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Effect Of Nanopolymer Modified Binder On Hot Mix Asphalt

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EFFECT OF NANOPOLYMER MODIFIED BINDER ON HOT MIX
ASPHALT

by

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A Thesis
Submitted to the Graduate Faculty

of the

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for the degree of

Master of Science
In Civil Engineering

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This thesis, submitted by Abolghasem Yazdani in partial fulfillment of the requirements for the Degree of Master of Science in Civil Engineering from the University of North Dakota, has been read by the Faculty Advisory Committee under whom the work has been done and is hereby approved.



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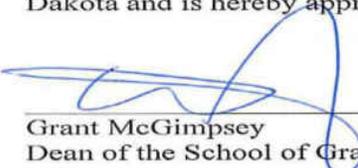
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ABSTRACT

There are some distresses in asphalt pavements such as high temperature permanent deformation (rutting), low-temperature (thermal) cracking, and load-associated fatigue cracking.

Rutting is caused by the accumulated plastic deformation (flow) in the asphalt mixture with repeated application of loads at upper pavement service temperatures. It is predominantly influenced by the aggregate and mix design. Modifiers can stiffen the binder and provide a more elastic material.

Thermal shrinkage cracking results from either a single thermal cycle where the temperature reaches a critical low temperature (Single Event Thermal Cracking) or from thermal cycling above the critical low temperature (Thermal Fatigue).

In this project, elastic polymer (SBS- styrene-butadiene-styrene) and nano TiO_2 (Titanium dioxide) were used to modify the virgin PG 58-28 binders. TiO_2 nanoparticles were used because they can accelerate the process of trapping and degrading organic and inorganic particles from the air while removing harmful air pollutants, such as NO_x , sulfur oxides (SO_x), and VOCs in the presence of UV light (sunlight).

Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV) were used to simulate short-term and long-term aging of the binder, respectively. Dynamic shear rheometer (DSR) was used to determine rheological properties of unaged, short-term and long-term aged binders. Optimum TiO_2 content in 12%SBS was determined based on binder test results. Hot mix asphalt (HMA) was designed with virgin binder (control) and binder modified with SBS and TiO_2 . Superpave gyratory compactor was used to compact mix specimens.

Asphalt Pavement Analyzer (APA), Disk-shaped compact tension (DCT), and semi-circular bending (SCB) was used to determine rutting, low-temperature cracking, and fatigue cracking resistance, respectively. Based on the results, 1wt.% TiO₂+12% SBS modified PG 58-28 binder and corresponding mix performed the best.

CHAPTER I

Introduction

1.1. General

Pavement is durable surfacing of a road, airstrip, or similar area. The primary function of a pavement is to transmit loads to the sub-base and underlying soil. Modern flexible pavements contain sand and gravel or crushed stone compacted with a binder of bituminous material, such as asphalt, tar, or asphaltic oil (flexible pavement). Such a pavement has enough plasticity to absorb shock. Concrete pavements (rigid pavement) are made of concrete, composed of coarse and fine aggregate and Portland cement, and usually reinforced with steel rod or mesh.

Composite pavement is a combination of flexible and rigid pavement. Asphalt pavement is one of America's building blocks. The United States has more than 2.7 million miles of paved roads and highways, and 94 percent of those are surfaced with asphalt (NAPA, 2016). Many of those are full-depth asphalt pavements; others are asphalt overlays used to restore the performance of deteriorating concrete pavements. Reclaimed Asphalt Pavement (RAP) from old pavement is also used in the construction (Chesner et al. 1998).

1.2. Asphalt Pavement Failures

Asphalt pavement structure consists of a surface course, an underlying aggregate base course and sub-base courses. Asphalt pavement layers are organized in the order of descending load bearing capacity with the highest load bearing capacity material on the top. Failure of any pavement layer can occur with accumulated distresses over time (Mallela et al. 2004).

Low temperature cracking is the most prevalent distress found in asphalt pavements built in cold weather climates. As the temperature drops the restrained pavement tries to shrink. The tensile stresses build up to a critical point at which a crack is formed. Thermal cracks can be initiated by a single low-temperature event or by multiple warming and cooling cycles and then propagated by further low temperatures or traffic loadings (Dave and Buttlar 2010).

Rutting is a linear, surface depression in the wheel path. Rutting is caused by deformation or consolidation of any of the pavement layers or subgrade. It can be caused by insufficient pavement thickness, lack of compaction, and weak asphalt mixtures (Chaturabong and Bahia 2017). In this research, additives were used to investigate the change in binder rheology and HMA mix performance.

1.3. Effect of Nanomaterials on Asphalt Binder Rheology

Nanotechnology has been explored to a considerable degree to address the problems in design, construction, and utilization of functional structures with at least one characteristic dimension measured in nanometers (Kelsall et al. 2005). A nanoparticle is a miniaturized particle that is measured in nanometers (nm) and is often defined as a particle with at least one dimension that is less than 100 nm. The physics and chemistry of nano-sized particles differ from those of conventional materials, primarily because of the increased surface area-to-volume ratio of nanometer-sized grains, cylinders, plates, and because of the quantum effects resulting from spatial confinement (Teizer et al. 2011a).

1.4. Effect of Polymers on Asphalt Binder Rheology

Polymers can be added, which modify the natural viscoelastic behavior of the asphalt cement; thus, affecting the ideal temperature range. There are two main classes of polymers used for this purpose: elastomers, which enhance strength at high temperatures, as well as elasticity at

low temperatures; and plasterers, which enhance strength but not elasticity. Three types of elastomeric copolymer modifiers are currently approved for use in Illinois: styrene-butadiene deblock (SB), styrene butadiene triblock (SBS); and styrene butadiene rubber (SBR). In addition to improving pavement performance at locations with extreme hot-cold temperature variations, there are other potential benefits of using polymer modified binders in HMA construction. Polymer-modified binders typically are more viscous (thicker) than unmodified binders, and tend to show improved adhesive bonding to aggregate particles (Yildirim 2007).

1.5. Problem Statement

A major distress mode of flexible pavements is permanent deformation, also known as rutting. Rutting is characterized by a depression that forms in the wheel paths and can be the result of permanent reduction in volume (consolidation/traffic densification), permanent movement of the material at constant volume (plastic deformation/shear), or a combination of the two. This mode of failure reduces serviceability and creates the hazard of hydroplaning because of accumulated water in the wheel-path ruts (Nguyen and Le 2016). Modifying high temperature rheology of an asphalt binder may affect the low and intermediate temperature rheology. The effect of polymers plus nanomaterial on low, intermediate and high temperature rheological properties and corresponding mixes were investigated.

1.6. Objectives

The objectives of this research are

1. To examine the effects of modified asphalt binder on the rheological properties of unaged, short term aged and long term aged binder.
2. Finding the optimum ratio of nanomaterial and polymer that can provide high performance as compared to virgin binder.

3. To investigate the effect of nanomaterial and SBS on mix performance in terms of rutting, fatigue cracking, and low-temperature cracking resistance.

1.7. Organization of the Thesis

Chapter I gives a brief introduction of HMA pavements, type of pavement failures, Nano-material, polymer and short background information on asphalt modification. Chapter II build on the information in Chapter I and provides more details about the major pavement failure types. It also provides information on the rheological properties of the binder and the HMA mix design. Chapter III describes the overall procedure of selecting materials, mix designs, mixing and compaction processes, testing methods and data analysis. Chapter IV includes the results and discussions from the research. Finally, conclusions, limitations and future work are included in chapter V.

CHAPTER II

Literature Review

2.1. Asphalt Cement

Asphalt is produced in a variety of types and grades ranging from hard-brittle solids to near water-thin liquids. The semi-solid form, known as asphalt cement, is the basic material used in asphalt concrete pavements (Speight 2015).

The consistency or viscosity of asphalt cement varies with temperature, and asphalt is graded based on ranges of consistency at a standard temperature. Careless temperature and mixing control can cause more hardening damage to asphalt cement than many years of service on a roadway. Air-blown asphalts typically use a softening point test. Purity of asphalt cement can be easily tested since it is composed almost entirely of organic-based asphalt, which is soluble in carbon disulfide. Refined asphalts are usually more than 99.5% soluble in carbon disulfide and any impurities that remain are inert. Because of the hazardous flammable nature of carbon disulfide, trichloroethylene, which is also an excellent solvent for asphalt cement, is used in the solubility purity tests (Koseoglu et al. 2016).

Asphalt cement must be free of water or moisture as it leaves the refinery. However, trucks that load the asphalt, may have moisture present in their tanks which can cause the asphalt to foam when it is heated above 100°C (212°F), which is a safety hazard. Specifications usually require that asphalts do not foam at temperatures up to 175°C (345°F). Asphalt cement, if heated to a high enough temperature, will release fumes which will flash in the presence of a spark or open flame. The temperature at which this occurs is called the flashpoint, and is well above

temperatures normally used in paving operations. Because of the possibility of asphalt foaming and to ensure an adequate margin of safety, the flashpoint of the asphalt is measured and controlled (Shell Bitumen 1995).

Another important engineering property of asphalt cement is its ductility, which is a measure of the ability of the asphalt to be pulled, drawn, or deformed. In asphalt cement, the presence or absence of ductility is usually more important than the actual degree of ductility because some types of asphalt cement with a high degree of ductility are also more temperature-sensitive. Ductility is measured by an extension test (AASHTO T51, 2014 ; ASTM D113, 2014), whereby a standard asphalt cement briquette molded under standard conditions and dimensions is pulled at a standard temperature (normally 25°C (77°F)) until it breaks under tension. The elongation at which the asphalt cement sample breaks is a measure of the ductility of the sample (Ruan et al. 2003).

2.2. Asphalt Pavement Distresses

The design of pavements is based on two major properties of materials. The first one is the stress-strain relationship of the material, which decides the critical values of stress and strain to be applied. The different forms of failure modes decide the second one. The two major failure modes of asphalt are fracture and permanent deformation (Tashman et al. 2005). These types of failure can be caused due to factors like rutting, fatigue, low temperature exposure, and moisture damage. These distress factors are explained in detail below.

2.2.1. Rutting

Rutting is another major distresses found in asphalt pavements that can significantly affect the pavement performance and reduce its service life. Rutting is of concern for at least five reasons: (a) if the pavement drainage system is not perfect, rutting traps water causing hydroplaning, which is a potential threat to road vehicles at high speeds; (b) as the rutting depth deepens, steering becomes increasingly difficult and sometimes dangerous; (c) if the rutting is covered with snow in cold regions, the resistance of the pavement to slippage will decrease; (d) rutting has a negative effect on the pavement roughness, generally leading to the drop of riding comfort; and (e) rutting often results in the reduction of the structural layer thickness and therefore lowers the pavement strength, inducing some damages. Therefore, the rutting depth is often used to evaluate the quality of a pavement (Henry 2000).

2.2.2. Low-temperature Cracking

Thermal or low-temperature cracking of asphalt concrete pavements is a serious problem in many regions of the United States. Low-temperature cracking is attributed to tensile stresses induced in asphalt concrete pavement due to temperature drops to extremely low levels. If the pavement is cooled to a low-temperature, tensile stresses develop as the pavement contracts. The friction between the pavement and the base layer resists the contraction. When the tensile stress induced in the pavement equals the strength of the asphalt concrete mixture at that temperature, a microcrack develops at the edge and surface of the pavement. At colder temperatures or repeated temperature cycles, the crack penetrates the full depth and width of the asphalt concrete layer as shown in Figure 2.1 (Haas et al. 1987).

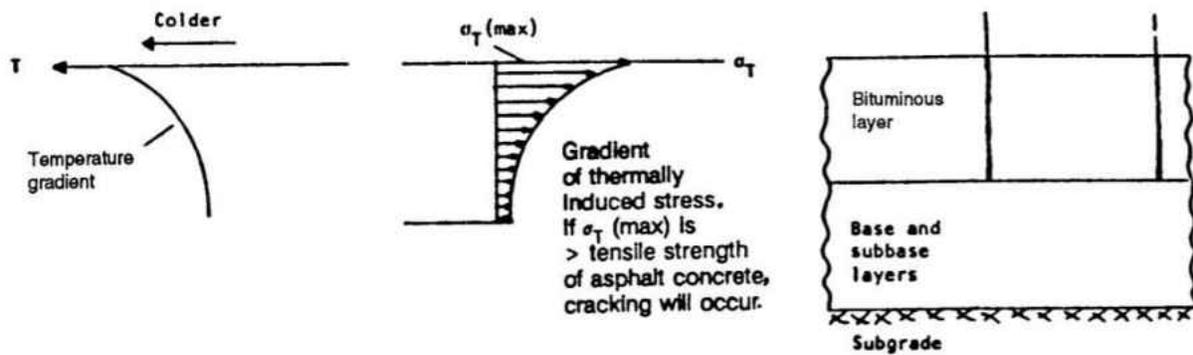


Figure 2.1. Cross section of cold pavement showing temperature and thermal stress gradient (Haas et al. 1987)

Sugawara research group reported that a typical microcrack initiates at the center or side lines, the edges of core sampling, and the corners of ditches, which are considered weak points in the pavement structure (Sugawara et al. 1982). The primary pattern of low-temperature cracking is transverse to the direction of traffic and is fairly regularly spaced at intervals of 30 m (100 ft) for new pavements to less than 3 m (10 ft) for older pavements. If the transverse crack spacing is less than the width of the pavement, longitudinal cracking may occur and a block pattern can develop as shown in Figure 2.2. Low-temperature cracks through the pavement structure create a conduit for the migration of water and fines into and out of the pavement. During the winter, the intrusion of deicing solutions into the base through the crack can lead to localized thawing of the base and a depression at the crack. Water entering the crack also freezes and forms ice lenses, which can produce upward lipping at the crack edge. Pumping fine materials through the crack will produce voids under the pavement and a depression at the crack upon loading. All of these effects result in poor ride quality and reduced pavement service life.

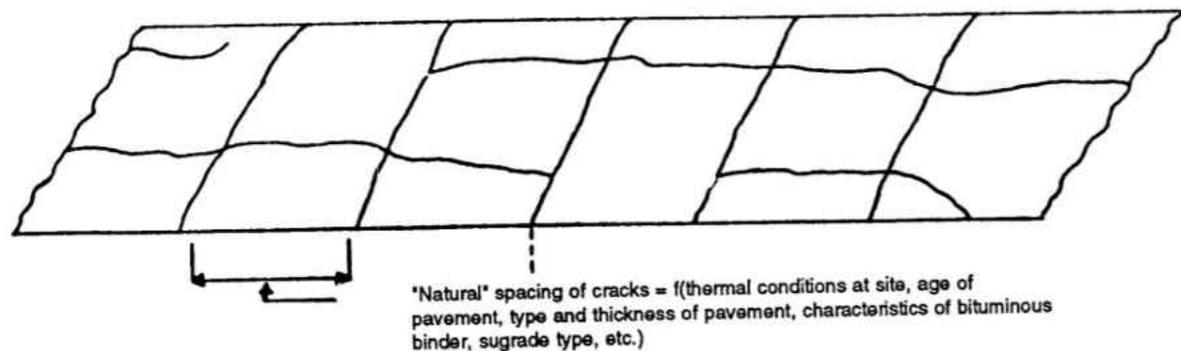


Figure 2.2. Plan view of runway with schematic illustration of typical thermal cracking pattern (Haas et al. 1987).

Factors that influence low-temperature cracking in asphalt concrete pavements may be broadly categorized as (1) material, (2) environmental, and (3) pavement structure geometry (Yoder and Witczak 1975).

2.3. Nanomaterials as Modifiers

A nanoparticle is a miniaturized particle that is measured in nanometers (nm) and is often defined as a particle with at least one dimension that is less than 100 nm. The physics and chemistry of nano-sized particles differ from those of conventional materials, primarily because of the increased surface area-to-volume ratio of nanometer-sized grains, cylinders, plates, and because of the quantum effects resulting from spatial confinement (Amiri 2011; Teizer et al. 2011b). The clay nanoparticles are the primary materials that could have application in asphalt construction based on a literature review of nanoparticles and nanomaterials. Carbon nanotubes (CNT), silica, alumina, magnesium, calcium, and titanium dioxide (TiO_2) nanoparticles can also have a significant effect on asphalt performance (Tanzadeh et al. 2017).

Photocatalysis compounds such as titanium dioxide (TiO_2) can trap and degrade organic and inorganic particles in the air and thus remove harmful air pollutants such as nitrogen oxides (NO_x) and volatile organic compounds in the presence of ultraviolet light (sunlight). Despite the rapid development of this technology, current applications are limited to concrete pavement surfaces, which represent only 6% of the national road network in the United States. About 94% of the road network in the United States is surfaced with hot-mix asphalt, a percentage that supports directing future research toward the use of TiO_2 coating in flexible pavements (Hassan et al. 2011).

2.4. Polymers as Modifier

The addition of polymers, chains of repeated small molecules, to asphalt has been shown to improve performance. Pavement with polymer modification exhibits greater resistance to rutting and thermal cracking, and decreased fatigue damage, stripping and temperature susceptibility. Polymer modified binders have been used with success at locations of high stress, such as intersections of busy streets, airports, vehicle weigh stations, and race tracks (King et al. 1999). Polymers that have been used to modify asphalt include styrene–butadiene–styrene (SBS), styrene–butadiene rubber (SBR), Elvaloy, rubber, ethylene vinyl acetate (EVA), polyethylene, and others. Desirable characteristics of polymer modified binders include greater elastic recovery, a higher softening point, greater viscosity, greater cohesive strength and greater ductility (King et al. 1999; Bates and Worch 1987).

Styrene–butadiene–styrene (SBS) is a block copolymer that increases the elasticity of asphalt (Becker et al. 2001). According to a 2001 review in *Vision Tecnologica* by Becker et al. (Becker et al. 2001), it is probably the most appropriate polymer for asphalt modification,

although the addition of SBS type block copolymers has economic limits and can show serious technical limitations. Although low temperature flexibility is increased, some authors claim that a decrease in strength and resistance to penetration is observed at higher temperatures.

Nonetheless, “SBS is the most used polymer to modify asphalts, followed by reclaimed tire rubber” (Becker et al. 2001). The Danish Road Directorate (Wegan and Nielsen 2001) found that an SBS-modified binder course showed no superior rut resistance compared to other Danish asphalt courses. Asphalt cores taken from the job site indicated that separation had occurred, and that the polymer phase was not homogeneously distributed, which might have been the cause of the poor performance of the pavement. As reported in the Journal of Material in Civil Engineering, transmission electron microscopy was used in 2002 to better understand the behavior of SBS in asphalt binders (Chen et al. 2002). Depending on the sources of asphalt and polymer, morphology varies: there can be a continuous asphalt phase with dispersed SBS particles, a continuous polymer phase with dispersed globules of asphalt, or two interlocked continuous phases. It is the formation of the critical network between the binder and polymer that increases the complex modulus, an indication of resistance to rutting.

A research group from Association of Asphalt Paving Technologists (AAPT) looked at the possibility of recycling SBS modified asphalt for resurfacing pavement (Mohammad et al. 2003). They found that the impact of the extraction and recovery process on the binder was minimal. Eight-year-old SBS modified binder was recovered from Route US61 in Louisiana, and was found to have experienced intensive oxidative age hardening. At low temperatures, the binder was quite brittle. Blends of virgin and recovered polymer modified binder were found to be stiffer than anticipated at both low and high temperatures. It was also found that as the percentage of recovered binder increased, rutting resistance increased, while fatigue resistance

decreased. In 2004, the Florida Department of Transportation and FHWA published a report (Roque et al. 2004) looking at the effect of SBS modification on cracking resistance and healing characteristics of Superpavee mixes. They found that SBS benefited cracking resistance, primarily due to a reduced rate of micro-damage accumulation. SBS did not, however, have an effect on healing or aging of the asphalt mixture. The possibility of using SBS-modified binders in India has been investigated recently (Shukla et al. 2003). Calculations indicated that the surface life of the Delhi–Ambala expressway would be almost doubled while the thickness of the bituminous layers would be reduced, although the cost per km would be greater for polymer modified binders.

2.5. Rheology of Asphalt

The flow properties of asphalts vary widely. Values of viscosity at 25°C. (77°F.) range from about 103 poises for the fluid petroleum residua to 109 poises and higher for hard asphalts. The type of flow varies from that of an essentially simple liquid (Newtonian flow) to highly complex (non-Newtonian) flow. Most asphalts are deformed continuously even by the smallest applied shearing stresses and thus do not exhibit measurable yield values. Exceptions to this are certain asphalts very high in wax content. When subjected to stress many asphalts undergo an elastic deformation in addition to the usually more predominate permanent flow. An accurate, complete and rapid evaluation of these different flow properties is the goal of rheological measurements of asphalts (Polacco et al. 2015).

The rheological properties of SBS modified asphalt blends have been investigated by many authors, including studies by Airey (Airey 2004; Airey 2003), Vlachovicova et.al. (Vlachovicova et al. 2007), Polacco et.al (Polacco et al. 2004a; Polacco et al. 2004b; Polacco et

al. 2007), Wolczyniak (Wolczyniak et al. 1997), Fawcett and McNally (Fawcett and McNally 2001), Becker et al. (Becker et al. 2001) and Adedeji et al.(Adedeji et al. 1996). The engineering properties of asphalt modified by SBS are enhanced (e.g., increased elastic response, lower creep stiffness) (Isacsson and Lu 1999)

2.6. Dynamic Shear Rheometer

The dynamic shear rheometer (DSR) (Anton Paar) SmartPave 92 (Figure 2.3) was used to characterize the viscous and elastic behavior of asphalt binders at medium to high temperatures.



Figure 2.3. Dynamic shear rheometer (DSR) used to analyze samples

At present the most commonly used method of fundamental rheological testing of bitumen is by means of dynamic mechanical methods using oscillatory-type testing, generally conducted within the region of linear viscoelastic (LVE) response. These oscillatory tests are undertaken using dynamic shear rheometers (DSRs), which apply oscillating shear stresses and strains to samples of bitumen sandwiched between parallel plates at different loading frequencies and temperatures (Airey 2002). The sinusoidally varying shear strain can be expressed as:

$$\gamma(t) = \gamma_0 \sin \omega t \quad (1)$$

and the resulting stress as:

$$\sigma(t) = \sigma_0 \sin(\omega t + \delta) \quad (2)$$

where γ_0 speak strain; σ_0 speak stress, Pa; ω = angular frequency, rad/s; t =time, seconds; and δ =phase angle, degrees.

The loading frequency, ω , also known as the angular frequency or rotational frequency is defined as:

$$\omega = 2\pi f \quad (3)$$

where f =frequency, Hz. The sinusoidally varying strain and stress can also be represented by the following complex notation as:

$$\gamma^* = \gamma_0 e^{i\omega t} \quad (4)$$

and

$$\sigma^* = \sigma_0 e^{i(\omega t + \delta)} \quad (5)$$

The complex shear modulus (G^*) is then defined as:

$$G^* = \frac{\sigma^*}{\delta^*} \quad (6)$$

The above equation for the complex modulus can also be written as:

$$G^* = \left(\frac{\sigma_0}{\delta_0}\right) \cos\delta + i\left(\frac{\sigma_0}{\delta_0}\right) \sin\delta = G' + iG'' \quad (7)$$

where G^* =complex shear modulus, Pa; G' =storage modulus, Pa; and G'' =loss modulus, Pa.

The in-phase component of G^* , or the real part of the complex shear modulus, is defined as:

$$G' = G^* \cos\delta \quad (8)$$

and the out-of-phase component, or the imaginary part of the complex shear modulus, as:

$$G'' = G^* \sin\delta \quad (9)$$

The principal viscoelastic parameters that are obtained from the DSR are, therefore, the magnitude of the complex shear modulus ($|G^*|$) and the phase angle (δ). $|G^*|$ is defined as the ratio of maximum (shear) stress to maximum strain and provides a measure of the total resistance to deformation when the bitumen is subjected to shear loading. It contains elastic and viscous components which are designated as the (shear) storage modulus (G') and (shear) loss modulus (G''), respectively. These two components are related to the complex (shear) modulus and to each other through the phase (or loss) angle (δ) which is the phase, or time, lag between the applied shear stress and shear strain responses during a test. Where G^* or the term 'complex modulus' are used in this paper, it is understood that they refer to the magnitude of the complex shear modulus ($|G^*|$) (Goodrich 1988; Petersen et al. 1994; Airey and Brown 1998)

2.7. Superpave[®] mix design

Marshall Mix design procedure has substantial drawbacks with respect to replicating the real or actual behavior of asphalt during construction and in actual in-service conditions (Jitsangiam et al. 2013).

The Strategic Highway Research Program (SHRP) has developed the Superior Performance Asphalt Pavements (SUPERPAVE) mix design procedure, which shifts to a large degree away from the empiricism of the Marshall Mix design to provide a more reliable and responsive solution to actual pavement conditions (Jitsangiam et al. 2013).

The Superpave design method for hot mix asphalt (HMA) mixtures consists of three phases: (1) materials selection for the asphalt binder and aggregate, (2) aggregate blending, and (3) volumetric analysis on specimens compacted using the Superpave gyratory compactor (SGC) (2). However, other than a final check for tertiary flow, there is no general strength or “push–pull” test to complement the volumetric mixture design method as there is for the more traditional Marshall and Hveem mixture design methods (Witczak 2002).

CHAPTER III

MATERIALS AND METHODS

3.1. Materials

Performance Grade (PG) 58-28 asphalt binder was used as the virgin binder in this study. The PG system is the method of categorizing an asphalt cement binder used in asphalt pavement relative to its rated performance at different temperatures. PG asphalt binders are categorized and selected to meet performance criteria at expected high and low temperature extremes with a certain level of reliability. The first two numbers (58) indicate that the binder meets high temperature physical properties up to 58 degrees Celsius (58°C = 136°F). The last two numbers (-28) indicate the binder meets low temperature physical properties down to -28 degrees Celsius (-28°C = -19°F). Paving asphalt PG 58-28 product meets AASHTO M320 specification requirements are shown in Table 3.1:

Table 3.1. AASHTO M320 specification for PG 58-28 (Lee et al. 2009)

Asphalt Property	Units	Method	AASHTO M320
Flash Point (C.O.C.)	°C	AASHTO T48	230 min
Apparent Viscosity at 135°C	Pa.s	AASHTO T316	3.00 max
Mass change, RTFO residue	mass%	AASHTO T240	1.00 max
DSRo (G*/sin δ) at 58°C	kPa	AASHTO T315	1.00 min
DSRr (G*/sin δ) at 58°C	kPa	AASHTO T315	2.20 min
DSRp (G* sin δ) at 19°C	kPa	AASHTO T315	5000 max
BBR (Stiffness) at -18°C	MPa	AASHTO T313	300 max
BBR (m-value) at -18°C	-	AASHTO T313	0.300 min

Aggregates used in this research were pre-selected by the mix design company, Knife River Materials. Due to the excellent engineering property and relatively low cost, styrene–butadiene–styrene (SBS) has been widely applied in the modification of binders. PG 58-28, mixed with 12% SBS was also chosen to investigate the effect of elastomers on rheology of asphalt binders.

Titanium dioxide (TiO_2) is the naturally occurring oxide of titanium. In nature, it is usually found in form of rutile, anatase, and brookite. Nano-scale titanium dioxide consists of 80% anatase and 20% rutile. It has been proved that nano- TiO_2 has very large surface area, very small diameter, and very low opacity compared to ordinary TiO_2 . Because of those unique properties, some researchers also utilized nano- TiO_2 to improve the performance of modified asphalt (Chen and Liu 2010). Rutile TiO_2 (99.5%, 10-30 nm) was purchased from SkySprings nanomaterials Inc. and used in this study as nano additive.

3.2. Asphalt Binder Modification

PG 58-28 and PG 58-28 mixed with 12 wt. % SBS were modified with different weight percentages of TiO_2 . Binder recipes were summarized in Table 3.2. Three series of five types of nanocomposite modified binders were made and tested.

Table 3.2. Summary of asphalt binder modification study

Series 1- Unaged					
Material	Type 1	Type 2	Type 3	Type 4	Type 5
SBS (wt.%)	0	12	12	12	12
TiO ₂ (wt%)	0	0	1	3	5
Series 2- Short-term aging					
Material	Type 1	Type 2	Type 3	Type 4	Type 5
SBS (wt.%)	0	12	12	12	12
TiO ₂ (wt%)	0	0	1	3	5
Series 3- Long-term aging					
Material	Type 1	Type 2	Type 3	Type 4	Type 5
SBS (wt.%)	0	12	12	12	12
TiO ₂ (wt.%)	0	0	1	3	5

3.3. Binder Test Methods

3.3.1. Rolling Thin Film Oven (RTFO) Test

This test is used to measure the effect of heat and air on a moving film of asphalt binder and to provide residue for additional testing. The effects of this treatment are determined from measurements of the properties of the asphalt binder before and after the test. The oven (figure 3.1) is equipped with a proportional temperature controller capable of maintaining a temperature of $163 \pm 1.0^{\circ}\text{C}$ ($325 \pm 1.8^{\circ}\text{F}$). Oven should be pre-heated for at least 2 hours before test. The samples should be free of water. Binders were put in a container with a loosely fitted cover in the oven. Mass of two empty containers should be recorded using an analytical balance with a readability of 0.001 g or better. Each glass container was filled with 35 ± 0.5 g of the sample in order to provide sufficient material for the tests. After pouring the sample into a glass container, they were turned to a horizontal position. Sample container was rotated for at least one full rotation. Sample containers were placed horizontally in a cooling rack for 1 to 3 hours. After

cooling down, the tests were conducted for 85 minutes at 163 °C and airflow of 4 L/min. This test was completed following the standard test method in ASTM D2872-04.



Figure 3.1. Rolling thin film oven (RTFO) and samples cooling rack

3.3.2. Pressure aging vessel (PAV) Test

Pressure Aging Vessel (PAV) (Figure 3.2) provides simulated long term aged asphalt binder for physical property testing. Asphalt binder is exposed to heat and pressure to simulate in-service aging over a 7 to 10 year period. The standard PAV procedure is found in AASHTO R 28. During the basic PAV procedure, RTFO aged asphalt binder samples were placed in stainless steel pans and then aged for 20 hours in a heated vessel pressurized to 305 psi (2.10 MPa or 20.7 atmospheres). Samples were then stored for use in physical property tests.



Figure 3.2. Pressure aging vessel (PAV) test assembly

3.3.3. Dynamic Shear Rheometer (DSR) Test

Dynamic shear rheometer (DSR) was used to characterize the viscous and elastic behavior of asphalt. The basic DSR test uses a thin asphalt sample sandwiched between two circular plates. The lower plate is fixed while the upper plate oscillates back and forth across the sample to create a shearing action. DSR tests were conducted on unaged, RTFO aged and PAV aged asphalt binder samples. Test temperatures greater than 46°C were used for samples that were 1 mm thick and 25 mm in diameter (Unaged asphalt binder and RTFO residue). Test temperatures between 4°C and 40°C were used for samples that were 2 mm thick and 8 mm in diameter (PAV residue) as presented in Figure 3.3.

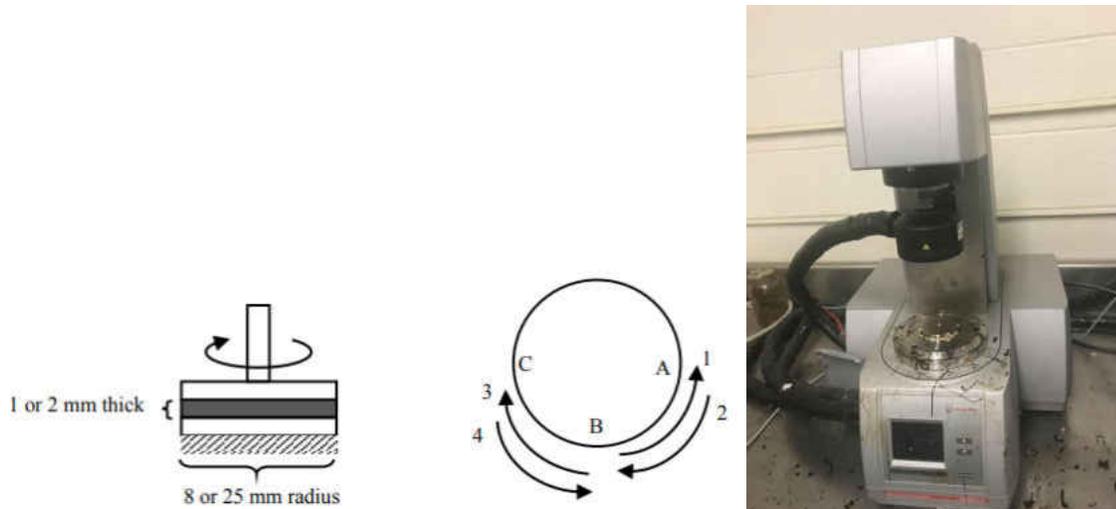


Figure 3.3. DSR instrument and oscillation diagram

In accordance with AASHTO M 320, the test is performed at 10 radians/second. This loading cycle simulates a wheel passing over the pavement surface at 55 mph. This test determines the complex shear modulus (G^*), phase angle (δ), and storage modulus (G'). After loading the sample in a silicon mold, it was then conditioned for 20 minutes at 30°C. Strain sweep test was performed to determine the linear viscoelastic area. Two frequency sweep were performed at 10 and 20 °C.

3.6. HMA Mix Design

The overall mix design procedure begins with evaluation and selection of aggregate and asphalt binder sources. Table 3.3 and Figure 3.4 show individual aggregates and blend gradation of PG 58-28 and picture, respectively. Table 3.4 shows mix design used in this study.

Table 3.3. PG58-28 Aggregate Gradation

	Natural fine	Rock	Washed dust	Dirty dust	Blend gradation	Lower control PI	Upper control PI
Sieve Size	% Passing	% Passing	% Passing	% Passing	% Passing	% Passing	% Passing
5/8" (16mm)	100.0	100.0	100.0	100.0	100.0	100	100
1/2" (12.5 mm)	100.0	100.0	100.0	100.0	99.6	90	100
3/8" (9.5 mm)	100.0	63.0	100.0	100.0	91.5		
#4 (4.75 mm)	90.0	2.0	81.0	81.0	66.2		
#8 (2.36 mm)	76.0	1.0	42.0	53.0	41.3	28	58
#16 (1.18 mm)	62.0	1.0	25.0	37.0	28.1		
#30 (0.6 mm)	47.0	1.0	13.0	28.0	18.5		
#50 (0.3 mm)	26.0	1.0	9.0	21.0	12.0		
#100 (0.15mm)	5.0	1.0	4.0	13.0	6.0		
#200 (0.075mm)	2.9	1.0	2.0	10.3	4.1	2.0	7.0

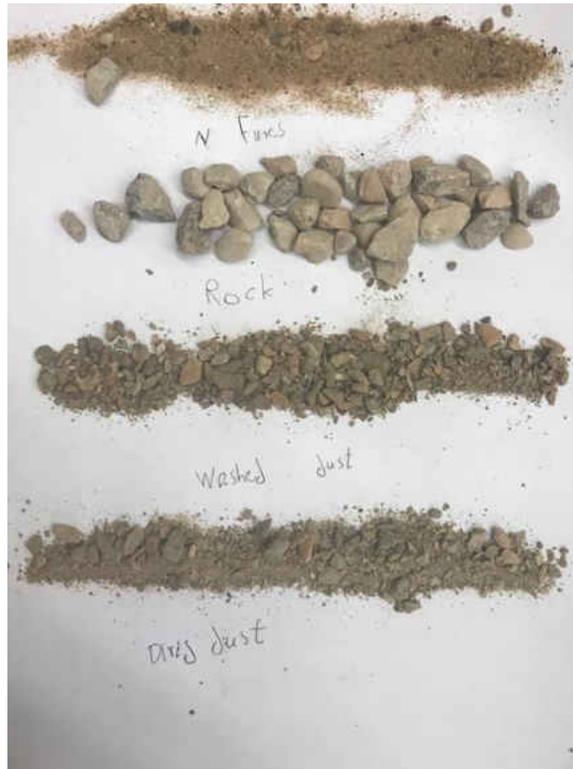


Figure 3.4. PG 58-28 aggregates

Table 3.4. Mix proportion for PG 58-28

Material	PG 58-28	
	Percentage (%)	Mass (g)
Natural fines	11.25	326.25
Crushed rock	22.5	652
washed dust	55	1595
Dirty dust	11.25	326.25
Binder	6.1	177

3.7. HMA Compaction

Superpave Gyrotory Compactor (SGC) was used to compact HMA mixes. Aggregates were heated to 163°C for at least 3 hours, asphalt binder along with the components of compaction were heated to 143°C. The mixing and compaction temperature for this research was 140°C and 135°C, respectively. The components of compaction were heated to the mixing temperature. The HMA was then tested for determining volumetric properties. The bulk specific gravity (G_{mb}), from AASHTO T166-13, was computed after recording the dry weight, saturated surface dry (SSD) weight and water submerged weight. With maximum specific gravity (G_{mm}) from the mix design and computed G_{mb} , the air voids were calculated. Table 3.5 shows the summary of the specimens with the weights and calculated air voids.

Table 3.5. PG58-28 virgin and modified specimens volumetric properties

Binder type	Mass (g)	Height (mm)	Gmm%	Dry (g)	Wet (g)	SSD (g)	Gmb	Air Void (%)
Virgin	2929.03	74.98	92%	2929.09	1668.3	2934	2.487	7.4
Virgin	2933	74.98	91.7	2933.07	1669.8	2937	2.487	7.35
Virgin	2940	74.97	91.58	2928	1628	2931	2.487	7.59
Virgin	2938	74.96	91.8	2938.04	1672.3	2941	2.487	7.39
Virgin+12%SBS	2928.8	74.97	91.7	2928.9	1669.3	2934.7	2.487	7.38
Virgin+12%SBS	2931.4	74.97	91.8	2931.9	1671.4	2936.4	2.487	7.31
Virgin+12%SBS	2927.8	74.96	91.6	2928.1	1667.8	2932.4	2.487	7.42
Virgin+12%SBS	2932.4	74.96	91.8	2932.8	1672.1	2936.1	2.487	7.62
Virgin+12%SBS+1% TiO2	2929.04	74.98	91.8	2929.09	1667.9	2934.04	2.487	7.37
Virgin+12%SBS+1% TiO2	2930.08	74.97	91.7	2931.02	1668.9	2935.08	2.487	7.42
Virgin+12%SBS+1% TiO2	2928.8	74.98	91.6	2929.01	1668.8	2933.9	2.487	7.48
Virgin+12%SBS+1% TiO2	2929.03	74.97	91.8	2929.09	1668.9	2933.08	2.487	7.43

3.8. Mix Test Methods

3.8.1. Asphalt pavement analyzer (APA)

AASHTO TP 63 utilizes pressurized rubber hoses laid across three dual-series sets of test specimens as shown in Figures 3.5 and 3.6.



Figure 3.5. AASHTO TP63 setup



Figure 3.6. AASHTO TP63 test specimen

Once the test specimens have been fabricated they are placed in the testing chamber and conditioned as desired under the pressurized rubber tube rack. The specimens compacted in the SGC were loaded into the cylindrical polyethylene molds at 150 ± 2 mm diameter and height of 75 ± 2 mm. The molds were placed in the machine under pre-pressurized hose reading of 100 ± 5 psi (695 ± 35 kPa) and load cylinder pressure under each wheel to achieve a load of 100 ± 5 lbf (445 ± 22 N). Since pavement rutting occurs at higher temperatures, test was carried out at the highest pavement temperature which is the upper temperature in the PG grade. Prior to beginning the test, the specimens were conditioned and stabilized at the testing temperature for 5 to 6 hours. The test consists of the wheels passing back and forth over the three sets of specimens up to 8,000 times. A raw data chart is generated during the course of the test indicating the number of completed cycles, rut depths and cabin temperature for each of the three sets of specimens.

3.8.2. Disk Shaped Compact Tension Test (DCT)

Figure 3.7 shows DCT test as specified in ASTM D7313(07). The test is generally used to obtain the fracture energy of asphalt mixture lab or field specimens, which can be used in performance-type specifications to control various forms of cracking, such as thermal, reflective, and block cracking of pavements surfaced with asphalt concrete. Standard testing is conducted at 10°C warmer than the PG low-temperature grade.



Figure 3.7. DCT test assembly

The DC(T) test is run in crack mouth opening displacement (CMOD) control mode at a rate of 1 mm/min. Typically, specimens are completely failed in the range of 1 to 6 mm of CMOD travel. Although the actual test takes only 1 to 6 minutes to perform, the actual amount of testing time per specimen is probably more close to 15 minutes, accounting for stabilization of test temperature, loading samples into the test apparatus, etc. Sample preparation involves sawing and coring operations. First, a water-cooled masonry saw (14 or 20 inch blade) is used to create the flat, circular faces, similar to the production of an indirect tension test specimen or simple performance test specimen. A single or dual saw system may be used. A dual saw system, while more costly, will produce more parallel faces and uniform thickness specimens, which may improve test repeatability. A marking template is then used to indicate the location of the 1.0 inch loading holes to be drilled.

3.8.3. Semi Circular Bend Test (SCB)

Similar to DCT, SCB test is used to obtain the fracture energy of asphalt mixture lab or field specimens, which can be used in performance-type specifications to control various forms

of cracking, such as thermal, reflective, and block cracking of pavements surfaced with asphalt concrete. Standard testing is conducted at 10°C warmer than the PG low temperature grade.

Similar to DC(T) test, the SCB test is run in crack mouth opening displacement (CMOD) control mode. However, the rate is 0.03 mm/min, 33 times slower than the DCT loading rate, which increases the duration of the test to as much as 30 minutes. Another significant difference is in the thickness of the specimen: DC(T) is 2” thick, while SCB is 1” thick.

Sample preparation is similar to DCT except that no coring is required. The only additional operation is gluing one IDT-type button on each face of the specimen, which are used to measure load line displacement (the displacement in the direction of the applied force) required to calculate fracture energy (Pocius 2012). Figures 3.8 and 3.9 shows the method of loading and state of the sample before and after SBC test, respectively.



Figure 3.8. SCB before test



Figure 3.9. SCB after test

Chapter IV

RESULTS AND DISCUSSIONS

The purpose of this study was to investigate the effect of polymeric nanocomposite on binder and mix performance. DSR was used to determine binder performance at medium and high temperatures whereas APA, DCT, and SCB were used to determine rutting, low-temperature, and fatigue cracking resistance, respectively of the mixes. Modified binders were mixed and tested along with virgin binder to compare the performance of both binder and mixes with the latter.

4.1. Binder Performance

4.1.1. Rutting Resistance ($G^*/\sin \delta$)

The higher $G^*/\sin \delta$ values from the DSR test indicate that the binders are less susceptible to rutting or permanent deformation at high pavement temperature. The $G^*/\sin \delta$ values of unaged, RTFO aged, and PAV aged binders were measured at 58°C and 64°C. The results are shown in Figures 4.1 to 4.4. As can be seen, the SBS modified binder resulted in the higher $G^*/\sin \delta$ than the virgin binder. However, the percentage improvement of rutting resistance due to the addition of 1% TiO_2 was observed much higher for control binders compared to polymer modified asphalt (PMA) binders, which in average, is 200% for virgin binders versus 50% for 12% SBS modified binder. The reason might be the PMA binder was already modified with SBS for high temperature performance so the addition of TiO_2 additives has less effect compared to the unmodified control binder. Although addition of nanoparticle improved the shear strain of the binder compared to the virgin binder, but high concentration of TiO_2 results in very porous structure in binders which appears to have negative effect on rutting

resistance property. However, 1% TiO₂ is considered as an optimum level for binder modification.

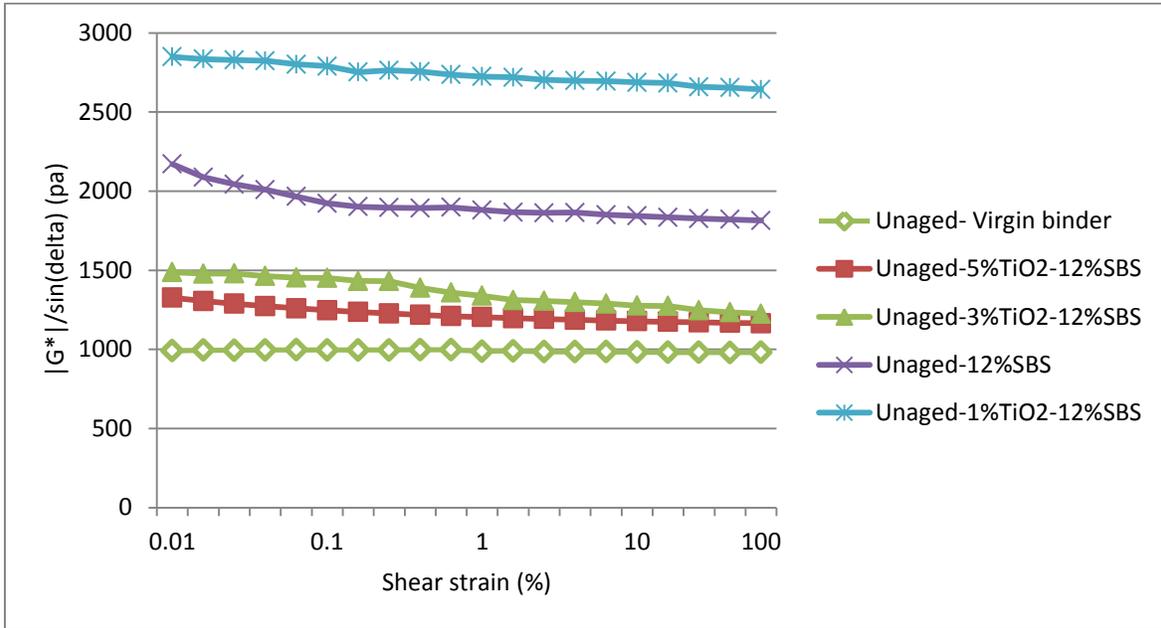


Figure 4.1. Rutting resistance of unaged virgin and modified binder at 58°C

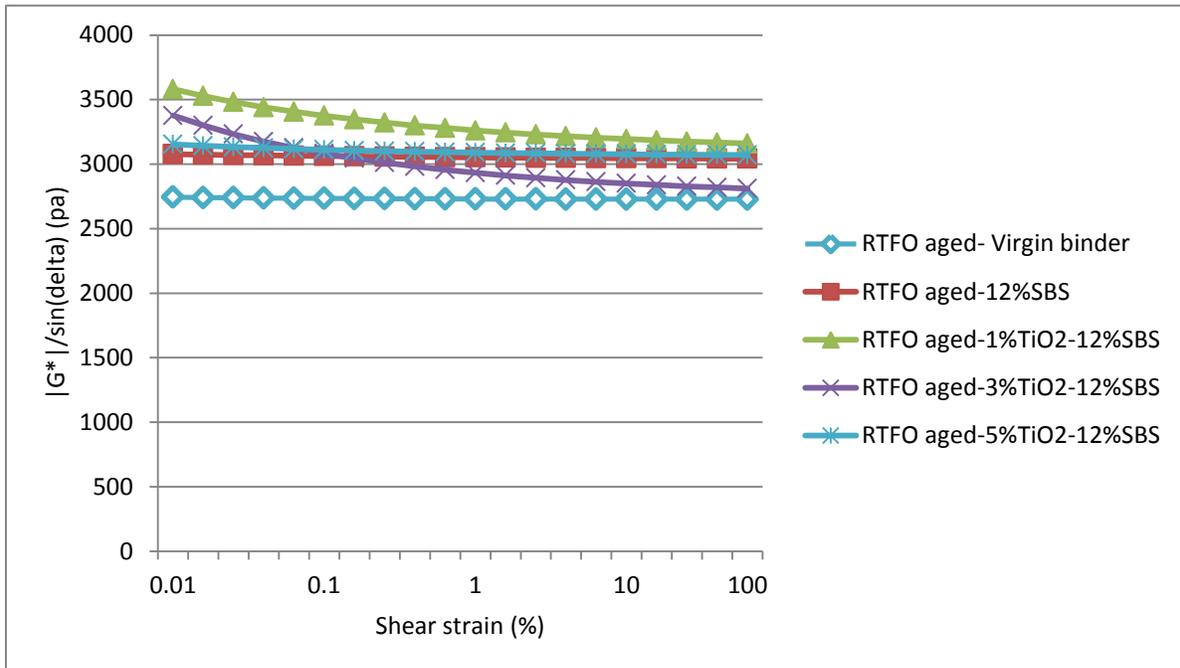


Figure 4.2. Rutting resistance of RTFO-aged virgin and modified binder at 58°C

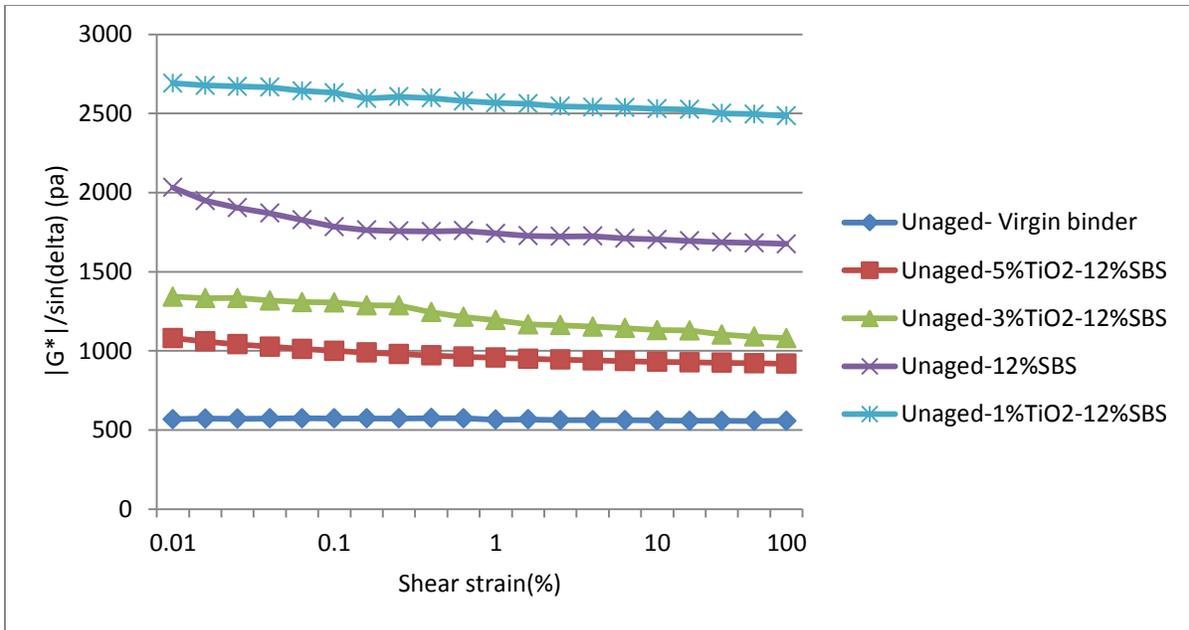


Figure 4.3. Rutting resistance of unaged virgin and modified binder at 64°C

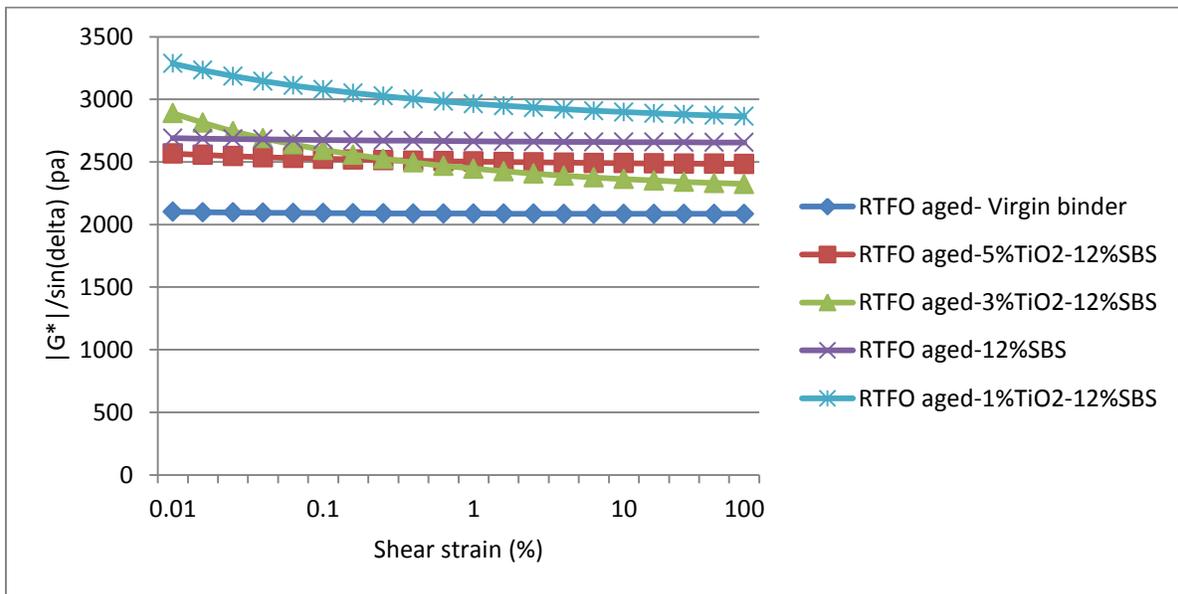


Figure 4.4. Rutting resistance of RTFO-aged virgin and modified binder at 64°C

4.1.2. Fatigue Cracking Resistance

The product of the complex shear modulus G^* and the sine of the phase angle, δ , is used in Superpave binder specification to help control the fatigue of asphalt pavements. The lower

values of $G^* \sin \delta$ are considered desirable attributes from the standpoint of resistance of fatigue cracking. The $G^* \sin \delta$ values of the binders were measured using the DSR at 58°C and 64°C and the results are illustrated in Figures 4.5 and 4.6. The SBS modified binder exhibited lower $G^* \sin \delta$ values than the virgin binder, indicating that the SBS polymer modification improved the resistance for fatigue cracking. The binders with 1% TiO_2 showed the lowest values within each modified binder type which is consistent with the rutting properties data. It needs to be mentioned that the addition of 1% TiO_2 into virgin and SBS modified binders reduced the $G^* \sin \delta$ values by 11% and 27.4%, respectively. It means that addition of polymer and nanoparticle has a positive effect on the cracking resistance at intermediate temperature. Also, all the values satisfied the maximum requirements of 5,000 kPa by Superpave.

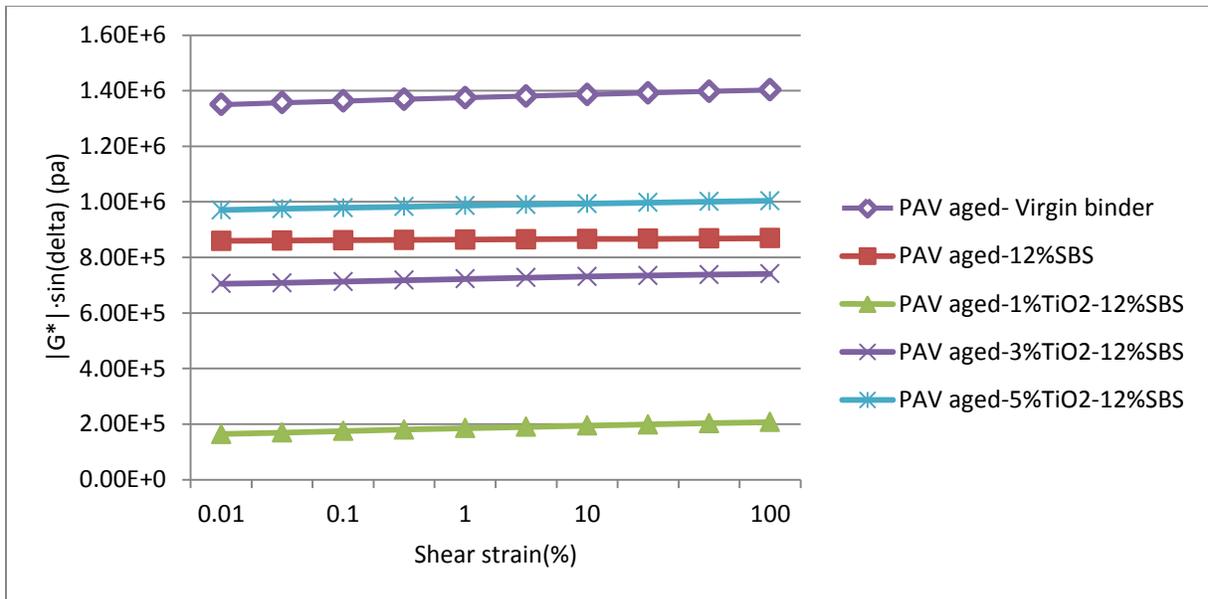


Figure 4.5. Fatigue cracking resistance of PAV-aged virgin and modified binder at 58°C

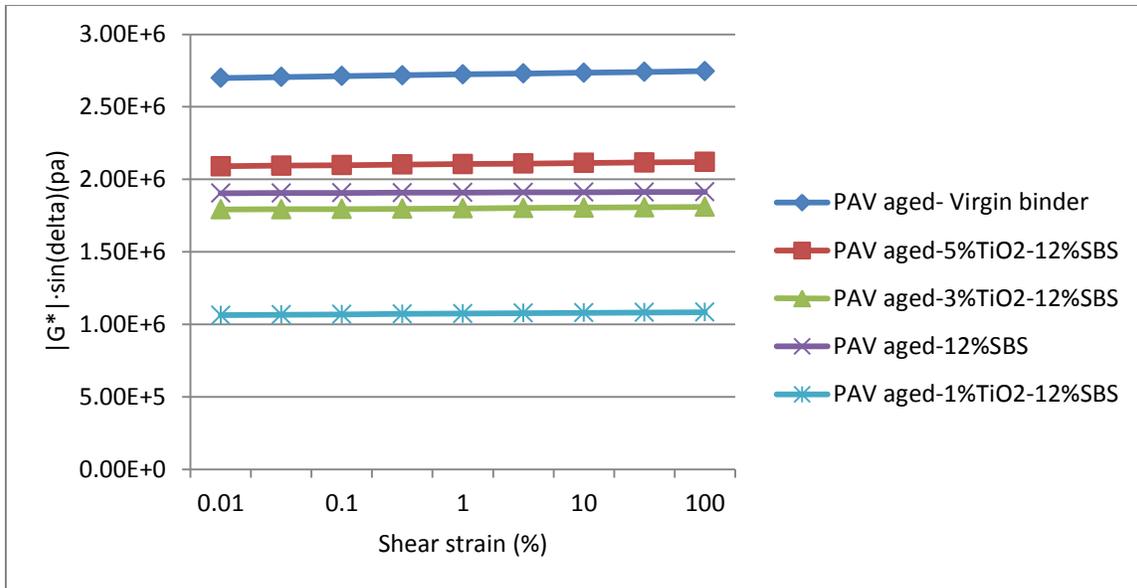


Figure 4.6. Fatigue cracking resistance of PAV-aged virgin and modified binder at 64°C

4.2. Mix Performance

4.2.1. Rutting Resistance

The layout of the APA test bed is shown in Figure 4.7. Six cylindrical specimens (150 mm (diameter) x 75 mm (tall)) were tested in each run of the APA. Table 4.1 shows the summary of the average rut depth, standard deviation and coefficient of variation for three types of selected binders. Each APA test was ran in duplicates and the average rutting depth is shown in Figure 4.10. Figure 4.11 display the progression of average rut depth from the opening to the end of the test at 8000 passes for virgin, 12%SBS modified, and 12%SBS+1%TiO₂ binders. As can be seen, virgin binder showed the highest slope compared to polymer modified and TiO₂+SBS modified binder which indicates significant level of resistance toward rutting upon modification of binder.

Table 4.1. APA Results Summary

Binder	2000			4000			6000			8000		
	Avg (mm)	St.D. (mm)	COV (%)									
Virgin	4.12	0.05	21	6.01	0.09	23	8.13	0.58	23.7	10.11	0.6	25
Virgin+12%SBS	3.05	0.10	17.5	4.01	0.12	12.35	5.02	0.65	1.35	5.78	0.15	19
Virgin+12%SBS +1%TiO ₂	1.85	0.06	25.3	1.91	0.25	24.1	1.96	0.25	15.01	2.01	0.23	19.87

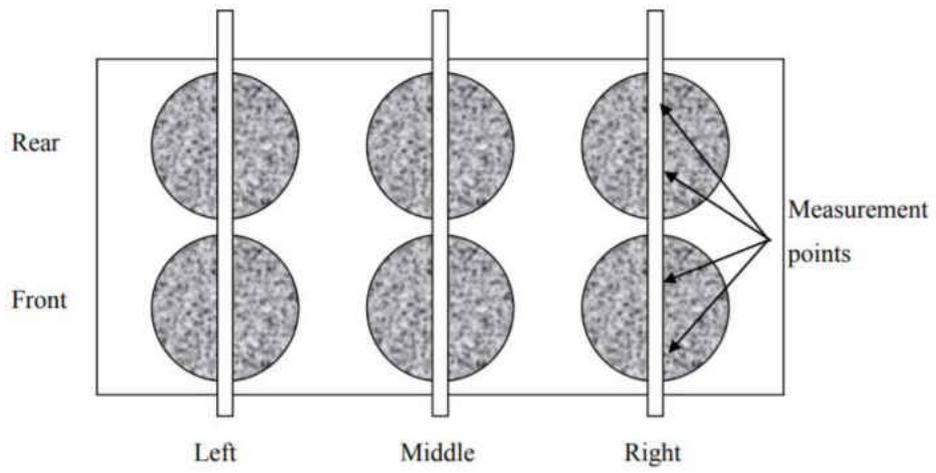


Figure 4.7. APA specimen layout

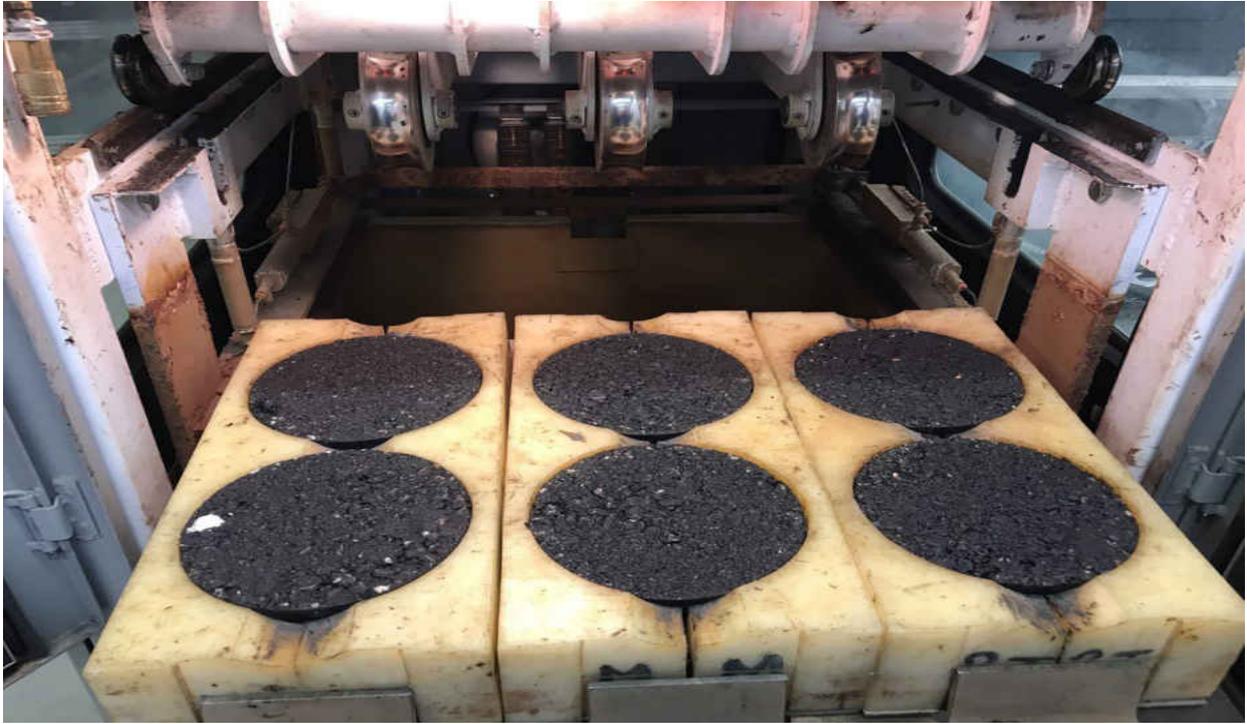


Figure 4.8. APA specimen layout before test

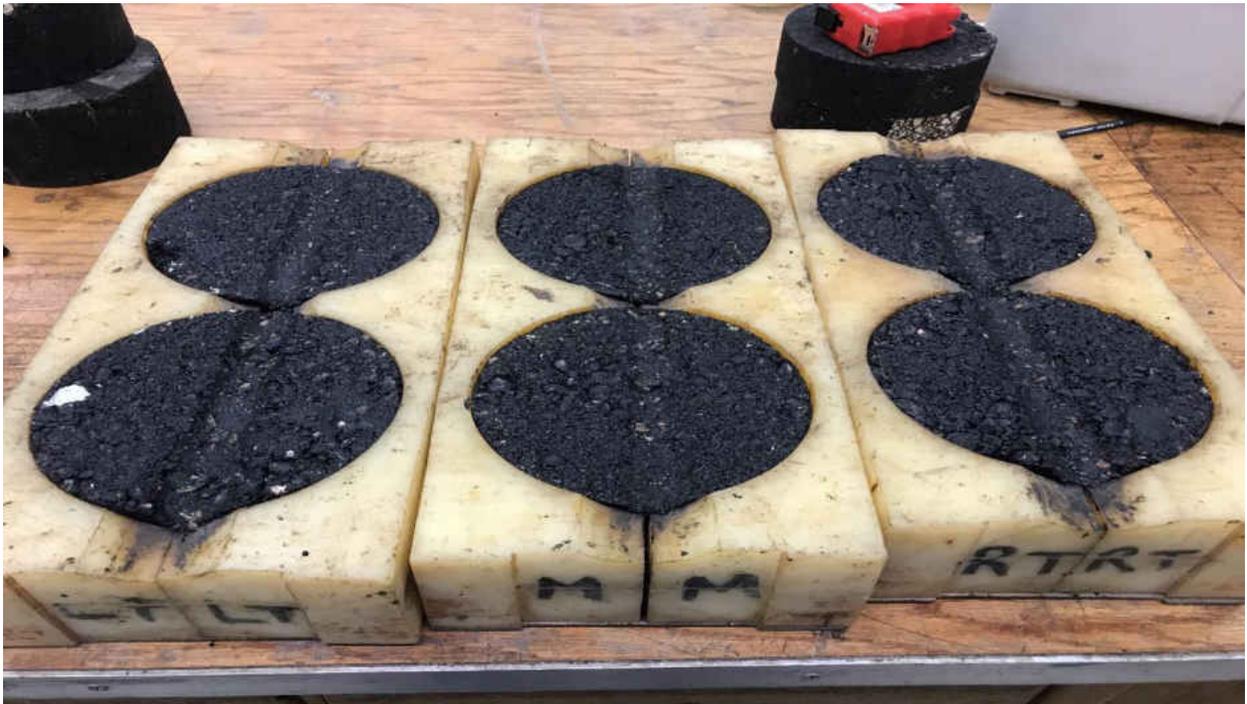


Figure 4.9. APA specimen layout after test

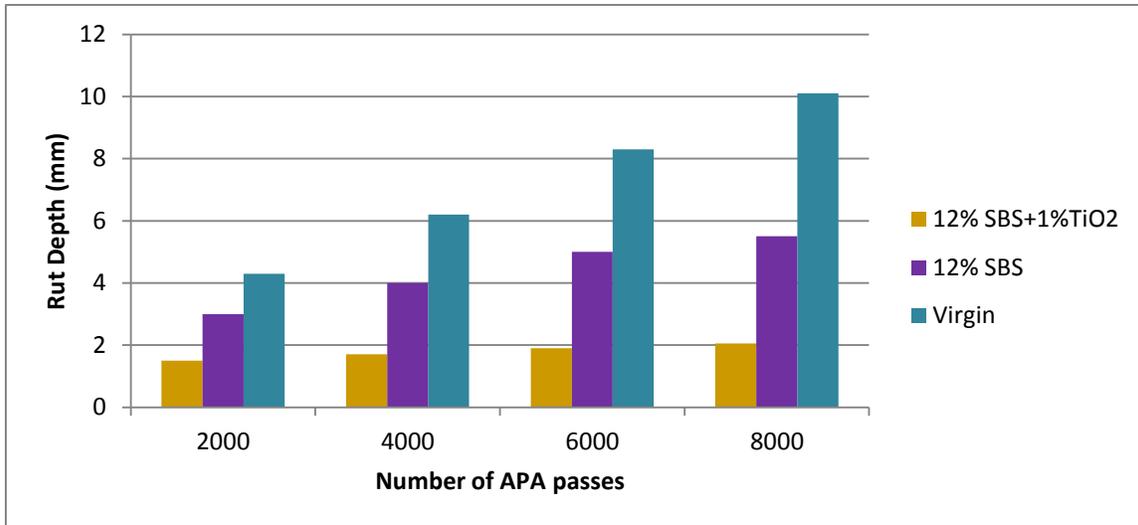


Figure 4.10. Rut depth vs. APA passes

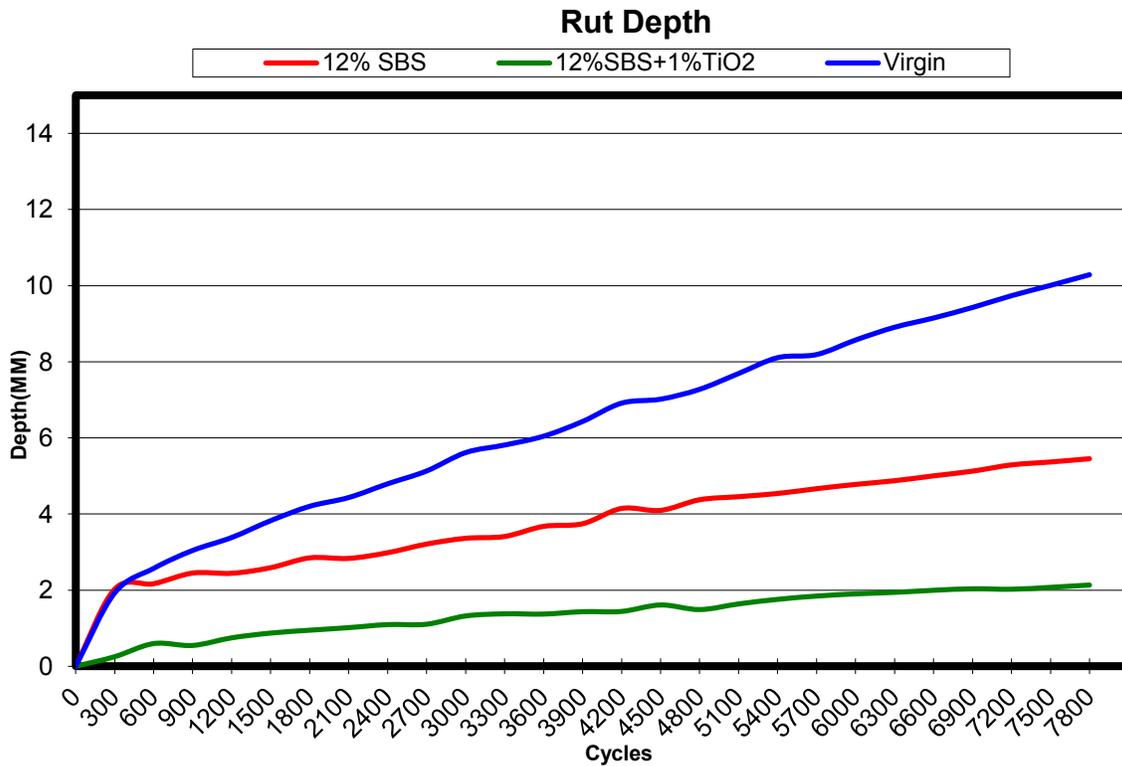


Figure 4.11. Rutting depth measurements for mixes with virgin and modified binders

4.2.2. Low-Temperature Cracking Resistance

The low-temperature cracking tests for the mixes were completed using the DCT test. Fracture energy is used to predict the performance of the mix against low temperature cracking. Fracture energy is calculated as the area under the curve of load versus Crack Mouth Opening Displacement (CMOD). Figures 4.12 to 4.14 show the graph plotted by the program within DCT machine. Tables 4.2 displays the average data of DCT test for mixes with virgin and modified binders in terms of fracture energy. As expected, nanocomposite modified binder requires higher energy to crack. Polymeric nanocomposites behave as filler in asphalt binder, especially addition of nanoparticles fill all the voids in the binder that results in more compact binder. Figure 4.13 and Figure 4.14 shows the tested samples before and after test, respectively.

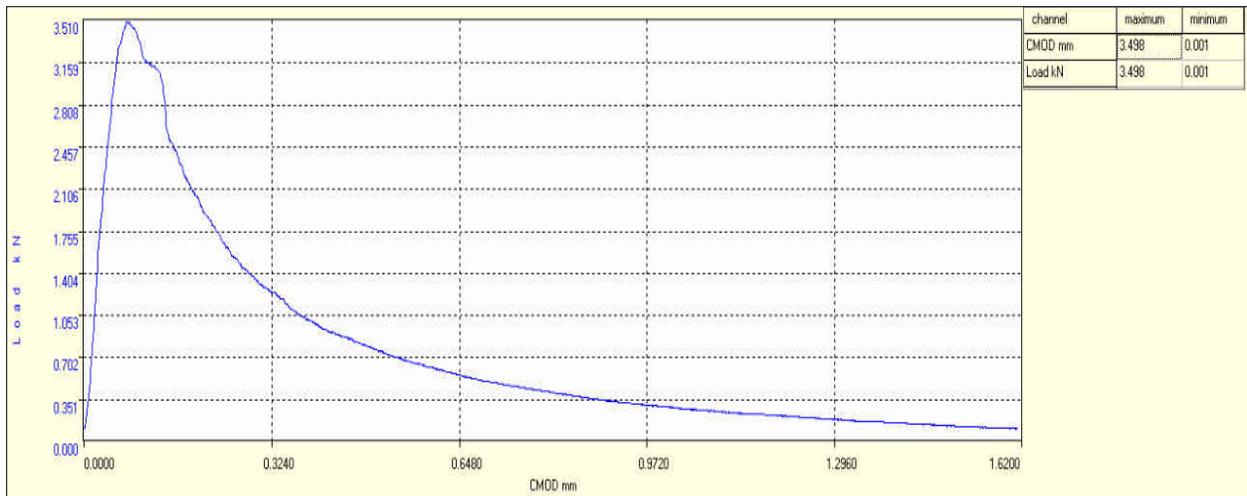


Figure 4.12. DCT graph for mix with virgin binder

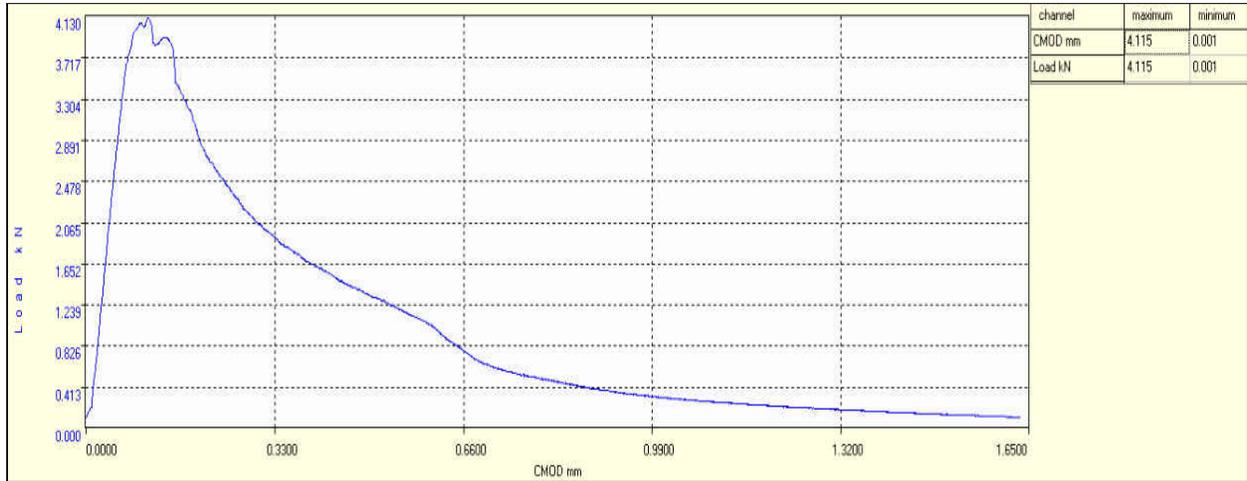


Figure 4.13. DCT graph for a mix with 12% SBS modified binder

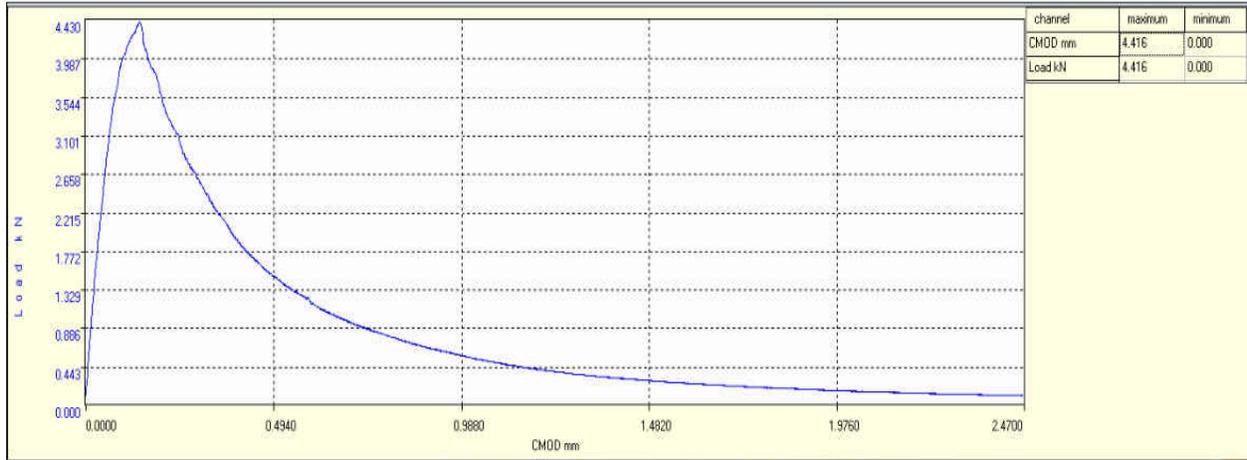


Figure 4.14. DCT graph for virgin binder modified with 12% SBS and 1% TiO₂

Table 4.2. Summary of DCT data

Binder Type	Energy(J/m ²)	St. Dev.(J/m ²)	COV(%)
Virgin binder	247	20	12
Virgin binder+12%SBS	334	12	7
Virgin binder+12%SBS+1%TiO ₂	515	23	9



Figure 4.15. Example of DCT specimen prior to test



Figure 4.16. Example of DCT specimen after the test

4.2.3. Fatigue Cracking Resistance

SCB test was used for this analysis in determining fatigue cracking resistance of the mixes. Fracture energy is the variable that is most commonly used in comparing and predicting the performance of the mixes. The measure of fracture energy is calculated as the area under the curve of load versus displacement. Due to the limited availability of materials, test samples were created in limited amount. The size of test specimen used was only the half i.e. 25mm instead of commonly used 50mm. Table 4.3 shows the summary of the results from SCB tests. As can be seen, fracture energy of modified binder increased with addition of polymer and nano-TiO₂. As a result of addition of TiO₂ nanoparticles the air void of the nanocomposite modified binder decreased by 210% compared to virgin binder.

Table 4.3. Summary of SCB results

Binder Type	Energy (J/m ²)	St. Dev. (J/m ²)	COV (%)
Virgin binder	321	25	7.5
Virgin binder+12%SBS	730	21	15.3
Virgin binder+12%SBS+1%TiO ₂	1023	18	6.3

CHAPTER V

CONCLUSIONS, RECOMMENDATIONS, AND FUTURE WORKS

5.1. Conclusion

In this research, PG 58-28 binder was modified with 12% SBS and various concentration (1 wt.%, 3 wt.%, and 5 wt.%) of nano TiO₂.

DSR test indicate that the binders are less susceptible to rutting or permanent deformation at high pavement temperature. It needs to be mentioned that higher concentration of TiO₂ will result in more porous and therefore fragile binder. Therefore, 1 wt.% of TiO₂ showed an optimum level of performance.

APA results showed that addition of nano-TiO₂ increases the rutting resistance of the pavement specimens compared to polymer modified and virgin samples. The nano-TiO₂ prevents tensile and vertical cracks from being easily generated by horizontal tensile stresses and prevents them from propagating.

DCT analysis results further verified the results that were produced from previous data collection method. Nano-TiO₂ modified asphalt binder showed a significant improvement in fracture energy as compared to virgin and polymer modified binder.

All in all, 1 wt.% TiO₂ modified asphalt binder showed an optimum level of strength in rutting and cracking properties, and successfully passed all the mechanical tests that were conducted on the specimens. It is worth mentioning that another obvious advantage of addition of nano-TiO₂ in asphalt binder can improve the NO_x reduction due to its photocatalytic effect.

Therefore, the modified nanocomposite samples have high potentials for capital investment in highway research.

5.2. Limitations

During this research, there were a few limitations on mixes which could have influenced the results. These experiments were conducted on PG 58-28 binder. However, it will be beneficial if other binder grades are evaluated. The specimen sizes used in the SCB test were not the standard size recommended by the I-FIT due to limited materials. Limited materials led to limited specimen preparation for the DCT and SCB tests which could be a reason for inconclusive results.

5.3. Future Works

In future, the photocatalytic effect of nano TiO_2 needs to be examined in the modified asphalt binder, since this TiO_2 nanoparticles can have high effect on NO_x reduction, and therefore can be used in long tunnels with limited air ventilation.

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