

Enhancement of Asphalt Performance by Graphene-Based Bitumen Nanocomposites

Sara Moghtadernejad, PhD

Ehsan Barjasteh, PhD

Ren Nagata

Haia Malabeh



Mineta Transportation Institute

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Mineta Transportation Institute College of Business
San José State University San José, CA 95192-0219

Tel: (408) 924-7560

Fax: (408) 924-7565

Email: mineta-institute@sjsu.edu

transweb.sjsu.edu/research/1918

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1. Introduction

As the population of California grows, the transportation infrastructure central to the way of life in the State deteriorates at an alarming rate, resulting in a constant need for costly repairs. California's highway and local roadway network support approximately 40 million residents and 35 million vehicles for livelihood. In the 2017 Asset Management Report, California Department of Transportation has estimated that out of 49,644 highway miles within its jurisdiction, 40.8% of these roadways are in "good" condition (Caltrans, 2017). California's transportation infrastructure, once a catalyst for growth, begins to threaten to undermine the integrity and reliability of the system due to a decline in the preservation of highway assets.

In addition to the billions of dollars spent annually on repairs stemming from materials and operations, surface rehabilitation of pavement precipitates the need to shut down the segment until repairs and maintenance are completed. Continued highway shutdowns exert monetary pressure on the State's \$2.6 trillion economy. According to the Texas Transportation Institute, within 494 US urban cities, travel delays due to motor vehicle congestion cost passenger vehicles an estimated hourly rate of \$20 (2017 dollars). Ultimately, the congestion that leads to added travel time and wasted fuel has resulted in an increase in the average economic loss from \$15 billion in 1982 to \$179 billion in 2017 (2017 dollars) (McFarland & Chui, 1986; Sweet, 2011; Texas A&M Transportation Institute, 2019). California's annual economic loss is estimated to be over \$61 billion due to excessive congestion resulting from necessary repairs and poor road conditions (ASCE, 2013). Significant efforts are taken to ease the perpetual cycle of deterioration and repairs. However, a demand for lengthened intervals between repairs and maintenance is essential to enhance the reliability of the transportation system and to mitigate these costs affecting California residents.

Millions of dollars are invested each year within the California State Highway System in the construction of asphalt concrete, commonly known as asphalt pavement or hot mix asphalt (FHWA, 1993). Asphalt cement is composed of two main components: (i) mineral aggregate, typically crushed stone that constitutes 90–95% of the asphalt cement mixture and (ii) asphalt binder or bitumen, a by-product of processing crude petroleum that comprises 5–10% of the total mixture (J. G. Speight, 2016; J. G. B. T.-A. M. S. and T. Speight, 1996a, 2016; Sweet & Texas A&M Transportation Institute, 2016). Although the asphalt binder mixture composition is volumetrically small, its impact on the overall asphalt pavement performance is abundantly significant (Islam, 2020). The asphalt binder serves as a waterproof barrier that adheres to, and holds the mineral aggregate in place. Asphalt binders are viscoelastic materials that embody response behavior of elastic and viscous elements (J. G. Speight, 2016; J. G. B. T.-A. M. S. and T. Speight, 1996b, 2016). Viscosity is heavily dependent on temperature changes, the binders gradually soften as the temperature rises and become brittle at low temperatures (Behnood & Modiri Gharehveran, 2018). High-quality binders exhibit performance properties such as resistance to cracking and oxidation from aging; broader upper and lower temperature limits that can withstand fluctuations in environment and climate conditions; and display rheological characteristics that allow constant structural stresses without permanent deformation or rutting (J. G. Speight, 2016; J. G. B. T.-A. M. S. and T. Speight, 1996a, 2016).

A method for enhancing the properties of asphalt binders includes changes in formulation by the addition of polymers. Asphalt binder modification has shown improvement in durability and strength, displaying excellent resistance to rutting and cracking, and temperature susceptibility. The addition of polymers to asphalt binders also improves performance by increasing resistance to stress due to failure (Behnood & Modiri Gharehveran, 2018). The asphalt's ability to contract and expand according to environmental conditions and mechanical stress is fundamental to its longevity. In the near future, Caltrans expects to navigate towards replacing usage of unmodified asphalt binders with performance grade (PG) modified binders in new pavement construction (Caltrans, 2021).

The most frequently used polymer for asphalt binder modification is styrene-butadiene-styrene (SBS) (J. G. Speight, 2016; J. G. B. T.-A. M. S. and T. Speight, 1996a, 2016; Yildirim, 2007). The enhanced characteristics of asphalt binders due to the addition of SBS include: reduced brittleness at lower temperatures, improvement in aging resistance and high temperature properties, and increased ductility from constant load application. Blending in small polymer particle size with the binder results in an efficient mixture and in improvements of rheological and morphological properties, and an increase in compatibility of the asphalt binder with SBS. Due to the significant molecular structure size and chemical nature difference of the SBS polymer and binder, a homogenous and stable mixture, free from molecular separation, is obtained when the polymer size is small. The incompatibility between the asphalt binder and SBS polymer can result in phase separation where the two components begin to separate as temperature rises (Behnood & Modiri Gharehveran, 2018).

As nanotechnology advances, additional materials for the development of improved asphalt become readily available. The addition of graphene nanoparticles to asphalt has been of great interest within the field of asphalt materials due to their enhancement of the asphalt binder's physical properties when compared to traditional pavement materials. The performance of graphene's role as reinforcement in polymer-modified asphalt binder is expected to yield added performance properties, which can render improved highway durability and a lengthened lifespan to infrastructure.

Although nano-modification of asphalt binder studies have been conducted, limited knowledge exists on the comparison of mechanical and rheological effects of several different methods of graphene nanoparticles and SBS polymer component incorporation within the asphalt (Ameri et al., 2016; Hasan et al., 2012; Mamun & Arifuzzaman, 2018; J. Yang & Tighe, 2013). Wang et al., concluded anti-rutting and fatigue properties of SBS modified asphalt binder was enhanced by the addition of as little as 0.5-1.0% carbon nanotubes. The modified asphalt samples within the study were manufactured by combination of nanoparticles and SBS polymer powders prior to being blended with the bitumen (P. Wang et al., 2017). Other studies indicate grafting polymers onto graphene nanoparticles, then mixing with additional polymers before being added to bitumen enhance overall performance due to its uniform distribution within the asphalt binder (Han et al., 2018). Evaluation of graphene nanoparticles added to SBS polymer-modified asphalt binder resulted in a significant decrease of phase separation between the SBS polymer and bitumen, when compared to the SBS polymer-modified asphalt binder absent of graphene nanoparticles (Goli et al., 2017).

This study aims to explore the differences in the rheological behaviors of various graphene-incorporated asphalt binder mixtures. In addition, the characteristics of alternative graphene

application methods to the asphalt binders and mixtures are reported. The fundamental objective is to evaluate whether production of a polymer first coated with graphene nanoparticles followed by mixing with bitumen will result in enhanced system compatibility when compared to the addition of a polymer to a graphene-modified asphalt binder mixture.

The binder mixture components were developed in the following four phases: (I) graphene-incorporated asphalt binder; (II) polymer-modified asphalt binder of varying polymer content; (III) graphene-coated polymers mixed with asphalt binder of varying nanoparticle composition; (IV) graphene-incorporated asphalt binder followed by the addition of polymers. These samples were tested for rheological properties including elasticity, susceptibility to pressure, aging, and temperature to determine the optimal mix design for enhanced elastic properties and improved rigidity. The data obtained from these test experiments were analyzed and are later discussed. Varying methods of adding graphene to polymer-modified asphalt were tested with a lab-scale process. Further research is required to effectively incorporate graphene into large-scale bitumen production.

2. Methodology

2.1 Material Selection

This study includes two different types of graphene nanoparticles and fixed sources of a polymer and asphalt binder. Graphene is an inorganic two-dimensional planar structure composed of dense carbon atoms in a hexagonal arrangement; this benzene-like structure is responsible for graphene's incredible rigidity even in small quantities (Dresselhaus & Dresselhaus, 2002; Park et al., 2015; Shenderova et al., 2002; Somasundaran & Xiao, 1990). Previous studies conducted on the addition of graphene nanoparticles to several polymer-modified asphalt samples have shown significant improvements in the materials' rheological and mechanical properties (Du et al., 2004; J. Wang et al., 2013; Z. Yang et al., 2010; Yin, 2010; Yoo et al., 2014; Zeng et al., 2017). Research indicates that the use of graphene nanoparticles specifically leads to an increase in the complex shear modulus (CSM) and a reduction of phase angle. Furthermore, graphene nanoparticles reinforce the distribution of polymers in the asphalt binder to achieve supplementary harmonious systems. Due to the small nature of graphene nanoparticles, their addition promotes compatibility and homogeneity between the polymer and binder system which can result in improved material properties. (Behnood & Modiri Gharehveran, 2018; P. Wang et al., 2017). Development of samples within this study include graphene nanoparticles nano-24 and nano-27, manufactured by Asbury Graphite Mills.

The polymer used in this application is SBS, selected for its ability to improve the rheology of asphalt binders and its ability to maintain its molecular structure throughout the mixing process. Multiple studies have concluded that SBS can increase resistance of asphalt mixtures to the effects of rutting due to its enhanced stiffness. Additionally, resistance to cracking at low temperatures results from the added flexibility SBS provides to the asphalt (Kim et al., 2013). In addition, SBS polymer has the advantage of a strengthened adhesivity between the aggregate and asphalt binder, which corresponds to improved mechanical properties such as shape retention of asphalt roadways (Behnood & Modiri Gharehveran, 2018). The studies conducted by Yang et al. have shown that mixing 3% graphene nanoparticles to SBS-modified bitumen increased the mixture strength by 80–150%. The enhanced properties include improvement in penetration, softening point, ductility, and high-temperature performance, which directly affect the durability of highway materials. Optimal performance results are typically seen in test samples incorporated with 3% to 7% polymer content (Behnood & Modiri Gharehveran, 2018; Han et al., 2018). This study will mainly focus on 3% SBS composition.

The SBS polymer, Kratom D1191ET, and the asphalt binder, PG64-10, were both supplied by San Joaquin Refining Co. The asphalt binder, PG64-10, was formulated to withstand a seven-day period of pavement temperatures between -10°C to 64°C , befitting California's varying climate (AASHTO, 2010).

2.2 Sample Production

Although the prepared samples varied in component composition, the mixing process remained constant throughout each of the four phases to ensure consistency. First, the asphalt binder, required for each sample, is prepared by being heated in a convection oven at a constant

temperature of 183°C for a minimum of 30 minutes to reduce its viscosity and achieve uniform mixing with the other components. Once heated, PG64-10 was added to a weighed empty container to approximately three-quarters full. Additional weights were recorded before adding the appropriate SBS and nanoparticle composition. The samples were first incorporated slowly by hand with a stirring rod and then transferred to a high-shear mixer operating at approximately 185°C and 6,500 RPM for the first hour; the speed was then reduced to 5,000 RPM for an additional hour of mixing. Occasionally, the mixture was checked to ensure a constant temperature and even mixing of the components. Subsequently, the sample was left in the convection oven at 183°C for a minimum of eight hours to allow the nanoparticles to network evenly. Finally, all samples were stored in one-quart tin containers. This method was applied to all samples within the following four phases.

Samples of Phase I, prepared to examine the rheological traits of the nanoparticles with the asphalt binder, consist of PG64-10 with 1% nano-24. An additional sample of Phase I was produced to be combined with Phase IV samples. Phase I samples will aid in the determination of whether altering the mixing methodology of the components during the production of a modified asphalt binder will lead to heightened properties. Sample properties are displayed in Table 1.

Table 1. Phase I Sample Components of Nanoparticles Mixed with Asphalt Binder

Phase I	Components
Nanoparticles + PG64-10	1% Nano-24

Phase II samples focus on the rheological traits of polymer-modified asphalt binder at two polymer ratios. Additionally, Phase II samples confirm whether the mixing methodology will produce samples consistent with industry standard results. Two samples were prepared consisting respectively of 1.0 wt.% and 3.0 wt.% SBS mixed with PG64-10, and additional properties are displayed in Table 2.

Table 2. Phase II Sample Components of SBS Polymer Mixed with Asphalt Binder

Phase II	Components
SBS Polymer + PG64-10	1% SBS polymer
	3% SBS polymer

The production of samples for Phase III involved mixing the nanoparticles in a solution of 40% acetone and deionized water. An ultrasonic bath was used for 15 minutes to achieve uniform mixing of the nanoparticle solution. Next, the appropriate amount of SBS was added before the sample was placed in the ultrasonic bath for another two hours. Excess acetone was removed, and the mixture was placed in the convection oven to fully dry at 110°C for one hour. Once dry, the graphene-coated SBS was mixed with PG64-10 using the high-shear mixer. Phase III samples were produced to examine whether the mixing methodology of various compositions of nanoparticles and polymer with the asphalt binder results in significantly enhanced properties. The samples were produced by coating the SBS with each nanoparticle type, followed by mixing the graphene-coated SBS in the PG64-10. Three batches of SBS-coated nanoparticles include: 1% nano-24, 3% nano-24, and 1% nano-27, seen in Table 3. Displayed in Figure 1 are Phase III samples prior to and after nanoparticle coated SBS is incorporated with the asphalt binder.

Table 3. Phase III Sample Components of Nanoparticle-Coated SBS Polymer Mixed with Asphalt Binder

Phase III	Components
Coated Polymer + PG64-10	1% Nano-24
	3% Nano-24
	1% Nano-27



Figure 1. Fully Dried 3% Nano-24-Coated SBS (left) & Heated Modified Asphalt Binder of 3% Nano-24-Coated SBS + PG64-10 after using a High Shear Mixture (right)

Phase IV samples were prepared by combining Phase I & Phase II samples. The samples were incorporated slowly over two days to ensure proper mixing and to prevent agglomeration or component separation. Additional SBS was introduced into the bitumen sample and mixed for two hours before samples entered the convection oven for eight hours at 183°C. The finalized sample properties for this phase are presented in Table 4.

Table 4. Phase IV Sample Components of Nanoparticles Mixed with SBS Polymer and Asphalt Binder

Phase IV	Components
Phase 1 + SBS Polymer	1% Nano-24 + PG64-10 + 3% SBS

2.3 Equipment & Experimental Testing

Multiple test methods were used to determine the thermodynamic limits and rheological properties of the samples, including a softening point temperature (SPT) test, dynamic shear rheometer (DSR) test, a bending beam rheometer (BBR) test, and a multiple stress creep recovery (MSCR) test. Additionally, the stability of the samples during low-temperature storage was tested in accordance with ASTM 5976 (American Society for Testing and Materials (ASTM), 2000).

2.4 Softening Point Temperature

The SPT test was used to analyze the thermodynamic limits of the asphalt binders; the higher the softening point temperature, the better suited the product is for use in high-temperature conditions. An asphalt pavement that softens at lower temperatures lacks the ability to maintain the appropriate rigidity in hot desert conditions. This can lead to roadway distortion and inelasticity due to a decrease in viscosity. Thus, the SPT test was conducted in accordance with ASTM D 36 using the ball-and-ring apparatus method. First, each sample was heated for thirty minutes at 183°C to liquify before being poured into the shouldered ring and allowed to cool for an additional thirty minutes. Simultaneously, a heated deionized water bath at approximately 5°C was prepared. Excess modified asphalt binder was removed with a heated spatula from the top of the shouldered ring and was assembled with the ring holder and ball-centering guide. The metal ball was placed on the shouldered ring and the thermometer placed in the center of the of the ring holder. This apparatus was placed into the heated water bath and monitored for a steady temperature increase. Once the metal ball touched the bottom shelf of the ring holder, the temperature of the water bath was documented as the SPT. A schematic of the ball-and-ring apparatus is documented in Figure 2.

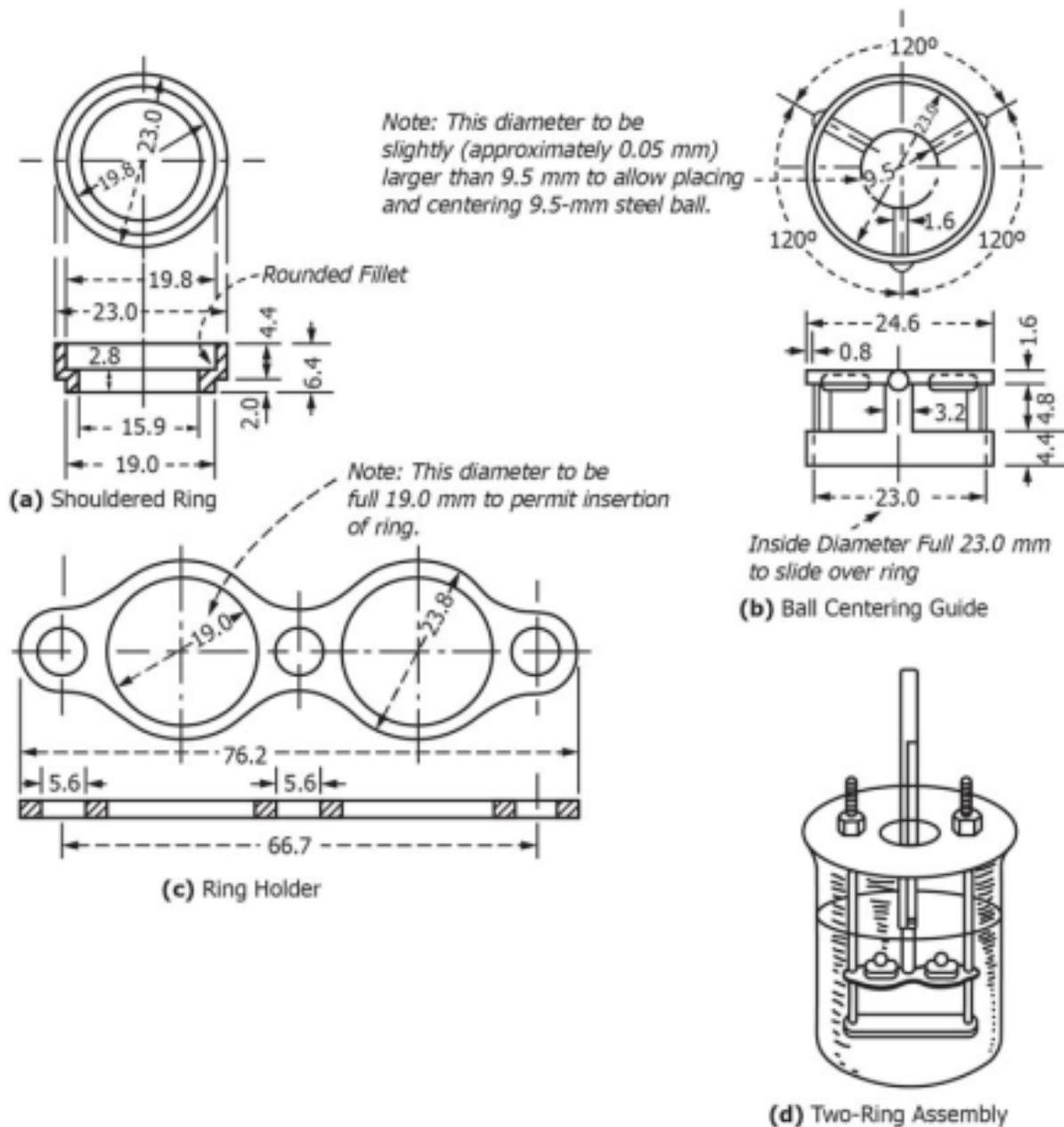


Figure 2. ASTM D 36 Ball-and-Ring Apparatus (Drews, 2008)

2.5 Dynamic Shear Rheometer

The objective of the DSR test is to determine the CSM ($G^*/\sin \delta$) and phase angle (δ), by applying a shear force to each sample. The phase angle measures the rheological property of viscoelasticity on a scale of 0° to 90° , following the trend that an asphalt binder with a lower phase angle possess a higher elasticity. Additionally, the DSR test determines the CSM parameter, defined as the resistance to deformation when an exerted shear force is applied to the asphalt binder; the higher the CSM, the greater the stiffness of the sample. Prior to testing, each sample was heated until liquified and a small amount was poured onto the rheometer plate. At temperatures ranging from 64°C to 76°C , a shear force was applied, and the resultant stress-strain response was measured using a Kinexus rheometer. The dynamic shear test was performed on non-aged and aged modified

asphalt binders to determine the effect extended time periods play on material brittleness and resistance to load deformation.

2.6 Bending Beam Rheometer

The BBR test determines the low-temperature properties dictating whether a beam of solid asphalt binder is brittle or if it will deflect force. The test apparatus incrementally increases the pressure load while the deflection of the beam is recorded. This test simulates the typical stress exerted onto a highway surface to determine whether the sample can withstand moments of stress and has the capacity to successfully return to its initial state. Sample failure appears as permanent cracks or ridges often seen on highway surfaces. Each sample is initially heated to 183°C (a liquid state) before being carefully poured into a specimen mold with the excess removed from the top to create a flat surface, seen in Figure 3. Once the sample has cooled and hardened, it is removed from the specimen mold and first conditioned in a methanol bath at approximately -6.0°C for one hour prior to testing, a second test was performed after the sample acclimated in a methanol bath condition of 0°C. A methanol bath was used in this experiment due to its ability to remain as a liquid at such low temperatures. As seen in Figure 4, the BBR test is performed by exerting a load shaft onto the center of the sample (2) to determine the mechanical properties of the solid asphalt binder (1).



Figure 3. Specimen Molds with Modified Bitumen for the Bending Beam Rheometer Test

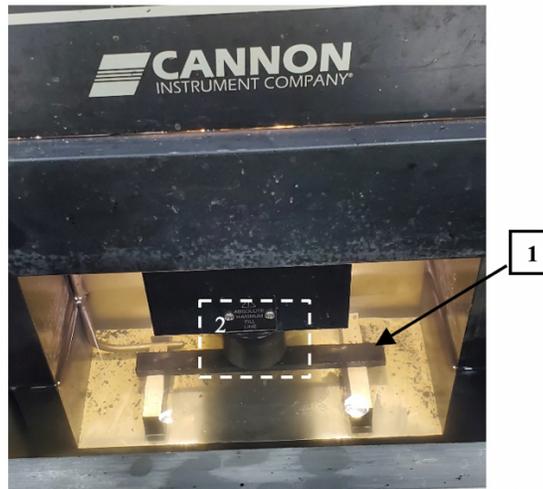


Figure 4. The Load Shaft (2) Applied Pressure onto Solid Beam of Modified Asphalt Binder (1) as it Undergoes BBR Testing

2.7 Multiple Stress Creep Recovery

The MSCR test, conducted in accordance with AASHTO T 350, is similar to the DSR test; however, instead of a single applied shear force, the MSCR test continually exerts varying levels of shear force to determine elasticity (AASHTO, 2019). The purpose of the MSCR test is to evaluate the sample's ability to retain elasticity over time and assess whether it will return to its original state after exertion or increased pressure. Again, the Kinexus rheometer was utilized throughout MSCR testing.

Samples that demonstrate the ability to withstand the force applied from the MSCR test went through simulated aging and were subjected to additional testing. The aging process (5–7 years) is simulated by the pressure aging vessel (PAV), and the rolling thin-film oven (RTFO) that can simulate oxidative aging of 5 years. The aging process is crucial in estimating the asphalt binder's potential lifespan and maintenance costs. The samples were prepared for the PAV by heating until liquified and were then evenly poured into a thin film oven pan and set onto a rack, as depicted in Figure 5. The apparatus was placed into the pressurized aging vessel for approximately twenty hours, to simulate aging of 5–7 years. Similarly, the samples prepared for the RTFO were liquified and poured into a glass cylinder, which was carefully rotated to ensure even coating of the glass interior surface, as depicted in Figure 6. The RTFO sample cylinders were placed in a convection oven for 85 minutes at 163°C to simulate asphalt binder aging up to 5 years in a harsh environment.



Figure 5. Thin Film Oven Pans for Pressure Aging Vessel Testing



Figure 6. Rolling Thin Film Oven Cylinders

2.8 Storage Stability

The storage stability test was performed to determine potential changes in asphalt binder characteristics due to long-term storage in sub-zero temperatures. Additionally, the researchers investigated whether all components were equally distributed after several phase changes during storage. Polymer degradation can occur at temperatures above 200°C; therefore, each sample was prepared for storage stability by heating the modified asphalt binder and then maintaining the temperature at 183°C for one hour to significantly reduce the viscosity, and to ensure the polymers cease to degrade. The softened samples were poured into test tubes and left in a convection oven for 48 hours at 165°C. Subsequently, the sample test tubes were promptly placed in a freezer for 3–5 hours to simulate long-term cold storage. The performance of the asphalt binder after long-term storage was observed by the SPT test.

The samples were divided into three pieces of equal length and heated separately until liquified to adequately cast the modified bitumen into the SPT test rings. Once cooled, the shouldered rings containing the top and bottom pieces were tested with the SPT in accordance with ASTM D 36.

3. Results & Discussions

Of the samples from Phase I–IV, seven samples were selected for testing. Henceforth, these samples will be referred to by the sample number as referenced in Table 5. Sample No. 4 was omitted from testing due to issues in evenly reducing the size of the SBS polymers.

Table 5. Sample Number and Corresponding Sample Components

Phase	Sample No.	Components
Nanoparticles + PG64-10	1	1% Nano-24 + PG64-10
SBS Polymer + PG64-10	2	1% SBS + PG64-10
	3	3% SBS + PG64-10
Coated Polymer + PG64-10	4	3% Nano-24 Coated Fine SBS + PG64-10
	5	1% Nano-24 Coated SBS + PG64-10
	6	3% Nano-24 Coated SBS + PG64-10
	7	3% Nano-27 Coated SBS + PG64-10
Phase 1 + SBS Polymer	8	1% Nano-24 + PG64-10 + 3% SBS

3.1 Softening Point Temperature

Testing of modified asphalt binder samples began with the SPT test to observe whether the mixing method used in Phase I or Phase II was more effective. An absence of change between samples No. 1 and No. 2 correlates to improper shearing or improper mixing of the SBS, causing the polymers to distribute unevenly in the sample. The SPT results also demonstrate the efficacy the nanoparticles contribute to the sample’s rigidity. Accordingly, an increase in the SPT of samples No. 6 and No. 7 correlates with the increase of the nanoparticle ratio. Furthermore, an increase in the SBS ratio from sample No. 2 and No. 3 caused the SPT performance to rise appropriately, so it is expected that the samples composed of higher SBS concentrations will exhibit higher softening point temperatures. Interestingly, these results also indicate that SBS coated with nanoparticles leads to a decrease in softening point temperature; however, SPT performance slightly improves after aging.

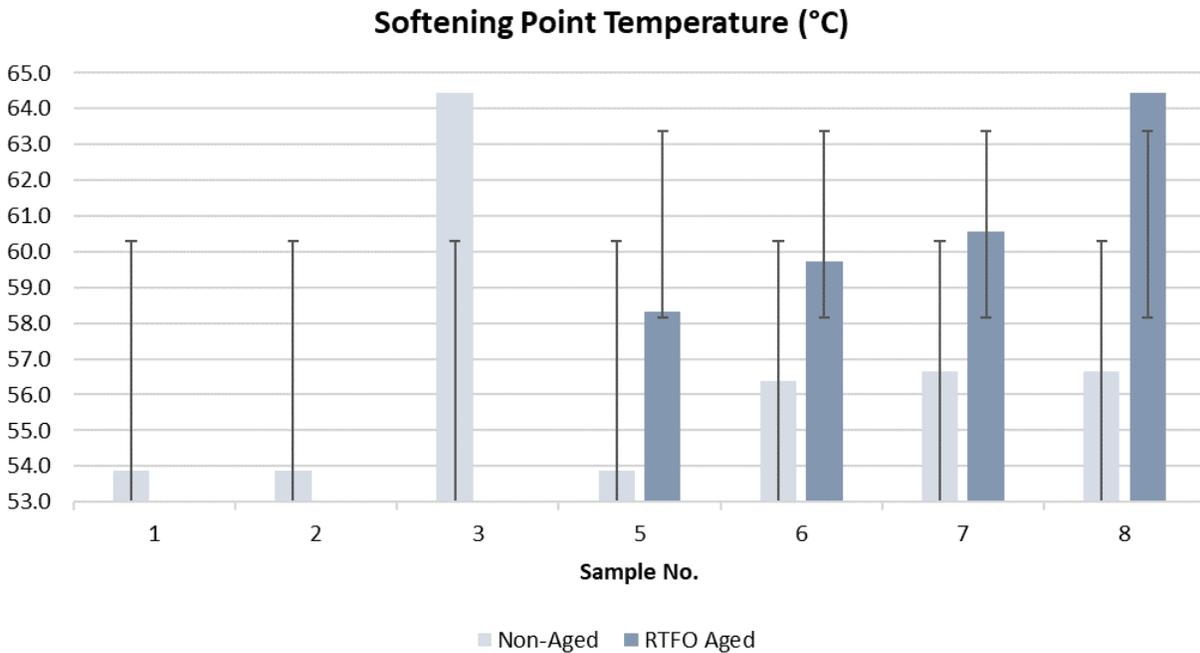


Figure 7. Softening Point Temperatures of Samples Tested in Accordance with ASTM D 36

After samples No. 5–8 went through the RTFO aging process, the respective SPT increased compared to the non-aged samples, seen in Figure 7. The trend of increased rigidity seen is due to the increase of graphene content within the samples, which follows the trend of previous studies and experiments; however, it is not certain whether there is a specific time frame when the SPT no longer increases. An interesting revelation is the dramatic difference nanoparticle composition and incorporation into the asphalt binder plays in the thermodynamic and rheological properties that can be seen between samples No. 5, 6, 8. Even though there is a small increase in the SPT of the non-aged 1.0 wt.% to 3.0 wt.% graphene, the enhanced sample properties due to the SPT increase is disproportionate to the addition of nanoparticle composition due to the steep cost increase of higher nanoparticle compositions. The sudden jump in SPT of sample No. 5 after RTFO aging may be evident of the sample’s increased lifespan. In the case of sample No. 8, although there is an increase in the SPT after aging, it is similar to the SPT of the non-aged sample No. 3; however, No. 8 exceeds all other bitumen samples with graphene-nanoparticle-coated SBS.

3.2 Dynamic Shear Rheometer

High-temperature properties and viscoelasticity were captured by plotting the logarithmic complex shear modulus and phase angle, respectively. The DSR test results in Figure 8 were performed prior to aging via RTFO or PAV. Initially, the samples were tested at 64°C, and samples were tested at higher temperatures of 70°C and/or 76°C until the resulting CSM fell below 1 kPa.

An attempt to examine the effects of normal use on a highway surface by subjecting samples No. 5–8 through RTFO aging concluded that in the case of sample No. 8, the elasticity has not changed, but its resistance to deformation increased due to a higher G^* after RTFO aging. These results are seen in Figure 9.

In addition, Figure 10 presents the DSR results of samples No. 5–8 that went through PAV aging at lower temperatures of 31°C to 37°C, as advised and recommended by the technicians from San Joaquin Refining Co. In sample No. 6 the phase angle follows a different trend, where it decreases as the temperature is increased, indicating an increase in elasticity. It is also difficult to assess the differences between the RTFO and PAV-aged samples due to a difference in the temperatures at which the samples were tested.

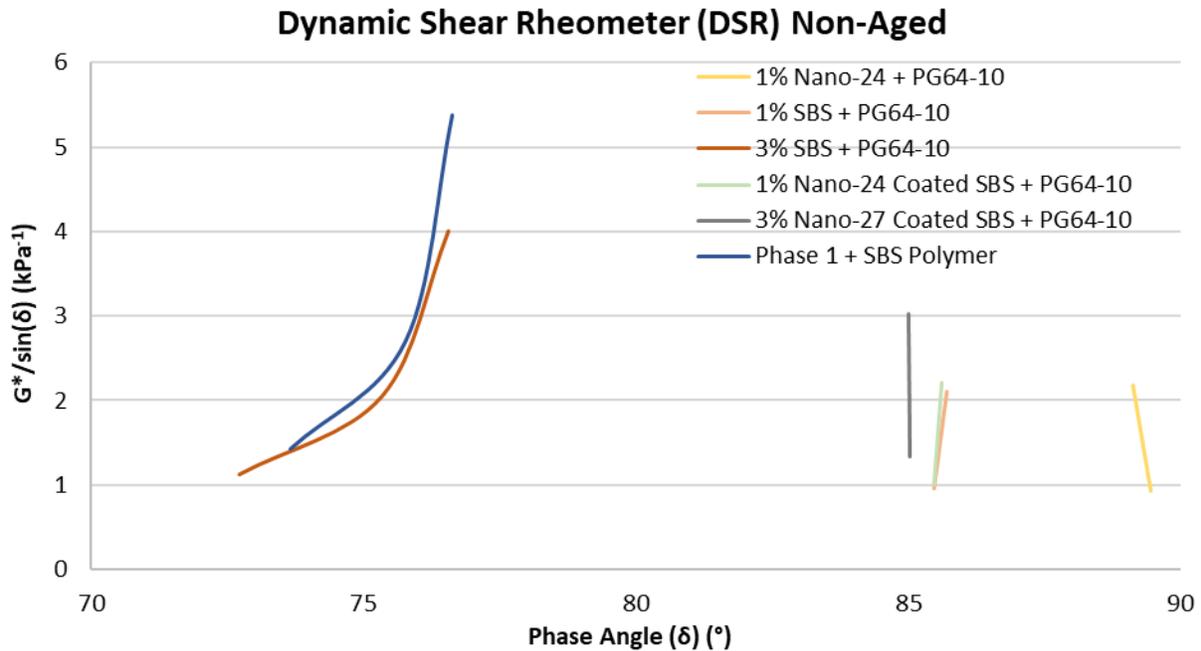


Figure 8. Complex Shear Modulus & Phase Angle of Non-Aged Samples at Temperature Range 64°C to 76°C

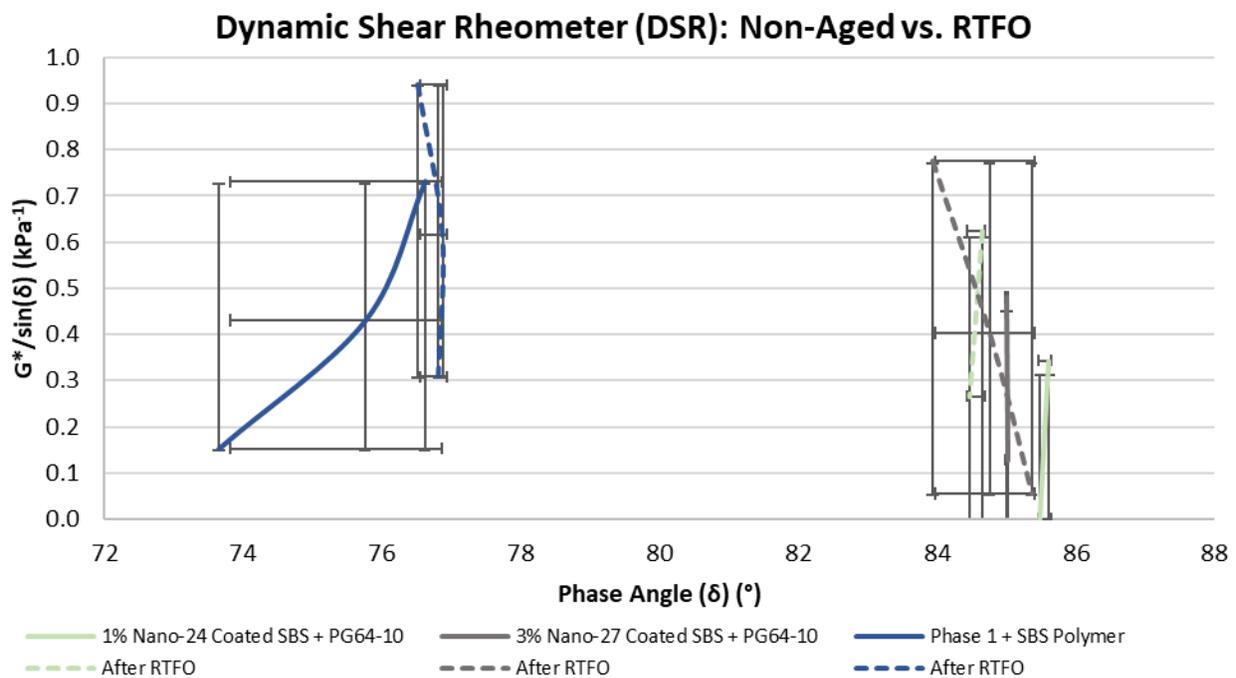


Figure 9. CSM & Phase Angle from 64–76°C of Samples No. 5, 7, 8 Non-Aged & after

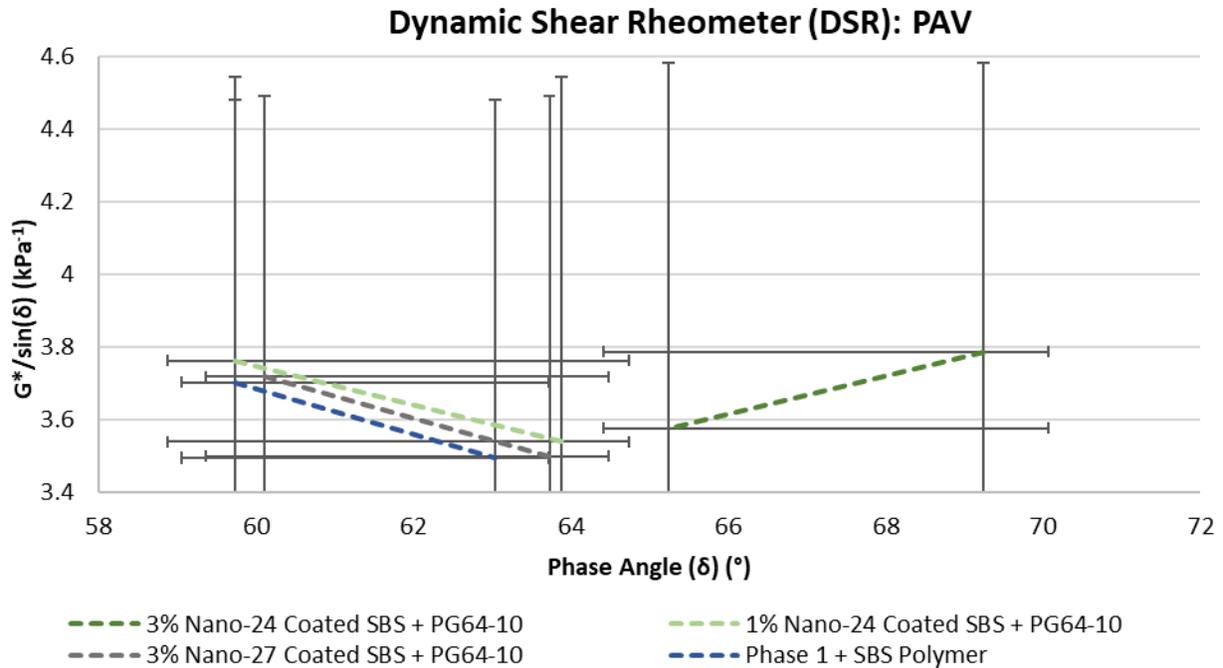


Figure 10. CSM & Phase Angle at Temperature Range 34°C to 37°C of Samples No. 5–8 after PAV Aging

The asphalt binder samples are expected to age at an acceptable rate; however, each may become susceptible to oxidative aging at higher temperatures. Although available methods to predict aging are imperfect and are strictly estimations, the data demonstrate that at higher temperatures, the CSM will decrease, causing the sample to be more susceptible to deformation. The data also suggest that as the temperature increases for the PAV-aged samples, the phase angle will increase, causing the sample to lose elasticity. It is expected that a highway surface will lose rigidity and elasticity over time due to aging; however, the results do not offer a conclusive timeline of roadway degradation or an approximate point when rutting will occur. The DSR test results concluded that all samples would lose their rigidity as temperatures increase. Overall, sample No. 8 exceeded the performance of the other samples with graphene nanoparticles in all stages of aging.

3.3 Bending Beam Rheometer

The results of the BBR tests focus on the investigation of the differences in rheological traits between non-aged samples and PAV-aged samples at lower temperatures. As seen in Figure 11, the samples were tested at -6.0°C and 0°C. The data suggest that both the non-aged and PAV-aged materials can withstand higher levels of pressure at lower temperatures. Furthermore, as the amount of nano-24 is increased from 1.0 wt.% to 3.0 wt.%, the rigidity of the sample increases slightly in both aged and non-aged states.

Interestingly, a modest yet apparent variability can be seen of the increase in PAV aged creep stiffness between the samples of No. 5, 6, 7, whose defining characteristics are different component compositions. This suggests that an increase in graphene nanoparticle composition notably decreases asphalt binder brittleness when compared to the characteristics of mix design alone, as in sample No. 8.

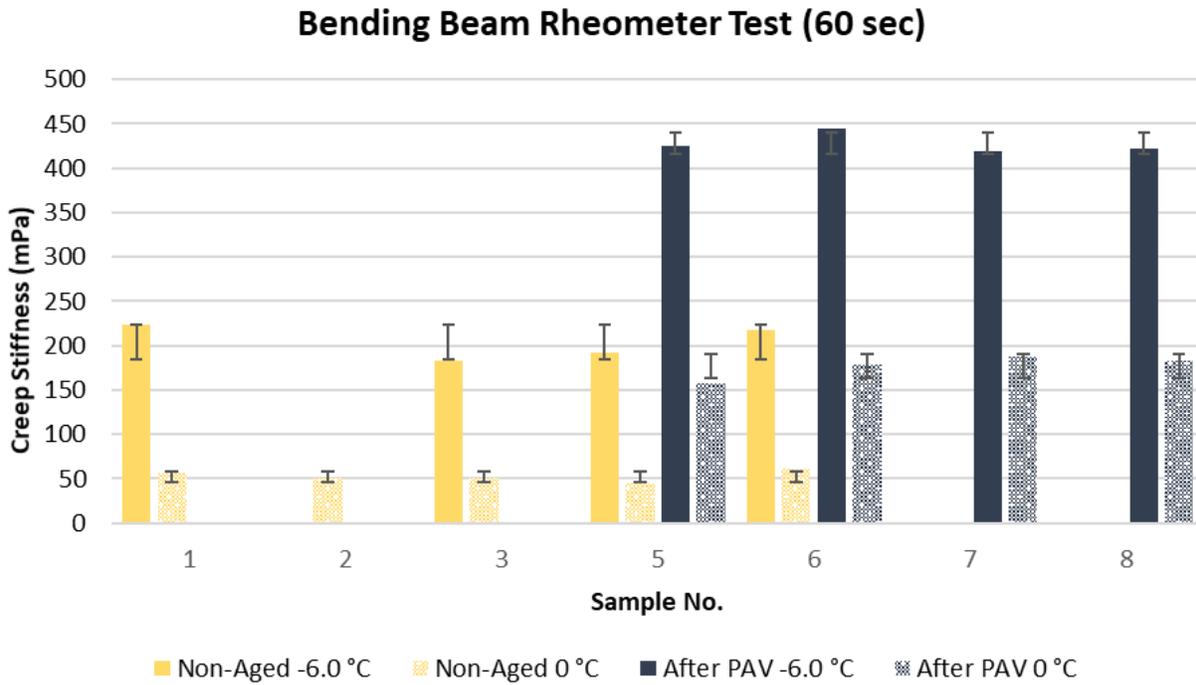


Figure 11. Bending Beam Rheometer Test Results at -6°C and 0°C of Non-Aged and PAV Aged Samples

3.4 Multiple Stress Creep Recovery (MSCR)

The MSCR test is essential in the exhibition of rheological traits that can be compared for potential use in real-world applications. As the complex modulus of an asphalt binder increases, an increase in stiffness is observed; this relates to the material’s ability to revert to its original form after subsection to a shear force. Ultimately, the MSCR test determines the creep compliance (J_{nr}), classified as the accumulation of strain caused by constant loading and unloading of stress to cause deformation.

In reference to Figure 12, the MSCR test was performed at a temperature of 64°C both before and after RTFO aging. Due to the desirability of a lower J_{nr} , results indicate that in the case of samples No. 5 and No. 6, the non-aged sample with higher nanoparticle composition, when tested at 0.1 kPa and 3.2 kPa, is better suited for roadway application. Similarly, after RTFO aging, samples No. 5, and 6 exhibit identical trends to the non-aged samples, and the higher nano-24 composition of No. 6 obtained a lower J_{nr} .

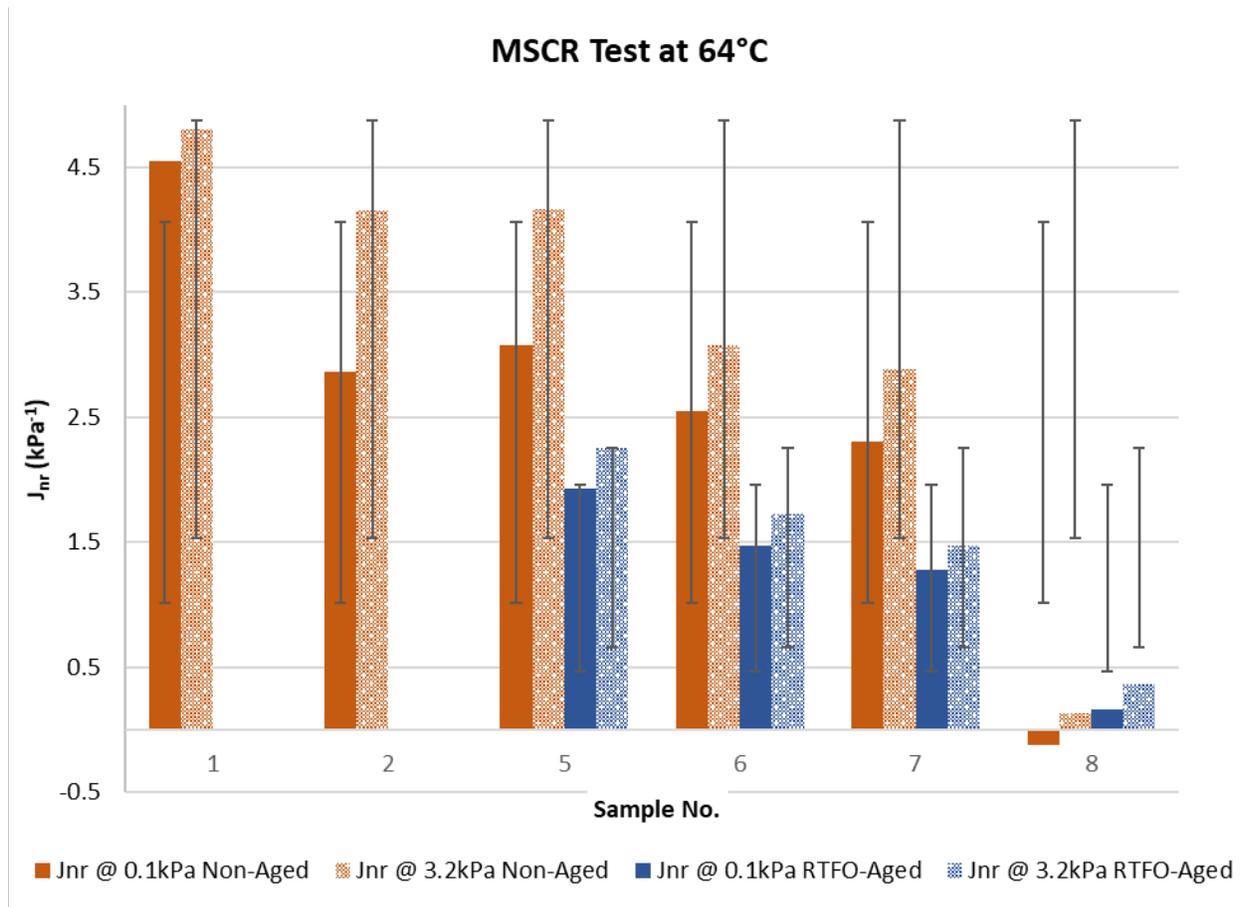


Figure 12. Multiple Stress Creep Recovery Test at 64°C

As can be clearly seen in Figure 12, the creep compliance decreases significantly in sample No.8, representing an improvement in the stiffness of asphalt binder prepared by separate mixing of the nanoparticles and SBS polymer. Addition of higher nanoparticle content slightly increases the stiffness; however, the non-coated graphene and SBS bitumen suggests an unquestionably lower susceptibility to rutting.

Analogous trends are seen in Figure 13, which presents the outcomes of MSCR results performed at 70°C. The ability of sample No. 8 to resist high stress indicates that coating SBS polymers with graphene is insignificant in the preparation of the asphalt samples to attain higher stress performance materials. Overall, MSCR test results demonstrate that sample No. 8 also outperforms sample No. 3, the SBS-modified asphalt binder. Elasticity in the SBS polymer improved as a result of the addition of nanoparticles. Since sample No. 8 contained a 1.0% nano-24 content, further studies are required to determine the optimal graphene nanoparticle and SBS content to enhance samples prepared by simultaneous mixing.

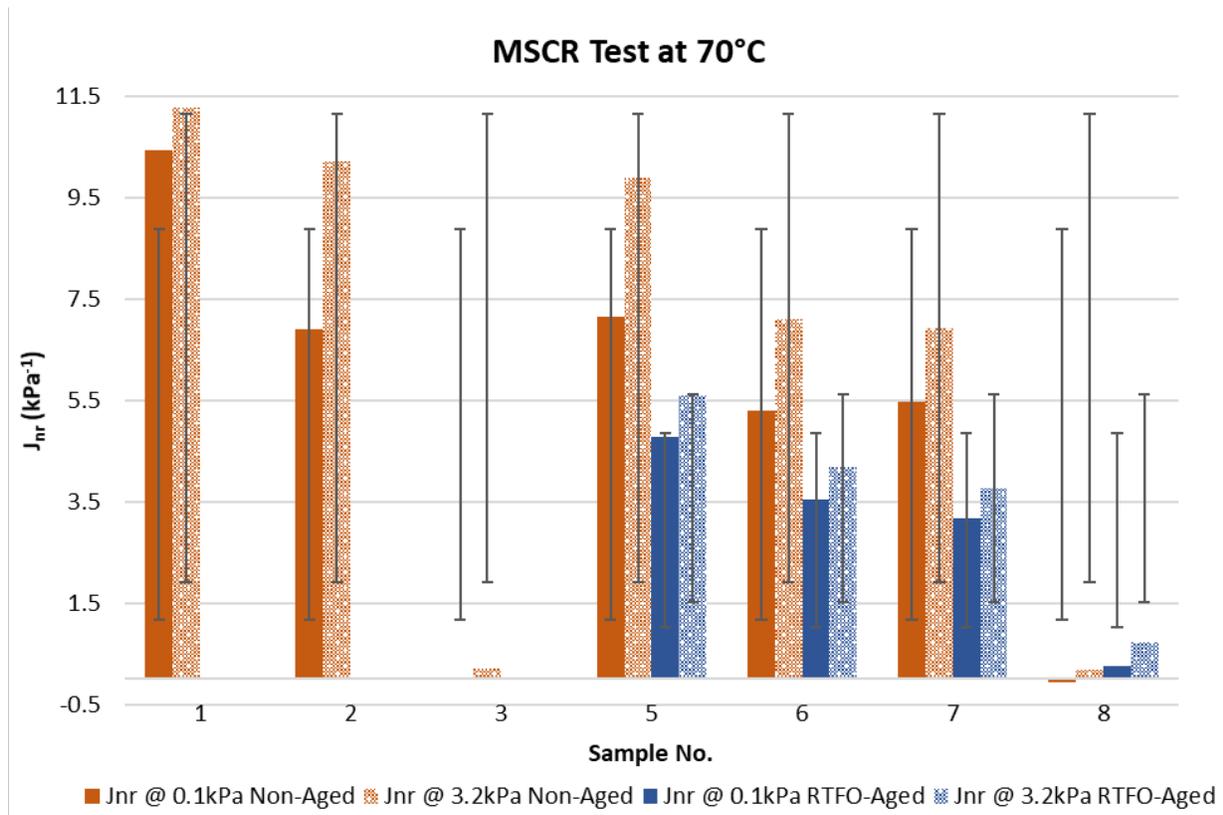


Figure 13. Multiple Stress Creep Recovery Test at 70°C

3.5 Storage Stability

The storage stability test indicates minimal deviation in SPT between the top and bottom of a stored sample under freezing temperature conditions. The data represented in Figure 14 suggest that the development of uniform component composition within the asphalt binder is feasible with this mixing methodology. The SPT of the samples subjected to freezing temperatures and the untreated samples is analogous—with the exception of sample No. 8, where a notable increase of SPT is seen in the samples exposed to colder temperatures. The workability of the asphalt binder mix design of graphene nanoparticles and SBS in sample No. 8 was enhanced and indicates that storage stability was not degraded.

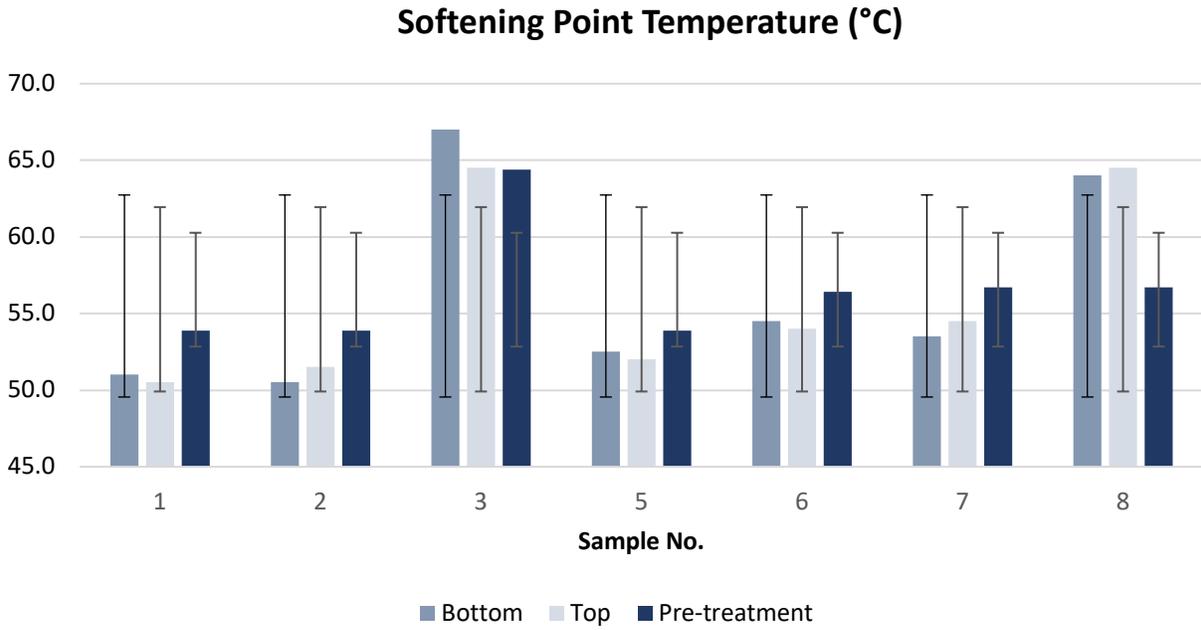


Figure 14. Softening Point Temperatures of Top and Bottom of Storage Stability Testing Procedures in Comparison to Prior to Storage Stability Testing

4. Conclusions

Upon testing of all samples for various rheological properties, it can be concluded that the introduction of nanoparticles into the asphalt binder slightly improves the sample properties by increasing the rigidity and reducing brittleness after aging. When compared to strictly SBS polymer samples, the graphene-modified polymers have shown themselves to be much more rigid while maintaining an acceptable amount of elasticity. Furthermore, it can be concluded that the nanoparticles have a positive effect on resilience to aging. The data suggest that after the PAV and RTFO aging processes, the samples maintain favorable rheological properties that have the potential to promote a longer lifespan.

An additional area requiring further investigation is whether the amount of nanoparticles causes significant improvements, considering the high cost of an increase in nanoparticle composition. When samples No.5, and 6 was compared, both showed an increase in rigidity while maintaining an acceptable level of elasticity. However, the increase of 1.0 wt.% to 3.0 wt.% is disproportionate to the level of enhanced characteristics. It should be noted that these conclusions are based on our investigation of various graphene nanoparticle compositions, but further studies are required to determine the optimal composition of these nanoparticles that leads to a proportional increase in favorable rheological properties.

Overall sample performance indicates the mixing methodology of graphene-incorporated asphalt binder followed by the addition of polymers possesses superior mechanical properties to the graphene-coated SBS samples. The mix design of sample No. 8 showed an enhancement in viscoelastic and high-temperature rutting from the DSR test. In addition, a decrease in the creep compliance from the MSCR test revealed the modified asphalt binder's ability to withstand added stresses leading to significant ability to resist permanent deformation. The durability of this Phase IV sample is attributed to the mixing procedure and can be more extensively investigated in further studies to determine the optimal graphene nanoparticle and SBS polymer compositions of the mix design within the asphalt binder for practical highway design applications.

Future plans for this study include expanding into the material properties of various nanoparticles, such as the nano-27 which was used only as a comparison for these trials. Seeing that the addition of nanoparticles has shown to be beneficial, further studies shall be done to determine which nanoparticles are most effective when used on highway road surfaces. This can also be said for the polymers used. Although these trials that have been conducted used a single polymer to maintain consistency of results, other polymers may prove to have better results especially in other climates. Upcoming plans include the incorporation of several different polymers to test whether they produce similar or much more favorable mechanical properties.

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List of Abbreviations

AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Society for Testing and Materials
BBR	Bending Beam Rheometer
Caltrans	California Department of Transportation
CSM	Complex Shear Modulus $G^*/\sin(\delta)$ (kPa)
DSR	Dynamic Shear Rheometer
PG64-10	Performance Grade Asphalt Binder
PAV	Pressure Aging Vessel
J_{nr}	Non-Recoverable Creep Compliance
RPM	Revolutions per Minute
RTFO	Rolling Thin Film Oven
SHS	State Highway System
SPT	Softening Point Test
SBS	Styrene-butadiene-styrene
TTI	Texas Transportation Institute
wt. %	Weight Percent

List of Nomenclature

δ Phase Angle (°)

Appendix A: Data About Mixture of Components

Sample No.	Components	Mass (g)				
		Container	Cont. + PG64-10	PG64-10	Mixture Components	
					Required	Actual
1	1% Nano-24 + PG64-10	93.42	821.46	728.04	7.2804	7.29
2	1% SBS + PG64-10	93.66	902.32	808.66	8.08	8.08
3	3% SBS + PG64-10	93.31	764.94	671.63	20.14	20.19
5	1% Nano-24 Coated SBS + PG64-10	96.03	698.99	602.96	6.02	8.05
6	3% Nano-24 Coated SBS + PG64-10	95.9	761.51	665.61	6.65	6.68
7	3% Nano-27 Coated SBS + PG64-10	95.92	737.5	641.58	6.41	6.42
8	1% Nano-24 + PG64-10 + 3% SBS	96.44	747.32	650.88	19.52	19.57

Appendix B: Softening Point Temperature Test Data

Non-Aged		
Sample No.	Components	Softening Point Temperature (°C)
1	1% Nano-24 + PG64-10	53.9
2	1% SBS + PG64-10	53.9
3	3% SBS + PG64-10	64.4
5	1% Nano-24 Coated SBS + PG64-10	53.9
6	3% Nano-24 Coated SBS + PG64-10	56.4
7	3% Nano-27 Coated SBS + PG64-10	56.7
8	1% Nano-24 + PG64-10 + 3% SBS	56.7

RTFO-Aged		
Sample No.	Components	Softening Point Temperature (°C)
5	1% Nano-24 Coated SBS + PG64-10	58.3
6	3% Nano-24 Coated SBS + PG64-10	59.7
7	3% Nano-27 Coated SBS + PG64-10	60.6
8	1% Nano-24 + PG64-10 + 3% SBS	64.4

Appendix C: Dynamic Shear Rheometer Test Data

Non-Aged Sample No.	Components	G*/sin(δ) (kPa ⁻¹)			Phase Angle (δ) (°)		
		64°C	70°C	76°C	64°C	70°C	76°C
1	1% Nano-24 + PG64-10	2.176	0.931		89.12	89.47	
2	1% SBS + PG64-10	2.106	0.955		85.71	85.48	
3	3% SBS + PG64-10	4.009	2.057	1.132	76.56	75.34	72.72
5	1% Nano-24 Coated SBS + PG64-10	2.204	0.999		85.62	85.49	
6	3% Nano-24 Coated SBS + PG64-10	2.836			84.79		
7	3% Nano-27 Coated SBS + PG64-10	3.019	1.341		85.00	85.02	
8	1% Nano-24 + PG64-10 + 3% SBS	5.379	2.694	1.422	76.62	75.76	73.65

RTFO Aged							
Sample No.	Components	G*/sin(δ) (kPa ⁻¹)			Phase Angle (δ) (°)		
		64°C	70°C	76°C	64°C	70°C	76°C
5	1% Nano-24 Coated SBS + PG64-10	4.20 8	1.84 5		84.6 6	84.4 7	
6	3% Nano-24 Coated SBS + PG64-10	5.54 5	2.37 8	1.07 4	84.2 5	85.2 2	85.9 4
7	3% Nano-27 Coated SBS + PG64-10	5.95 8	2.53 2	1.13 5	83.9 4	84.7 6	85.3 7
8	1% Nano-24 + PG64-10 + 3% SBS	8.72 4	4.13 8	2.03 2	76.5 2	76.8 8	76.8 2

PAV Aged							
Sample No.	Components	G*/sin(δ) (kPa ⁻¹)			Phase Angle (δ) (°)		
		31°C	34°C	37°C	31°C	34°C	37°C
5	1% Nano-24 Coated SBS + PG64-10	5774	3458		59.74	63.88	
6	3% Nano-24 Coated SBS + PG64-10		6115	3768		69.24	65.24
7	3% Nano-27 Coated SBS + PG64-10		5209	3149		60.11	63.73
8	1% Nano-24 + PG64-10 + 3% SBS		5037	3114		59.74	63.04

Appendix D: Bending Beam Rheometer Test Data

Non-Aged			
Sample No.	Components	Creep Stiffness (MPa)	
		-6.0 °C	0 °C
1	1% Nano-24 + PG64-10	223	56.1
2	1% SBS + PG64-10		50.2
3	3% SBS + PG64-10	183.5	51.1
5	1% Nano-24 Coated SBS + PG64-10	192.5	45.4
6	3% Nano-24 Coated SBS + PG64-10	217	60.8

PAV Aged			
Sample No.	Components	Creep Stiffness (MPa)	
		-6.0 °C	0 °C
5	1% Nano-24 Coated SBS + PG64-10	424.7	157.0
6	3% Nano-24 Coated SBS + PG64-10	445.0	179.0
7	3% Nano-27 Coated SBS + PG64-10	419.5	188.0
8	1% Nano-24 + PG64-10 + 3% SBS	422.0	183.5

Appendix E: Multiple Stress Creep Recovery Test Data

Non-Aged							
Sample No.	Components	J _{nr} @ 0.1 kPa			J _{nr} @ 3.2 kPa		
		64°C	70°C	76°C	64°C	70°C	76°C
1	1% Nano-24 + PG64-10	4.55	10.44		4.8	11.26	
2	1% SBS + PG64-10	2.859	6.892		4.152	10.21	
3	3% SBS + PG64-10	106.6	0.00717	0.07691	90.9	0.2145	0.513
5	1% Nano-24 Coated SBS + PG64-10	3.076	7.144		4.16	9.892	
6	3% Nano-24 Coated SBS + PG64-10	2.55	5.297		3.08	7.092	
7	3% Nano-27 Coated SBS + PG64-10	2.302	5.465		2.888	6.927	
8	1% Nano-24 + PG64-10 + 3% SBS	-0.1207	-0.06505	-0.06092	0.127	0.1865	0.3975

RTFO Aged							
Sample No.	Components	J _{nr} @ 0.1 kPa			J _{nr} @ 3.2 kPa		
		64°C	70°C	76°C	64°C	70°C	76°C
5	1% Nano-24 Coated SBS + PG64-10	1.926	4.668		2.251	5.52	
6	3% Nano-24 Coated SBS + PG64-10	1.469	3.55	7.826	1.728	4.195	9.411
7	3% Nano-27 Coated SBS + PG64-10	1.282	3.162	7.291	1.471	3.756	8.771
8	1% Nano-24 + PG64-10 + 3% SBS	0.1638	0.2691	0.3259	0.36	0.7264	2.333

Appendix F: Storage Stability Test Data

Sample No.	Components	Softening Point Temperature (°C)		
		Top	Bottom	Pre-treatment
1	1% Nano-24 + PG64-10	50.5	51.0	53.9
2	1% SBS + PG64-10	51.5	50.5	53.9
3	3% SBS + PG64-10	64.5	67.0	64.4
5	1% Nano-24 Coated SBS + PG64-10	52.0	52.5	53.9
6	3% Nano-24 Coated SBS + PG64-10	54.0	54.5	56.4
7	3% Nano-27 Coated SBS + PG64-10	54.5	53.5	56.7
8	1% Nano-24 + PG64-10 + 3% SBS	64.5	64.0	56.7

About the Authors

Sara Moghtadernejad, PhD

Dr. Sara Moghtadernejad joined the CSULB Department of Chemical Engineering as an Assistant Professor in Fall 2018. Dr. Moghtadernejad's Multiphase Flow Lab (MFL) pursues various interdisciplinary and multidisciplinary research directions in Chemical Engineering. She studies processes and phenomena involving fluid and powders that depict phase changes and have multiple states. All of her research arises from practical applications, with the aim of solving challenging and contemporary industrial issues. MFL is a result-oriented laboratory and is equipped with state-of-the-art tools to closely reproduce real-life conditions and to design various interesting experiments. Those are followed by distinct numerical and statistical analyses to verify experimental observations and to provide solutions, enhancements, and corrections.

Ehsan Barjasteh, PhD

Dr. Ehsan Barjasteh joined the CSULB Departments of Mechanical & Aerospace Engineering and Chemical Engineering (joint position) as an Assistant Professor in Fall 2015. His research focuses on development and optimization of advanced polymer and composite materials and processes. His research interests include manufacturing advanced composite materials using out-of-autoclave (OOA) processes, self-healing and repair of composite structure using smart materials, toughening advanced composite materials, life-time prediction and aging, 3D-printing of polymers for high-temperature applications, and progressive damage detection and analysis.

Ren Nagata

Ren Nagata attended California State University Long Beach for an undergraduate degrees and for graduate studies in the Chemical Engineering department.

Haia Malabeh

Haia Malabeh attended California State University Long Beach for an undergraduate degree and for graduate studies in the Chemical Engineering department. Haia also holds a Professional Engineering License in Mechanical Engineering.

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