researchers to enhance polymer properties since its small size gain it new promising properties when added to other material (Richard et al., 1993).

Recently this nano material attracted the attention of the asphalt pavement researchers and with the expected of more positive result, small amount of nanoclay was added as a third part to enhance the compatibility between the polymer and asphalt binder, the results showed remarkable improvement in modified binder’s properties comparing with the unmodified one (Markandy et al., 2010, Saeed et al., 2010, Zhang et al., 2009, Polacco et al., 2008, Yu et al., 2008, Jianying et al., 2007).

The statement mentioned above gave the motivation to few researchers to adopt this nanomaterial to modified asphalt binder where two types of nanoclay: nanofil and cloisite tested by(Ghilie, 2006), and the results shown an improvement in various asphalt binder and mixture properties including stiffness, rutting,
aging resistance and indirect tensile strength, dynamic creep, and fatigue resistance for mixture. (Jianying, 2009), in his study investigated the effect of two types of montmorillonite nanoclay: inorganic and organic montmorillonite the thermo-oxidative and ultraviolet (UV) aging properties of asphalt, the test result indicated that the addition of montmorillonite had restrained the effect of thermo-oxidative and ultraviolet aging on modified binder properties. The effect of Nanofil-15 and Cloistore-15A on asphalt properties were explored by (Jahromi, 2009); properties like the stiffness, and aging resistance where clearly enhanced after adding the nanoclay to asphalt bitumen. Organic nano-montmorillonite has chosen by (Shaopeng, 2010) to prepare the nanoclay modified bitumen, the study carried out to evaluate the fatigue resistance property of the modified bitumen, the research results showed that the modified bitumen with nanoclay exhibited better fatigue resistance. Different contents of organic montmorillonite were selected by (Gang Liu, 2009), the morphology and the thermal properties studied before and after aging, the research results indicated that the availability of nanoclay improved aging resistance of asphalt binder. A lot of studies have been done on nanoclay modified polymers and other materials but the research studies on this promising material to modified asphalt binder still unclear due to lack of published research on this aspect. Also, very little information is available regarding the effect of nano materials on the characteristics, rheological properties, and viscoelastic behavior of nano material modified asphalt (nanocomposite). However, the characteristics of this material can significantly influence the properties of the nanocomposite, and thus can have a significant effect on mixture performance.

To better understand and characterize the interaction of nanoclay with the asphalt binder, more effort and studies need to be carried out on nanoclay modified asphalt binder to determine its performance. It is clear that a fundamental understanding of rheological properties will be vital of importance in practical application of nanoclay modified asphalt. Thus, besides the physical properties, this paper will focus on investigation of rheological and deformation properties of unaged and aged bituminous binders using Dynamic Shear Rheometer, which is considered to be an effective and common approach to obtain the desired purpose mentioned above.

**MATERIALS AND METHODS**

**Asphalt Binder:**

80/100 penetration grade asphalt binder was use in this study as base asphalt. Most agencies would normally specify a higher performance grade (PG) such as a PG 76 instead of the 80/100 penetration grade for highway construction projects. Since the performance of the nanoclay particles were of concern in this study, the commonly used 80/100 penetration grade soft binder was intentionally selected. This is to make sure there are no additional properties derived from additives if modified binders such as 60/70 and PG 76 were used. The properties of base asphalt binder are shown in Table 1.

**Table 1:** Properties of base asphalt binder 80/100 penetration grade.

<table>
<thead>
<tr>
<th>Binder Properties</th>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Penetration at 25°C (dmm)</td>
<td>ASTM D5</td>
<td>80</td>
</tr>
<tr>
<td>Softening Point (°C)</td>
<td>ASTM D36</td>
<td>46.5</td>
</tr>
<tr>
<td>Viscosity at 135°C (Pa.s)</td>
<td>ASTM D4402</td>
<td>0.379</td>
</tr>
<tr>
<td>Ductility at 25°C (cm)</td>
<td>ASTM D113</td>
<td>&gt;100</td>
</tr>
<tr>
<td>Specific Gravity (g/cm³)</td>
<td>ASTM D70</td>
<td>1.03</td>
</tr>
<tr>
<td>G'/sin δ at 64°C (kPa)</td>
<td>AASHTO TP5</td>
<td>1.21</td>
</tr>
<tr>
<td>RTFOT aged G'/sin δ at 64°C (kPa)</td>
<td>AASHTO TP5</td>
<td>3.93</td>
</tr>
</tbody>
</table>

*American Society for Testing and Material
**American Association of State Highway and Transportation Officials

**Nanoclay:**

One type of treated commercially available organic montmorillonite nanoclay (OMMT) particles (N3) was utilized in this study. The OMMT was treated to improve the properties of additives for bitumen; the nanoclay has to be modified by surface treatment to ensure separation of layers. This makes an intensive interaction between the nanoclay and bitumen (László et al., 2006). Two percentages (1 and 7% by weight of the base binder) of nanoclay particles and the control binder were employed, and these particles were blended with base asphalt binder. Nanoclay used in this study refers to organically modified montmorillonite (OMMT). The nanoclay properties are shown in Table 2.

**Table 2:** Nanoclay properties.

<table>
<thead>
<tr>
<th>OMMT Treatment</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Montmorillonite</td>
<td>Dimethyl benzyl (hydrogenated tallow alkyl) ammonium cations</td>
</tr>
<tr>
<td>CEC</td>
<td>90-120 mmol/100g</td>
</tr>
<tr>
<td>Color</td>
<td>white</td>
</tr>
<tr>
<td>Appearance</td>
<td>fine</td>
</tr>
<tr>
<td>X Ray Diffraction Properties d002 (nm)</td>
<td>d = 2.8</td>
</tr>
</tbody>
</table>
Preparation of OMMT/Asphalt Binders Composite:

At first, bitumen was heated to 20°C higher than the target mixing temperature of 150±5°C in order to facilitate mixing. The appropriate mixing temperatures were determined following ASTM D4402 using the rotational viscometer. Nanoclay/asphalt binder composites were prepared by blending the predetermined nanoclay content with asphalt binder in aluminum can; the aluminum can was placed in a container that filled with fine material in order to prevent heat lost via the conductivity of aluminum can outer layer. The whole setup was then placed on a hot plate to provide uniform heating. A mechanical mixer module IKA Labortechnik, RW 20 DZM.n was used to blend the nanoclay and asphalt binder at the established mixing temperature. Then, nanoclay with appropriate contents (around 1 gram per minute) was added to the preheated bitumen in a shear blender. An "X" shaped propeller is used to stir the nanoclay/asphalt binder composites, the propeller speed was kept fixed at approximately 500 revolutions per minute (rpm) to reduce splashing and eliminate the air bubbles entrapped inside the sample and the mixing temperature was kept constant to produce homogeneous mixtures during the mixing process. After the completion of nanoclay addition the operated speed increased to 2000 rpm for entire 1 hour to ensure good dispersion of OMMT in binder. At the end of the mixing operation, the nanoclay/asphalt binder composites were used to prepare specimens for the penetration, softening point, viscosity, and DSR tests. The RTFO short term aged properties of nanocomposites were compared with unaged and original binder.

Aging Process of Asphalt Binder:

Hardening due to oxidation which is the reaction with oxygen from the environment and due to volatilization which is the loss of light oils in the asphalt is a main cause of aging (Read et al., 2003). According to Strategic Highway Research Program; asphalt aged during the mixing, manufacturing, lay down process and during service life (SHRP-A-367, 1994). For simulation the short term aging process that occurs in asphalt binder during manufacturing and construction of asphalt pavement, the Rolling Thin Film Oven test (RTFO) was adopted in this study by measuring the effect of heat and air on a moving film of asphalt binder according to the requirement of (ASTM D2872) and (AASHTO T-240) standard. In order to determine the influence of short term laboratory aging resistance of samples, the organic montmorillonite nanoclay (OMMT) modified asphalt binder and the non-modified samples were aged at standard aging temperature of 163 °C and air flow 4000 ml/min for 85 minute using the Rolling Thin Film Oven test. When bitumen gets aged it becomes harder and the increment in hardness due to aging in relation to their fresh value can be assessed by retained penetration (RP) and increment in softening point (ISP) values. They are defined as follow:

\[ RP(\%) = \frac{\text{aged penetration}}{\text{unaged penetration}} \times 100 \]  

\[ ISP(°C) = \text{aged softening point} - \text{unaged softening point} \]  

In addition, temperature susceptibility of bituminous binders has been calculate din terms of penetration index (PI) using the results obtained from penetration and softening point tests. The classical approach related to PI calculation has been given in the Shell Bitumen Handbook (Read et al., 2003) as the following equation:

\[ PI = \frac{1952 - 500 \times \log (Pen \ 25) - 20 \times SP}{50 \times \log (Pen \ 25) - SP - 120} \]  

Where Pen25 is the penetration at 25°C and SP is the softening point temperature of bituminous binders, respectively.

Physical Properties of Base and Modified Asphalt Binder:

The physical properties before and after short term aging of the both base and OMMT modified asphalt binders were characterized using penetration test at 25°C in accordance to ASTM D5, softening point test ASTM D36 and viscosity at two test temperature 135°C and 165°C using Brook field Viscometer in accordance to ASTM D4402.

Rheological Properties of Base and Modified Asphalt Binder:

The DSR is generally used to characterize both viscous and elastic behaviors of bitumen by measuring the complex shear modulus (G*) and the phase angle (δ) where the G* represent the total resistance of a binder to deformation while exposed to repeated pulses of shear stress, while the δ is an indicator of the relative amounts of the recoverable and non-recoverable deformation. Different sizes of plate diameters and different thickness of
bituminous binders are considered for different test temperatures, which were based on the expected stiffness of bituminous binders in the temperature ranges, on preliminary testing and past experience with DSR device. The standard thickness of bituminous binders for different plate sizes over different temperature ranges were: 2 mm for 8 mm diameter plate over a temperature range of -10°C to 20°C and 1 mm for 25 mm diameter plate over a temperature range of 30°C to 60°C (The Asphalt Institute, 2007, Richard, 2009).

In dynamic mechanical analysis, the temperature susceptibility of bitumen may be evaluated by measurements of various viscous and elastic parameters (e.g., storage and loss modulus, dynamic, and complex viscosities) at different temperatures and frequencies (Lu and Isacsson, 1998). Asphalt mixtures containing the binders with lower temperature susceptibility should be more resistant to cracking and rutting at low and high temperatures, respectively. In order to get a good knowledge of the rheological properties of bituminous binders, HAAKE, Rheo Stress RS1, Phoenix, Dynamic Shear Rheometer has been used for characterizing the both viscous and elastic behavior of unaged and short term aged unmodified base and modified OMMT asphalt binders.

In this present study, 1 mm thick for 25 mm diameter plate at fixed frequency of 10 rad/sec and controlled stress mode of 0.12 kPa and 0.22 kPa for unaged and short term aged unmodified base and nanoclay modified asphalt binders, respectively is used in accordance to the Strategic Highway Research Program asphalt binder specifications (SHRP). The DSR rutting parameter \( G^* / \sin \delta \), was used as an indicator of the stiffness and rutting resistance of asphalt binders to permanent deformation. According to the SHRP Superpave PG binder specification, the minimum values for \( G^* / \sin \delta \) is limited to 1000 Pa for original asphalt binder, 2200 Pa after Rolling Thin Film Oven aging and 5000 kPa after Pressure Aging Vessel (PAV) procedure (AASHTO TP5, 1993). Temperature susceptibility is usually defined as the change in binder properties as a function of temperature. Since binder properties may be characterized by means of various parameters, different approaches have been proposed to evaluate temperature susceptibility. Since rutting is more prevalent at high temperatures than at intermediate or low temperatures, the properties related to rutting should therefore be measured in the upper range of pavement service temperatures. A DSR Oscillation Temperature Steps was performed at intermediate and high temperature ranged from 40 to 82°C with 6 degree increment to determine the change in binder properties as a function of temperature. The SHRP rutting factor \( G^* / \sin \delta \), elastic (storage) modulus (G) represents the elastic storage of energy, because the strain is recoverable in an elastic solid and can be defined as \( G = G^* \cos \delta \). In addition, viscous (loss) modulus (G’) is described as viscous dissipation of energy through permanent deformation in flow and is defined as \( G = G^* \sin \delta \) were measured at temperature of 64°C for unaged and RTFO aged base and modified OMMT asphalt binders.

RESULTS AND DISCUSSION

Physical Properties of Nano composite:

Consistency of OMMT/Asphalt Binders Composite:

The penetration test provides a measure of consistency or hardiness of bitumen, and softening point test determines the temperature at which a specific viscosity of bitumen is reached at some point during its transition from solid to liquid. The softening point of a bituminous material does not take place at any definite temperature, but rather involves a gradual change in consistency with increasing temperature. The variation in softening point and penetration values before and after RTFO aging for both base and OMMT modified asphalt binder can be seen in Fig.1 (a-b). Under the same aging condition; it can be clearly notice that; the softening point value increased and the penetration value of modified binder decreased by increasing the nano particles content due to formation of exfoliated (layered) structure. In addition, the RTFO aged modified binder has a higher softening point and lower penetration values compared to both base and unaged modified binder. The increase in softening point and decrease in penetration are a result of the stiffening effect of nanoclay. The increase in softening point temperature is favorable since bituminous binders with higher softening point may be less susceptible to permanent deformation (rutting).

The effect of adding OMMT on aging properties of asphalt binder can be determined using 'Eq. (1), (2), and (3)’ above. A lower retained penetration and lower Penetration index (PI) value and higher increment in softening point reflect more aging of the binder. The results in Table 3 indicated that when content of OMMT reaches to 7%, the difference between softening point after and before aging is decreased by 14% after adding OMMT. Also, retained penetration increased by 11.18% as well as penetration index (PI) by 19.74%. In another word, nanoclay modified binder exhibit less temperature susceptibility compared to base asphalt binder with increasing OMMT content which confirms once again that adding the OMMT reduces/or improve aging resistance of bituminous binders and improve (reduce) temperature susceptibility.
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Fig. 1(a-b): Penetration and Softening point of the modified asphalts with various concentration of OMMT (a) before and (b) after short term aging.

Table 3: Retained Penetration, Increment in Softening Point, and Penetration Index of base and RTFO aged asphalt binder.

<table>
<thead>
<tr>
<th>Nanoclay content (%)</th>
<th>Retained Penetration (RP) (%)</th>
<th>Increment in Softening Point (ISP) °C</th>
<th>Penetration Index (PI) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>62.50</td>
<td>5.0</td>
<td>-0.988</td>
</tr>
<tr>
<td>1</td>
<td>65.75</td>
<td>4.7</td>
<td>-1.003</td>
</tr>
<tr>
<td>7</td>
<td>69.49</td>
<td>4.3</td>
<td>-1.135</td>
</tr>
</tbody>
</table>

Viscous Flow:
The viscosity of asphalt is used to determine the flow characteristics of an asphalt binder to provide some assurance that it can be pumped and handled at the hot mixing facility. The viscosity values of unaged and RTFO binders with or without nanoparticles were measured at intermediate and high temperatures of 135°C and 165°C respectively, are shown in Fig. 2. It can be seen that the addition of the nanoparticles increased the viscosity of the binders at both temperatures regardless of the aging state. In addition, the RTFO aged modified binder exhibit higher viscosity value than the unaged modified binder. Moreover, the binder with a higher percentage of nanoparticles has a noticeably higher viscosity value. The reason might be that, the very high surface area of clay nanoparticles results in a stronger interaction between asphalt binder and nanoparticles which make the movement of bitumen molecule chains is limited by the layers of OMMT at high temperature during formation of an exfoliated (layered) structure. This indicates that the nanoparticles make the binders stiffer at the maximum pavement service temperature, which is beneficial in improving resistance to permanent deformation.

Fig. 2(a-b): Viscosity at 135°C and 165°C of the modified asphalts with various concentration of OMMT (a) before and (b) after short term aging.
**Dynamic Rheological Properties of OMMT/Modified Binders:**

**Dynamic Modulus and Phase Angle:**

In Superpave specifications, phase angle is defined as the time lag between strain and stress under the traffic loading, and is highly dependent on the temperature and frequency of loading. It can be used as an indicator of viscosity and elasticity of binders. Under normal pavement temperatures and traffic loadings, asphalt binders act with the characteristics of both viscous liquids and elastic solids while the complex shear modulus ($G^*$) represent the total resistance of a binder to deform while exposed to repeated pulses of shear stress (The Asphalt Institute, 2007).

In this study, all specimens, unaged and RTFO residues containing nanoclay particles were tested at the high performance temperatures of 40 to 82°C and thus exhibit viscoelastic properties. The isochronal plot in Fig. 3 and Fig. 4 displays the influence of temperature variation on the complex modulus and phase angle values of various samples from base and OMMT modified binder (in unaged and aged states) at 40 to 82°C. As shown in Fig. 3 and Fig. 4, phase angles of all RTFO binders were consistently reduced as the percentage of nanoparticles increased from 1 to 7%, and the dynamic modulus were increased across the entire temperature domain of the plots compared to unaged modified binder, this consistent with the results obtained from the physical tests which showed that 7% concentration of nanoclay has caused the highest stiffening effect regardless of aging state. As expected, phase angle values of all modified binders have increased by increasing the temperature, contrary the complex modulus decreased regardless aging state. In addition, it seems that the RTFO phase angle of modified binders is lower; where as unaged binders have higher phase angle values. Although the samples with various percentages of nanoclay particles have similar trend, the binder containing 7% nanoclay particles presents the lowest phase angle and the highest complex modulus values. This indicates that the addition of nanoparticles increases the elastic characteristics of the binder. It was concluded that, for the binders tested, the addition of nanoparticles causes an increase in the elastic property of binders after RTFO aging, and has an influence on the elastic and viscous characteristics of an asphalt binder. In addition, the existing of the nanoclay layers lead to minimize the volatilization of oily asphalt’s component while subjected to RTFO aging which might strength the bonding with asphalt and thus led to improve its rheological properties.

![Fig. 3: Isochronal plot of the complex shear modulus ($G^*$) and phase angles ($\delta$) vs. temperature variation before aging.](image-url)
Rutting Factor ($G^*/\sin \delta$) of OMMT/Modified Binders:

Owing to the physical characteristics of a typical asphalt material, asphalt concretes are more susceptible to rutting at a high-service temperature when asphalt binder has a lower viscosity and are more likely to creep under heavy traffic loads. In Superpave specifications, $G^*/\sin \delta$ is used as a key factor to define the permanent deformation of an asphalt binder at a high performance temperature (SHRP-A-410, 1994, Airey et al. 2008). As shown in Fig. 5, the $G^*/\sin \delta$ value increases as the percentage of nanoclay particles increases, regardless of the aging state. In Fig. 5, all RTFO $G^*/\sin \delta$ values are higher than 2.2 kPa at 64°C, and at 7% is even greater than 6.0 kPa. At 64°C, the $G^*/\sin \delta$ values of unaged binders with 1% and 7% nanoclay particles are higher than 1.0 kPa. The RTFO with 7% nanoclay exhibits a significantly higher $G^*/\sin \delta$ value which increased the rutting resistance by almost two times at 64°C compared to base and 1% modified binders. Obviously, the addition of nanoclay particles is beneficial in improving the rutting resistance of RTFO asphalt binder. It was concluded that the huge surface area and the existing of layer silicate of nanoparticles results in a stiffer asphalt binder.

Deformation Resistance of OMMT/Modified Binders:

Unlike purely elastic substances, asphalt binder, a viscoelastic substance, has an elastic component and a viscous component. Purely elastic materials do not dissipate energy (heat) when a load is applied and then removed, whereas a viscoelastic substance loses energy. The binder should have a large value of storage
modulus $G$ at high temperatures for deformation resistance because $G$ measures the binder elasticity (Lu X. and Isacsson U. 1998). High elasticity is convenient at high temperatures to avoid viscous flow of the binder.

The elastic and viscous modulus values of base and nanoclay modified binders tested at temperature of 64°C before and after short term aging are shown in Fig. 6(a-b). Similar to previous results, the addition of nanoparticles resulted in an increase in elastic modulus for all binders regardless aging state and RTFO binder with 7% nanoclay particles has the highest value. In addition, the RTFO binder exhibits a higher elastic modulus value at 64°C in comparison with an unaged base and nanoclay modified binder at the same temperature. This increase in elastic modulus may be because of the interactions between nanoparticles and asphalt binders. It is clear from the figures that the viscous (loss) modulus and elastic (storage) modulus of nanoparticles modified binder are higher than that of base binder regardless aging state.

![Fig. 6(a-b): Relationship between (a) elastic (Storage) modulus and (b) viscous (Loss) modulus with nanoclay concentration at 64°C and frequency of 10 rad/s (1.59 Hz) for OMMT modified asphalt binder.](image)

It can be concluded from Table 4 that the modification index (storage shear modulus, $G'$) of [Gnanoclay/G'binder] is higher than deformation resistance, $G^*$, [G* nanoclay /G* binder] at 64°C in both unaged and aged states. By increasing OMMT content $G^*$, $G$, and $G'$ are increased. This means that a continuous uniform nanoparticles network exfoliated structure is formed and nanoclay tends to show more elastic behavior at high temperatures, and this leads the elastic behavior to be dominant gradually.

<table>
<thead>
<tr>
<th>Nanoclay Content (%)</th>
<th>Elastic Modulus</th>
<th>RTFO aged</th>
</tr>
</thead>
<tbody>
<tr>
<td>1%</td>
<td>$G^{\text{nanoclay}} / G^{\text{binder}}$</td>
<td>2.450</td>
</tr>
<tr>
<td>7%</td>
<td>$G^{\text{nanoclay}} / G^{\text{binder}}$</td>
<td>3.217</td>
</tr>
<tr>
<td></td>
<td>$G^\text{RTFO aged}$</td>
<td>1.267</td>
</tr>
<tr>
<td></td>
<td>$G^\text{RTFO aged}$</td>
<td>1.806</td>
</tr>
</tbody>
</table>

**Temperature Steps Tests of OMMT/Modified Binders:**

The temperature steps tests were performed under stress control and frequency of 10 rad/s (1.592 Hz) and employed a range of temperature from 40 to 82°C. The overall temperature steps tests were run with a 25 mm diameter and 1 mm testing gap geometry complex modulus and phase angle were evaluated along with elastic and viscous modulus in accordance with various temperatures. Fig. 7 displays the influence of temperature variation on the rutting parameter ($G' / \sin\delta$) values of various samples from base and OMMT modified binder (in unaged and aged states) at 40 to 82°C. An increase in temperature yields decrease in the modulus values for base and OMMT modified binders, regardless of aging state or the presence of nanoparticles. Moreover, the RTFO samples generally have higher rutting parameter values than unaged samples.

**Failure Temperature:**

Fig 7 illustrates that addition of nanoparticles results in an increase in failure temperature for all modified binder specimens, regardless of the aging state. However, this increase is generally different for various RTFO binder specimens. As presented in Table 5, it can be noted that RTFO with 7% nanoclay particles has the highest failure temperature. Whereas base binder has the lowest among all OMMT modified binders. The failure properties test results in Table 5 indicated that; for unaged nanocomposite with 7% nanoclay content increased the high temperature stiffness by one grade. As for RTFO aged nanocomposite with 7% nanoclay content increased by one grade i.e the high temperature grade was one grade higher than the base asphalt binder for both
unaged and RTFO aged, this means introducing nanoparticles to the mix reduces the aging effect. As a result, one might conclude that nanoclay concentration plays an important role in determining the influence of nanoparticles on binder failure temperature and in reducing deformation affect and enhance aging resistance. This is consistent with the results obtained from RP and ISP test results.

**Fig. 7:** Rutting parameter (G*/sin δ) measured at 64°C and frequency of 10 rad/s (1.592 Hz) as a function of temperature for 80/100 asphalt modified with OMMT before and after ageing.

**Table 5:** PG-Grading of OMMT modified asphalt binder.

<table>
<thead>
<tr>
<th>OMMT failure property</th>
<th>Aging state</th>
<th>Unaged OMMT</th>
<th>RTFO aged OMMT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>OMMT content</td>
<td>0%</td>
<td>1%</td>
</tr>
<tr>
<td></td>
<td>OMMT content</td>
<td>0%</td>
<td>1%</td>
</tr>
<tr>
<td>Failure temperature at G*/sin δ = 1kPa</td>
<td></td>
<td>64.7</td>
<td>67.6</td>
</tr>
<tr>
<td>SHRP PG (high temperature grading)</td>
<td></td>
<td>64</td>
<td>64</td>
</tr>
<tr>
<td>Failure temperature at G*/sin δ = 2.2kPa</td>
<td></td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SHRP PG (high temperature grading)</td>
<td></td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

**Conclusions:**

The following conclusions are drawn based on the results obtained from this limited study:

- Temperature steps results show that the addition of a higher percentage of nanoparticles results in greater complex modulus and lower phase angle values in both RTFO aged and unaged states.
- Results illustrate that the nanoparticles can effectively improve the deformation resistance of an asphalt binder, owing to the increase of rutting factor value in both unaged and short term aged state.
- The addition of nanoparticles increases the viscosity of the binders at intermediate and high temperature regardless of the aging state. In addition, the short term aged binder has a noticeably higher viscosity value than the unaged. The results indicate that the nanoparticles make the binders stiffer at the high pavement service temperature and are beneficial in improving resistance to permanent deformation.
- Moreover, the results illustrate that the increase of nanoparticle content results in higher viscous and elastic modulus values, regardless of aging state. A low elasticity is suitable at low temperatures to facilitate dissipation, but high elasticity is convenient at high temperatures to avoid viscous flow of the binder. The study has confirmed that modification of the binders by nanoclay facilitates this phenomenon at higher temperatures.
- Compared to the unaged samples, the short term aged samples generally have higher complex modulus and lower phase angle values, owing to the oxidation of the binder regardless of nano content. Moreover, the binder with 7% nanoparticles exhibits the lowest phase angle and the highest elastic modulus, and thus its elastic characteristics are the most remarkable.
- Adding the OMMT reduces the deformation and improves resistance to aging of bituminous binders as illustrated by the results of failure temperature of short term aged and unaged nonoclay modified binders which gave the same SHRP high performance grade and this was consistent with the RP and ISP test results.
- From the test results, it is suggested that; in farther studies to try more OMMT proportions to settle the gap and to minimize the difference in the performance between 1 and 7% of OMMT to gain more knowledge about the behavior of this promising material with asphalt binder.
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