NEAR EAST UNIVERSITY

INSTITUTE OF GRADUATE STUDIES

DEPARTMENT OF CIVIL ENGINEERING

# EVALUATION OF THE PERFORMANCE CHARACTERISTICS OF ASPHALT BINDERS AND MIXTURES CONTAINING WARM ADDITIVE MODIFIED WITH NANO-SILICA

**IN PARTIAL FULFILMINT OF THE REQUIRMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN CIVIL ENGINEERING**

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**NICOSIA**

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**Ph.D. THESIS**

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## Declaration

I hereby declare that all information, documents, analysis, and results in this thesis have been collected and presented according to the academic rules and ethical guidelines of the Institute of Graduate Studies, Near East University. I also declare that as required by these rules and conduct, I have fully cited and referenced information and data that are not original to this study.

Dania Alothman

17/06/2022

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**Dania Alothman**

Dania

**To my parents, husband, and lovely daughter…**

## Abstract

**Evaluation of the Performance Characteristics of Asphalt Binders and Mixtures Containing Warm Additive Modified with Nano-Silica**

**Dania Alothman**
**Ph.D., Department of Civil Engineering**
**June, 2022, 187 pages**

The impact of base and ZycoTherm binders modified with various proportions of Nano-Silica (i.e., 2%, 4%, and 6%) based on the weight of the binder at high, moderate, and low temperatures performance was investigated. Brookfield viscosity, penetration, softening point, and storage stability tests were all conducted on base and modified binders. Moreover, the rheological properties were examined by bending beam rheometer (BBR), Superpave rutting parameter ($G^*/\sin \delta$), master curves, isochronal plots, multiple stress creep recovery (MSCR), and Superpave fatigue parameter ($G^*.\ \sin \delta$). Furthermore, resilient modulus test and moisture susceptibility to evaluate the moisture resistance of bituminous mixtures were investigated. By observing the outcomes of the modified binder tests, the incorporation of Nano-Silica into the base and ZycoTherm binders improved temperature susceptibility. Based on the outcomes of the Superpave rutting parameter, the inclusion of Nano-Silica enhanced the stiffness of the modified binders up to 130% for 6% of Nano-Silica, while the improvement was caused by 6% of Nano-Silica with ZycoTherm was nearly 92% at 70 $^{\circ}$C. The MSCR test revealed that the base and ZycoTherm binders' recovery *(R %)* was 27.72% and 25.99% for 6% of Nano-Silica and 6% Nano-Silica with ZycoTherm at 100 MPa, respectively. The results show that the incorporation of Nano-Silica is capable of improving the rutting potential regardless of the levels of stress. The rutting resistance of modified base and ZycoTherm binders were improved at all concentrations of Nano-Silica, with the optimal concentration being 6% Nano-Silica. Moreover, it was observed at different temperatures that the values for the complex shear modulus raised when the phase angle values were reduced. Furthermore, at low temperatures, it is presumed that the performance of Nano-Silica modified bitumen binders will have minimal performance as opposed to the binders prepared with ZycoTherm with or without Nano-Silica particles are able of changing the performance grade for the base bitumen. The laboratory assessment findings revealed that all of the modified mixes utilized in this research enhanced adhesion qualities and raised the resistance of the bituminous mixes to moisture susceptibility. Among all the mixes investigated in this study, the bituminous mixes both ZycoTherm and Nano-Silica demonstrated the greatest resistance to moisture deterioration. Furthermore, ZycoTherm and Nano-Silica combinations showed a significant rise in resilient modulus values. In addition, economic analysis revealed that the ZycoTherm /Nano-Silica modified bituminous mix was more cost effective.

***Key Words:*** WMA mixture, ZycoTherm, Nano-Silica, Rheological behavior, Moisture susceptibility.

# Abstract

## Evaluation of the Performance Characteristics of Asphalt Binders and Mixtures Containing Warm Additive Modified with Nano-Silica

**Dania Alothman**
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***Key Words:*** WMA mixture, ZycoTherm, Nano-Silica, Rheological behavior, Moisture susceptibility.

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## List of Abbreviation

| | |
|---|---|
| **AASHTO:** | American Association of State Highway and Transportation Officials |
| **ASTM:** | American Society for Testing and Materials |
| **BBR:** | Bending Beam Rheometer |
| **CAA:** | Coarse Aggregate Angularity |
| **CER:** | Coast Effectiveness Ratio |
| **DP:** | Dust to Binder Ratio |
| **DSR:** | Dynamic Shear Rheometer |
| **ESAL:** | Equivalent Single Axle Load |
| **FAA:** | Fine Aggregate Angularity |
| **FHWA:** | Federal Highway Administration |
| **GHGs:** | Greenhouse Gases |
| **HMA:** | Hot Mix Asphalt |
| **ITS:** | Indirect Tensile Strength |
| **JO PETROL:** | Jordan Petroleum Refinery Company |
| **LVDT:** | Linear Variable Differential Transformer |
| **LVE:** | Linear Viscoelastic |
| **$M_R$:** | Resilient Modulus |
| **MSCR:** | Multiple Stress Creep Recovery |
| **NAPA:** | National Asphalt Pavement Association |

**NCHRP:** Nacional Cooperative Highway Research Program

**NMAS:** Nominal Maximum Aggregate Size

**NRL:** Natural Rubber Latex

**NS:** Nano-Silica

**OBC:** Optimum Binder Content

**OGFC:** Open-Graded Friction Courses

**PAV:** Pressure Aging Vessel

**PCS:** Primary Control Sieve

**PER:** Performance Enhancement Ratio

**PG:** Performance Grade

**pH:** Power of Hydrogen

**PI:** Penetration Index

**PR:** Price Ratio

**RPM:** Round per minute

**RV:** Rotational Viscometer

**RTFO:** Rolling Thin Film Oven

**SARA:** Saturates, Aromatics, Resins, and Asphaltenes

**SE:** Sand Equivalent

**SGC:** Superpave Gyratory Compactor

**SHRP:** Strategic Highway Research Program

| | |
|---|---|
| **SMA:** | Stone Matrix Asphalt |
| **SSD:** | Saturated Surface Dry |
| **Superpave:** | Superior Performing Asphalt Pavements |
| **TSR:** | Tensile Strength Ratio |
| **TTSP:** | Time–temperature Superposition Principle |
| **VFA:** | Voids Filled with Asphalt |
| **VMA** | Voids in Mineral Aggregates |
| **WMA:** | Warm Mix Asphalt |
| **ZT:** | ZycoTherm |
| **ZSV:** | Zero Shear Viscosity |

**List of Symbol**

| | |
|---|---|
| $\sigma$**:** | Applied stress |
| $\delta$**:** | Phase angle |
| $\varepsilon_r$**:** | Strain value at the end of the recovery stage in MSCR test |
| $\varepsilon_0$: | Strain value at the beginning of the creep stage in MSCR test |
| $\varepsilon_c$**:** | Strain value at the end of the creep stage in MSCR test |
| $\varepsilon_1$**:** | Strain rate at the end of the creep stage in MSCR test |
| $\varepsilon_{10}$ | Corrected strain value at the end of the recovery period for every cycle in MSCR test |
| $\varepsilon$**:** | Strain during the load cycle |
| $\nu$ **:** | Poisson ratio |
| $\delta(t)$: | Deflection in mm at time |
| **CO:** | Carbon monoxide |
| $CO_2$**:** | Carbon dioxide |
| **D:** | Sample diameter |
| $G^*$**:** | Complex shear modulus |
| $G^*/\sin\delta$**:** | Rutting resistance parameter |
| $G^*.\sin\delta$**:** | Fatigue resistance parameter |
| $G_{mb}$**:** | The bulk specific gravity |
| $G_{mm}$**:** | Theoretical maximum specific gravity |
| $G_{sa}$**:** | Apparent specific gravity |

| | |
|---|---|
| **$G_{sb}$:** | Dry specific gravity |
| **H:** | Reversible deformation |
| ***$J_{nr}$*:** | Non-recoverable creep compliance |
| **N:** | Number of recovery and creep cycles in MSCR test |
| **$NO_X$:** | Nitrogen oxides |
| **P:** | Maximum load |
| **$P_{0.075}$:** | Aggregate% passing sieve No.200 (0.075mm) |
| **$P_{be}$:** | Effective bitumen binder |
| **Ps:** | Aggregate% of the overall weight of the mix |
| ***R%*:** | Percentage recovery |
| **S1:** | Average tensile strength of the dry subset |
| **S2:** | Average tensile strength of the conditioned subset |
| **$SiO_2$:** | Silicon dioxide |
| **$SO_2$:** | Sulfur dioxide |
| ***S* (t):** | Creep stiffness at time |
| **t:** | Sample thickness |
| **Va:** | Voids of air in a compacted mix |
| ***$W_c$*:** | the amount of work that is dissipated each load cycle |

# CHAPTER I
# Introduction

## 1.1 Research Background

Asphalt pavement is extensively utilized across the world because of its great road performance. Bitumen binder is regarded as having significant effects and benefits to pavement performance as an adhesive component in the pavement. Bitumen is a viscoelastic substance whose rheological properties are mostly influenced by temperature. Because bitumen binder is temperature sensitive, it is susceptible to being soft at elevated temperatures (rutting distress) and stiff at cold temperatures (cracking distress) on the pavement. Furthermore, the black surface of bitumen absorbs a large amount of heat from solar radiation, causing the average surface temperature of the asphalt pavement to be substantially higher than the surrounding air temperature in the warmer months. This worsens the permanent deformation (rutting) of pavement surface caused by vehicle loads in the summer (Zhang, et al., 2020). The asphalt pavement must resist a wide variety of environmental variables and traffic volumes while in service. In many situations, the traditional penetration grade of bitumen can no longer provide the appropriate performance during the service life, necessitating early conservation or repair (Crucho, et al., 2019). The asphalt pavements are subjected to a variety of distresses, the most significant of which are elevated temperatures (rutting), moderate temperatures (fatigue), and low temperatures cracking from the perspective of flexible pavement design. Vehicle Loads, temperature changes, mixture characteristics, and the rheological characteristics of the bitumen all impact the nature and severity of these distresses (Bhat & Mir, 2021). Furthermore, bitumen is a substance that deteriorates with age, and its characteristics deteriorate over time. The old bitumen stiffens and becomes extremely brittle, reducing the performance of the bituminous mixtures (Hunter, et al., 2015). As a result, the aging resistance of the bituminous mixtures determines their service life (Apostolidis, et al., 2017). Additionally, pavements should have excellent durability as well as sustainability throughout their service life in order to perform efficiently. This feature is dependent on bitumen characteristics including cohesion and adhesion between the bitumen binder and aggregates as well as varied conditions such as air, temperature, and water (Mirabdolazimi, et al., 2021). Greater production temperatures of hot mix asphalt (HMA) lead to higher fuel costs, toxic gases, and increasing gaseous emissions

(Xu, et al., 2017). Therefore today, eco-friendly solutions in production technology have been highlighted as a result of the numerous environmental issues that engineers have experienced in construction projects. Warm mix asphalt (WMA) technology is one method that researchers have investigated for lowering pollutant emissions during the manufacturing and construction of bituminous mixtures. In order to address the aforementioned issues, different modifiers are being developed and incorporated into bitumen binder to enhance the high- and low-temperature properties of bituminous mixes. Numerous types of additives have been investigated throughout the years to modify the bituminous mixes. The most often studied additives, were fibers, rubber, WMA technology, as well as a broad range of polymers (Porto, et al., 2019). Although researchers have been conducted to investigate the impact of various types of nanomaterials, there are few studies on the utilization of Nano-Silica for the modification of bitumen (Sukhija, et al., 2021). Therefore, the findings of this study are expected to give a comprehensive knowledge of the influence of ZycoTherm/Nano-Silica modification on bitumen performance that will be valuable to the asphalt construction industry.

## 1.2 Problem Statement

For several decades, since pavement loads weren't really severe, various base bitumen binders were blended to enhance their qualities. Nevertheless, as traffic volumes have raised, the usage of larger axle loads and greater tire pressures have intensified the demands on roadway pavements and asphalt pavement surface layers. Conventional pavement materials fail to fulfill the practical needs of today's and tomorrow's highway pavement constructions based on earlier field observations and studies undertaken in the literature. Pavements distresses decrease the pavement's lifetime; therefore, these issues must be addressed if the pavement's lifetime is to be maintained. The most prevalent pavement distress is rutting deterioration, which occurs most frequently at high temperatures. Furthermore, moisture damage caused by a lack of bonding between the bitumen and aggregate can cause a variety of durability issues such as stripping, and a variety of many other distresses including rutting and fatigue cracking. Furthermore, the utilization of HMA in pavements has had a detrimental impact on the environment throughout the years owing to significant $CO_2$ emissions as well as energy usage. Additionally, HMA is manufactured and constructed at high temperatures (150-170 ˚C). This has resulted in a search for solutions that can minimize fuel and energy consumption while still producing bituminous mixes with the required efficiency and workability. The employment of WMA technology is a positive step to achieve this goal. As a result, superior quality, safer, more durable, and ecologically friendly paving materials are highly in demand. The use of ZycoTherm (WMA additive) has lately established as the preferred alternative in the pavement industry for addressing the stripping problems of bituminous mixes caused by moisture. In general, a mixture containing a chemical-based agent such as ZycoTherm has equivalent rutting resistance to HMA. As a result, Nano-Silica is employed to improve the rutting resistance of bitumen binder and bituminous mixtures containing WMA additive. An excellent modification method is one that maximizes the rutting resistance of the bitumen binder while costing the least amount of money. Nanomaterials such as Nano-Silica, among many other types of bitumen modification technologies, give a unique approach for improving the rheological characteristics of bitumen with lower cost than other type of modifiers according to the literature. Despite the impressive improvement in bitumen characteristics achieved by utilizing Nano-Silica, the effectiveness of the modification method has been limited by some constraints such as the negative effect on the low-temperature performance of bitumen as well as the elevated mixing and compaction temperatures owing to higher viscosities. It should also be emphasized that no perfect additive exists to enhance all of the mechanical and rheological properties of bitumen at the same time. Usually, focusing on improving one aspect of bitumen comes at the risk of missing another. Besides the advantages of these materials on improving the bitumen performance, it has been noted in various research that when employed as single additives, they are restricted in their ability to address several difficulties at the same time. On the other hand, the use of ZycoTherm material has many positive effects, such as low manufacturing temperatures leading to lower energy usage, thus lowering production costs, and it is a superior material in terms of resistance to moisture damage but does not have a greater positive effect in resisting rutting. Individually, ZycoTherm and Nano-Silica are effective bitumen binder modifiers. The following research focuses on examining the mechanical and rheological features of bitumen modified with ZycoTherm/Nano-Silica given that there have been few studies conducted on the multiple stress and creep recovery (MSCR) and frequency sweep tests of ZycoTherm-modified binders. Furthermore, studies on Nano-Silica have been carried out in recent years, but the effect of ZycoTherm/Nano-Silica concerning the rutting resistance, low-temperature cracking, and moisture susceptibility has yet to be determined. Hence, it is decisive to analyze the performance of ZycoTherm modified with several percentages of Nano-Silica utilizing these approaches. However, their compatibility as a couple is an area of research that needs to be looked into more.

### 1.3 Research Objectives

The major purpose of this research was to assess the influence of Nano-Silica on the mechanical, rheological, and physical properties of bitumen containing ZycoTherm material. The following sub-objectives can achieve the main objective:

1) Examine the effects of ZycoTherm, Nano-Silica, and ZycoTherm /Nano-Silica on the physical properties.
2) Evaluate the effects of adding ZycoTherm, Nano-Silica, and ZycoTherm /Nano-Silica on various rheological characteristics.
3) Investigate the effect of adding ZycoTherm, Nano-Silica, and ZycoTherm/Nano-Silica on the low-temperature performance using BBR.
4) Evaluate the mechanical performance of asphalt mixtures modified with ZycoTherm, Nano-Silica, and ZycoTherm /Nano-Silica.

### 1.4 Research Questions

The research questions deriving from the research statement problem that should be addressed in order to satisfy the study objectives are as follows;

1) What is the impact of ZycoTherm containing different concentrations of Nano-Silica on the physical and rheological properties of bitumen?
2) What effect does ZycoTherm with varying concentrations of Nano-Silica have on the low-temperature performance of bitumen?
3) What effect do Zycotherm, Nano-Silica, and ZycoTherm having varying concentrations of Nano-Silica have under ageing conditions?
4) What is the influence of ZycoTherm, Nano-Silica, and ZycoTherm including varied concentrations of Nano-Silica on mechanical properties of asphalt mixtures?

5) Does the presence of ZycoTherm in a combination comprising both ZycoTherm and Nano-Silica lower the mixing and compaction temperatures?

**1.5 Research Scope**

The scope of this research was decided early, even before laboratory work began. The specified scope must be reasonably possible within the timeline specified. Therefore, the study is limited to conduct the experiments on base and modified bitumen binders to characterize the conventional properties such as penetration, softening point, and viscosity, characterize the rheological properties using both dynamic shear rheometer (DSR) and bending beam rheometer (BBR), and evaluate the temperature sensitivity of the bitumen using storage stability test. Moreover, perform appropriate aggregate tests to fulfill the Superpave standards and design the aggregate gradation to meet the Superpave mix design requirements. Additionally, determination of the optimal binder content of HMA and WMA that meets 4% air voids, followed by mechanical testing on the bituminous mixes such as moisture susceptibility using the Lottman technique and resilient modulus test. Finally, carry out an economic study on both base and modified bituminous mixes to assess if the modified mixes containing both ZycoTherm and Nano-Silica may be considered economically effective.

**1.6 Research Significance**

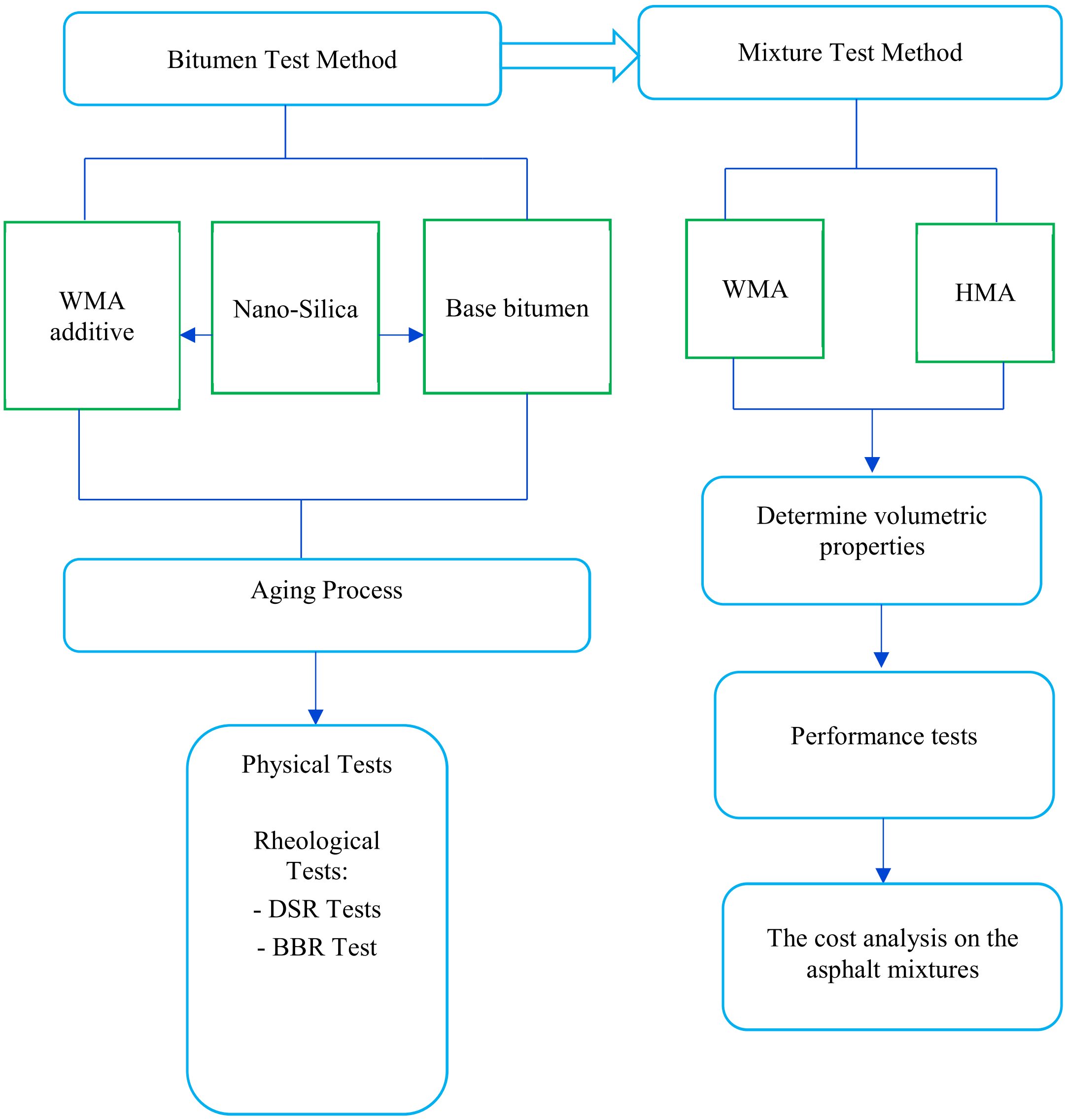
Pavement distresses reduce pavement lifetime; hence, these concerns must be addressed if pavement lifetime is to be maintained. Rutting deterioration is the most common type of pavement deterioration. As a result, Nano-Silica was applied to modify the base bitumen in order to improve its characteristics, as it is one of the best modifiers for boosting rutting resistance. Despite the significant increase in bitumen properties achieved by using Nano-Silica, the efficacy of the modification process has been restricted by various restrictions, such as greater mixing and compaction temperatures due to higher viscosities. As a result, the use of Nano-Silica modified mixes in pavements employing HMA technology has had a negative influence on the environment over the years due to considerable $CO_2$ emissions and energy consumption. As a consequence, the use of WMA technology is the solution to environmental preservation. The use of ZycoTherm as a WMA additive has recently established itself as the superior option in the pavement industry for attempting to address moisture-induced stripping problems in asphalt mixtures, lowering mixing and compaction temperatures, lowering fuel consumption, and lowering toxic gas emissions. Because the effect of ZycoTherm as an individual modifier on rutting resistance is considered ineffective when compared to base bitumen, the application of Nano-Silica in ZycoTherm binders resulted to an improvement in the characteristics of ZycoTherm modified binders. Many studies have been conducted on Nano-Silica and ZycoTherm as separate modifiers, but their compatibility as composite nanomodifiers is an area of research that requires further investigation. The use of ZycoTherm/Nano-Silica composite has many benefits, including maximizing the rutting resistance of the modified bitumen at a low cost due to the use of Nano-Silica, which is considered one of the modifiers with a lower cost compared to other modifiers according to literature, minimizing the mixing and compaction temperatures and providing safe and eco-conditions to the labor, and reducing fuel usage due to the ZycoTherm presence in the composite bitumen, and extending the lifetime of asphalt pavement by reducing stripping and rutting resistance distresses caused by the presence of both ZycoTherm and Nano-Silica therefore, lowering the cost of replacement and maintenance.

## 1.7 Research Methodology

To determine the appropriateness of Nano-Silica for bitumen modification, a set of experiments were conducted on various mixes of unmodified and modified bitumen binders. The research was concerned with the influence of Nano-Silica at three various concentrations (i.e., 2%, 4%, and 6%) combined with 0.1% ZycoTherm by bitumen weight and a base bitumen with 60/70 penetration grade as the base bitumen. The research methodology was carried out in three phases. The bitumen test technique is covered in the first phase, the mixture test method is covered in the second phase and the cost analysis of the various mixes is provided in the third phase. The bitumen testing technique takes into account all of the physical and rheological properties of the unmodified and modified binders. Starting with standard properties such as penetration, softening point, and viscosity. Considering the results of the softening point readings, temperature sensitivity tests such as temperature susceptibility and storage stability were performed. Following that, rheological features were assessed using the DSR and BBR. The DSR test protocol was conducted on unaged and aged (RTFO and PAV-aged) bitumen binders to evaluate Superpave rutting resistance, Superpave fatigue resistance, frequency sweep, and MSCR tests. The second phase of the research was carried out on HMA and WMA mixtures to analyze the mechanical performance of various combinations. Finally, a cost analysis of the mixes was undertaken to assess the economic benefits and cost effectiveness in order to determine whether the mixtures used in this study are economically feasible and provide an efficient benefit throughout the life of the asphalt pavement. The research methodology represented in Figure 1 was employed to fulfill the research goals.

**Figure 1**

*Research Outline*



## 1.8 Thesis Structure

This thesis is structured into five chapters, which are as follows;

- Chapter one includes a research introduction outlining the study problem statement, objectives, research questions, and a brief explanation of the research technique used to accomplish goals.
- Chapter two provides a detailed literature review describing earlier work in this area as well as the various approaches and strategies now in use. Furthermore, this section highlights previous research conducted on the modifiers utilized in this study as an individual modifier and their influence on the physical and rheological characteristics of bitumen, as well as their effect on the mechanical properties of the mixtures.
- The materials and experimental techniques are described in chapter three. This chapter also includes a full explanation of the methodology used in this work to assess the impact of the used modifiers on the physical, rheological, and mechanical properties of bitumen binders and mixes.
- Chapter four offers findings of the data analysis from the experimental procedures, as well as accompanying remarks. Furthermore, economic analysis of various mixtures was carried out.
- Finally, chapter five summarizes the study findings, recommendations and suggestion for future research.

# CHAPTER II
# Literature Review

## 2.1 Asphalt Pavement

The durability and resilience of asphalt pavement are well-known. Asphalt pavement is the ideal solution for almost all paved surfaces because of its strength. Along with its dependability and long life, asphalt pavement is preferred by most state and federal governments. It is not necessary to replace it for twenty to twenty-five years if it is correctly installed. Asphalt pavement is still the most preferred choice for driveways, highways, parking lots, airstrips, and other applications. Asphalt pavements are regarded as the best technique of paving highways because of characteristics such as excellent skid resistance, good stability, low noise, resistance to water damage, better comfort, and ease of maintenance (Luo, et al., 2019). Asphalt may be found almost anywhere. Aggregate, filler, sand, various kinds of additives, and liquid petroleum asphalt produce asphalt pavements. Asphalt pavements are bound together with liquid asphalt, a black sticky material. It has a viscous consistency and comes in semi-solid forms. Bitumen is another frequent word for asphalt. Once mixed, the pavement contains 90 to 95 percent aggregate and filler or sand, and 5 to 10 percent bitumen.

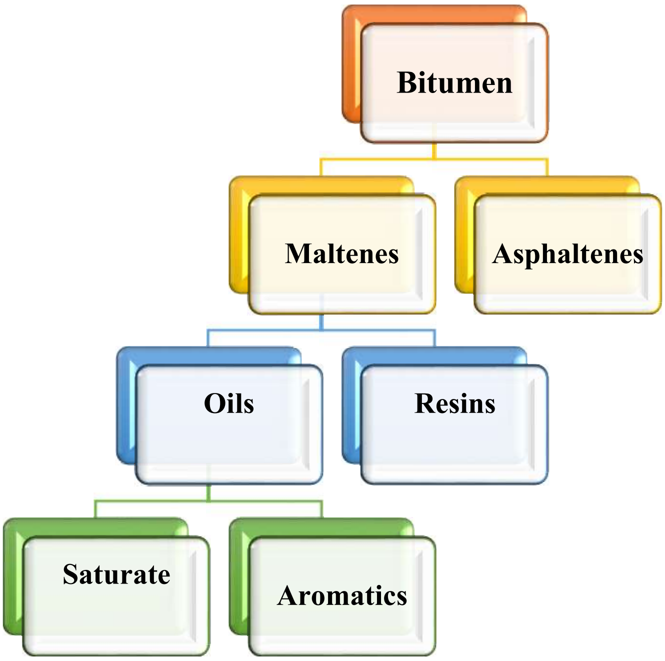
One of asphalt pavement's greatest qualities is its flexibility, which allows it to respond to various conditions caused by weather and thus the continually changing surface under it. Asphalt's capability to repel water is another important feature. The proportion of constituents and the quality of asphalt depends on the source from which it is obtained as well as the production and processing procedures of crude oil (Zhang, et al., 2015). Asphalt is a thermoplastic and viscoelastic material that is used in flexible pavement construction due to its initial low cost of construction (Jain, et al., 2013). Bitumen binder is a viscoelastic substance whose rheological properties are primarily influenced by both temperature and time. As a result, the bitumen must be resistant to weather and increasingly demanding traffic loads, so rheological qualities have an important role in several areas (Laukkanen, 2015). Higher stiffness at elevated temperatures and low frequencies and higher elasticity at low temperatures and high frequencies are favorable properties of bitumen to improve its performance characteristics which are under the influence of the climatic conditions and dynamic vehicular loading (Al-Mansob, et al., 2016). Due to its temperature sensitivity, asphalt binder is vulnerable to becoming soft at higher temperatures and stiff at lower temperatures, resulting in pavement rutting and cracking, respectively. Asphalt is indeed an organic combination made up of hydrocarbons and their byproducts that ages quickly when exposed to environmental conditions such as oxygen, water and heat. Asphalt pavements turns exceedingly stiff and brittle as its ages, resulting in low-temperature and fatigue cracking. These problems shorten the pavement's service life and raise maintenance expenditures. The use of additives in asphalt mixtures to improve their qualities has increased dramatically in recent years. Asphalt pavements are a valuable resource for both economic growth and daily living in both industrialized and growing countries. Because of the constantly increasing traffic volume, it is known that bitumen binders and mixes with enhanced properties are required to ensure the lifetime of the road infrastructure (Lancaster, 2016). Developing various bitumen modifiers has been the subject of numerous published studies. Numerous researches have been dedicated to modifying plain bitumen with nanomaterials to produce better performing and longer- lasting pavement roads (Martinho & Farinha, 2019; Filho, et al., 2020; Saboo, et al., 2021). Furthermore, the improvement of energy-efficient and ecologically friendly paving technology looks to be of particular importance (Xiong, et al., 2019). Many experiments have been conducted over the years to produce cleaner asphaltic mixes by lowering the temperatures of manufacture, mixing, and compaction. Many aims may be recognized under this purpose, such as enhanced ecological sustainability, decreased energy usage, and improved workability (Chen, et al., 2019). The current study's primary premise is to investigate the modification of warm mix asphalt utilizing Nano-Silica to generate asphalt with excellent performance and durability.

### *2.1.1 The Chemical Ccomposition of Bitumen*

Bitumen is a frequently utilized product for road construction all over the world. Bitumen is typically created by refining crude oil, as well as its ultimate characteristics are determined by the crude oil source and refining procedures (Crucho, et al., 2019). Bitumen is composed primarily of hydrocarbons and their compounds, which are completely soluble in toluene, is non-volatile, as well as softens slowly when heated (McNally, 2011). Asphalt molecules are primarily made up of carbon and hydrogen, but they also comprise one or more heteroatoms (oxygen, sulphur, and nitrogen) as well as small quantities of metals, primarily nickel and vanadium. The percentages of these elements vary based on the source of the bitumen, but usually, asphalt comprises approximately 82-88% carbon by weight, up to 6% sulphur, and about 10% hydrogen (Lesueur, 2009). The majority of bitumen created from a range of crude oils has a high amount of carbon. Any particular bitumen binder's polar fractions are usually divided into two main categories: asphaltenes and maltenes, which are two chemical families. The maltenes are split into two categories: resins and oils (Robert, 2000). Figure 2 depicts the major chemical constituent groups of bitumen.

**Figure 2**

*Groups of Bitumen Chemical Components*




(Robert, 2000)

SARA (Saturates, Aromatics, Resins, and Asphaltenes) is a common abbreviation for the separated fractions. Asphaltenes have the greatest polarity, while saturates have the least; aromatics and resins' polarity is somewhere in the middle (Sultana & Bhasin, 2014). Asphaltenes are extremely polar, with a higher molecular weight than maltenes, and have a substantial impact on the bitumen's overall characteristics. Higher asphaltene concentration bitumen materials have greater softening points and viscosity values with lower penetration values (Robert, 2000). Furthermore, asphaltene has a specific influence on the rheological performance of bitumen (Mozaffari, et al., 2017). Asphaltenes are solid black powdered particle that makes up around 8-20% of the weight of bitumen, which is the soluble component of bitumen. Another component of asphalt is resins, which seem to be semi-solid portions with dark brown color that operate as a dispersion agent, preventing the asphaltene particles from coagulating. Moreover, resins give adhesive characteristics and ductility for bitumen due to their polar nature (Yarranton, et al., 2007).

The oil part of the bitumen is made up of both saturates and aromatics. The dispersion environment for asphaltenes is dominated by aromatics, which are composed of the smallest molecular weight aromatic molecules in bitumen. They are made up of non-polar carbon chains with a great capacity to dissolve other significant molecular weight hydrocarbons (Whiteoak, 1990). Saturates are viscous non-polar oils with a white color. Saturates generally make about 0% – 15% of the total weight of the bitumen fractions. Bitumen is known to have fluid attributes due to saturate.

Generally, bitumen is a hydrocarbon petroleum substance composed of three major elements (Saha & Biligiri, 2019; Zhao, et al., 2021):

1) Asphaltene, a solid component of bitumen that aids to its stability
2) Resins, the adhesion element in bitumen
3) Oils, the thermal sensitivity element in bitumen

### *2.1.2 Asphalt Pavement Distresses*

Pavement distresses can arise as a result of the single or combined impacts of asphalt material quality, quality of construction, traffic loads, and moisture condition (Tamrakar, 2019). Fatigue cracking, rutting, and thermal cracking are common distresses in the pavement, and they are caused by low-quality materials and inadequate asphalt pavement maintenance (Martinho & Farinha, 2019). FHWA (2009) classified distresses in asphalt pavement into four groups: mat problems, deterioration, surface deformation, and surface cracking, as shown in Table 1.

**Table 1**

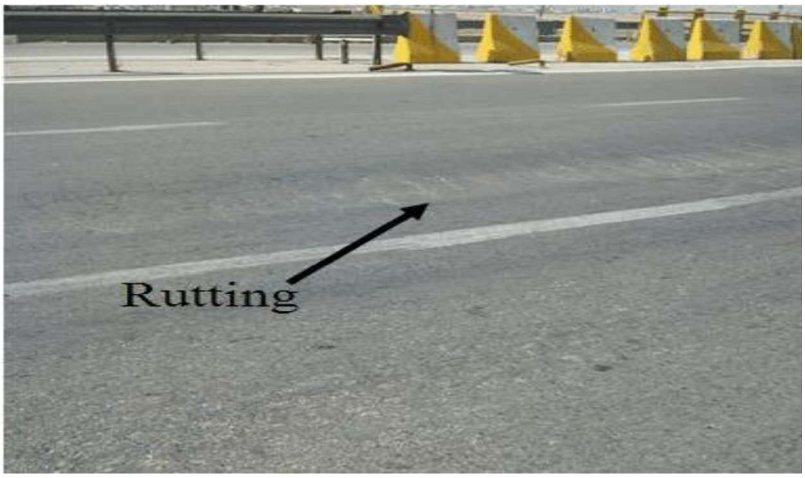
*Distresses of Asphalt Pavement*

| Major classifications | Varieties |
|---|---|
| Mat problems | Delamination, segregation and bleeding |
| Surface deformation | Shoving, depression, rutting and swell |
| Deterioration | Stripping, patching, polishing, pumping and pot holes |
| Surface cracking | Fatigue, longitudinal, block, reflection cracks and transverse |

Surface deformation refers to the deterioration of structural layers as a result of excessive traffic. Moreover, the weakness of underlying layers might be a minor consideration (Tamrakar, 2019). Rutting has been found as a key source of distress in pavements as well as is one of the primary design characteristics taken into account while constructing flexible pavements (Bhat & Mir, 2019). Rutting is a term used to describe the accumulation of permanent deformation in the pavement top layer over time, as seen by the engraving of the wheel path in the paved road. Figure 3 shows rutting distress on asphalt pavement. Deficiency of compaction, inadequate pavement thickness, and poor asphalt blends can all cause rutting. When it comes to preventing permanent deformation (rutting), the binder's characteristics have a significant role. The total rutting resistance of asphalt pavement mixtures is influenced by the asphalt binder's inherent capacity to resist rutting. The total rutting resistance of the bituminous mix at high temperatures is influenced by the bitumen binder, which acts as a bonding material for aggregate particles (Liu, et al., 2021). As a result, before using bitumen in the fields, it is critical to do an adequate evaluation of its ability to resist rutting. The requirements on road asphalt material layers have grown as a result of rising traffic volumes, necessitating the need to improve the performance of existing asphalt pavements. In addition, progress has been achieved in the use of technology and the development of innovative materials, and advances in our understanding of asphalt characteristics and performance have made it simpler to evaluate the benefits of utilizing new modifiers in the development of hot asphalt mixes (Zghair, et al., 2019). Severe loads, temperature fluctuations, and stresses are impossible for bitumen to withstand on its own. As a result, using modifiers and additives to increase the binder quality is critical. Furthermore, to increase the quality of the bitumen binder, the appropriate bitumen binder parameter that limits rutting problems in flexible pavement must be identified.

**Figure 3**

*Rutting Distress*




(Khabiri & Saberian, 2013)

Surface cracking is another form of distress, in which surface cracks are developed regardless of their placement. Most the surface cracking varieties are induced by climatic factors excluding the alligator (fatigue) cracking, which is generated by load (Ragnoli, et al., 2018). Fatigue is one of the primary distresses in pavements, and it occurs when the pavement is subjected to repetitive strains and stresses as a result of traffic loads (Al-Qadi, et al., 2005). Under repetitive traffic load, fatigue cracks, also known as alligator cracks, are a sequence of interconnecting irregular polygonal fractures. Once the drainage quality is inadequate, alligator (fatigue) cracks can occur owing to faults in the base layer, and they can appear anywhere on the asphalt pavement (Tamrakar, 2019). Fatigue cracking indicates that the pavement is nearing the end of its lifetime, which is far below the required standard (Ragnoli, et al., 2018). The qualities of the aggregates, the bitumen, and the proportional amounts of these two constituents in the mix determine a mixture's capacity to resist fatigue cracking. Aggregate particle characteristics such as shape, aggregate gradation, and angularity determine the stress state encountered by the bitumen within the mix, which affects the mixture's ultimate fatigue cracking resistance. The bitumen, on the other hand, is responsible for holding the particles of aggregate together and, as a result, the mix's fatigue cracking resistance is determined by bitumen’s intrinsic capacity to resist stresses before actual failure. (Hajj & Bhasin, 2018). Safaei et al. (2014) reported that fatigue cracking starts in the bitumen phase and spreads throughout the entire mix. Figure 4 illustrates fatigue cracking distress.

**Figure 4**

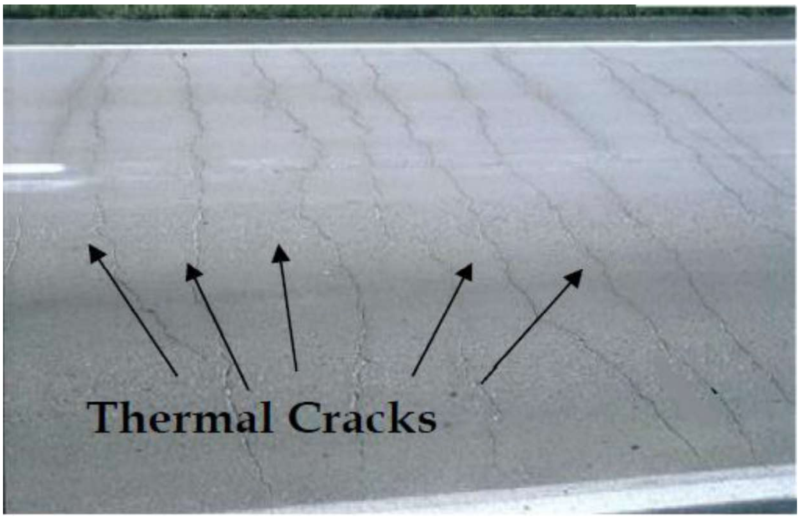
*Fatigue Cracking Distress*

(Aziz, et al., 2016)

Thermal cracking, which is not load-related, is another type of surface cracking distress. Thermal cracking develops once the thermal stress that accumulates in the pavement layer during cooling periods surpasses the tensile strength of the bitumen, which is typical in cold areas. Therefore, water infiltrates bottom pavement layers through cracks in the pavement, thereby weakening it and resulting in a harsher ride and significantly shorter lifetime (Rahbar-Rastegar, et al., 2018). Figure 5 shows a common pattern of thermal cracking on pavements surface.

**Figure 5**

*Thermal Cracking Distress*




(Behnia, et al., 2018)

The thermal shrinkage discrepancy between aggregate particles and the surrounding bitumen is another cause of thermal stresses in the pavement. As the temperature of the pavement falls, bitumen contracts further than aggregates, creating increased thermally stresses in the asphalt pavement. Furthermore, as the temperature drops, the bitumen becomes extremely brittle and more susceptible to fracture. As a consequence, micro-cracks form in the bitumen when thermally generated tensile stresses exceed the bitumen's fracture resistance (Behnia, et al., 2018). As previously said, there are various significant factors in the development of low-temperature (thermal) cracking, but the bitumen acts as the most crucial function (Liu & Wang, 2021).

## 2.2 Rheological Properties of Bitumen

The analysis of material deformation and flow is referred to as rheology (Taylor & Airey, 2015). Asphalt binder is a viscoelastic, thermoplastic material that acts as elastic-solid under short loading durations at low-temperatures while under long loading times at high -temperatures like a viscous liquid. Rheology may be defined as the study of substances that have solid and liquid properties. HMA pavement output is influenced by the deformation and flow of the asphalt binder in the mix. Rutting can occur when HMA pavements deform and flow excessively, while fatigue cracking can occur when they are stiff. Since the rheological features of asphalt binder change with temperature, rheological properties require two steps: (1) to completely characterize a binder, its rheological properties must be studied over a broad temperature range; (2) the rheological properties of various asphalt binders must be assessed at a standard reference temperature to compare them. It is more consistent to identify the viscous and elastic elements of bitumen as opposed to analysing the chemical and physical features to understand the rheological attributes that affect the overall performance features of the flexible pavement roads (Crucho, et al., 2019). The rheological characteristics of bitumen are generally measured using a dynamic shear rheometer, which is an oscillation form testing instrument (DSR). The viscoelastic behavior of materials is measured using the DSR when they are exposed to a certain load phase and temperature. The load may be applied in two modes: oscillatory, creep and recovery.

Several separate tests were performed under a variety of frequencies and temperatures to determine the rheological properties of asphalt using the DSR instrument. Multiple stress creep recovery test, and Superpave rutting resistance parameter. These assessments are capable of measuring the rutting resistance specifications of bitumen at high temperatures by determining the various specifications in unaged and short-term states. Furthermore, by evaluating various parameters, the frequency sweep test can evaluate the rutting resistance of bitumen at elevated temperatures over a range of frequencies. On the contrary, Superpave fatigue resistance specifications are capable of determining the fatigue resistance for bitumen at moderate temperatures by determining various parameters in long-term conditions. At very low temperatures, another instrument such as a bending beam rheometer (BBR) is intended to determine the rheological characteristics of bitumen. These tests are discussed in depth in the sections that follow.

### *2.2.1 Rutting Bitumen Testing*

In-wheel pathways, rutting generally shows itself as a surface depression. Horizontal loading caused by vehicle tire-pavement contact friction during repeated vehicle braking and acceleration causes substantial shear stress and strain in asphalt pavement, particularly at bus stops and intersections on urban roadways (Wang & Al-Qadi, 2010). Traffic and weather circumstances, including slow-speed traffic, heavy loads, and elevated temperatures, have a significant impact on rutting performance (Morea, et al., 2011). Furthermore, the inadequate material (Zhang, et al., 2017) and construction (Zhang, et al., 2018) quality of the mixture are important contributors to rutting buildup. There are plenty of pavement issues that are linked to rutting distress. Once water accumulates in grooves, for instance, the water penetration into the pavement layers might damage the pavement further (Tian, et al., 2017). Sybilski et al. (2013) revealed that bitumen characteristics contributed around 40% to the rutting performance of the bituminous pavement. Moreover, several researchers showed that the qualities of bitumen binder have a significant impact on pavement performance (Liu, et al., 2019; Mamvura, et al., 2020). Rutting distresses start in the bitumen matrix's binder phase and spread from there. As a result, the bitumen binder's temperature dependability has a considerable impact on the stiffness of the asphalt pavement and hence performance (Sukhija, et al., 2021). Dynamic mechanical evaluation with a dynamic shear rheometer (DSR) may be used to assess the rheological characteristics of bitumen binder, which are predictive of its performance (Elkholy, et al., 2018). Bitumen binder test that may be linked to rutting resistance include conventional linear viscoelastic evaluation utilizing DSR by analyzing the Superpave rutting parameter (*G*/* sin *δ*). Multiple stress creep recovery (MSCR) test is now widely used to investigate bitumen binder rutting resistance (Zhou, et al., 2018; Bhat & Mir, 2019; Sukhija, et al., 2021).

**A. Superpave Rutting Resistance Parameter.** Early in 1987, a new bitumen binder standard named Superior Performing Asphalt Pavements was launched as part of the Strategic Highway Research Program (Superpave). The dynamic shear rheometer test may be used to measure the two parameters, complex shear modulus (*G**) and phase angle (*δ*). At moderate to elevated temperatures, the DSR is utilized to assess the viscous and elastic characteristics of the bitumen binder. *G** represents the specimen's overall resistance to deformation when frequently sheared, whereas *δ*, represents the time lag between both the applied shear stress and the generated shear strain. The temperature where the *G**/sin *δ* at 10 rad/sec equals 1.0 kPa for unaged (fresh) bitumen binders (AASHTO T315, 2012) or 2.2 kPa for short-aged asphalt binders (AASHTO T315, 2012), known as the high- temperature PG of bitumen binder (AASHTO M320, 2010), are utilized to differentiate rutting possibility of bitumen binder. The Superpave rutting resistance parameter method presumes that rutting occurs more frequently in softer, greater viscous bitumen (lower *G**, higher *δ*) than stiffer, greater elastic bitumen (higher *G**, lower *δ*) (Karki & Zhou, 2018). Bitumen must be stiff (not excessively deformed) and elastic (able to recover to its original form after load distortion) to prevent rutting.

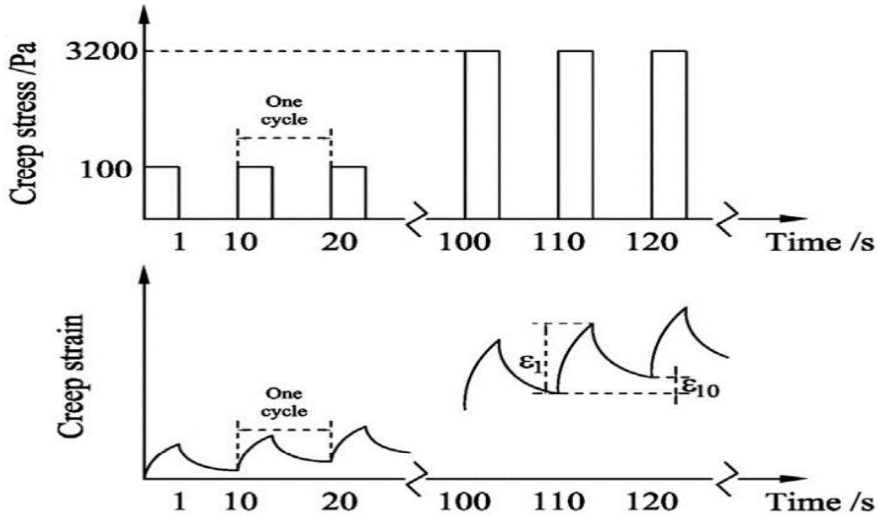
*G**/sin *δ* is also measured by testing binders in the linear viscoelastic domain at a constant temperature and frequency,10 rad/s is the frequency at which it is measured (Subhy, 2017). The measured property is associated with the viscous constituent of the bitumen binder, which is determined by the sine of the phase lag but also considers the binder stiffness by incorporating the complex shear modulus (Southern, 2015). A minimum value of the complex shear modulus is identified when permanent deformation is a major problem of HMA pavement. Therefore, the tendency to persistent deformation is lessened as this parameter's value rises (Southerm, 2015). The rutting potential of bitumen binder was measured using the *G**/sin *δ* parameter (Laukkanen, et al., 2015; Saltan, et al., 2017; Shi, et al., 2018; Shafabakhsh, et al., 2019; Bhat & Mir, 2021).

The $G^*/\sin \delta$ parameter has proven to relate well with the inclination of permanent deformation in unmodified bitumen. However, this parameter is less effective in binders containing large quantities of polymers and various modified binders. Moreover, $G^*/\sin \delta$ does not always accomplish this objective, and can be seen in the condition of the polymer-modified bitumen binders (Dongre & D'Angelo, 2006). There have been numerous forms of criticism regarding the Superpave parameter ($G^*/\sin \delta$) due to the lack of association with asphalt mixture or pavement functioning (Amini, et al., 2018). Researchers have addressed the deficiency of $G^*/\sin \delta$ by using parameters measured through the unrecoverable strain and percent recovery parameters that are determined by the multiple stress creep recovery (MSCR) test for bitumen binders (AASHTO T350, 2014), which have shown good associations with the asphalt mixture rutting ability (Zhou, et al., 2014; Zhang, et al., 2015). Zhou et al. (2014) discovered that the MSCR test parameters distinguish the rutting ability of bitumen better than the existing PG test parameter ($G^*/\sin \delta$), particularly for the highly modified bitumen binders.

**B. Multiple Stress Creep Recovery.** Through field observations, it has been proven that rutting is one of the main failure forms of asphalt pavements. Rutting can be found in the form of longitudinal depression down the wheel paths due to recurring traffic loads (Liu, et al., 2020). One key strategy in enhancing the rheological features of asphalt binders is the rutting mitigation approach (Domingos & Faxina, 2016). The MSCR test incorporates the application of repetitive creep and recovery of shear stress under short durations of one second and then removing the stress for nine seconds to enable recovery of the material. The process is repeated for ten cycles through the application of various stress levels. Figure 6 shows the creep stress and strain during the test.

**Figure 6**

*The MSCR Test Loading and Unloading Cycle*




(Wen, et al., 2017)

The test is normally implemented on RTFO aged samples to imitate the ageing throughout mixing and construction. The test is performed on samples that are placed between two parallel plates that have a diameter of 25 mm by applying the dynamic shear rheometer (DSR) equipment, which is described in detail in the ASTM D7405 or AASHTO TP 70-12 standards. The MSCR test stress levels are 100 Pa and 3200 Pa, which represent both high and low-speed traffic conditions. According to the test, a new rutting specification identified as the non-recoverable creep compliance was acquired by dividing the residual strain at the end of the recovery portion with the applied stress at the creep portion. The evaluations proved that the non-recoverable creep compliance has a much better correspondence with the rutting on loading sections as opposed to the Superpave rutting specification (D'Angelo, et al., 2007). The non-recoverable creep compliance obtained from the MSCR test is the most promising assessment to characterize the rutting ability of asphalt binders. Numerous studies have shown that the non-recoverable creep compliance correlates better to rutting when analyzed with $G^*/\sin\delta$, where the rutting performance of unmodified and modified asphalt binders are well predicted (Laukkanen, et al., 2015). Essentially, the MSCR test employs a number of creep and recovery cycles at assorted stress levels. The concept of this application is that the viscoelastic strain induced within the creep portion is recovered after the shear stress is removed. This allows the permanent strain to be separated from the total strain, which is better associated with the field rutting

(Liu, et al., 2020). Based on the AASHTO TP 70 and ASTM D7405 standards, the non-recoverable creep compliance was applied to measure the resistance of asphalt binders against permanent deformation under constant load. Moreover, a novel parameter known as the percent recovery was used to detect the presence of elastic response of the tested asphalt binders. As per the test protocol represented in AASHTO T 350–14 and ASTM D7405–15, two major specifications acquired from the MSCR test are the non-recoverable creep compliance, $J_{nr}$, and the percent recovery, *R%*. To determine the value of the parameters ($J_{nr}$ and *R)* Eqs. 1-2 are applied whereas the outcome of the testing is computed using the correlation shown in Figure 7.

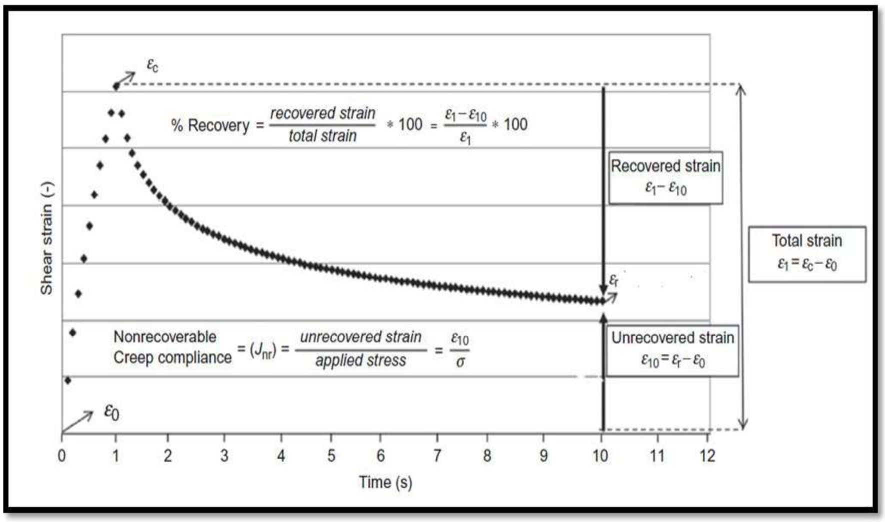
$$J_{nr}(\sigma, N) = \frac{\varepsilon_r - \varepsilon_0}{\sigma} = \frac{\varepsilon_{10}}{\sigma} \quad (1)$$

$$R(\sigma, N) = \frac{\varepsilon_c - \varepsilon_r}{\varepsilon_c - \varepsilon_0} \times 100 = \frac{\varepsilon_1 - \varepsilon_{10}}{\varepsilon_1} \times 100 \quad (2)$$

Where; $J_{nr}$ (σ, N) is the non-recoverable creep compliance at applied stress with N denotes the number of recovery and creep cycles, $\varepsilon_r$ denotes as the strain value at the end of the recovery stage, $\varepsilon_0$ is the strain value at the beginning of the creep stage, $\varepsilon_{10}$ is the corrected strain value at the end of the recovery period for every cycle, which is after 10 seconds, R (σ, N) is the % recovery at applied stress with N denotes the number of recovery and creep cycles, $\varepsilon_c$ denotes as the strain value at the end of the creep stage, $\varepsilon_1$ denotes the strain rate at the end of the creep stage.

**Figure 7**

*A Typical Creep –Recovery Cycle*




(Southern, 2015)

Once the MSCR test was incorporated into the AASHTO and ASTM standards, the next step required the establishment of a parameter criterion for the non-recoverable creep compliance. D'Angelo (2010) developed a correlation between the Superpave binder criterion and the non-recoverable creep compliance. The non-recoverable creep compliance ($J_{nr}$) has the potential to predict the improvement conveyed through modification and is appropriate for the specification of both neat and modified bitumen. The binder grade at certain climatic temperatures may also be categorized according to traffic designation and loading rate based on the AASHTO MP19-10 principle, which is illustrated in Table 2.

**Table 2**

*Grade Category of $J_{nr}$ at 3200Pa Based on Traffic Loading*

| Grade category | Jnr at 3200Pa, (1/kPa) | Traffic loading level (ESALs) |
|---|---|---|
| E (Extremely loading) | 0.0 < 0.5 | > 30 million and speed of traffic<20Km/h |
| V (Very high loading) | 0.5 < 1.0 | > 30 million or speed of traffic<20Km/h |
| H (High loading) | 1.0 <2.0 | 10-30 million or speed of traffic 20-70Km/h |
| S (Standard loading) | 2.0 < 4.0 | <10 million and speed of traffic >70Km/h |

The $J_{nr}$ at different stress levels is proposed as an indicator of the sensitivity of permanent deformation and stress dependence for the bituminous binders (Southern, 2015). Presently, the multiple stress creep recovery (MSCR) test has been extensively applied universally to analyze the influence of modification on the performance of asphalt binders at high temperatures as a result of its success in rutting characterization (Ling, et al., 2017; Ghanoon, et al., 2019; Bhat & Mir, 2021; Sukhija, et al., 2021). The indication of a good relationship with asphalt mixture rutting is the most crucial criterion for an asphalt binder high-temperature performance assessment. The test results proved that the non-recoverable creep compliance ($J_{nr}$) calculated at 3.2 kPa delivered the best relationship with field rutting in comparison to those measured at different stress levels (Liu, et al., 2020).

Furthermore, laboratory studies are more commonly applied to validate the correlation of the MSCR test with asphalt mixture rutting. Current studies performed for laboratory confirmation; (Laukkanen, et al., 2015; Zhang, et al., 2015; Behnood, et al., 2016), portrayed the ability of the MSCR test in precisely anticipating the binder's influence on asphalt mixture rutting. The tests used include the wheel tracking test, unconstrained dynamic creep test, flow time assessment, repetitive loading permanent deformation assessment and flow number test. By comparing the non-recoverable creep compliance against the other binder rutting parameters (e.g. $G^*/\sin\delta$, Shenoy's parameter, and ZSV), the MSCR test proved to be the best test technique available that quantifies the rutting sensitivity of asphalt binders (Radhakrishnan, et al., 2018). It can be established that both field and laboratory studies validated the good relationship of the MSCR test with asphalt mixture rutting. This fact specified a solid foundation for the application of the MSCR test as a specification assessment.

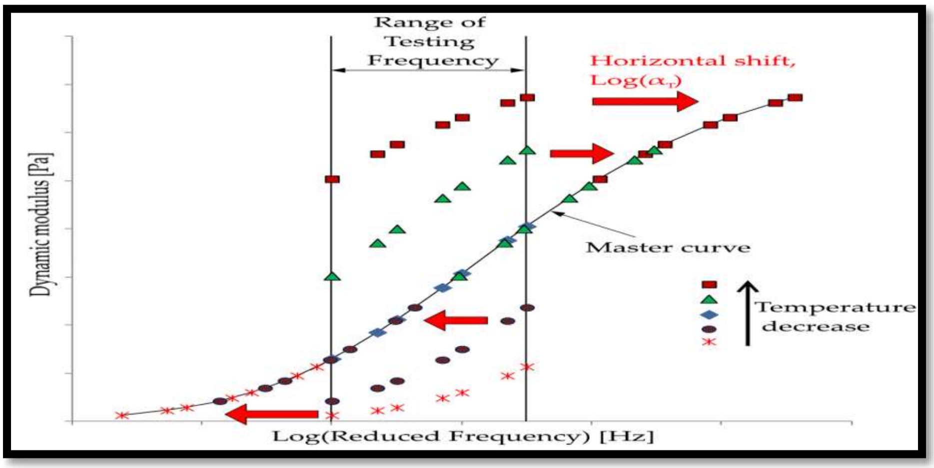
### *2.2.2 Frequency Sweep Test*

The asphalt pavement structure had a dynamic loading impact beneath traffic loads, as well as the bitumen substance had varied viscoelastic characteristics at various loads frequencies (Huang, et al., 2019). Asphalt binder is composed of a viscoelastic material where its rheological performance can be analyzed in oscillatory mode. Numerous researchers advocated the oscillatory mode of assessment for asphalt binders by using the dynamic shear rheometer DSR. Dynamic Shear Rheometer (DSR) can quantify the elastic and viscous characteristics of the asphalt binder (ASTM D7175). The behavior of the bitumen is identified through the complex shear modulus ($G^*$) and phase angle ($\delta$). The variable $G^*$ is the materials stiffness and is associated to the materials resistance against the permanent deformation (rutting) when recurring shear loading is employed. The time interval between the applied stress and the resulting strain is associated to the phase angle, $\delta$. Generally, for a perfectly elastic material, an applied load produces an immediate reaction; therefore, the time lag is zero. On the contrary, a viscous material has a comparatively large time lag between the applied stress and reaction; in which the phase angle moves towards 90 ˚ (Asphalt Institute, 2003). Phase angles of zero and $90^{\circ}$ indicate pure elastic and viscous material respectively. At extremely low temperatures, asphalt has total elasticity, but at relatively high temperatures, it becomes completely viscous. Therefore, high values of $G^*$ and low values of δ are desirable for rut resistance (Abedali, 2017). Throughout the years, researchers have effectively applied DSR to distinguish asphalt binders (Nazari, et al., 2018; Shafabakhsh, et al., 2019; Sukhija, et al., 2021). A frequency sweep test is an assessment performed under the linear viscoelastic (LVE) parameter at various temperatures that can be used to determine several rheological characteristics of asphalt binders. Linear behavior is accomplished at low temperatures and short loading durations (high frequencies), where the material acts as an elastic solid. The linearity is also fulfilled at high temperatures and long loading durations (low frequencies), where the materials completely act as a Newtonian fluid. Non-linearity is only prominent when temperatures are within a moderate range and loading times (field circumstances). Therefore, it is critical when testing in these conditions to guarantee that the bitumen binders will remain within the LVE range by constraining the applied strain (Lamperti, 2011). The frequency sweep test is utilized to determine the viscoelastic bitumen binder functions by evaluating the binders' rheological characteristics in a strain-controlled oscillatory loading regime (Nazari, et al., 2018). A frequency sweep test is carried out by applying a DSR on the asphalt binders. In the first stage of the test, a strain sweep test is performed to determine the LVE strain constraint for the asphalt binders at various frequencies and temperatures. The magnitude of strain in the frequency sweep test is selected to guarantee that the asphalt binders are within the LVE domain (Saboo, et al., 2019). The gap and the diameter were 2 mm and 8 mm when the testing temperature was under 40 $^{\circ}$C. However, when the temperatures were above 40 $^{\circ}$C, the values changed to 1 mm and 25 mm (Nazari, et al., 2018; Sukhija, et al., 2021).

The time-temperature superposition principle (TTSP) is primarily applied to signify the rheological characteristics of bituminous materials over an extensive range of frequencies. There have been several studies performed investigating the viscoelastic properties of binders, which have proved the existence of an interrelationship among the temperature and loading time (Subhy, 2017). For example, rheological characteristics found at high temperature and frequency are found at low temperature and frequency. This consistent correlation between various conditions can be specified by a shift factor (Zhang, et al., 2018). The viscoelastic behavior of binders at a specific temperature over an identified range of loading durations can be equivalent to the behavior analyzed at various temperatures during the same loading time by multiplying the loading times by a shift factor. Hence, the viscoelastic measurements, i.e. complex modulus $G^*$ and phase angle $\delta$, when tested at various temperatures, can be transferred to a reference temperature to yield a continuous curve at a decreased frequency, which is referred to as the master curve. An example of such a concept that applies the time-temperature superposition principle is graphically illustrated in Figure 8.

**Figure 8**

*Time –Temperature Superposition Principle*

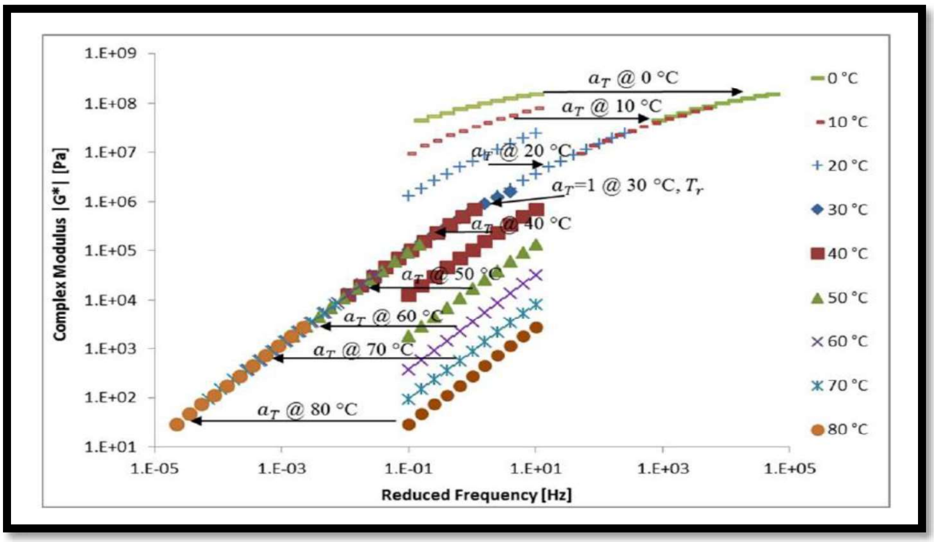



(Lee, 2013)

A master curve is formed at a selected reference temperature by horizontally shifting the other curves that are tested at various temperatures to correspond with the reference curve, which results in creating a single curve. Figure 9 explains the shifting method applied to combine the curves into a smooth and constant master curve.

**Figure 9**

*The Master Curve's Construction*

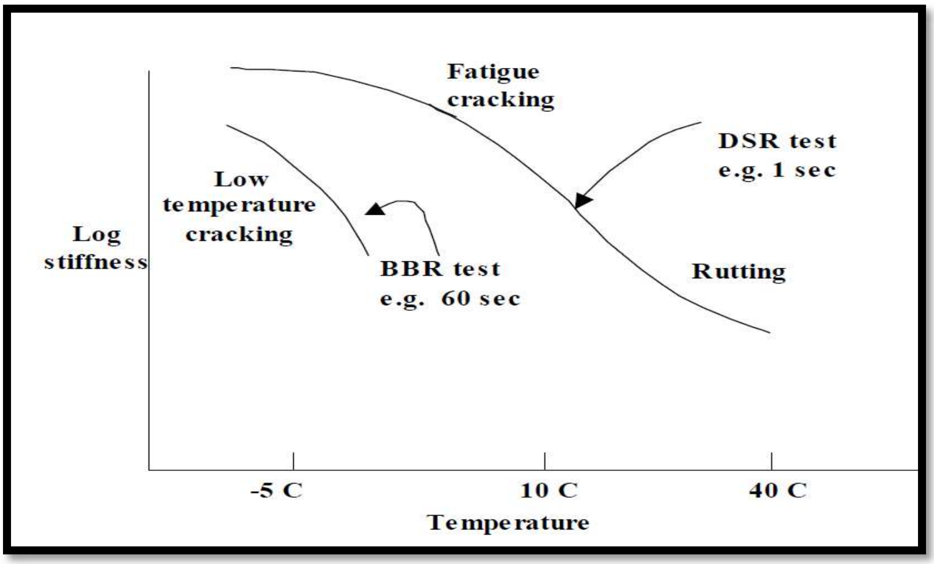



(Subhy, 2017)

The rheological statistics of bitumen are portrayed and evaluated by applying the isochronal plot. The isochronal plot is a basic plot of some viscoelastic functions, such as the complex modulus and phase angle, against the temperature at a constant frequency or loading duration (Alhamali, et al., 2016). As a result, viscoelastic statistics can be displayed over a range of temperatures at a certain frequency applying an isochronal plot. The simplest advantage of isochronal plots is the evaluation of complex modulus or phase angle at various temperatures. Furthermore, a number of bitumen characteristics, for instance, temperature susceptibility, can be inferred from this plot form. Temperature susceptibility, which is usually a major performing criterion for bitumen, can be identified as the modification of consistency or viscosity, as a function of temperature. Temperature susceptibility should be established on measurements at various temperatures but comparable loading durations since the rheological characteristics of bitumen are a function of both time and temperature. The general form of an isochronal plot at a continuous loading time is illustrated in Figure 10.

**Figure 10**

*Schematic Presentation for an Isochronal Plot*




(Rahimzadeh, 2002)

### *2.2.3 Superpave Fatigue Cracking Resistance Parameter*

For many years, characterizing the fatigue resistance of asphalt binders and enhancing this characteristic through modification has been a focus of intense research. One of the major load-related distresses of asphalt pavements is known as fatigue cracking. Fatigue cracking is linked to increasing tensile strains, formation of micro-cracks, as well as their expansion at a moderate-temperature range (Nejad, et al., 2017; Nazari, et al., 2018). It is highly reliant on the rheology of bitumen, form, and gradation of aggregates and filler constituents, in addition to the loading and environmental circumstances (Nejad, et al., 2017; Hajj & Bhasin, 2018). Fatigue of the asphalt mixtures is considered an essential failure approach in the future life of the pavement. Fatigue is the word specified to describe the decrease of stiffness in asphalt mixtures resulting from repetitive traffic loads (Southern, 2015). Moreover, the fatigue factor, ($G^*$·sin $\delta$), was established by The Strategic Highway Research Program (SHRP) during the 1990. The factor proved to be an effective indicator for pavement fatigue performance and is generally applied to distinguish the asphalt binder grade in SHRP qualification (Hajj, et al., 2012). PG binder qualification applies the $G^*$. sin $\delta$ parameter to distinguish the fatigue resistance of asphalt binders. Fatigue cracking is stress or strain-controlled state. Loads exerted on the pavement surface are a result of the repetitive loading of traffic cycles, in which most of these forces recover from rebounding, whereas the rest leads to irreversible cracking deformation. Smaller modulus ($G^*$) allows the binders to dissipate work energy more effectively without producing significant stresses, while lower phase angle ($\delta$) allows the binders to recover their original form with the least amount of energy wasted (Subhy, 2017). During Superpave, fatigue specification is tested on the pressure aging vessel (PAV)-aged binder at moderate temperatures. The DSR is used in the oscillatory shear condition with an 8 mm diameter for the parallel plates and a 2 mm gap to determine the complex shear modulus ($G^*$) and the phase angle ($\delta$) concerning the Superpave principles, which has indicated a maximum value of 5000 kPa for the fatigue cracking resistance parameter. The energy wasted in any particular cycle once bitumen binders are exposed to cyclic loading in a sinusoidal pattern is defined by Eq. 3.

$$W_c = \pi G^* \epsilon_0^2 \ Sin\ \delta \tag{3}$$

Where; $W_c$ is the amount of work that is dissipated each load cycle, $\epsilon_0$ is the strain amplitudes.

As a result, $G^*$.sin $\delta$ may be utilized to evaluate wasted energy for various materials exposed to sinusoidal loading for a particular strain amplitude. In the situation of bitumen exposed to cyclic loading, a bitumen with a larger wasted strain energy each cycle (a greater value of $G^*$. sin $\delta$) suffers more cumulative damage each cycle (fatigue) than a bitumen with a lower cumulative damage (Hajj & Bhasin, 2018). Researchers have questioned the correspondence between the $G^*.\sin\delta$ parameter and the fatigue characteristics of asphalt binders. Conversely, various researches have advocated that the existing SHRP fatigue parameter does not essentially signify the true binder influence associated with the mixture or pavement function (Zhou, et al., 2013). The causes leading to poor binder-mixture interrelation for the SHRP fatigue parameter are primarily credited to the fact that determining $G^*$ and $\delta$ under comparatively small strain in the linear viscoelastic section does not signify the actual variability of strains or stresses that take place within the binder films of pavements. This provides inadequate information regarding the behavior of binder films under different environmental and loading circumstances (Subhy, 2017). In general, fatigue cracking is correlated to the aged binder, and as a result, the testing for this property is normally performed on binder or mixtures that have been exposed to an aging or conditioning procedure. There are not many standardized test approaches are available to determine the fatigue characteristics of bituminous binders, and the approaches available are not globally agreed upon to measure this characteristic correctly. Regardless, the parameter for Superpave fatigue resistance is applied in specifications nowadays (Southern, 2015). There have been assorted researches carried out to study the fatigue performance behavior of bituminous constituents by applying the Superpave fatigue parameter using the DSR (Zhou, et al., 2013; Saltan, et al., 2017; Nejad, et al., 2017).

### *2.2.4 Bending Beam Rheometer*

When the temperature decreases, asphaltic binders become stiffer. Since that would result in increased asphalt mixture stiffness. One major distress form encountered by asphalt pavements created in cold areas is thermal cracking. Due to the decreased temperature, the asphalt mixture layer is compressed in cold weather conditions. However, the abrasion between the lower layers and the asphalt hinders the displacement of the asphalt layer, which leads to tensile stresses within the asphalt. While the temperature falls, the values of these stresses rise, and when the stresses extend to the tensile strength of the asphalt mixture, the main cracks are produced at the bottom layer of the asphalt mixture (Kakar, et al., 2020; Wang, et al., 2021). This form of cracking is generally referred to as “thermal crack” or transverse crack due to the direction of cracking concerning the traffic direction (Lamperti, 2011).

Furthermore, there are various efficient factors resulting from the occurrence of low temperature cracking, but asphalt binder is the most vital function (Liu & Wang, 2021). The probability of low-temperature cracks increases with higher creep stiffness of bitumen and its decreased viscous response, which is the rate at which the stiffness of bitumen shifts over time (Nejad, et al., 2017). For the resistance of thermal cracking, the following two factors are vital; the stiffness or resistance to deformation and the potentiality to reduce stresses. These parameters can be determined at decreased temperatures by applying the BBR and providing an indication regarding the asphalt binder's capability to withstand low-temperature cracking. Since low-temperature cracking is an occurrence that is mostly found in older pavements, the test is performed on the long-term aged (PAV). The BBR has three main elements listed below and are illustrated in Figure 11.

1) Testing frame unit,
2) Temperature-controlled bath, and
3) Data acquisition system (computer).

Based on Figure 11, the elements of the testing frame consist of specimen supports, an air bearing and a loading cell. The load and displacement are measured using the linear variable differential transformer (LVDT), which has been connected to a data acquisition system. A rectangular bitumen sample is exploited as the test model in this examination. The dimensions of the sample are 127 mm long, 12.7 mm wide and 6.3 mm thick, and is illustrated in Figure 12.

**Figure 11**

*A Schematic Diagram of BBR*

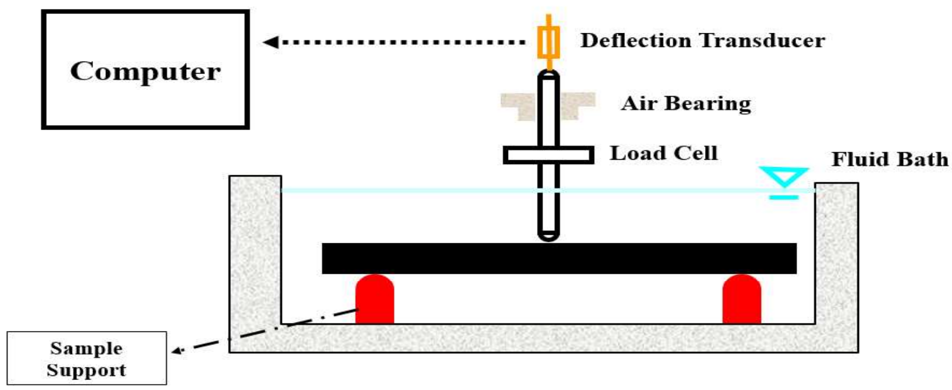
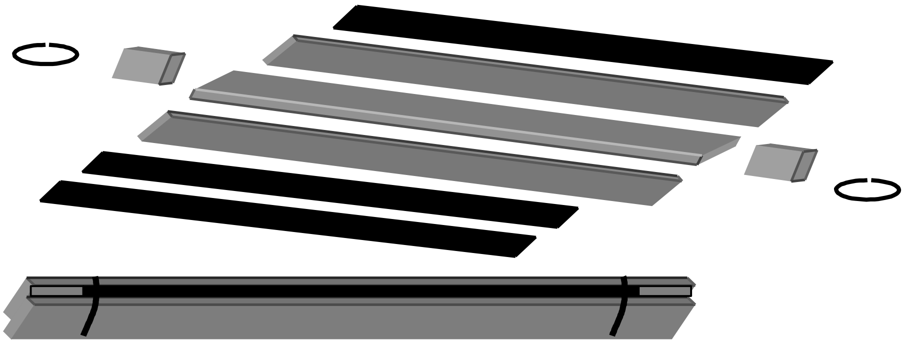


**Figure 12**

*Mold for BBR Specimens*



(Rahimzadeh, 2002)

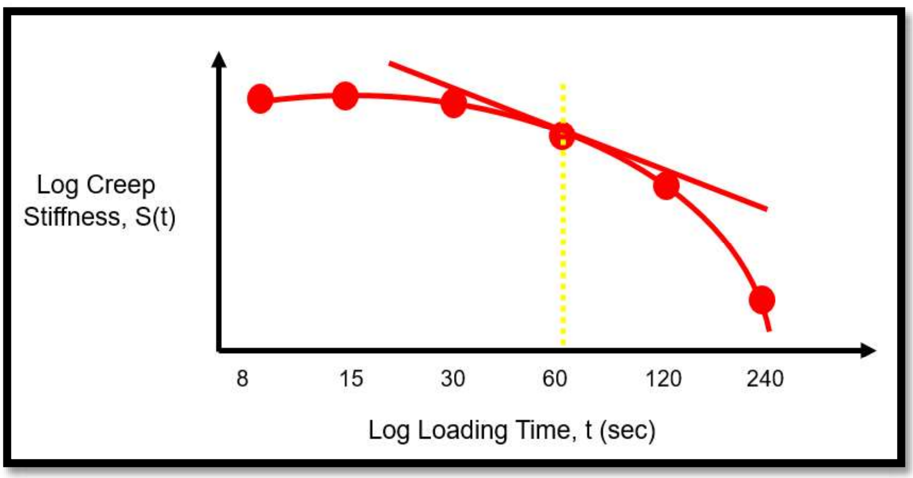
The time-reliant creep stiffness is computed using the following equation, Eq.4. The creep stiffness and m-value are the criteria used for the BBR test. The m-value is the tangent of the creep curve at 60 s load time, as presented in Figure 13. For thermal cracking, a maximum stiffness of 300 MPa and a minimum "m" value of 0.3 at a loading time of 60 s are selected for the Superpave specification criteria.

$$S\,(\mathrm{t}) = \frac{Pl^3}{4bh^3\delta(t)} \qquad (4)$$

Where; $S$ (t) is the creep stiffness at the time (t), P is the constant load that applied at the mid-span, L is the distance between the supports of a beam (102mm),b denotes the width of a beam (12.5mm),h is the thickness of the beam (6.25mm) and $\delta(t)$ denotes as the deflection in mm at the time (t).

**Figure 13**

*BBR Parameters at Low- Temperature*



The attempts resulting from studies conducted by the Strategic Highway Research Program (SHRP) have determined that cracking resistance of asphalt binders significantly affects the development of non-load- related cracking in asphalt pavements. The latest researches imply that the non-load-related cracking is linked to the oxidation of bitumen (Moraes & Bahia, 2018). To prolong the service life of pavements, adjustment was extensively applied to enhance the performance of bitumen binders. Nevertheless, the occurrence of aging in neat bitumen is already a complicated process, and its complication intensifies when adjustment is included (Zhang, et al., 2019). Rheological approaches including the Bending Beam Rheometer (BBR) test was commonly applied to assess the paving function and rheological characteristics of bitumen binders. Many researchers (Subhy, et al., 2018) have determined that when the aging duration increased, the creep stiffness (S) of bitumen binders increased while the alteration ratio of creep stiffness (m-value) decreased. In the matter of the paving performance of bitumen binders, low-temperature cracking resistance will decrease with aging.

## 2.3 Modifiers and Additives for Bitumen Binder /Mixture

Bitumen, being a viscoelastic substance, is crucial in influencing many characteristics of pavement performance. A bitumen mixture, for instance, must be flexible at low temperatures to avoid asphalt pavement cracking and stiff at elevated temperatures to avoid rutting. Moreover, because of rising performance-related demands on the road pavement, traditional bitumen (unmodified) does not behave as intended. As a result, modified bitumen has been embraced and widely utilized as a commercial product to enhance the bitumen qualities. The selection of an appropriate bitumen binder is one of the greatest critical factors that influence the quality of asphalt pavements. Expected traffic volume circumstances and asphalt pavement temperature, are taken into account while selecting the best bitumen for a certain area. Because elevated asphalt pavement temperatures and high traffic loads are frequent on many roadways, modified bitumen has been utilized in the pavement's top layer surfaces to enhance asphalt pavement performance (Abedali, 2017). When bitumen materials fail to meet the standards for constructing a high-performing bitumen structure, one of the most desirable ways for achieving the needed qualities of the utilized materials is to modify them (Enieb & Diab, 2017). Researchers have attempted to use many sorts of additives to improve the performance of bitumen (Crucho, et al., 2019). The pavements must be able to withstand a wide variety of environmental circumstances and traffic volume while in use. In many situations, traditional penetration grade bitumen no longer provides the appropriate performance over time, necessitating urgent conservation or repair. Various kinds of additives have been investigated throughout the years to adjust the characteristics of bituminous mix, with an emphasis on improving mechanical performance (Crucho, et al., 2019). Adhesion enhancers, rubber, warm mix asphalt (WMA) technology, fibers, and a range of polymers were the most often studied additives (Porto, et al., 2019). Numerous bitumen binder modification approaches have been suggested to address major distresses like fatigue cracking and rutting (Porto, et al., 2019; Ramadhansyah, et al., 2020). An optimal modification strategy maximizes the bitumen binder's fatigue and rutting resistance while reducing costs. Despite polymer modification of bitumen binder being widely used in many areas, its high price and poor storage stability render it challenging to utilize (Zhu, et al., 2014). Nanomaterials, among other types of bitumen modification approaches, offer a unique alternative for improving the rheological characteristics of the bitumen binder (Sukhija & Saboo, 2020). Following advances in nanotechnology, the research of nanomaterials has expanded, and their use as a bituminous mixture's additive has been investigated in the last two decades.

### *2.3.1 The Basics of Nano-Silica*

In recent years, nanotechnology has grown in popularity amongst those industrial sectors and researchers. The number of nanomaterial-containing products entering the market has also risen rapidly, and this growth is expected to continue in the years ahead. Nanotechnology has given rise to a slew of new products and uses. Along with numerous benefits in the manufacturing sector, the construction industry has seen tremendous development in the field of nanotechnology over the last few years. Nanomaterials may contribute to conventional construction materials with additional functionalities, such as improved toughness and mechanical properties, due to the dimensions regulated in the transition region among atoms and molecules (Lee, et al., 2009). Nanoparticles are typically classified as particles with a diameter of less than 100 nanometers, but there is no definite size cut-off (Napierska, et al., 2010; Martinho & Farinha, 2019). Silica is a generic term for products made up of silicon dioxide ($SiO_2$), which can be crystalline or amorphous. Crystalline silica comes in a variety of shapes and sizes (Napierska, et al., 2010). The industrial use of various forms of amorphous silica is currently increasing. Through the use of $SiO_2$ in a Nano dispersed shape, such as sols (colloidal silica), gels, and Nano powders, becomes more and more common in technology sectors (Potapov, et al., 2018). Colloidal silica, also known as silica sol, is used in this study. It is most commonly manufactured in a multi-step procedure whereby an alkaline silicate solution is partly counterbalanced with a mineral acid. Silica sols and Nano powders are useful materials with a wide range of applications. The cost of silica depends greatly on its properties and area of use in industry (Potapov, et al., 2018). However, the cost of Nano-modified bitumen is cheaper (by around 20–30%) when compared to polymer-modified bitumen (Hossain, et al., 2015). The inexpensive cost of manufacture and strong performance qualities of Nano -Silica is its benefits. Many features of Nano-Silica include outstanding stability, a huge specific surface area, excellent adsorption, and great dispersion ability (Yao, et al., 2013; Nazari, et al., 2018; Bhat & Mir, 2019). The construction sector is a viable approach for applying the obtained Nano-silica, and there are numerous applications of sols and Nano powder silica as Nano-modifiers for concrete to improve strength, water resistance, sulfate resistance, and other properties. Although several researchers have utilized nanomaterials in Portland cement, nanomaterial-modified bitumen is a comparatively recent development (Taherkhani & Afroozi, 2016). One of the nanomaterials that beneficial in improving the mechanical characteristics of bitumen binder is Nano-Silica (Zghair, et al., 2019).

**A. Effects of Nano-Silica on Bitumen Binder Conventional and Rheological Characteristics.** Many researchers have conducted a variety of experimental tests to determine the appropriateness of Nano-Silica for bitumen modification. The underlying literature in this section will be confined to penetration, softening point, and ductility as conventional testing, with the viscosity, DSR, and BBR tests carried out in this research for rheological features.

Taherkhani and Afroozi (2016) investigated the performance of bitumen modified with 1, 3, and 5% Nano-Silica in terms of penetration and softening point; the outcomes revealed that as Nano-Silica content rose, penetration reduced and softness point increased. A reduction in penetration indicates an increase in stiffness, while an improvement in softening point indicates a decrease in temperature sensitivity, which enhances resistance to permanent deformation at elevated temperatures, the same findings were observed previously in the study conducted by

(Zghair, et al., 2019). Ezzat et al. (2016) investigated the characteristics of bitumen binder treated with various percentages of Nano-Silica (3, 5, and 7%). The results indicated that Nano -Silica generated from silica fume had a lower penetration and a higher softening point. Prior studies indicate that the incorporation of Nano-Silica in bitumen binder, regardless of Nano-Silica dose, results in a drop-in ductility and penetration (Taherkhani & Afroozi, 2016; Zghair, et al., 2019).

Permanent deformation is one of the most common types of asphalt pavement deterioration. The primary cause of asphalt rutting has been recognized as "accumulated strain," which is caused by traffic loads (Hajikarimi, et al., 2015). However, bitumen characteristics are also essential, particularly for modified bitumen, which is mentioned to be utilized to increase bitumen rutting resistance. Several studies (Shafabakhsh, et al., 2019; Ghanoon & Tanzadeh, 2019; Bhat & Mir, 2021) recommend the inclusion of Nano-Silica in bitumen binder to improve rutting resistance. This enhancement, however, is dependent on the Nano-Silica dose and type. Increased Nano-Silica doses may increase rutting resistance but have a negative impact on low-temperature performance (Moeini, et al., 2020). Shafabakhsh et al. (2019) discovered that the inclusion of 3% Nano-Silica did not affect the elevated temperature performance of bitumen binder while the inclusion of 5% and 7% Nano-Silica improved the bitumen binder's elevated temperature performance by one and two grades, respectively. According to recent research, the $G^*/\sin \delta$ parameter cannot reliably estimate the rutting performance in the site (Amini, et al., 2018). As a result, the MSCR test is recommended to further anticipate the performance of elevated temperature bitumen binder (Ling, et al., 2017). Arshad et al. (2017) incorporated Nano-Silica at various concentrations (1-5%), and the results revealed that 2% Nano-Silica was the optimal dose with greater effect in terms of non-recoverable creep compliance ($J_{nr}$) and percentage recovery (*R%*).

At low temperatures, the performance might be a disadvantage of modifying bitumen with nanomaterials. Onochie et al. (2013) used a BBR to test the neat bitumen with 2% and 4% Nano-Silica and observed that the adjustments resulted in 6% and 14% greater creep stiffness and comparable to and 2% lower m-value, respectively. Nejad et al. (2017) reported the same findings, the incorporation of Nano-Silica into the bitumen caused higher creep stiffness and lowered the m-value, demonstrating the inefficiency of Nano-Silica on the low-temperature cracking resistance. Table 3 shows a more comprehensive review of the conventional and rheological properties of Nano-silica modified binder.

**Table 3**

*The Impact of Nano-Silica on the Physical and Rheological Properties of the Bitumen Binder*

| Inferences | The improvement in conventional/rheological properties | Reference |
|---|---|---|
| In summary, the Nano-Silica modified binder showed a reduction in penetration, an elevation in viscosity, and a rise in softening point. | Conventional | Alhamali, et al., 2015; Crucho, et al., 2018 |
| Utilizing the (DSR), the modified bitumen had a greater complex modulus ($G^*$) and a lower phase angle ($\delta$). | Rheological | Nejad, et al., 2017; Shi, et al.,2018 |
| Utilizing the (DSR), the modified bitumen exhibited better fatigue resistance. | Rheological | Baldi-Sevilla, et al., 2016; Nazari et al., 2018 |
| Utilizing the (DSR), the modified bitumen with Nano-Silica had a greater complex modulus and lower phase angle which increased $G^*.\sin\delta$, indicating poorer fatigue resistance. | Rheological | Nejad, et al., 2017; Moeini, et al., 2020 |
| In summary, Nano-Silica can improve rutting resistance regardless of the dose, type, or approach utilized to evaluate rutting resistance. | Rheological | Saltan, et al., 2017; Ghanoon & Tanzadeh, 2019; Bhat & Mir, 2021 |
| The addition of Nano-Silica raises the viscosity of the Nano-Silica modified bitumen. | Rheological | Zghair, et al., 2019; Sukhija, et al., 2021 |
| Increased in viscosity values are related to increased stiffness of Nano-Silica modified bitumen as well as increased Nano-Silica dispersion and adsorption in bitumen material | Rheological | Enieb & Diab, 2017 |

**B. Effects of Nano-Silica on Asphalt Mixtures Mechanical Performance.** To perform optimally, Pavements must have excellent durability and sustainability during the serviceability, the trait which is depending on pavement qualities including adhesion and cohesion between particles and the bitumen. Moisture in asphalt mixtures can induce a reduction of adhesion between aggregates and the bitumen, as well as a lack of bitumen cohesiveness, resulting in stripping and bleeding in pavement (Behiry, 2013). The type of bitumen utilized in asphalt mixtures, as well as the rheological qualities of bitumen, are two of the most influencing factors on the moisture sensitivity of bitumen mixtures (Mirabdolazimi, et al., 2021). In this research, the method for assessing asphalt moisture sensitivity in compacted mixes is used by applying the Lottman method. The Resilient Modulus ($M_R$) is another significant test for evaluating the mechanical performance of asphalt mixes. In mechanistic-empirical for the design of pavement, $M_R$ is an essential feature. $M_R$ of asphalt mixes is a measure of stiffness as well as resistance to repeated loads.

The researchers that performed research on Nano-Silica modified asphalt mixes observed significant increases in the mixture's mechanical performance. The mechanical tests most commonly used in this literature to assess the performance of asphalt mixtures are indirect tensile strength (ITS) and resilient modulus because both of these tests were performed in this research.

Hasaninia and Haddadi (2017) investigated an asphalt mixture that had been modified with (2-8%) Nano-Silica. The 60/70 binder served as the reference binder. The Marshall approach was used to estimate the OBC, and the findings revealed that the reduction in OBC was related to the rise in Nano-Silica dose. In terms of performance testing, the effects gained with the adjustments were susceptible to the Nano-Silica dose, with the 8% Nano-Silica adjustment having the greatest effects. In terms of the 8% adjustment, the modified mixture showed a 37% rise in resilient modulus.

Enieb and Diab (2017) revealed that adding Nano-Silica at varying concentrations (2, 4, and 6%) raised the resilient modulus and lowered the moisture susceptibility of the modified mixes, allowing for the construction of more durable pavements. Furthermore, Cai et al. (2018) prepared an asphalt mixture with 1% Nano-Silica. The following effects were observed by the researchers: an improvement in (ITS) by 9%; slight enhancement in (TSR) from 80% to 82 %. Moreover, Sezavar et al. (2019) evaluated the influence of the addition of Nano -Silica on moisture susceptibility. It was shown that the samples modified with Nano-Silica have a greater ITS, and compressive strength than the control mix, therefore a higher moisture susceptibility

Ezzat et al. (2020) investigated an asphalt mixture containing 7% Nano-Silica. The Nano-Silica modified mixture demonstrated an 11% improvement in (ITS) and an improvement in the (TSR) ratio from 88% to 93%. Ganesh and Prajwal (2020) observed that the asphalt mixes modified with Nano-Silica were stiffer than the control mix, as demonstrated by greater resilient modulus ($M_R$) values.

Shafabakhsh et al. (2021) showed that bitumen mixes modified with Nano-Silica had superior cracking resistance than control mixes, owing to improved adhesion between the aggregates and the bitumen modified with Nano-Silica. Mirabdolazimi et al. (2021) observed that the addition of Nano-silica to (60/70 and 80/100) bitumen enhanced the moisture sensitivity of two asphalt mixes. Moreover, Nano-Silica can be utilized to minimize the moisture sensitivity of asphalt mixes under various traffic loading situations. Furthermore, the ITS findings of all samples revealed that the modified bitumen with Nano-Silica greatly enhances the ITS values of the specimens in both dry and wet circumstances.

Because of the ability of Nano-Silica to enhance the mechanical characteristics of asphalt mixes, as demonstrated in the literature, this research aims to improve the qualities of both HMA and WMA mixtures.

### *2.3.2 Hot Mix Asphalt (HMA)*

Pavement construction with asphaltic concrete is quite common all over the world. Transportation infrastructures and road systems are important for a country's growth. The growth of the worldwide population and the need for economic prosperity had remarkably contributed to the construction of road networks, particularly paved roads and highways. Asphaltic concrete is used to pave more than 4.68, 3.8, 3.68, 0.41, and 0.17 million km of roads in Europe, Asia, the United States, Canada, and Mexico, respectively (Shi, et al., 2019). For road construction, HMA is the most extensively utilized infrastructural material. HMA is a multi-phase heterogeneous substance made up of a viscoelastic bitumen binder, irregular hard aggregate particles, and a small proportion of air gaps (Gopalakrishnan & Kim, 2011). A combination of bitumen binder and graded aggregate that is blended at a high temperature and compacted to generate a dense asphalt pavement surface layer with approximately 5% bitumen and 95% aggregate. Under a range of traffic and environmental circumstances, HMA pavement mixes are anticipated to operate for long periods (FHWA & NAPA, 2001). Stone Matrix Asphalt (SMA), Open-Graded Friction Courses (OGFC), and Dense - Graded are examples of HMA types. Dense-Graded mixtures are developed to withstand a wide range of distresses caused by applied forces (stresses). Rutting, thermal cracking, fatigue cracking, and moisture damage are examples of these problems. They are predicted to reduce service life, durability and raise the cost of maintenance of asphalt pavements (FHWA & NAPA, 2001). SMA is a Gap-Graded HMA with a solid stone-on-stone structure linked together with bitumen, filler, as well as stabilizing additives, like fibers and/or bitumen modifiers, optimizes rutting resistance and durability (Abd Al Kareem & Albayati, 2022). OGFC combinations are developed to be water permeable, as opposed to Dense -Graded and SMA mixes, which are generally impermeable. Either a crushed stone or, in some situations, crushed gravel with a relatively small quantity of synthetic sand is used in these combinations (Abd Al Kareem & Albayati, 2022). In wet conditions, OGFC combinations decrease tire spray and produce a smooth texture of asphalt pavements more than Dense-Graded HMA (Ghani, et al., 2020). When evaluating the rutting performance of bitumen pavements, the aggregate gradation is the most important factor that influences the entire behavior of the pavements (Ghani, et al., 2020; Majeed & Sarsam, 2021). Due to the combined influence of the environment and traffic volume, pavements degrade over time. If appropriate materials are not employed in the design and manufacture of HMA, the serviceability of road pavement might be cut in half. During the design life of a correctly constructed pavement, the distresses will not surpass the permissible limitations (Klinsky, et al., 2018). A mix design's purpose is to figure out what mixture of bitumen and aggregate can provide long-term performance. Mix design is a set of laboratory processes for determining the quantities of ingredients to be used in a bituminous mixture. These methods involve identifying an acceptable blend of aggregate to achieve adequate gradation of aggregates, as well as selecting the kind and proportion of bitumen to be utilized as a binder for that selected gradation. Well-designed bitumen mix can be anticipated to last for several years (MS-2 Asphalt Institute, 2014). The general goal of designing bitumen pavement mixes is to achieve a gradation of aggregate and bitumen binder content that results in a mixture that:

1) Adequate mix stability to meet traffic needs without causing deformation;
2) Enough bitumen to provide a long-lasting pavement;
3) An optimum void content to restrict the penetrability of harmful air and water into the mix;
4) Aggregate texture and toughness to provide adequate skid resistance in undesirable (bad) weather; and
5) Adequate workability to allow efficient mix placement without segregation.

The ultimate objective of mixture design is to find a specific design bitumen content that balances all of the required attributes. Stiffness, impermeability, workability, stability, durability, and fatigue resistance are all factors that influence asphalt performance. There is no one bitumen content that can optimize all of these qualities in this scenario. Alternatively, the bitumen percentage is chosen according to the attributes that are required for the given conditions (MS-2 Asphalt Institute, 2014).

The destruction of bitumen pavements by moisture is a big concern all around the world. Moisture intrusion into the pavement from various sources damages the bitumen by reducing the cohesive link inside the bitumen binder and deteriorating the bonding between both the bitumen and aggregates (Ahmad, et al., 2020). Assessing the permeability of HMA is the first step in discovering difficulties caused by the presence of moisture in the bitumen pavement (Abou-foul, et al., 2018). Moisture deterioration in asphalt pavement can be minimized by enhancing the bitumen pavement's stripping strength. Moisture can migrate through the permeability pathway within the bitumen pavement. As a result, if the permeability of the bitumen pavement can be controlled, moisture deterioration can be avoided or mitigated (Ahmad, et al., 2020). As a result, while developing an HMA, permeability and the proportion of air voids are stated together (Nejad, et al., 2010). The purpose of bituminous mixes design is to obtain the lowest possible air voids while maintaining long-term performance. When there are too many air spaces, the bitumen pavement becomes water and air accessible, resulting in shorter service life. While, when there aren't enough air spaces, the pavement becomes deformed (Zaltuom, 2018).

The ability of a substance to disperse the imposed traffic loads across a larger region for long durations without being impacted by nearby circumstances is measured by the durability of bitumen pavement (FHWA, et al., 2017). Greater pavement layer stiffness and durability lead to broader resistant regions, which restrict the amount of strain encountered somewhere at bottom of the pavement structure, but cracking probability rises as the stiffness of the bituminous mixes rises (Al-Mansoori, et al., 2020). The serviceability of pavement is generally planned to be between 10 and 15 years (Yang, et al., 2015). The aging of the bitumen binder is well acknowledged as having a significant impact on the longevity of asphalt pavements and causing early degradation. This aging mechanism is permanent, and it can dramatically alter the rheological characteristics of bitumen binder, causing pavement stiffening, as well as a reduction in the lifetime of road pavement (Himan, et al., 2018). The industry and research are working to improve the pavement's durability. Distresses will be decreased as a result of improving the pavement's durability, increasing the roadway network's service life. Researchers, industry, and government organizations have been trying to improve design techniques, set higher requirements, and use higher-quality materials to achieve this aim (Espinoza-Luque, et al., 2018). Providing an acceptable level of service on the road network helps not only the direct users by lowering the likelihood of incidents and damage to the vehicle, but also benefits the neighboring society. Because less maintenance is required, traffic jam is reduced, resulting in lower greenhouse gas emissions, disturbance, noise, and long delays (Kang, et al., 2014). As a result, to maximize pavement durability, distresses should be reduced to a minimum. Heavy rutting can reduce rider. Hard aggregate, polymer modification, stiff bitumen binder, and decreased binder concentration have all been used in the mixture design to minimize rutting distresses (Wu, et al., 2016). There are several forms of cracks that develop in pavements, namely low temperature, block, and fatigue cracking, each with its initiation process. Several researches have been conducted to reduce pavement cracking by applying bitumen modification, the use of additives, aggregate gradation adjustments, and raising binder content (Pszczoła, et al., 2017).

The workability of HMA is another key aspect that should be taken into consideration. The ease at which a pavement mix may be put and compact is referred to as workability. Mixes with excellent workability are simple to put and compacted, whereas those with low workability are hard to place and compacted. According to research on the workability features of HMA mixes, aggregate gradation and kind have a considerable impact (MS-2 Asphalt Institute, 2014). Harsh mixes (those with a greater ratio of coarse aggregate and/or insufficient bitumen) tend to separate during handling and can be hard to compact. Furthermore, too fine particles in HMA can cause the asphalt to become sticky, rendering it hard to deal with (Gudimettla, et al., 2004). Workability is extremely crucial during the hand placement of HMA around manhole covers, severe bends, and other impediments. It's critical that the mixes applied in these locations be workable (MS-2 Asphalt Institute, 2014).

The primary goal of HMA design techniques is to calculate the percentage of bitumen to add to a certain aggregate gradation to achieve good performance in the site. These processes and tests are frequently developed from laboratory testing. To develop HMA mixes for flexible pavements, the Marshall Technique is used. Bruce Marshall created the Marshall Mix Design process for the first time in 1939. Marshall Method utilized virtually all across the world, the American Society for Testing and Materials (ASTM) and the American Association of State Highway and Transportation Officials (AASHTO) have established the Marshall Test protocols. Several researchers have criticized the classic Marshall Compaction method, claiming that it does not match the one used in the field (NCHRP, 2011). Furthermore, the Marshall Mix design technique has many shortcomings, including its method of compaction, which does not mimic field compaction (Varma, et al., 2019). In 1987, the Federal Highway Administration (FHWA) began five-year research to enhance the performance of HMA pavements. The Superpave (SUperior PERforming Asphalt PAVEments) system was developed as a consequence. Bitumen binder requirements, volumetric mix design, and performance testing are all available from Superpave (Lee, et al., 2018). Superpave isn't simply a set of software; it's also a binder specification and a tool for mixing and analyzing mixtures. Superpave is a system that includes all of these aspects.

The construction of new roadways, as well as their rehabilitation, reconstruction, and maintenance, as well as the protection of old pavements, necessitates a massive amount of materials and the use of non-reproducible energy sources, all of which have significant cost implications for the economy. Toxic gas emissions into the environment as a result of new road construction, maintenance and repair operations are harmful and life-threatening aspects of the ecosystem (Rafiq, et al., 2021). WMA technology is utilized to mitigate these adverse effects.

### *2.3.3 Warm Mix Asphalt (WMA)*

HMA manufacturing and usage consumes a lot of fossil fuels and emits a lot of greenhouse gases (GHGs) like $CO_2$, CO, $NO_X$, $SO_2$ etc. (Behnood, 2020). Asphalt industries and sectors all over the globe have taken significant efforts to reduce GHG emissions and excessive fossil fuel usage to prevent climate change. The asphalt industry has attempted to reduce the mixing and compaction temperatures of HMA without adversely impacting the mixed qualities. WMA technology has recently been developed and evaluated as a potential alternative to traditional HMA (Teh & Hamzah, 2019). WMA is mixed normally at temperatures ranging from 100 to 140 ˚C, whereas HMA is mixed at temperatures ranging from 150 to 180 ˚C (Wang, et al., 2018). WMA refers to a set of additives and technology that may be applied to produce asphalt mixes at temperatures lower than those utilized to produce HMA (between 20 and 60 ˚C) (Behnood, 2020). WMA has received a lot of awareness in recent years because of its superior environmental, economic, and technical advantages, like emissions reductions, lowered bitumen aging, better working conditions (due to lower toxic gas emissions), improved workability, decreased energy usage, and longer construction lifetime (Yang, et al., 2019). Furthermore, the reduction in production temperatures results in decreased energy usage, lowering production costs (Hasan, et al., 2017; Ranieri, et al., 2017). WMA methods demand reduced mixing and compaction temperatures than traditional HMA, which reduces energy usage by 18- 30 percent (Almeida-Costa & Benta, 2016). WMA tends to make paving simpler by improving workability, longer hauling distances, allowing for early traffic opening, and an extended construction season. WMA technologies are also cost-effective, as they may reduce fuel consumption by up to 20 to 25 percent (Pérez-Martínez, et al., 2014). Various researchers have revealed contradictory outcomes about the performance of WMA technologies in certain cases, which could be likened to the form and percentage of WMA technology and additive, respectively, form and percentage of other additives in WMA, and different test techniques applied to evaluate WMA binders and mixtures (Behnood, 2020). The employment of WMA technologies has several environmental, technological, and economic advantages. Table 4 lists some of the advantages of WMA technology.

**Table 4**

*Benefits of WMA Technology*

| **Observed effect** | **Reference** |
|---|---|
| In the construction field, there has been an enhancement in working, safety, and health conditions for employees and workers. | Hurley & Prowell, 2005 a and b |
| WMA was shown to be related to an 18 percent decrease in fossil fuel use and a 24 percent decrease in air pollutants through a lifecycle study. | Hassan, 2010 |
| Because of the lower greenhouse gas emissions, WMA plants may be situated close to urban locations. | Capitão, et al., 2012 |
| WMA's cost-effectiveness in terms of both economic and environmental expenses has been confirmed through recent studies. | Jamshidi, et al.,2018 |
| WMA contributes to a more ecologically friendly pavement manufacturing process. | Song, et al., 2018 |
| WMA processes resulted in a 40% reduction in greenhouse gas emissions and energy usage. | Wang, et al.,2018 |
| WMA has been shown to lead to significant reductions in emissions and energy savings. | Yang, et al.,2019 |
| WMA technique reduces manufacturing temperatures to around 20–40 ˚C while maintaining the bituminous mixture's technical performance. | Rahman, et al., 2020 |

Reduced production temperatures in WMA mixtures may be accomplished using three basic methods: foaming technology, organic additives, and chemical agents

(Kassem & Chehab, 2021; Cheraghian, et al., 2021). Foaming technologies occur by applying cold pulverized water to the heated bitumen binder or injecting it into the mixing container (Hasan, et al., 2017). The water vaporizes as the temperature of the bitumen rises, and the steam is encased in the binder. The encased steam can temporarily expand the volume of the bitumen binder and lower its viscosity, improving the bituminous mixture's workability (Behnood, 2020). Waxes and fatty amides are the most common organic additives (Kheradmand, et al., 2014). Because waxes are present, organic additives reduce the viscosity of the neat bitumen binder. Based on their chemical structure, many types of organic additives that lower the viscosity of the bitumen binder have been given in an abundance of literature (Solouki, et al., 2015). These additives typically melt between 80 and 120 ˚C, lowering the viscosity of the bitumen binder and lowering manufacturing temperatures (Hainin, et al., 2015). These additives can be combined directly with the bituminous mixtures or with a bitumen binder. Based on organic WMA technology, Asphaltan A, Asphaltan B, Sasobit, Sasobit Redux, and Licomont BS100 are some examples of this technology (Almeida & Sergio, 2019).

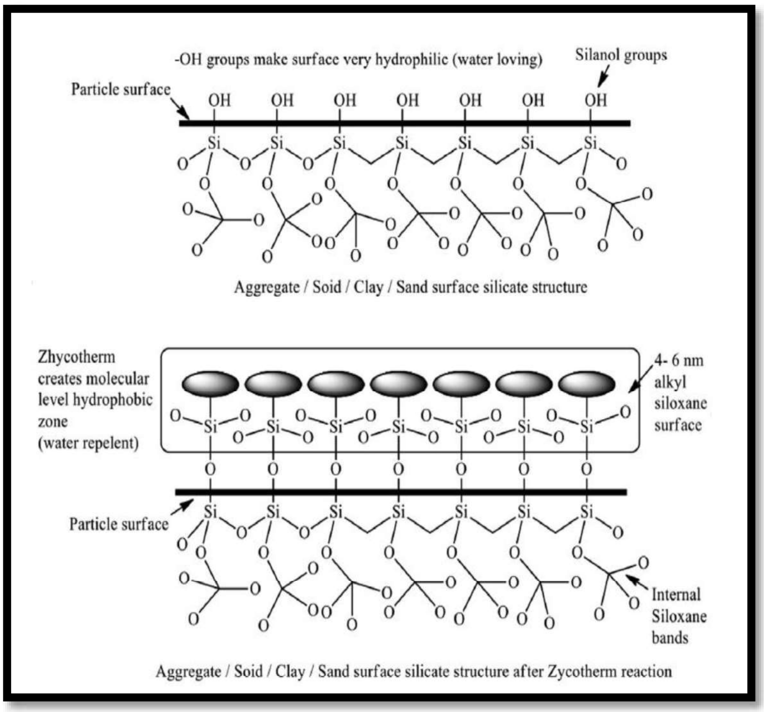
Chemical agents have a distinct functioning mechanism than foaming and organic-based methods. Chemical WMA agents can be applied to the bitumen during the manufacturing phase to promote adhesion and increase the binder's coating of the aggregates. Chemical additives improve both the workability and compaction of the mixture, as well as the aggregate coating by the bitumen binder (Yu, et al., 2020). The purpose of these agents is to decrease the surface tension between the bitumen binder and the aggregate. As a result, the bitumen-aggregate system interacts more smoothly, resulting in lower production temperatures without compromising performance (Capitão, et al., 2012; Abed, et al., 2020; Caputo, et al., 2020). Furthermore, when chemical-based WMA methods are used, the viscosity and rheological characteristics of the neat bitumen binder typically remain unaltered (Xiao, et al., 2012). Chemical agents like Zycotherm, Evotherm, Cecabase, Rediset are commercially accessible (Yang, et al., 2017; Khani Sanij, et al., 2019; Cheraghian, et al., 2020).

Wang et al. (2018) found that almost of WMA investigations utilized mixed design techniques identical to those used in traditional HMA. The efficiency of compacted WMA may be greatly influenced by material qualities like bitumen binder and aggregate type. For field and laboratory investigations of WMA, the majority of the available research has employed Dense-Graded mixes (Li, et al., 2018). Many studies have compared and analyzed the volumetric characteristics of WMA with those of traditional HMA (Ghuzlan & Ar'ar, 2016). The impact of warm mix additives on volumetric characteristics of asphalt mixes, such as optimum binder content (OBC) and voids in mineral aggregate (VMA), has been widely researched. In WMA, the OBC remains the same as in the conventional HMA (Singh, et al., 2018). Furthermore, in the majority of investigations, there was no alteration in VMA in WMA (Raghavendra, et al., 2016).

**A. The Basics of Nano-ZycoTherm.** ZycoTherm is an odorless nanotechnology-based product that has a great potential to increase bitumen binder and aggregate adhesion in asphalt mixes. This additive enhances the moisture resistance of asphalt mixes. It allows for full coating. Furthermore, ZycoTherm leads to a decrease in mixing and compaction temperatures, rendering it an environmentally beneficial warm mix additive (Tripathi & Ray, 2020). Zydex Industries, based in Gujarat, India, created ZycoTherm, a chemical warm mix additive that offers substantial advantages because of WMA technology. ZycoTherm is an antistrip warm-mix chemical additive used to ensure asphalt pavements' moisture resistance. ZycoTherm is a warm mix additive that is incorporated at 0.1 percent to 0.15 percent of the weight of the bitumen binder at room temperature. ZycoTherm is a new type of organo-silane that is made up of organo groups. ZycoTherm's silanol group interacts with silicon on aggregate surfaces, converting hydrophilic aggregate (water- loving) surfaces to hydrophobic aggregate (water repellent) surfaces, as shown in Figure 14.

**Figure 14**

*Aggregate Surface with and without ZycoTherm*




(Ameli, et al., 2020)

Not only does this reaction render them hydrophobic, but it also strengthens the link between bitumen binder and aggregate. Moreover, without ZycoTherm creating polar-polar contact between bitumen binder and aggregate, thus 5–15 percent of bitumen is involved in bonding. The bitumen binder is loosely attached to the aggregate without Zycotherm. Meanwhile, with Zycotherm, the contact is non-polar-non-polar, and 85–95 percent of the bitumen is involved in bonding (Ameli, et al., 2020). Bitumen is tightly bound with Zycotherm and maintains that connection even when stressed (Aghapour & Babagoli, 2020; Norouzi, et al., 2021). The use of liquid antistripping chemicals, such as ZycoTherm, has lately been established as the preferred alternative in the asphalt sector for addressing the stripping problem caused by moisture in asphalt mixes. The essential characteristics of ZycoTherm are depicted in Figure 15.

**Figure 15**

*Features of ZycoTherm*


**B. Effects of ZycoTherm on Bitumen Binder Conventional and Rheological Characteristics.** Several traditional bitumen tests, such as penetration, softening point, and ductility tests, are widely used to study the physical performance of bitumen. The rotational viscosity, DSR, and BBR tests are also used to assess the rheological performance of bitumen binders. This section provides an overview of the performance of the ZycoTherm-modified bitumen binder in terms of physical and rheological characteristics.

The traditional characteristics of base bitumen with (80/100) grade modified with ZycoTherm were studied by (Mirzababaei, et al., 2017), and the findings showed that the penetration values were reduced and the softening point was enhanced. In addition, the enhanced penetration index (PI) revealed that the binder was more resistant to temperature susceptibility. In comparison to neat bitumen, (Ameli, et al., 2020) revealed that adding ZycoTherm to the bitumen binder resulted in a 19% reduction in penetration and an 11% increase in softening point. Tripathi and Ray (2020) revealed that the addition of ZycoTherm reduced the penetration value of plain bitumen and increased the softening point somewhat, indicating that it tended to improve the traditional properties of bitumen. Moreover, the ductility has been increased, and the pavement service is improved.

Mirzaaghaeian and Modarres (2019) investigated the storage stability of bitumen binder modified with ZycoTherm and reported that bitumen specimens containing 0.07 and 0.14 percent ZycoTherm had satisfactory stability. Sani et al. (2020) performed a study to determine the impact of ZycoTherm on the performance of natural rubber latex (NRL) modified bitumen binder, the modified binders with ZycoTherm outperformed the plain and NRL-modified bitumen without ZycoTherm in terms of penetration and softening point outcomes. Furthermore, Raufi et al. (2020) investigated the status of phase bonding between the bitumen binder and the modifier while kept at elevated temperatures using three different dosages of ZycoTherm 0.1, 0.2, and 0.3%. According to their findings, the softening point temperature differential between the top and bottom sections of the tested specimens remained below 2.5 $^{\circ}$C.

A bitumen binder should be stiff sufficiently to prevent rutting and not distort too much. It should, on the other hand, be elastic sufficiently to recover to its original form after being deformed by a load. As a result, $G^*/\sin \delta$, should be considered. As a result, several studies have utilized this parameter to evaluate the performance of binders against rutting. Mirzababaei et al. (2017) revealed the results of a study done on bitumen binder modified with various percentages of ZycoTherm to assess rutting, the inclusion of 0.1% of ZycoTherm had the greatest impact on rutting resistance of bitumen binders with (80/100) grade. In a study on the modified binder with ZycoTherm, Ibrahim and Mehan, (2015) observed that adding 0.5% ZycoTherm increased $G^*/\sin \delta$ (more resistance to rutting) and decreased $G^*$. Sin $\delta$ (more resistance to fatigue cracking) as compared to neat bitumen. Raufi et al. (2020) assessed the rheological properties of ZycoTherm binders in terms of the Superpave rutting parameter in short-term aging at low and high frequencies. The results showed that the rutting performance of the ZycoTherm modified bitumen increased, resulting in greater $G^*/\sin \delta$ values at high and low frequencies when compared to the plain bitumen. Moreover, by evaluating the Superpave rutting parameter, Ameli et al. (2020) observed that adding ZycoTherm resulted in an improvement in the performance compared to the neat bitumen. Furthermore, the addition of ZycoTherm had no significant effect on viscosity reduction, which is similar to the findings of Mirzababaei et al. (2017) which can be attributed to their chemical characteristics as this type of WMA additive does not influence bitumen viscosity. In contrast, a study conducted by Raufi et al. (2020) revealed that the viscosity of bitumen modified with ZycoTherm resulted in a 76.5% drop in viscosity when compared to the plain bitumen binder at 135 ˚C using a Brookfield rotational viscometer. Sani et al. (2020) conducted a research to investigate the rheological characteristics of both unaged and short term-aged. The findings for NRL modified bitumen binders with ZycoTherm revealed that adding ZycoTherm to the NRL binder resulted in a greater improvement.

On the other hand, applying multiple stress creep recovery test is another effective approach to assess rutting resistance. Mirzababaei et al. (2017) found that adding ZycoTherm to the bitumen binder (80/100) reduced the $J_{nr}$ values at both 100 and 3200 Pa stress levels, and all concentrations of ZycoTherm increased the performance of bitumen binder against rutting. Conversely, Ameli et al. (2020) observed that the inclusion of ZycoTherm increased the value of $J_{nr}$ and decreased the recovery percentage $R\%$ followed by the conventional binder. The explanation for this might be due to the sample's low stiffness and poor elastic nature.

Many research has shown that chemical agents such as ZycoTherm do not affect rutting resistance (Bairgi, et al., 2018) or rheological properties (Xiao, et al., 2012), but that rutting performance is a function of the kind and source of original bitumen binder (Singh, et al., 2017; Bairgi, et al., 2018). Moreover, there are still differences of opinion among researchers in terms of opponents and proponents of ZycoTherm's ability to modify rheological characteristics, including rutting. And, although ZycoTherm can enhance rutting resistance, the improvement isn't significant, thus this study aimed to improve ZycoTherm's rheological characteristics, particularly rutting resistance, using Nano-Silica.

**C. Effects of ZycoTherm on Asphalt Mixtures Mechanical Performance.** The field performance of asphalt pavement is directly related to providing the appropriate mechanical performance of asphalt mixes. This section compares and reviews the laboratory performance of ZycoTherm in comparison to the traditional HMA in terms of moisture deterioration, and rutting resistance. Many tests, including resilient modulus, moisture susceptibility, and indirect tensile test, have been performed extensively on WMA modified with ZycoTherm. Furthermore, many studies show that the features and performance of WMA are comparable to conventional asphalt mixes, if not superior in some situations (Wang, et al., 2019).

Moisture damage, often known as stripping, is one of the most common durability issues. Water and repetitive traffic loads are generally the causes of this deterioration. When water enters between the aggregate and bitumen layer, it breaks and weakens the adhesive connection between the aggregate and the binder bitumen, causing the bitumen layer to separate and remove from the aggregate surface. Using anti-stripping chemicals such as Nano-ZycoTherm to strengthen the binding between aggregate and bitumen while enhancing the aggregate's surface charges is a more widely recognized strategy (Haghshenas, et al., 2015). Mirzababaei (2016) investigated the effects of ZycoTherm in terms of stripping (moisture damage) on asphalt mixtures made with calcareous and siliceous with two different gradations. Many tests were performed to evaluate the stripping including, resilient modulus, modified Lottman method, Texas boiling test, and fracture energy. The findings revealed that ZycoTherm improved moisture susceptibility performance regardless of type or gradation of aggregate. Sharma et al. (2019) found that ZycoTherm performed better regards moisture resistance in comparison to Sasobit and Evotherm. Sanij et al. (2019) conducted a study on asphalt mixes modified with ZycoTherm, the creep, moisture susceptibility, and resilient modulus were all enhanced. Ameli et al. (2020) revealed the findings of a research done on asphalt mixes modified with ZycoTherm to assess the ITS, resilient modulus, and fracture energy, the ZycoTherm enhanced the results due to greater binder-aggregate adhesion. The fracture energy outcomes showed that ZycoTherm modified mixtures had the highest values when compared to other WMA additives and the control mix, indicating that the inclusion of ZycoTherm improves bitumen and aggregate adhesion and cohesion, so the greater energy is required to break the aggregate-bitumen bond. As a result, the mixture's resistance to moisture susceptibility improves. Almost all investigations have shown the ZycoTherm favorable benefits regards to the moisture resistance and durability of asphalt mixtures (Sharma, et al., 2019; Ameli, et al., 2020).

Due to the low manufacturing temperatures, rutting is one of the key issues for WMA. It is an essential criterion for determining how well an asphalt pavement performs during its serviceability. Rutting properties are influenced by a range of elements, such as additive kind and dose, bitumen binder grade, and gradation of aggregate (Bairgi, et al., 2018). Many researches in WMA looked at the influence of production temperature and concluded that WMA is expected to be less resistant to rutting than HMA, and this is because WMA ages more slowly at lower mixing and compaction temperatures (Zhao, et al., 2012). In general, the rutting resistance of a mix containing a chemical warm mix additive is the lowest. This could be attributable to chemical agents softening and lubricating the binder (Bairgi, et al., 2020). Hasan et al.

(2017) conducted a study utilizing the Hamburg Wheel Tracking method to measure the rutting resistance of asphalt mixtures containing ZycoTherm. The findings revealed that the ZycoTherm seemed to have a comparable outcome to conventional HMA (60/70 grade) in terms of rut depth. Furthermore, despite the reduced temperature, the addition of ZycoTherm resulted in improved workability than HMA. The amount of energy needed to compact the WMA mixture was less than that needed to compact HMA. The addition of ZycoTherm also reduced the amount of effort required to compact the warm mix specimens. Ameri et al. (2018) conducted research on the performance properties of asphalt mixes including ZycoTherm by measuring rutting resistance using dynamic creep, moisture susceptibility using the Lottman technique, and resilient modulus. According to the findings, the mixtures comprising of 0.1 percent ZycoTherm had higher rutting resistance compared to the control mix. This is because bitumen and aggregates have a stronger bond. The mixes including ZycoTherm contribute a 15% rise in ITS when analyzing the ITS.  Since ZycoTherm is an antistripping chemical agent, few studies have looked at the behavior of WMA produced with ZycoTherm in terms of rutting resistance of asphalt mixtures.

# CHAPTER III
# Materials and Experimental Procedures

The experimental procedures and the materials utilized in the research, are detailed in this chapter. All types of asphalt binders, conditioning procedures, and specimen preparation techniques are covered in detail. The research to be carried out is separated into two sections, as stated in Chapter I, physical and rheological characterisation of bitumen binder and mechanical characterisation of asphalt mixture. The DSR was used as the primary rheological characterisation method for all study binders. All study binders were subjected to a DSR experiment, as well as a frequency sweep test and MSCR. A detailed description of the two approaches, as well as their subsequent examination, is included. The experimental methodology for Indirect Tensile Strength (ITS) and Resistant Modulus ($M_R$) tests, as well as the associated data analysis, are described.

## 3.1 Materials

A penetration-grade bitumen binder of 60/70 was used in this research as a base bitumen. Bitumen binder was obtained from the Jordan Petroleum Refinery Company (JO PETROL). The physical characteristics of this binder were determined through laboratory testing, which included both conventional and rheological asphalt tests. In this chapter, the rheological features of this asphalt binder were described in detail. The conventional properties of base bitumen are shown in Table 5. In the modification process, Nano-Silica (NS) was used. Nano-Silica is produced by Nouryon Chemicals, Bohus, Sweden. Table 6 lists the characteristics of Nano-Silica. Furthermore, ZycoTherm (ZT), a chemical warm mix additive was used to produce WMA. ZycoTherm is established by Zydex Industries, Gujarat, India. Table 7 presents the physical features of ZycoTherm.

**Table 5**

*Physical Properties of Base Bitumen*

| Test | Result | Criteria | Standard Method Procedure |
|---|---|---|---|
| Penetration at 25 °C-0.1 mm | 67 | 60-70 | ASTM D5 |
| Softening point - °C | 50 | 48-56 | ASTM D36 |
| Ductility at 25 °C-cm | 100 | 100 min | ASTM D113 |
| Flash point °C | 322 | 230 min | ASTM D92 |
| Specific gravity at 25 °C- g/cm$^3$ | 1.02 | 1.01-1.06 | ASTM D70 |
| Rotational viscosity at 135 °C- Pa.s | 0.5672 | 3Pa.s max | ASTM D2196-10 |

**Table 6**

*Physical Properties of Nano-Silica*

| Property | Value |
|---|---|
| Appearance | Slightly Milky Transparent |
| PH | 6.71 |
| Viscosity (20 °C) | 3.05 mPa.s |
| Particle Size | 11 nm |

**Table 7**

*Physical Properties of ZycoTherm*

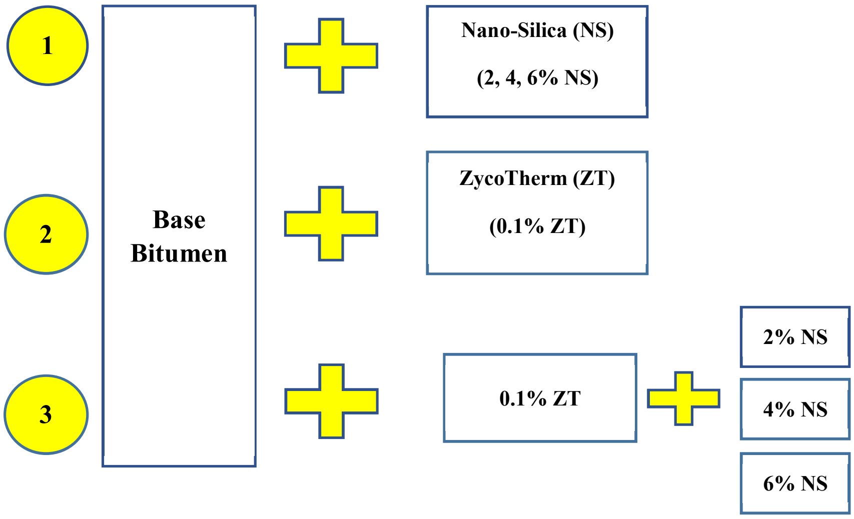
| Property | Value |
|---|---|
| Ingredients | Alkoxy, Hydroxyalkyl, alkylsilyl compounds, ethylene glycol, Benzyl alcohol. |
| Form | Liquid |
| Color | Pale yellow |
| Viscosity | 1-5 Pa.s |
| Specific gravity (25 °C) | 0.97g/cm$^3$ |
| Odor | Odorless |
| Flash point | >80 °C |
| pH | 10 % solution in water neutral or slightly acidic |

## 3.2 Modified Bitumen Sample Preparation

Construction of pavements and road networks for adverse weather conditions and heavy traffic require specially formulated bitumen grades to improve the performance. It is feasible to develop new types of bitumen that provide a considerably more cohesive combination by adding special materials such as Nano-Silica to enhance the overall characteristic features of base bitumen. The modification process of base bitumen with two types of modifiers, namely; Nano-Silica and ZycoTherm, and the material percentages used are shown in Figure 16.

**Figure 16**

*Modification Process*




The 60/70 bitumen penetration grade was utilized. To develop Nano-Silica modified bitumen, the binder was mixed with 2-6% NS in 2% increments of the base bitumen weight. To achieve the required viscosity, the binder was heated to 160 °C, whereas the bitumen was modified by the use of a mechanical mixer. A cylindrical container containing approximately 400 g of binder was placed on a heated plate. The hot plate was fixed at 160 °C to maintain the binder's viscosity during the mixing operation. While stirring the mixture with a mechanical mixer, the heated binder was gradually mixed with Nano-Silica for 60 minutes and the stirring speed was set at 1800 rpm. The modified binder was placed in the oven before forming the test sample to avoid any bubbles. The NS modified binders were labeled using the abbreviations 2% NS, 4% NS, and 6% NS to make sample identification easier. Figure 17 shows the blending mechanism when Nano -Silica was added to a heated bitumen.

**Figure 17**

*The blending Process of Adding Nano- Silica to Heated Bitumen*

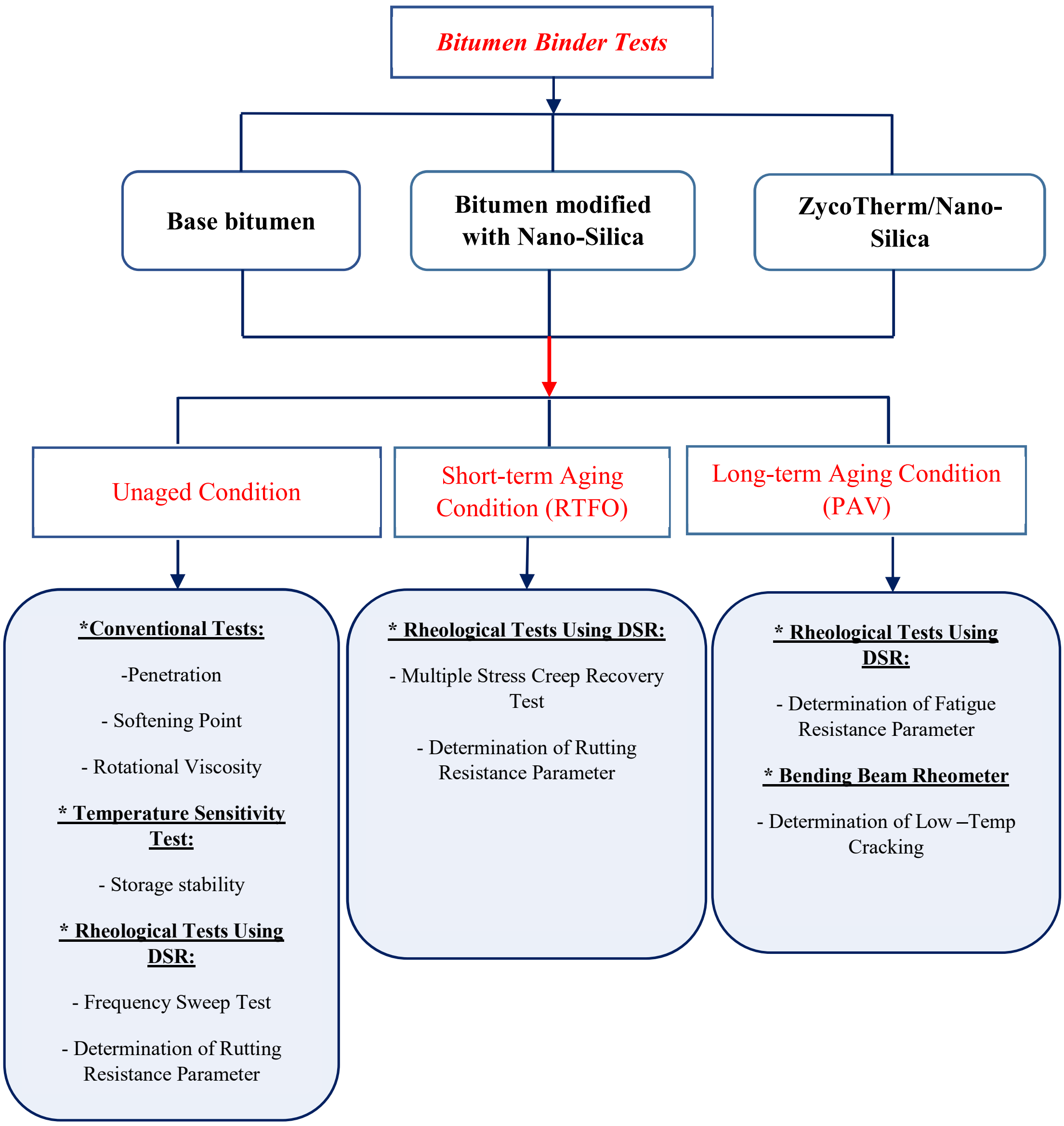
ZycoTherm binder was produced with a concentration of 0.1% of the binder's weight, as stated by the manufacturer's recommendation. The mechanical stirrer was adjusted to establish a 15-30 mm deep vortex inside the melted bitumen at a desirable speed. The ZycoTherm was then dropped into the middle of the vortex, whereas the bitumen was agitated at 130 °C. The mixing procedure was completed after 15 minutes of stirring. Furthermore, to prepare WMA modified with Nano-Silica (ZycoTherm/ Nano-Silica), the base bitumen was blended first with 0.1% ZycoTherm as mentioned previously, and then the Nano-Silica was gradually added to the mix by 2, 4, and 6% and blended with bitumen binder at 1800 rpm. The modified binders were labeled as, 0.1%ZT+2%NS, 0.1%ZT+4%NS, and 0.1%ZT+6%NS.

### 3.3 Experimental Methods: Bitumen Tests

While the damage resistance of asphalt mixes is substantially connected to the characteristics of bituminous binders, test techniques are employed to evaluate a specific property of binders to identify the best relevant binder tests and parameters to characterize the binder influence on damage resistance. Determining these tests and characteristics would fundamentally and logically lead pavement engineers in optimizing and selecting the best binder for a particular situation. As a result, this would help to maximize the value of pavements while also improving their quality of life. The kind of modifier, modifier dosage, and blending circumstances are all factors related to bitumen modification. The following sections will focus on the requirements and testing that are most important to bitumen binder performance. The characteristics of the binder are also tested at high, intermediate, and low service conditions. Figure 18 shows the tree diagram displaying the experimental system applied to demonstrate the binder tests of this research study.

**Figure 18**

*Framework of Bitumen Tests*




### *3.3.1 Conventional Tests*

The tests were used to determine the basic characteristics of the base bitumen and the modified binders: penetration test (ASTM D5), softening point test (ASTM D36), and viscosity test with a Brookfield Rotational Viscometer (ASTM D4402).

The bitumen binder is made of thermoplastic material, which means it softens as the temperature rises and hardens as the temperature drops. The temperature susceptibility of base and modified binders was examined using a penetration index (PI). To determine the PI, the temperature susceptibility of binders was estimated using penetration at 25 °C and softening point findings, as given in Eq. 5.

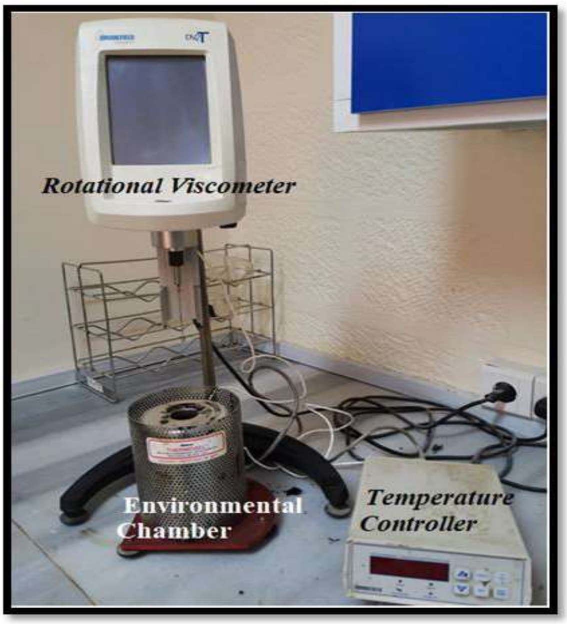
$$PI = \frac{1952 - 500\log(Pen_{25}) - 20SP}{50\,log(Pen_{25}) - SP - 120} \quad (5)$$

Where; ($Pen_{25}$) is the value of asphalt binder penetration in 0.1 mm at 25 °C, and (*SP*) is the value of bitumen binder softening point in °C.

In the elevated-temperature of pavement construction, the Rotational Viscometer (RV) is applied to figure out how viscous binders are. The Superpave bitumen binder standard takes this measurement into account. The RV test may be performed at varying temperatures, the viscosity test was carried out in this study at temperatures ranging from 135 to 195 °C with a 10 °C increment. The rotational viscometer is shown in Figure 19.

**Figure 19**

*Rotational Viscometer*




### *3.3.2 Storage Stability Test*

The bitumen high-temperature storage stability was assessed using a hot storage stability method. This method is used to assess a binder's stability to withstand phase separation at elevated temperatures. Separately prepared binders were placed into a 140-mm-high, 30-mm-diameter aluminium foil tube. For 48 hours, the tube was maintained upright in a 163 °C oven. Following the previous conditioning time, the aluminium tubes were placed vertically in a -10 °C freezer. The tubes were then divided into three halves of similar lengths. The softening points of the upper and bottom portions then were tested in line with ASTM D36. Once the difference between both the softening points of the upper and bottom portions reaches a maximum of 2.5 °C, the blend is deemed stable (Zani, et al., 2017).

### *3.3.3 Bitumen Binder Aging Conditions*

Short-term and long-term aging occur throughout the manufacturing and forming phases of the HMA and WMA. During these two phases, a rolling thin film oven (RTFO) was used to imitate the circumstances of short-term aging, and a pressure aging vessel (PAV) was used to simulate the circumstances of long-term aging. The RTFO aging method is utilized to simulate aging throughout mixing and placing, whereas PAV aging method is utilized to simulate aging during the in-service lifetime. As a result, bitumen tests involving mixing and installation qualities, namely the DSR, are carried out on RTFO aged samples, whilst bitumen tests involving in-service performance, the DSR and BBR are carried out on samples that have been aged in the RTFO and then the PAV.

To mimic manufacture and placement aging, the asphalt binder is subjected to elevated temperatures. One of the cornerstones of the Superpave PG binder standard is that testing should be as near as feasible to field performance. Because the bitumen binder in HMA ages significantly throughout the manufacturing and installation processes, a technique to mimic aging is critical for researching and forecasting early age hot mix asphalt pavement performance and distresses. The RTFO technique in its most basic form as part of ASTM D 2872, unaged bitumen samples weighing 35 grams were placed in cylindrical glass bottles and rotated in an oven. The carriage moves within the oven while the samples were aged at 163 °C by rotating at 15 rpm under air-blown pressure for 85 minutes. After that, samples were kept for use in rheological property testing and long-term aging. Base bitumen and modified binders were aged under RTFO. Figure 20 shows the RTFO used in this study.

**Figure 20**

*Rolling Film Thin Oven*

The Pressure Aging Vessel (PAV) offers a simulated long-term aged bitumen binder. Heat and pressure are applied to the bitumen to imitate in-service aging during a 7 to 10-year timeframe. In aged pavements, many HMA distresses begin or grow very severe. To investigate and anticipate these sorts of distresses, a method to mimic aged asphalt binder is necessary. Long-term aged bitumen binder must be tested at moderate and cold temperatures to evaluate fatigue and low-temperature cracking resistance, according to the Superpave PG binder standard. 50 g of RTFO bitumen samples were placed in pans. A PAV that has been preheated to the test temperature of 100 $^{\circ}$C without being pressured, then the PAV was set to 2.1 MPa as it approaches the test temperature. The samples were removed after 20 hours. The samples were then kept for future assessment, in accordance with the ASTM D 6521. Unmodified and modified binders were aged under PAV. Figure 21 shows the PAV used in this research.

**Figure 21**

*Pressure Aging Vessel*


### *3.3.4 Dynamic Shear Rheometer*

This technique was first presented in 1993 as part of the Strategic Highway Research Program (SHRP). The DSR is a highly effective instrument for determining bitumen's elastic, viscoelastic, and viscous characteristics across a broad temperatures and frequencies range. This instrument creates a dynamic oscillatory load by applying a sinusoidal shear stress or strain in the pattern of a sinusoidal function of time. Various binder characteristics were measured using a DSR instrument before and after the adjustment. The findings of tests conducted at moderate and high temperatures can be used to estimate resistant to fatigue and rutting in asphalt pavements, respectively. A thin bitumen sample is placed between two circular discs, the lower disc remains stationary whereas the upper disc oscillates over the sample. The type of test conducted on the bitumen being evaluated determines the test temperature, which can fluctuate from 30 $^{\circ}$C to 80 $^{\circ}$C, as well as the sample size and disc diameter. A sample of 1 mm thickness and 25 mm in diameter is used to evaluate unaged binder and RTFO aged binder at the high-temperature standard for a specified performance grade (PG).

PAV aged samples are evaluated at lower temperatures. The sample becomes extremely rigid as a consequence of the lower temperatures. As a result, a 2 mm thick sample with an 8 mm diameter is employed. The DSR determines the complex shear modulus (G*) and phase angle (δ) of a specimen. Once repeatedly sheared, the complex shear modulus (G*) may be thought of as the sample's overall resistance to deformation, whereas the phase angle (δ) represents the lag between both the applied shear stress as well as the resultant shear strain. The frequency sweep test, multiple stress creep recovery test (MSCR), Superpave rutting resistance determination, and Superpave fatigue resistance determination were all done using the DSR. Figure 22 shows the DSR device used in this research.

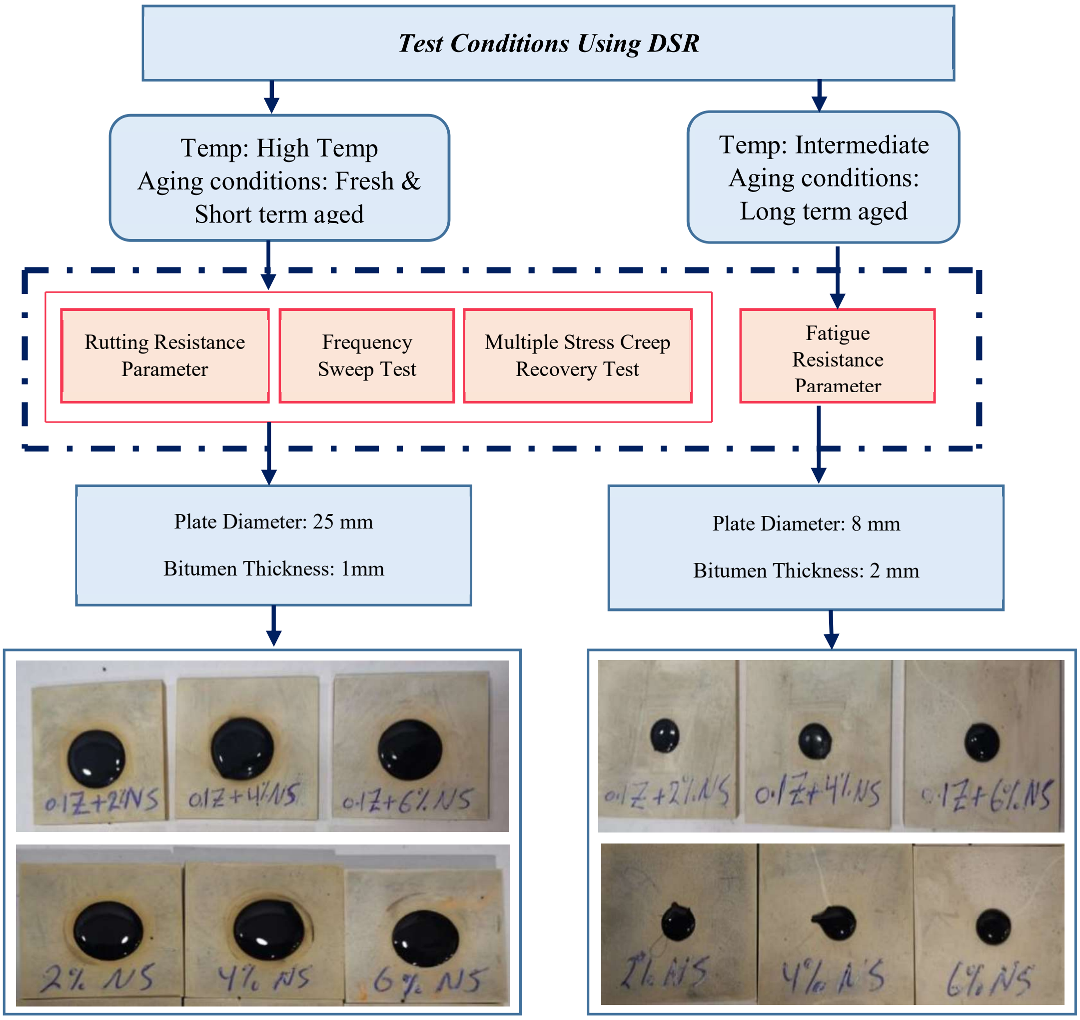
**Figure 22**

*Dynamic Shear Rheometer*


**A. Geometry of a sample.** The geometry of the sample was chosen based on the assessment type, circumstance, and requirement. To avoid melting, the sample geometry at elevated temperatures should have a large plate diameter. To avoid brittle cracking at low temperatures, the sample should have a small plate diameter with large bitumen thickness. Using the dynamic shear rheometer (DSR), two types of a plate are utilized. For high-temperature testing, the sample plate geometry is a 25 mm diameter with a 1mm spacing while the sample plate has an 8mm diameter with 2 mm spacing is utilized at intermediate temperature. Figure 23 illustrates a tree diagram of DSR testing and the geometry test samples that were used.

**Figure 23**

*DSR Testing and Test Samples*




**B. Rutting Resistance Determination Using Superpave Parameter.** Strategic Highway Research Program (SHRP) created the rutting resistance parameter, this rheological characteristic, which is incorporated in the Superpave binder standard as the high service temperature feature following AASHTO T315 and ASTM D7175. A DSR in an oscillatory state is used to test the property under defined temperature and frequency circumstances. The value of this parameter is calculated by dividing the complex shear modulus ($G*$) by the sine of the phase angle ($\delta$). Another perspective is that rutting is essentially a cyclic loading mechanism. Work is done to distort the asphalt pavement surface with each loading cycle (traffic loading). The elastic component of the asphalt pavement recovers some of this effort, while the rest is dissipated by the bitumen viscous component as permanent deformation and cracking (Abedali, 2017). As a result, the amount of effort wasted every loading cycle should indeed be reduced to minimize the rutting. Therefore, the rutting parameter $G^*/\sin\delta$ should be increased as much as possible. The work dissipated each loading cycle is represented in Eq. 6. The existing Superpave elevated-temperature binder specification requires the binder that short –term aged using RTFO to be more than 2.2 kPa and 1.0 kPa for fresh (unaged binder). On both fresh (unaged) and short-term aged binders, the assessment was carried out utilizing a dynamic shear rheometer and a parallel plate with a diameter of 25 mm and a spacing of 1 mm at 10 rad/s frequency.

$$W_c = \pi\sigma^2(1/\frac{G*}{Sin\delta}) \quad (6)$$

Where:

$W_c$: is the amount of work that is dissipated each load cycle

σ: is the applied stress

G*: is the complex modulus

δ: is the phase angle

**C. Frequency Sweep Test.** Among the most popular methods for assessing the rheological properties of an asphalt binder is the frequency sweep test. The frequency sweep test may be used to mimic the speed of a vehicle on the highway. A loading frequency of 10 Hz mimics a 60 km/h speed, whereas a loading frequency of 15 Hz mimics a 90 km/h speed (Huang, et al., 2019). The viscous and elastic characteristics of the evaluated binder may be determined using two major rheological parameters obtained from frequency sweep tests conducted across a variety of frequencies. Complex shear modulus ($G^*$) and phase angle ($\delta$) are these two parameters. The master curve and isochronal plots for the tested specimen are constructed using the frequency sweep test. The behaviour of a system at a constant frequency is represented by an isochronal plot, which is a curve on a graph (time of loading). Isochrones are, for example, curves of complex modulus as a function of temperature at a fixed frequency. The change of $G^*$ and $\delta$ with temperature for constant frequency or loading time is shown by the isochronal curve of $G^*$ and δ for the specimens. The master curve is a tool for describing sample rheological characteristics. The time-temperature superposition concept is used to construct the master curve of $G^*$ and/or $\delta$. To establish a master curve, a reference temperature is chosen, and the data obtained via frequency sweeps at many other temperatures is moved by shift factors to one at the selected reference temperature (Ali, et al., 2015). The frequency sweep was evaluated for both base and modified samples, with the load frequency being adjusted from 1 Hz to 15 Hz. Furthermore, the tests were carried out in a strain-controlled state of 12 percent, with temperatures ranging from 10 to 82 degrees Celsius, with a temperature differential of 6 degrees Celsius. For temperatures below 40 degrees Celsius, an 8 mm spindle with a gap of 2 mm was used, whereas for temperatures over 40 degrees Celsius, a 25 mm spindle with a gap of 1 mm was used. As indicated in Table 8, the test variables and circumstances are provided.

**Table 8**

*Test Requirements for Frequency Sweep*

| Frequency Sweep Test Parameters | Test Conditions Situation | | Standard Unit |
|---|---|---|---|
| Test Temperatures | $10^{+6}$ - 34 | $40^{+6}$ - 82 | ˚C |
| Diameter of the Spindle | 8 | 25 | mm |
| Gab Thickness | 2 | 1 | mm |
| Test Frequencies | From 1Hz to 15Hz | | Hz |

**D. Multiple Stress Creep Recovery (MSCR).** Rutting is typical pavement distress that occurs as a result of the pavement's accumulated permanent deformation caused by high temperatures and repetitive vehicle loads. MSCR test is suitable for evaluating the rutting performance of binder because it can replicate actual loading conditions of pavement. MSCR test was recently adopted to enhance the Superpave performance grading PG system's rutting evaluation. The American Association of State Highway and Transportation Officials (AASHTO) and the American Society of Test Methods (ASTM) have both standardized the MSCR test: AASHTO T 350 and ASTM D7405, respectively. A DSR in the creep phase at a given temperature is used to test the MSCR. The test includes applying a series of static stresses on a binder specimen, following a period of recovery at various levels of stress. The MSCR test is performed by applying a creep-recovery loading consisting of a 1second loading time and 9 second unloading time to a bitumen sample placed between parallel discs for 10 cycles; during each creep stage, a specific shear stress of 0.1 kPa and 3.2 kPa is applied to a bitumen sample.

The MSCR test yields two key components: non-recoverable creep compliance ($J_{nr}$) and percent recovery (*R %*). The non-recoverable creep compliance of an asphalt binder is a measurement of its resistance to rutting under repetitive loading conditions. For determining the rutting capability of bitumen binders, the non-recoverable creep compliance gained from the MSCR test is the most effective. The rutting potential index within the MSCR test is non-recoverable creep compliance ($J_{nr}$), $J_{nr}$ is determined by dividing unrecoverable strain at the end of the recovery phase by the utilized shear stress as shown by Eq. 7. The % recovery is used to determine whether or not the tested bitumen binders have an elastic response. *R %* is defined as the ratio of the recoverable strain at the end of the recovery phase to the produced shear strain at the end of the creep phase as shown by Eq. 8.

$$J_{nr}(\sigma, N) = \frac{\varepsilon_r - \varepsilon_0}{\sigma} \quad (7)$$

$$R(\sigma, N) = \frac{\varepsilon_c - \varepsilon_r}{\varepsilon_c - \varepsilon_0} \times 100 \quad (8)$$

Where; $J_{nr}(\sigma, N)$ is the non-recoverable creep compliance at applied stress with N denotes the number of recovery and creep cycles, $\varepsilon_r$ denotes as the strain value at the end of the recovery stage, $\varepsilon_0$ is the strain value at the beginning of the creep stage, R (σ, N) is the % recovery at applied stress, $\varepsilon_c$ denotes as the strain value at the end of the creep stage.

The purpose of the MSCR assessment in this study is to assess the $J_{nr}$ and *R%*. The test parameters and conditions are listed in Table 9.

**Table 9**

*MSCR Test Requirements*

| MSCR Test Parameters | Test Conditions Situation | Unit |
|---|---|---|
| Aging Condition of Sample | Short Term Aged Sample-RTFO | ---- |
| Test Temperature | 64 | ˚C |
| State of Loading | Stress Controlled | ---- |
| Stress Levels | 0.1 and 3.2 | kPa |
| Creep Duration | 1 | Second (s) |
| Recovery Duration | 9 | Second (s) |
| Diameter of the Spindle | 25 | mm |
| Gab Thickness | 1 | mm |

**E. Fatigue Resistance Determination Using Superpave Parameter.** It is widely understood that the characteristics of bituminous binders have a major impact on the fatigue resistance of asphalt mixes. Fatigue cracking generally begins in the binder and spreads from there. At intermediate temperatures, the Superpave fatigue parameter ($G^*$. sin $\delta$) is frequently utilized to characterize and manage the fatigue characteristic of binders. Lower ($G^*$. sin $\delta$) is preferable since it reduces the amount of energy wasted every loading cycle (Subhy, 2017). Fatigue cracking is less frequent when the quantity of energy wasted every loading cycle is minimal. As a result, the amount of effort wasted every loading cycle should be reduced to decrease fatigue cracking. At a constant strain, the work wasted each loading cycle can be represented in Eq. 9. The DSR was employed in the oscillatory shear state with 8 mm diameter of the parallel plates and 2 mm gab to determine the complex shear modulus ($G^*$) and the phase angle ($\delta$) to evaluate viscoelastic characteristics of asphalt binder in the moderate range of temperatures. All the tested specimens were aged for short-term and long-term aging using RTFO and PAV, respectively with failure criteria is set at a maximum value of 5000 kPa. The test was conducted at 10rad/s frequency at temperatures ranging from 16 °C to 34 °C.

$$W_c = \pi \varepsilon^2 \{(G^*) \times (\sin\delta)\} \quad (9)$$

Where:

$W_c$: denotes the amount of work that is dissipated each load cycle.

$\varepsilon$: indicates the strain during the load cycle.

G*: is the shear complex modulus.

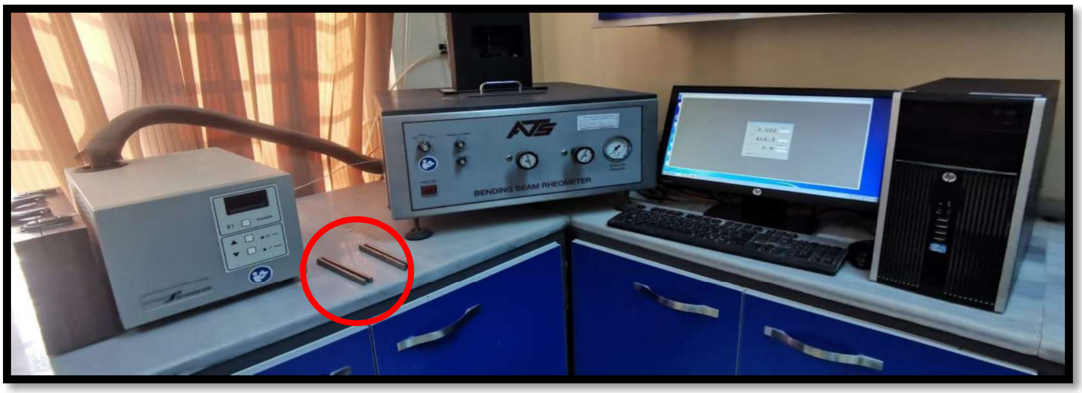
δ: is the phase angle.

### *3.3.5 Bending Beam Rheometer*

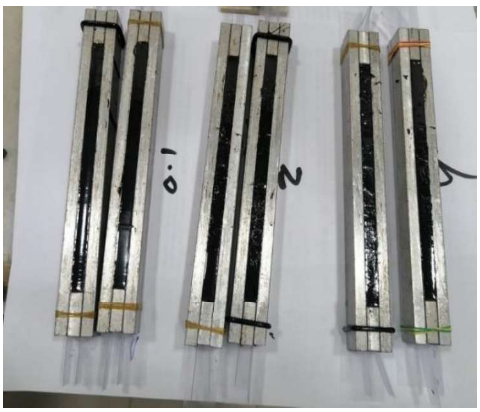
Low-temperature characteristics are linked to a cracking susceptibility. Because asphaltic binders stiffen when they react with ambient oxygen, tests to assess low-temperature characteristics are frequently performed on binders that have undergone an aging or conditioning treatment. To determine the stiffness and relaxation characteristics of the bitumen binder, the bending beam rheometer (BBR) test is utilized. These characteristics indicate an asphalt binder's capability to overcome low -temperature cracking and are used to evaluate the low-temperature performance grade (PG) of an asphalt binder. The BBR technique was used following ASTM D6648. BBR test method requires applying a load equal to 0.98 N on the center of the beam of an asphalt binder, a thin asphalt beam is supported and immersed in a cold liquid bath. The deflection of the beam is evaluated versus time after a load is placed at its center. The BBR assessment is used to establish two key parameters: the creep stiffness of an asphalt binder as a function of the time as well as the slope of the stiffness curve at 60 s (also known as the m-value). The test is primarily handled by software. At different temperatures, the creep stiffness and m-value were determined using the BBR technique: 0 $^{\circ}$C, - 6 $^{\circ}$C, -12 $^{\circ}$C, and -18 $^{\circ}$C. The creep stiffness must have a value of 300 MPa as a maximum, and the m-value must be at a minimum of 0.3, according to the Superpave specification sets. Base and modified binders were all tested to calculate the creep stiffness and m-value. The BBR test is performed on a binder that has undergone both short-term and long-term aging using RTFO and PAV, respectively. Figure 24 shows the used bending beam rheometer in this study.

**Figure 24**

*Bending Beam Rheometer*



*(a) Bending Beam Rheometer*



*(b) Asphalt Binder Beam Samples*

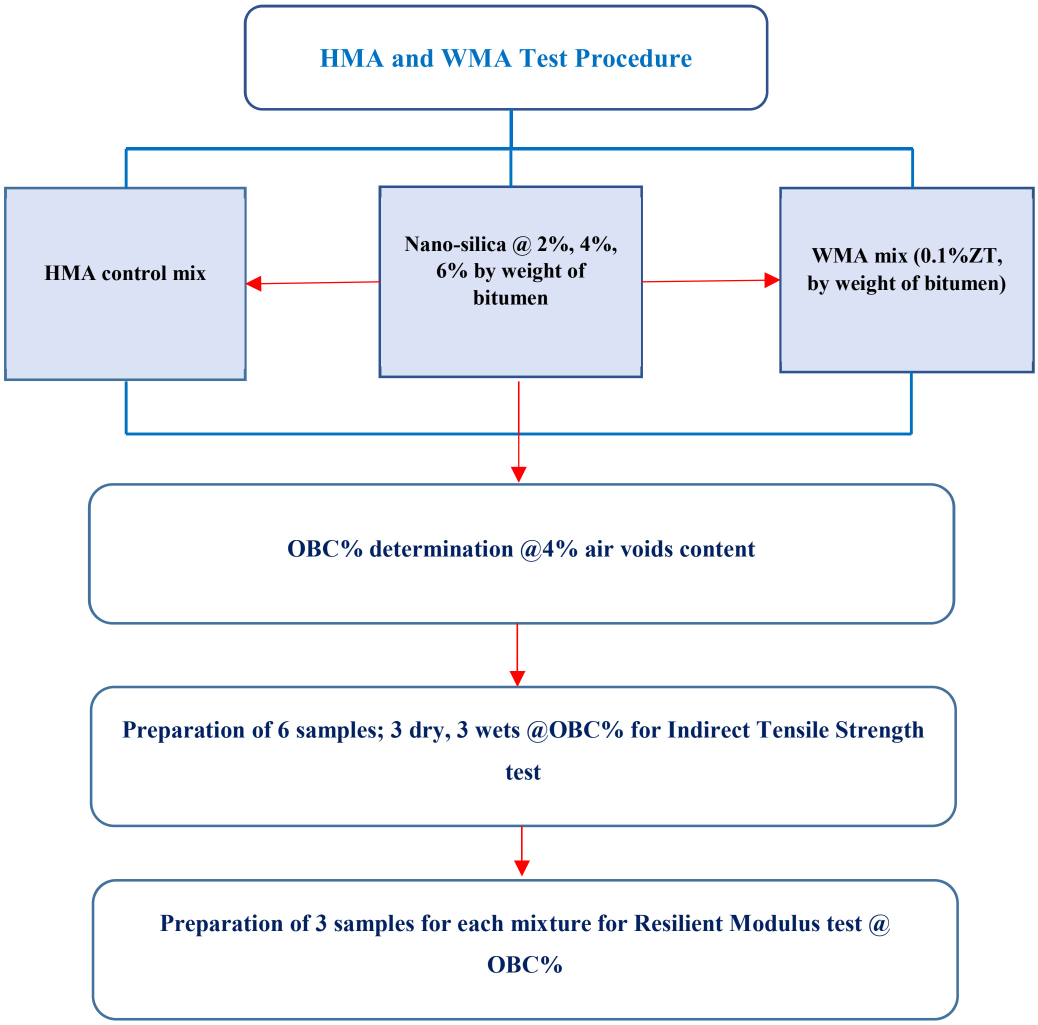
*(c) The Removed Samples after Testing*

### 3.4 Designing of Bitumen Mixtures

One of the objectives of this study is to figure out how Nano-Silica and ZycoTherm affected the mechanical characteristics of asphalt mixes. This section goes into the raw materials needed to prepare the samples, including aggregates. To ensure long-term efficiency in pavement construction, asphalt mix design approaches seek to balance the mixture of aggregate and bitumen. Mineral aggregates, bitumen, as well as air are known to make up the majority of an asphalt mixture. A mix design's major goal is to create mixes that are resistant to distortion and cracking. The physical qualities of the materials used determine the attributes of the resulting mixes. Each one of the materials must be carefully chosen and assessed to ensure that the asphalt mixes and desired performance are met. The control and modified asphalt mixes were designed using the Superpave mix design technique. The Superpave design process is divided into three phases: mineral aggregate and bitumen selection, design of aggregate gradation, and volumetric analysis of the compacted samples with the Superpave Gyratory Compactor. The American Association of State Highway and Transportation Officials (AASHTO) and the American Society for Testing and Materials (ASTM) were used to design the asphalt mixture. The Superpave mix design approach was used to develop all of the asphalt mixes in this study. The Asphalt Institute's (MANUAL SERIES NO. 02 (MS-2) Seventh Edition 2014) Superpave mix design process was utilized as a guide to design various types of mixes. Moreover, the laboratory methods for the tests that were done have been evaluated. In this research, resilient modulus and indirect tensile strength tests were applied. The following sections go over the test method assessments in depth. Figure 25 shows a tree diagram to demonstrate the asphalt mixture experimental design method.

**Figure 25**

*The Asphalt Mixture Experimental Design Method*




### *3.4.1. Aggregate Tests and Gradation*

Aggregate refers to a group of mineral aggregates that includes gravel, sand, and crushed stone. By weight, aggregate makes approximately 90 to 95 percent of asphalt mixtures. According to their size, aggregate in asphalt mixtures can be separated into three types: coarse aggregate, fine aggregate, and mineral filler. The coarse aggregates are those that are retained on a 4.75mm sieve (No.4). Fine aggregates pass through the 4.75 mm but are retained on the 0.075 mm sieve (No.200). Four stockpiles according to aggregate sizes of basalt were used in this research. Figure 26 shows the coarse and fine aggregates used in this study.

**Figure 26**

*Aggregate Types (Coarse and Fine Aggregates)*


Pavement specialists from all over the world have collaborated with the industry to properly define the future of aggregate production by focusing on aggregate characteristics that are critical for long-term road performance. Because aggregate characteristics are so important in overcoming persistent deformation, a lot of work has gone into creating two types of aggregate properties: consensus and source properties. Basic aggregate characteristics such as specific gravity and water absorption should also be established as one of the main aggregate features. The tests which should be done on aggregates to guarantee that they satisfy needed requirements and therefore will result in asphalt mix with the desired performance have been recommended by the Superpave asphalt mix design. The aggregate characteristics listed below should be assessed.

(I) Consensus Properties

The traffic volume and position within the pavement determine the consensus properties criteria. The following are consensus properties:

- Coarse Aggregate Angularity (CAA).
- Fine Aggregate Angularity (FAA).

- Sand Equivalent (SE).
- Flat & Elongated Particles (F &E).

The requirements for these qualities are affected by the volume of traffic and where it is located within the asphalt pavement structure. Materials that are close to the pavement surface (less than 100 mm) are exposed to intense levels of stress and require better-quality materials than mixes further down the pavement structure (more than 100 mm). Because the design traffic (ESALs) in this research is Heavy 10-30 million, and the layer location for design purposes is less than or equal to 100 mm, the following requirements in Table 10 are the Superpave aggregate consensus properties standard requirements.

**Table 10**

*Superpave Criteria for Aggregate Consensus Properties*

| Property | Superpave Criteria | Standard Method |
|---|---|---|
| Coarse Aggregate Angularity (CAA), percent | Min. 95/90 | AASHTO T 335 |
| Fine Aggregate Angularity (FAA), percent | Min .45 | AASHTO T 304 |
| Sand Equivalent (SE), percent | Min .45 | AASHTO T 176 |
| Flat & Elongated Particles (F &E), percent | Max .10 | ASTM D4791 |

(II) Source Properties

Other aggregate features are essential in addition to the consensus aggregate attributes. As a result, a collection of source characteristics is suggested. While these qualities are important to consider throughout the mix design stage, they are as follows:

- Toughness.
- Deleterious materials.

Set limitations on aggregate source attributes were defined in the Superpave mix design system. Certain recommended source attribute assessments and common criteria are shown in Table 11.

**Table 11**

*Superpave Criteria for Aggregate Source Properties*

| Property | Superpave Criteria | Standard Method |
|---|---|---|
| Toughness (Los Angeles Abrasion), percent | Max.30 | AASHTO T 96 |
| Deleterious materials (clay lumps), percent | <2 | AASHTO T 112 |

(III) Basic Properties

Before designing the asphalt mixture, fundamental aggregate characteristics like as specific gravity and water absorption should be evaluated. The following are the commonly recognized categories of aggregates specific gravities that are used in asphalt mix: dry specific gravity ($G_{sb}$) and apparent specific gravity ($G_{sa}$). The basic features that should be determined are as follows:

- Dry Specific Gravity for both Coarse & Fine Aggregate.
- Apparent Specific Gravity for both Coarse & Fine Aggregate.

Dry specific gravity takes into account the aggregate particle's total volume, including all the capillaries (pores) that fill with water following a 15- to 19-hour soaking. The volume of the aggregate is considered by apparent specific gravity to equal the volume of the aggregate. It excludes the volume of any capillaries that fill with water just after a 15- to 19-hour immersion. AASHTO T 85 /ASTM C127 is used to determine the $G_{sb}$ and $G_{sa}$ for coarse aggregate, whereas AASHTO T 84 /ASTM C128 is used to determine the $G_{sb}$ and $G_{sa}$ for fine aggregate. Dry and apparent specific gravities for both coarse and fine aggregate were determined using Eqs. 10-13. Table 12 shows the coarse and fine aggregate properties used in this study.

$$G_{sb} = \frac{A}{B-C} \quad (10)$$

Where;

Gsb: is the dry specific gravity for coarse aggregate

A: is the oven-dry sample’s mass

B: is the saturated surface –dry sample mass in the air

C: is the water-saturated sample mass

$$G_{sb} = \frac{A}{B+D-C} \quad (11)$$

Where;

$G_{sb}$: is the dry specific gravity for fine aggregate

A: is the oven-dry sample's mass

B: is the saturated surface –dry sample mass in the air

C: is the mass of the flask with the sample and water to the mark

D: is the flask's mass filled with water

$$G_{sa} = \frac{A}{A-C} \quad (12)$$

Where;

$G_{sa}$: is the apparent specific gravity for coarse aggregate

A: is the oven-dry sample's mass

C: is the water-saturated sample mass

$$G_{sa} = \frac{A}{A+D-C} \quad (13)$$

Where;

$G_{sa}$: is the apparent specific gravity for fine aggregate.

A: is the oven-dry sample's mass.

C: is the mass of the flask with the sample and water to the mark.

D: is the flask's mass filled with water.

**Table 12**

*Physical Properties of Basalt Aggregates*

| Test | Aggregates (hot bins) | | | | Superpave Criteria | Method |
|---|---|---|---|---|---|---|
| | Agg (1) | Agg (2) | Agg (3) | Agg (4) | | |
| Bulk Specific Gravity ($G_{sb}$) | 2.75 | 2.72 | 2.70 | NA | NA | AASHTO T 85 /ASTM C127<br>AASHTO T 84 /ASTM C128 |
| Apparent Specific Gravity ($G_{sa}$) | 2.79 | 2.76 | 2.74 | 2.71 | NA | AASHTO T 85 /ASTM C127<br>AASHTO T 84 /ASTM C128 |
| Consensus Properties | | | | | | |
| CAA (%) | 98/97 | 97/95 | NA | NA | Min. 95/90 | AASHTO T 335 |
| FAA (%) | NA | NA | 50 | 48.7 | Min .45 | AASHTO T 304 |
| F&E (%) | 3.21 | 3.14 | NA | NA | Max .10 | ASTM D 4791 |
| SE (%) | NA | NA | 72 | 73 | Min .45 | AASHTO T 176 |
| Source Properties | | | | | | |
| L.A. Abrasion (%) | 24.6 | 25.0 | 27.5 | NA | Max.30 | AASHTO T 96 |
| Deleterious materials (%) | Free | Free | Free | Free | <2 | AASHTO T 112 |

The gradation of the aggregate has been one of the variables which must be properly addressed in the construction of asphalt mixture. The objective of developing and regulating aggregate gradation is to ensure that there are enough voids in the binder-aggregate mix to satisfy the correct asphalt layer thickness upon every aggregate particle. Superpave requirements for aggregate gradations with a nominal maximum aggregate size of 19 mm were used to choose the aggregate gradation. Moreover, the design of the aggregate blend was achieved by using Eq.14. Table 13 shows the criteria for Superpave grading and Figure 27 shows the used aggregate gradation in this study according to AASHTO M 323.

$$P = [A*a] + [B*b] + [C*c] + [D*d] \qquad (14)$$

Where;

P: is the combined passing percentage for a particular sieve.

A, B, C, and D: the percentage passing through a specific sieve for each individual aggregate (stockpile).

a, b, c, and d: the proportion for each aggregate to be added to the design blend.

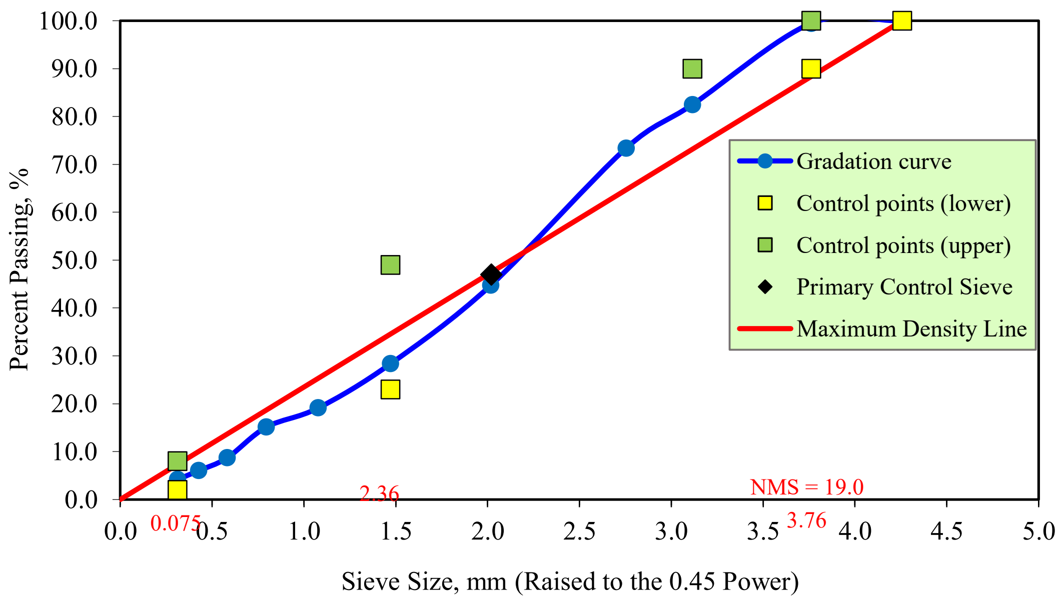
**Table 13**

*Gradation of Superpave Specifications for a Nominal Maximum Size of 19 mm*

| Sieve No. | Sieve (mm) | Min. (% passing) | Max. (% passing) |
|---|---|---|---|
| 1" | 25 | 100 | --- |
| 3/4 | 19 | 90 | 100 |
| 1/2" | 12.5 | --- | 90 |
| 3/8" | 9.5 | --- | --- |
| No.4 | 4.75 | --- | --- |
| No.8 | 2.36 | 23 | 49 |
| No.16 | 1.18 | --- | --- |
| No. 30 | 0.6 | --- | --- |
| No.50 | 0.3 | --- | --- |
| No.100 | 0.15 | --- | --- |
| No.200 | 0.075 | 2 | 8 |

**Figure 27**

*The Target Aggregate Gradation*



### *3.4.2 Bitumen Binder Type*

Bitumen serves as adhesive, binding aggregate particles together. Temperature and loading time impact the viscoelastic properties of bitumen binder. The type and source of base bitumen, as well as the modifier types, were previously discussed in section 3.1 while the bitumen physical and rheological tests were discussed in section 3.3.

### *3.4.3 Mixing and Compaction Temperatures*

Regarding laboratory mixing and compaction temperatures, asphalt mixture design techniques have utilized equiviscous temperature ranges. The viscosity of the bitumen binder was measured using rotational viscometer (RV) at three different test temperatures 135 ˚C, 165 ˚C, and 195 ˚C, demonstrating a connection between temperature and viscosity. The rotational viscometer test is used to measure a bitumen's workability throughout the mixing and compaction phases. At which the viscosity-temperature line passes the compaction viscosity limit of 0.28 $\pm$ 0.03 Pa.s, compaction temperatures were found. Where the viscosity-temperature line passes the mixing viscosity limit of 0.17 $\pm$ 0.02 Pa.s, mixing temperatures were found. For base bitumen, this approach currently performs well. In the scenario of using modified bitumen in the mixtures, the mixing and compaction temperatures may be too significant due to the higher viscosity of the modified bitumen, which is undesirable in terms of energy usage during the production phase.

### *3.4.4 Designing the Bitumen Content and Mixing Process for Asphalt Mixtures*

To achieve 4% air voids, lab specimens were compacted at varying bitumen contents. Two bitumen binder contents were chosen to be above the predicted optimum with two different levels, one being higher than the expected with 0.5 and the other being 1.0, Pb+0.5 and Pb+1, respectively. The other bitumen content was chosen to be less than the predicted optimum Pb-0.5. Control HMA specimens with four different bitumen contents were prepared, with three duplicates for each bitumen content. Three replicates weighing about 4700 g of aggregate were prepared at every testing bitumen content. The aggregates and asphalt were preheated in the oven for around 4 hours at a mixing temperature. The wire whips and spatulas, as well as the mixing bowl were preheated to the mixing temperature. When the aggregate and asphalt were reached the appropriate mixing temperature, they were immediately taken from the oven and combined in the bowl that is fixed in the mechanical mixer until the aggregate had gained a uniform and full coating. The procedure of determining the optimal asphalt content for HMA samples is long, beginning with the selection of an estimated or predicted optimum bitumen and ending with the determination of the specified bitumen content. Four bitumen contents were selected based on the estimated optimal bitumen content. The estimated optimum content was 4.7%. The other asphalt content was: 4.7%-0.5, 4.7%+0.5and 4.7%+1.0. The procedure for determining optimum asphalt content for control WMA was the same as for HMA.

Finally, several procedures should be completed to determine the desired optimal asphalt content for the mixes, such as determining the volumetric parameters at N design (the design number of gyration) at each bitumen binder level. The next sections will go over these processes in depth.

### *3.4.5 Compaction Procedure with Superpave Gyratory Compactor*

After mixing the necessary number of specimens with various bitumen contents, the mixture was placed in the oven for two hours at a temperature equivalent to the mixture's defined compaction temperature. According to AASHTO R 35, laboratory compaction of asphalt mixes was done at two distinct levels of gyration based on predicted traffic demand. These levels, known as $N_{ini}$ and $N_{des}$, relate to the anticipated number of gyrations required to achieve various levels of field densification. Table 14 shows the Superpave Gyration Numbers (Compaction Effort) based on the design used in this study (20 -year design ESALs).

**Table 14**

*Superpave Gyrations at Different Designate Levels*

| 20-Year Design ESALs (millions) | N(initial) No. of Gyration | N(design) No. of Gyration |
|---|---|---|
| 3 to <30 | 8 | 100 |

All the asphalt mixes were compacted by a Superpave Gyratory Compactor (SGC) with 600 kPa compaction pressure and the speed of gyration was set to be 30 rpm, Figure 28 shows the used SGC in this research. For volumetric samples compaction as stated in Table 14, the SGC was adjusted to the correct number of gyrations at ($N_{in}$ and $N_{des}$). The molds were placed in an oven at the appropriate compaction temperature for each mix about 60 minutes before the compaction of the first sample. After that the mold was removed from the oven and the lower plate was covered with a filter paper (to avoid the mixture from sticking). Then, the conditioning mixture at the compaction temperature was placed inside the mold and the top surface of the mix was leveled and covered with a filter paper. In the final step of compaction, after completing the prescribed number of gyrations the compacted sample in the mold was removed from the SGC, and carefully the sample was released from the mold. The compacted specimens had a diameter of 150 mm and a height of 115±5 mm.

**Figure 28**

*Superpave Gyratory Compactor*

*(a) Superpave Gyratory Compactor* *(b) Compacted Sample*

The bulk specific gravity ($G_{mb}$) and the theoretical maximum specific gravity ($G_{mm}$) tests were performed on compacted samples and loose mixes (not compacted), respectively. The SSD technique should be used to estimate the bulk specific gravity of all compacted mixes, as per AASHTO T 166 or ASTM D2726. Weighing the sample in air and water is part of the bulk specific gravity measurement. The mass of the saturated surface dry sample in the air was determined in the last stage. By rapidly blotting the specimen with a moist cloth to dry the surface without removing the water from the surface gaps (voids), the saturated surface dry (SSD) mass was determined. For determining $G_{mb}$ Eq. 15 was applied.

ASTM D2041 and AASHTO T 209 describe the theoretical maximum specific gravity. To determine the maximum theoretical specific gravity, two specimens were prepared. These specimens are un-compacted, and their size is determined by the mixture's maximum aggregate size, the used samples weighed 2500 g. $G_{mm}$ samples were combined with a certain amount of bitumen binder. Following the mixing procedure, the loose specimen was spread on a piece of paper to separate the particles from one another. The mixture was then stirred to get cool to avoid clumping. The mixture was then allowed to cool to room temperature before the test. In the following step, the mass of the flask while immersed in water was determined while the water was kept at 25±1 ˚C. The empty flask was then placed on the scale, and the specimen was then added (the mass of the dry specimen was recorded). To thoroughly cover the specimen, an amount of water was added. The flask was attached to the vacuum for 14±1 min to remove the trapped air, and it was shaken every 2 minutes to assist release the air bubbles. After the vacuum procedure was completed, the flask and specimen were submerged in water for 10 minutes at a temperature of 25±1 ˚C (the mass of the flask and specimen in water were determined). For determining $G_{mm}$ Eq. 16 was utilized. Figure 29 shows some of the compacted samples and Figure 30 shows the loose sample.

$$G_{mb} = \frac{A}{B-C} \qquad (15)$$

Where;

A: the dry sample's mass in the air.

B: the saturated surface –dry sample mass in air.

C: the water-saturated sample mass at 25 ˚C.

$$G_{mm} = \frac{A}{(A-(C-B))} \qquad (16)$$

Where:

A: the specimen dry mass, g.

B: the mass of the immersed flask in water, g.

C: the mass of the immersed flask and sample in water, g.

**Figure 29**

*Compacted samples*


**Figure 30**

*Loose Mixture Sample*

### *3.4.6 Volumetric Characteristics of the Compacted Samples*

The volumetric characteristics of a compacted pavement mix are critical criterion for determining the performance of an asphalt pavement mixture. Following the compaction, volumetric parameters for the mixes were obtained. These parameters are as follows and they were determined using the Eqs. 17-19.

- Air voids (%) (Va).
- Voids in the mineral aggregate (VMA).

- Voids filled with asphalt (VFA).

$$Va = 100 - \frac{100 \times Gmb}{Gmm} \quad (17)$$

Where;

Va: the voids of air in a compacted mix.

$G_{mb}$: the bulk S.G of the compacted sample.

$G_{mm}$: the maximum S.G of the loose sample.

$$VMA = 100 - \frac{Gmb \times Ps}{Gsb} \quad (18)$$

Where;

Ps: the aggregate as a proportion of the overall weight of the mix.

$G_{sb}$: is the dry S.G of the aggregate blend.

$$VFA = 100 \times \frac{VMA - Va}{VMA} \quad (19)$$

Where;

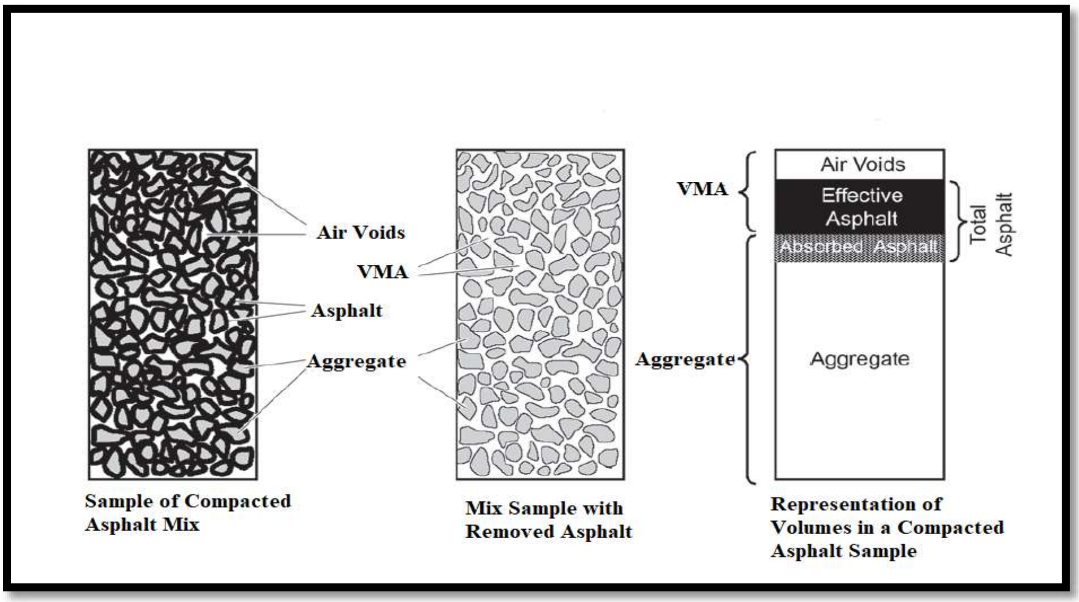
VMA: is the voids in the mineral aggregate.

Va: is the voids of air in a compacted mix.

In a compacted mixture, the volume of air voids refers to % of air voids. The mix gets less permeable as the air spaces decrease. A too extremely high void content creates pathways through the mix, allowing undesirable air and water to enter. Rutting and bleeding might occur if the air void content becomes too low. For a design that optimizes the necessary performance attributes, 4% air voids are usually regarded the optimum as estimate. VMA is considered as the inter - granular voids between mineral aggregates in a compacted pavement mix, including air voids and effective (non-absorbed) bitumen binder, expressed as a proportion of overall volume. The VMA value must be kept high enough to maintain a sufficient asphalt layer thickness, resulting in a long-lasting pavement surface. Asphalt mixes with VMA values below the threshold will have thin asphalt layers and create a low-durability pavement, Figure 31 shows the air voids in compacted mixtures.

**Figure 31**

*Voids in a Compacted Asphalt Mixture*




(Asphalt Institute's Manual, 2014)

The proportion of the VMA which is contained or filled with the effective binder is termed voids filled with asphalt (VFA). VFA is included in the mix to guarantee that the asphalt film thickness is adequate. The mixture will have insufficient endurance if the VFA value is too low, and it will be unstable if it's too high. The permissible VFA values ranged based on the volume of traffic. The dust to binder ratio (DP) is another crucial metric to specify for an asphalt mixture. Is defined as the proportion of aggregate that passes through 0.075-mm sieve to the effective binder as shown in Eq. 20. This characteristic is concerned with the workability of asphalt mixes in particular. A low value of dust to binder ratio tends to move underneath the roller and this typically results in a delicate mix that lacks cohesiveness and is hard to compact in the field. Since the design traffic utilized in this study is10< 30 million and the NMAS is 19 mm, Table 15 indicates the Superpave mixture needs based on traffic volume and NMAS.

$$DP = \frac{P_{0.075}}{P_{be}} \tag{20}$$

Where:

$P_{0.075}$: % of aggregate passing sieve No.200 (0.075mm).

$P_{be}$: is the effective binder.

**Table 15**

*Superpave Mix Specification*

| 20-Year Design ESALs (millions) | VMA, (Percent) Minimum | VFA Ranges, (Percent) | DP Ranges |
|---|---|---|---|
| 10<30 | 13.0 | 65-75 | 0.6-1.2 |

## 3.5 Performance Testing of Asphalt Mixtures

The experimental procedure was separated into two sections that assist to understand and anticipate the characteristics of asphalt binder and mixtures. Based on a comparison of modified and base binders, the first stage mixed additives with a bitumen binder to determine the improvement in bitumen binder characteristics. In the second step, base bitumen was mixed with aggregate after being modified with additives. The developed mixes were subjected to a thorough laboratory assessment. As a result, a series of mechanistic studies were conducted to investigate how asphalt mixes behaved under varied loading and environmental circumstances. The following mechanical tests were used to evaluate asphalt mixtures: indirect tensile strength (ITS), and resilient modulus ($M_R$).

### *3.5.1 Indirect Tensile Strength*

Moisture damage, which takes place as a result of a deficit of bonding between the bitumen and aggregate, can cause a variety of integrity issues, including potholing, stripping, and a variety of other deteriorations like fatigue and rutting (Haghshenas, et al., 2018; Sobhi, et al., 2020). The serviceability of asphalt pavements will be reduced as a consequence of moisture damage, and maintenance costs will rise (Ameri, et al., 2021).

There are several techniques for determining a mix's moisture sensitivity. The indirect tensile strength test was conducted in this research to evaluate the moisture sensitivity of an asphalt mix. The AASHTO T-283 test technique was used to assess the stripping resistance of asphalt mixtures using the Lottman test. Six samples were compacted to a height of 95±5 mm at a design bitumen content, 3700 g of aggregate was required to achieve the requirements of this test. The six specimens were compacted at 7% of air voids, according to the test protocol. AASHTO T 283 sample preparation limits of ± 0.5% air spaces are recommended by the Asphalt Institute. After that, the compacted specimens were divided into two sets: conditioned and unconditioned. It's critical to ensure that the two groups have similar characteristics and that the variation of air void values in each group is generally ± 0.2%. For the moisture condition group (condition state), three compacted samples were saturated by immersing them in water in the vacuum container until at least 25 mm of water is covered the specimens and applying a partial vacuum of 525 mm Hg for 5 to 10 min. The saturation level for the conditioned samples was then measured by measuring the volume of absorbed water. The specimen saturation level of 70% to 80% is recommended by AASHTO T 283. The partially soaked samples were then immersed in a water bath at 60 ±1 ˚C for 24 ±1 h. Lastly, the partially soaked samples were immersed in a water bath at 25 ˚C for two hours to reach the required assessment temperature. Samples in the unconditioned subgroup are kept at room temperature just before the indirect tensile strength test begins. A water bath was used to adapt the unconditioned group of samples to 25 ˚C for 2 hours directly before assessment. Figure 32 shows the prepared samples for the ITS test.

**Figure 32**

*Base and Modified Samples for ITS*


The conditioned and unconditioned specimens were subjected to indirect tensile strength measurement. Figure 33 shows the loaded sample into the loading device. The specimen was diametral loaded at a rate of 50 mm/min till it achieved its maximum load.

**Figure 33**

*ITS Loading Apparatus*

*(a) Lottman Breaking Head* *(b) Load Frame*

For every sample, the indirect tensile strength and tensile strength ratio were computed using the formulas below:

$$\text{ITS} = \frac{2000 \times P}{\pi t D} \quad (21)$$

Where;

ITS: denotes as the indirect tensile strength, kPa.

P: denotes as the maximum load, N.

t: is the sample thickness just before the tensile strength test, mm.

D: denotes as the sample diameter, mm.

$$\text{TSR} = \frac{S2}{S1} \quad (22)$$

Where;

TSR: is the tensile strength ratio.

S1: is the average tensile strength of the dry subset, kPa.

S2: is the average tensile strength of the conditioned subset, kPa.

### *3.5.2 Resilient Modulus ($M_R$)*

The resilient modulus of the bituminous mix indicates the stiffness of the used materials. The resilient modulus values may be utilized to assess the quality of materials and to create information for pavement design or assessment and analysis. The resilient modulus test was performed as a nondestructive test on asphalt mixtures.

To conduct the experiment based on ASTM D4123, three samples for each mix were prepared at 25 ˚C. A diametrical haversine loading with 1000 N was applied to the asphalt mixture samples at a 1 Hz frequency. The samples should have a minimum thickness of 51mm and a minimum diameter of 102 mm for a 25 mm maximum aggregate size. The samples in this study had a diameter of 150 mm and a height of 83 mm as shown in Figure 34. Before testing, the test samples were placed in a controlled cabinet at the required test temperature for a minimum of 24 hours. Start the computer and the associated software to collect and analyze the data gathered during the test. In the software, the specimen identifiers were entered (thickness and diameter). Following that, the sample was placed in the loading apparatus as shown in Figure 35 and a repeated haversine load was applied to the sample. The resilient modulus in MPa is computed using Eq. 23.

$$M_R = \frac{P(\nu+0.2734)}{tH} \tag{23}$$

Where;

P: the maximum load in (N).

$\nu$ : Poisson ratio.

t: the thickness of the sample (mm).

H: reversible deformation (mm).

**Figure 34**

*$M_R$ Samples*

**Figure 35**

*Sample Position for Resilient Modulus Test*


The resilient modulus is a based on the mathematical estimate of the pavement response to traffic loading that may be expressed as the ratio of repeated applied stress to the associated recovered strain after a specific number of loading cycles .The higher the modulus of resilience of bituminous mixtures, the greater the expected performance of the pavement at a given temperature, because the mix can recover after releasing the imposed stresses (Enieb & Diab, 2017).

**3.6 Summary**

The asphalt binders utilized in the study, as well as their conditioning methods, are explained in this chapter. The different tests on bitumen binder and mixes that were done as part of the research, as well as the test circumstances, are described. This chapter also includes a summary of the testing procedures. Furthermore, each test was repeated at least three times, then average results were recorded to enhance the validity of the data obtained. Tables 16 and 17 represent the number of experiments performed on mixes and bitumen binder (testing matrix), respectively.

**Table 16**

*Testing Matrix for Bituminous Mixtures*

| Variable | | Number | Total number of samples |
|---|---|---|---|
| Aggregate type: Basalt | | 1 | 72 samples |
| Test | ITS and $M_R$ | 2 | |
| Temperature | 25°C | 1 | |
| Samples | Base bitumen (60/70)<br>Base bitumen + (2, 4, and 6% NS)<br>Base bitumen +0.1% ZT<br>0.1%ZT+ (2, 4, and 6% NS) | 8 | |
| Replicates | 3 samples ITS $_{Dry}$<br>3 samples ITS $_{Wet}$<br>3 samples $_{MR}$ | 3<br>3<br>3 | |

**Table 17**

*Testing Matrix for Bitumen Binders*

| Bitumen type | | Number | Total number of samples |
|---|---|---|---|
| 60/70 | | 1 | 8 |
| Base bitumen + NS (2, 4, and 6%)<br>Base bitumen +0.1%ZT<br>0.1%ZT+ NS (2, 4, and 6%) | | 3<br>1<br>3 | |
| **Tests** | | | **Total number of samples** |
| (I) Conventional | Penetration<br>Softening point<br>Rotational viscosity | 3<br>3<br>3 | 240 |
| (II)Temperature Sensitivity | Storage stability | 3 | |

**Table 17** (Continued).

| | | Unaged | RTFO-aged | Pav-aged | |
|---|---|---|---|---|---|
| (III) Rheology | | | | | |
| | *G**/sin *δ* | 3 | 3 | ---- | 6 |
| | MSCR | ----- | 3 | ---- | 3 |
| | Frequency sweep | 3 | ------ | ---- | 3 |
| | *G**.sin *δ* | ------- | ------ | 3 | 3 |
| | BBR | ------ | ------ | 3 | 3 |

# CHAPTER IV
# Results and Discussion

This chapter discusses the influence of Nano-Silica on the physical and rheological properties of base bitumen and ZycoTherm modified binders. The dynamic shear rheometer was utilized to evaluate the effect of adding Nano-Silica on rheological characteristics using frequency sweep test under unaged condition and to evaluate the rutting potential of the base and modified binders using the Superpave rutting resistance parameter and MSCR under unaged and short-term aging (RTFO-aged) conditions. Moreover, it discusses the evaluation of the fatigue resistance of the base and modified binders utilizing DSR under long-term aging conditions (PAV-aged). The BBR was used to evaluate the low-temperature performance of bitumen binders containing ZycoTherm modified with different percentages of Nano-Silica. The Asphalt mixes were prepared using the Superpave mix design method. The optimal bitumen binder contents (OBC) for both HMA and WMA mixes were identified. All of the volumetric properties were determined in order to meet the standard limitations for the design of a bituminous wearing course. Furthermore, the moisture susceptibility of asphalt mixtures was evaluated using indirect tensile strength (ITS), and a resilient modulus ($M_R$) test was performed on asphalt mixtures since it is used as an indicator of the stiffness of the mixtures.

## 4.1 Physical Properties

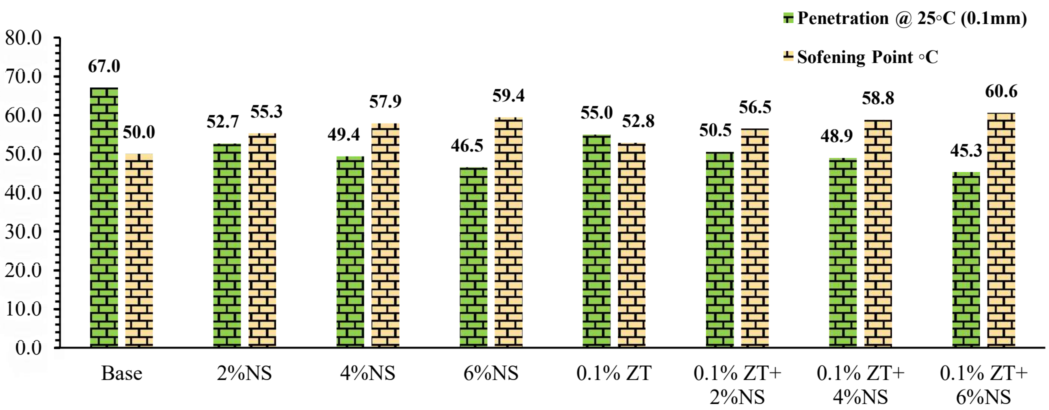
The physical characteristics of the base and modified binders were assessed using the testing techniques such as penetration, softening point, and viscosity. The temperature sensitivity of the binders was investigated. Moreover, the storage stability of the bitumen binders was evaluated using the softening point test.

### *4.1.1 Penetration and Softening Point*

The penetration and softening point of the bitumen used in road construction are the most essential characteristics. The bitumen penetration test determines the stiffness or softness of the bitumen. The consistency is determined by the penetration result obtained from the bitumen penetration test. Therefore, this test is used to categorize penetration grade bitumen into typical penetration ranges. Softening point is an empirical test that determines the temperature at which the bitumen phase transitions from semi-solid to liquid. Generally, a bitumen specimen with a lower penetration has a higher softening point. Figure 36 shows the effect of adding Nano-Silica to the base and ZycoTherm binders. The penetration decreases while the softening point increases. In comparison to the base and ZycoTherm modified binders, each Nano-modified bitumen sample showed reduction in penetration and an increment in softening point. The reduction in penetration values indicate the stiffening effect of the binder, whereby the stiffening increases in line with the increased Nano-Silica concentration. The increased stiffness was a desirable attribute for bitumen binder since its improved resistance to rutting failures at high temperatures. A decrease in bitumen penetration value might result in improved adhesive bonding between the bitumen and the aggregates, resulting in more durable bituminous mix (Pasandín & Pérez, 2014).

**Figure 36**

*Penetration and Softening Point of Base and Modified Bitumen Binders*



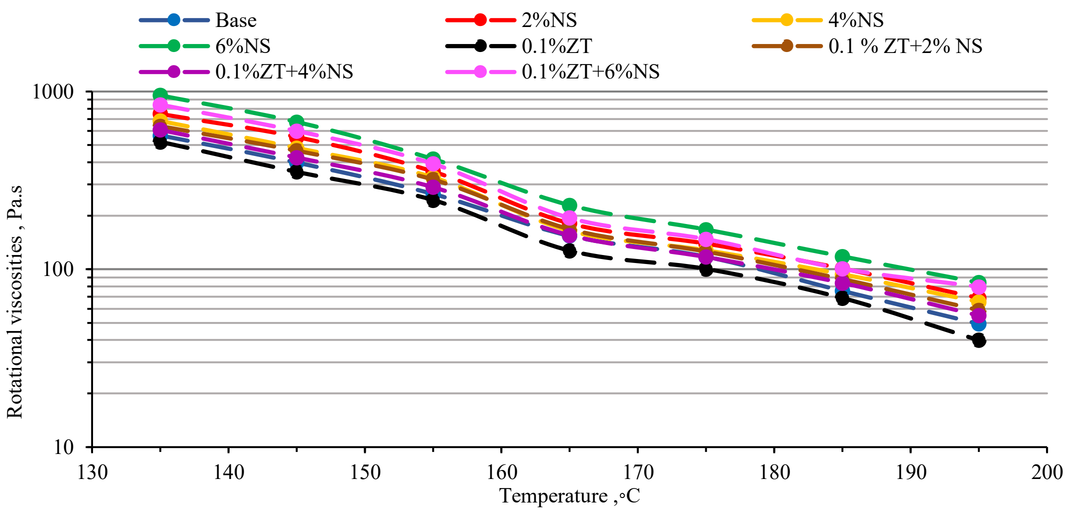

### *4.1.2 Rotational viscosity*

The viscosity is another physical attribute of a fluid, which is a measurement of the resistance to flow. The viscosity test ensures that the asphalt binder has enough pump-ability, mix-ability, and workability. The capability to pump bitumen between storage tanks or into a bitumen producing facility for hot mixes is referred to as pump-ability. The ability of bitumen to be correctly mixed in the manufacturing facility with aggregates or other bitumen constituents of the hot mix is referred to as mix-ability. Workability refers to the ability to apply and compact hot mix asphalt in the field with acceptable effort (Jadidirendi, 2017). As demonstrated in Figure 37, all of the bitumen binder specimens had low viscosity values at elevated temperatures. The addition of

Nano-Silica to the base binder causes the increment of the viscosity of the Nano-Silica modified binders. Furthermore, according to the Superpave criteria, at 135 ˚C the viscosity values were within the limits. However, regarding to the 4% NS, chemical changes and physical diffusions may occur during the process of mixing the Nano-Silica modified binders, resulting in the development of a new structure and a reduction in viscosity as compared to other percentages. Additionally, when stress is applied, Nano-Silica can strengthen the base binder and improve the recovery capabilities. However, when Nano-Silica was added to ZycoTherm binder, the viscosity was decreased. The viscosity lowering effect of ZycoTherm as a modifier binder is substantial. ZycoTherm is a useful characteristic for an addition, especially when assessing its efficacy because it lowers operating temperatures, which assists in the development of potentially cost-effective and sustainable pavements. Greater viscosities lead to a poor workable HMA because of greater stiffness, which leads to increased energy usage in the manufacturing plant due to higher mixing temperatures, as well as more effort required to compact the asphalt mix. As a result, the ZycoTherm resulted in a reduction in viscosity values, for example; a viscosity reduction of roughly 11.5% and 15% at 135 and 165 ˚C, respectively, in the binder containing both 0.1% ZycoTherm and 6%NS. Furthermore, low viscosity in the bitumen is beneficial since it helps the bitumen in the WMA mixture to effectively coat the aggregate surface (Sanij, et al., 2019). As a result, when ZycoTherm is in a liquid state, it is one of the factors that led to decrease the viscosity (Raufi, et al., 2020).

**Figure 37**

*The Viscosity of Base and Modified Bitumen Binders*



### *4.1.3 Temperature Susceptibility*

The relationship between penetration value and softening point temperature is utilized to assess the temperature susceptibility of bitumen binder (Penetration Index, PI). Eq.5 shows the formula for calculating the PI for bitumen. PI is a technique for calculating the temperature susceptibility of bitumen. The PI value for bitumen ranges from (-3) to (+7), with lower values suggesting more temperature-sensitive bitumen and higher values indicating less temperature-sensitive bitumen. According to Table 18, the addition of Nano-Silica stiffened the base and ZycoTherm binders, with maximum and minimum PI values of 0.900 and -0.495, respectively. Increased PI values indicate a drop-in penetration value and an increase in softening point. Furthermore, the higher the penetration index value, the lower the temperature susceptibility of the bitumen binder indicates greater resistance to low-temperature cracking as well as rutting deformation especially in summer months (Ghasemi & Marandi, 2013). Because all of the PI values in this study are between +1 and -1, all of the Nano-Silica percentages used in this study to modify both the base and ZycoTherm binders are suitable for road construction (Arshad, et al., 2016).

**Table 18**

*PI of Base and Modified Bitumen Binders*

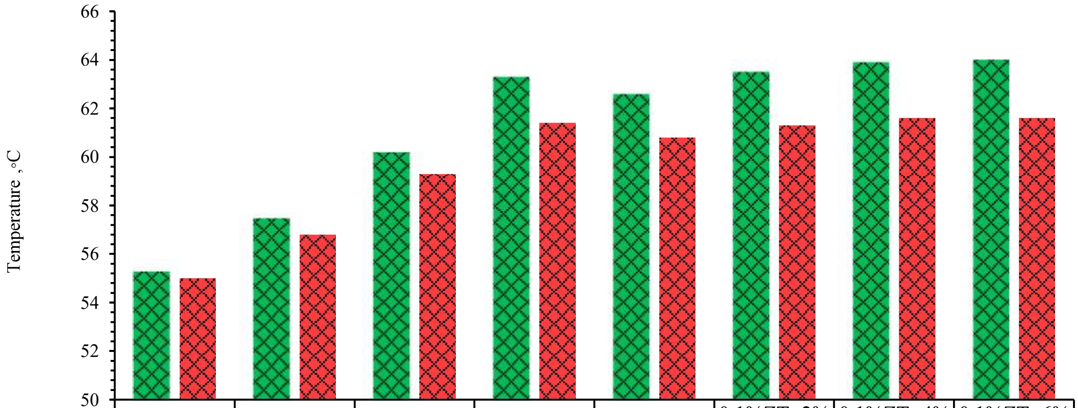
| Binder | Base | 2 % NS | 4 % NS | 6 % NS | 0.1 % ZT | 0.1 % ZT + 2 % NS | 0.1 % ZT + 4 % NS | 0.1 % ZT + 6 % NS |
|---|---|---|---|---|---|---|---|---|
| PI | -0.495 | 0.165 | 0.555 | 0.727 | -0.296 | 0.350 | 0.719 | 0.900 |

### *4.1.4 Storage Stability*

The phase separation or storage stability test was performed to determine the compatibility of the bitumen and the modifier, considering this is one of the primary indications showing the efficacy of the bitumen modification process. Storage stability was conducted to determine the modifier's stability and suitability with a bitumen binder to store at high temperatures. To assess storage stability, the difference in softening points between the top and bottom parts of bitumen specimens taken from aluminium foil tubes was determined, the disparity in softening points should not be greater than 2.5 ˚C (Zani, et al., 2017). The softening points of the base and the modified binders are shown in Figure 38.

**Figure 38**

*The Storage Stability of the Base and Modified Bitumen Binders*




| | base | 2%NS | 4%NS | 6%NS | 0.1%ZT | 0.1%ZT+2% NS | 0.1%ZT+4% NS | 0.1%ZT+6% NS |
|---|---|---|---|---|---|---|---|---|
| Top | 55.3 | 57.5 | 60.2 | 63.3 | 62.6 | 63.5 | 63.9 | 64 |
| Bottom | 55 | 56.8 | 59.3 | 61.4 | 60.8 | 61.3 | 61.6 | 61.6 |

When compared to base bitumen, the storage stability of Nano-Silica modified samples was increased at all Nano-Silica concentrations, which is consistent with other findings (Bhat & Mir, 2019). Concerns have been raised about the usage of liquid additives in bitumen, such as their limited life span and tendency to separate from bitumen during storage (Rubio, et al., 2012). As a result, the storage stability of ZycoTherm binder was assessed. The stability of bitumen samples with 0.1% ZycoTherm was acceptable. The addition of Nano-Silica to ZycoTherm binders increased storage stability values. However, the variation in softening point between the upper and bottom parts was below 2.5 °C for all binders, as shown in the Figure 38, demonstrating that all these binders able to kept at high temperatures and remain stable.

## 4.2 Rheological Characteristics

Bitumen rheology may be described generally as the essential measures relating to bitumen flow as well as deformation properties (Taylor & Airey, 2015). Bitumen binder is a viscoelastic, thermoplastic, complicated substance that changes behaviour depending on temperature and loading time. At elevated temperatures and/or at slow moving loads, it is entirely viscous; under these conditions, bitumen becomes susceptible to rutting. Bitumen is also completely elastic and subsequently cracking at low temperatures and/or high rapid moving, and as a result, bitumen becomes susceptible to low- temperature cracking distress. Furthermore, the predominant mechanism of distress is fatigue cracking at in-service asphalt pavement temperatures ranging from 10 to 35 °C, when the asphalt pavement is subjected to a significant portion of its repeated traffic loads (Subhy, 2017). The DSR is commonly used to characterize the viscosity and elastic characteristics of binders across a large temperature range. For base and modified binders, $G^*/\sin\delta$, MSCR, Frequency sweep test, and $G^*.\sin\delta$ were evaluated. Furthermore, the BBR test was utilized to evaluate the low temperature cracking for binders.

### *4.2.1 Superpave Rutting Resistance Parameter*

The Superpave rutting parameter ($G^*/\sin\delta$) was evaluated at various temperatures to evaluate the improvement in the binder's rutting resistance following the addition of Nano-Silica to base and ZycoTherm binders, as shown in Figure 39. The determined $G^*/\sin\delta$ parameter for unaged binders is shown in Figure 39 a. Nano-Silica modified binders have greater $G^*/\sin\delta$ values than base and ZycoTherm binders. Moreover, as compared to the base bitumen at 70 °C, the ZycoTherm modified binder improved potential rutting resistance by 8.14%. Previous research showed similar results, demonstrating that the addition of ZycoTherm has an effect on rutting resistance (Ibrahim & Mehan, 2015; Mirzababaei, et al., 2017), whereas this value increased considerably with the inclusion of Nano-Silica at all percentages. The addition of Nano-Silica to base and ZycoTherm binders resulted in significant improvement levels at all temperatures, according to the findings of the modified binders.

The $G^*/\sin\delta$ parameter for RTFO-aged binder is represented in Figure 39 b; $G^*/\sin\delta \geq 2.2$ kPa is used to assess the binder's rutting performance. All Nano-Silica modified binders had a higher $G^*/\sin\delta$ value than the base and ZycoTherm modified binders, which is comparable to the unaged state. The highest $G^*/\sin\delta$ value was found in base and ZycoTherm binders containing up to 6% NS by weight of the bitumen, followed by binders containing 2% NS, while the lowest $G^*/\sin\delta$ value was found in binders containing 4% NS. Meanwhile, 4% NS has the lowest $G^*/\sin\delta$ despite passing the Superpave criteria under unaged and RTFO aged conditions. It's possible that the chemical reactions of Nano-Silica were to cause (Alhamali, et al., 2016). It is essential to note that the Superpave rutting parameter is affected by the stiffness of the bitumen binder. The inclusion of Nano-Silica stiffens bitumen binders, which is beneficial for rutting. The improved rutting is due to Nano-Silica dispersion inside the bitumen binder, where the Nano-Silica particles adhere to the binder's surface, resulting in a new Nano-Silica modified binder network structure. This strengthened structure is capable of absorbing and transmitting additional load to the bitumen binder, resulting in greater rutting resistance (Yao, et al., 2013). The results reveal that both base and ZycoTherm binders have a high-performance grade (PG) at 64 °C. The highest temperature at which $G^*/\sin\delta \geq 1.0$ kPa is defined as high–temperature performance grade. The $G^*/\sin\delta$ findings at various temperatures demonstrate that adding Nano-Silica to the base and ZycoTherm binders at any concentration (2 –6%) improves the binder's performance grade at high temperatures by one grade.

**Figure 39 a**

*The Superpave Rutting Parameter (G*/sin δ) of Unaged Base and Modified Bitumen Binders*

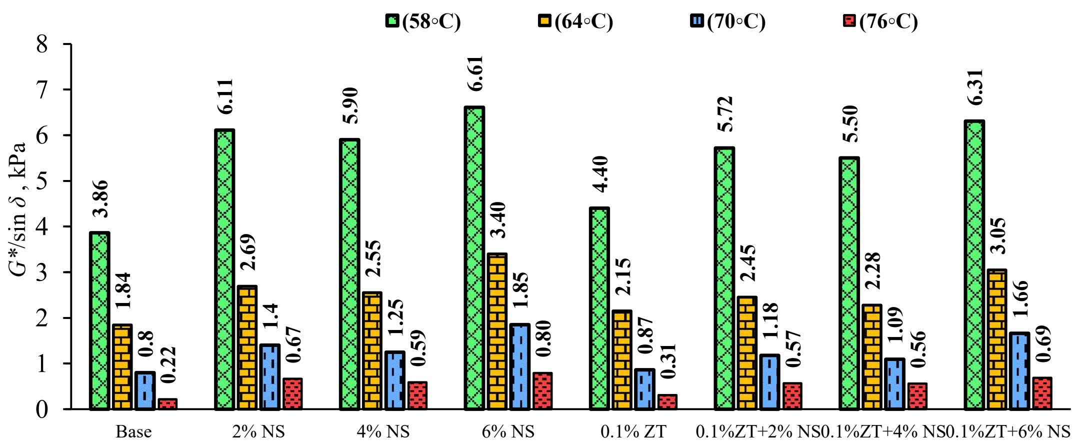
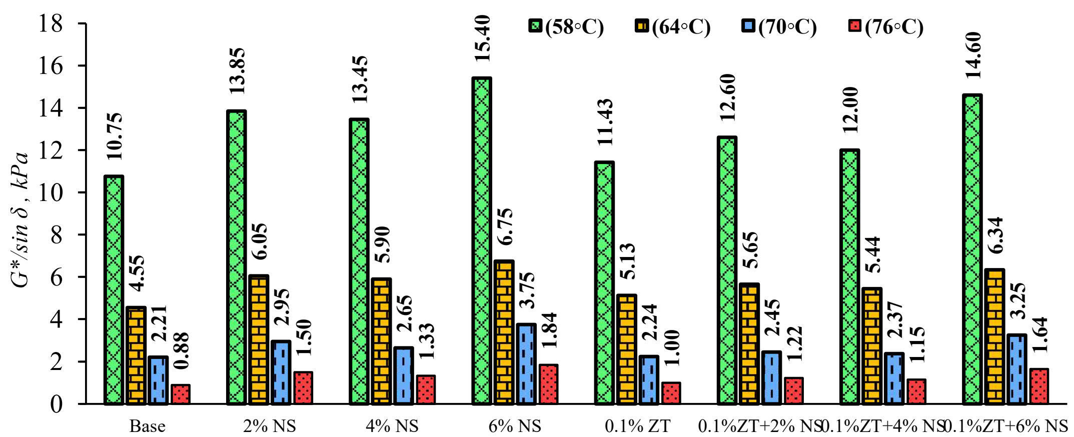



**Figure 39 b**

*The Superpave Rutting Parameter (G*/sin δ) of RTFO-aged for Base and Modified Bitumen Binders*



### *4.2.2 MSCR Test Rutting Potential*

The MSCR test is a common method for evaluating the rutting resistance of bitumen binder. As previously stated in chapter three, two separate parameters from the MSCR were examined to evaluate the resistance to rutting of bitumen binder, non-recoverable creep compliance ($J_{nr}$) and percentage recovery (*R%*). Furthermore, the cumulative strain was another result of the MSCR test.

Figures 40 a and 40 b show the typical strain outputs from the MSCR test of base and modified bitumen at 64 $^{\circ}$C with two levels of stress. Figure 40 a display the low-stress level (0.1 kPa) during the first 100 seconds, whereas Figure 40 b displays the high-stress level (3.2 kPa) for the next 100 seconds. The MSCR results comprise of two stages, namely the creep stage and the recovery stage, to complete a cycle. Analyzing the cumulative strain and stress levels reveals that the accumulated creep compliance at 0.1 kPa is lower than at 3.2 kPa, indicating that as stress levels increase, so does the accumulated strain. According to the results, the addition of 0.1% ZycoTherm to the base bitumen resulted in a drop in the accumulated strain, which is consistent with the previous findings (Mirzababaei, et al., 2017). However, when Nano-Silica was added to the binders, the accumulated strain was significantly reduced when compared to the base and 0.1% ZycoTherm binders. As a consequence, at high temperatures, the Nano-Silica can enhance the stiffness of the binder. In contrast to other percentages, it is revealed that 6% NS has the lowest cumulative strain, followed by 0.1% ZT +6% NS.

**Figure 40 a**

*The Accumulated Creep Compliance at 0.1 kPa*

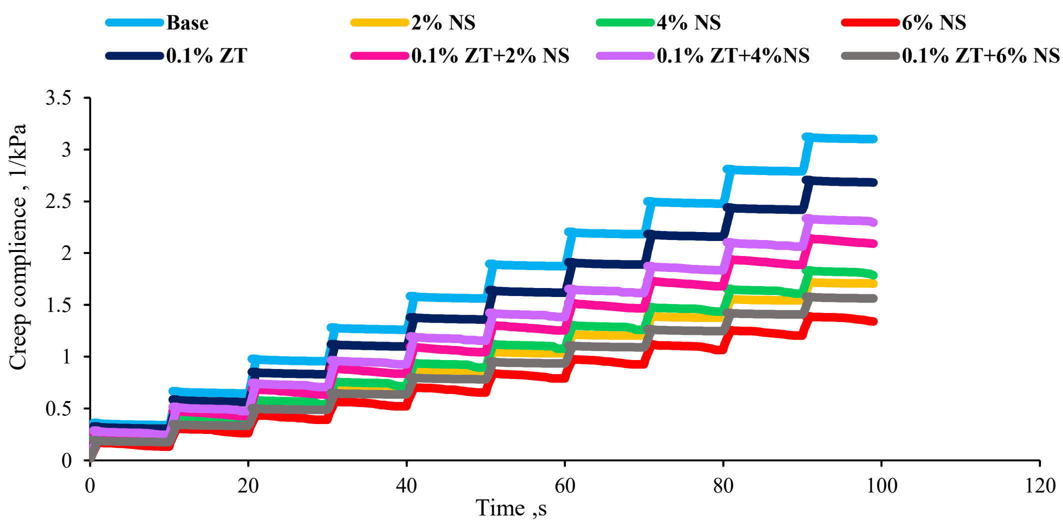
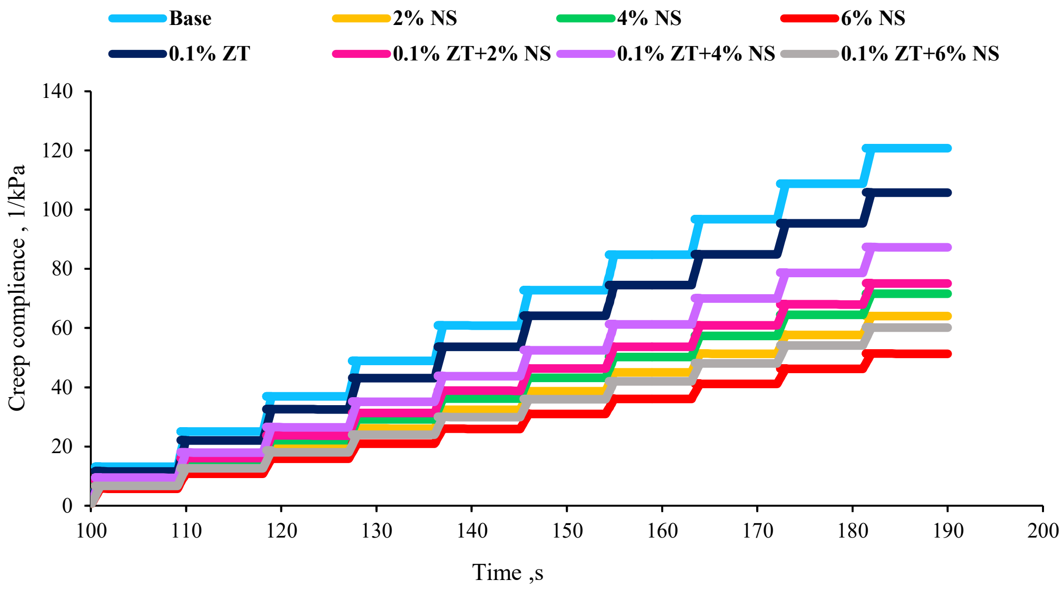


**Figure 40 b**

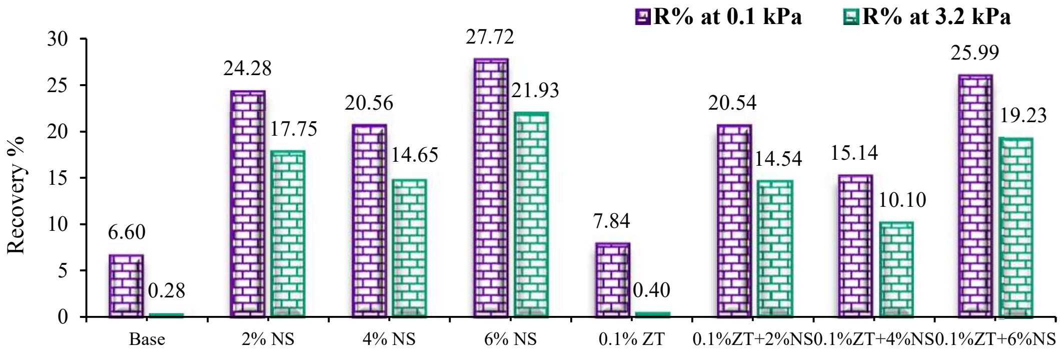
*The Accumulated Creep Compliance at 3.2 kPa*




The recovery percentages for the base and modified binders at 0.1 kPa and 3.2 kPa stress levels are presented in Figure 41. An evaluation of the collected findings at each stress level revealed that the value reduced at 3.2 kPa compared to 0.1 kPa. The recovery of the base binder was 6.6% according to the data obtained at 0.1 kPa stress level, and it increased to 24.28%, 20.56%, and 27.72% with the addition of 2%, 4%, and 6% of Nano-Silica, respectively. Whereas 0.1% ZycoTherm modified binder demonstrated 7.84% recovery, it increased to 20.54%, 15.14%, and 25.99% with the addition of 2%, 4%, and 6% Nano-Silica, respectively. A comparable improvement was seen at 3.2 kPa stress level. The addition of Nano-Silica to base and ZycoTherm modified binders enhanced the elasticity of the bitumen binder, resulting in better recovery performance. According to the results acquired, the addition of Nano-Silica increases the recovery value. The elastic recovery increased at all concentrations, with 6% NS exhibiting the greatest improvement; however, when 4% NS was added, the improvement was minor. This is also consistent with the results of (Arshad, et al., 2017).

**Figure 41**

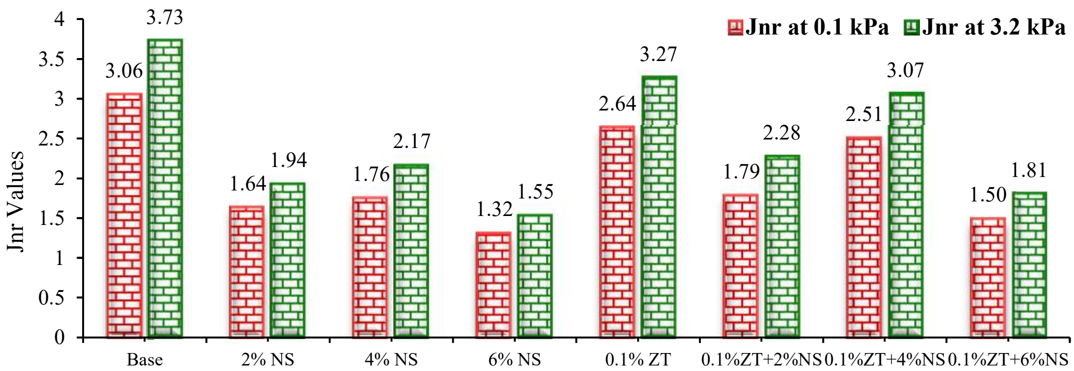
*R% at 0.1 and 3.2 kPa Stress Levels*




$J_{nr}$ constraint was used to evaluate the effect of the binder on rutting performance. Figure 42 shows how the addition of Nano-Silica to bitumen affects the $J_{nr}$. In terms of rutting, a lower $J_{nr}$ value is preferred. The inclusion of Nano-Silica improves rutting resistance. This is due to the increased surface area of Nano-Silica, which causes an interconnected network to develop within the underlying bitumen binder, increasing stiffness.

**Figure 42**

*$J_{nr}$ at 0.1 and 3.2 kPa Stress Levels*




According to the results, the $J_{nr}$ increases as the degree of stress increases from 0.1 kPa to 3.2 kPa. The efficiency of modification has been shown to improve with rising degrees of stress. This illustrates the effectiveness of Nano-Silica in improving the stress sensitivity of bitumen binders (Sukhija, et al., 2021). At 0.1 kPa, the reductions in $J_{nr}$ in comparison to the base bitumen were 46.27%, 42.44%, and 56.87% when Nano-Silica was added at 2%, 4%, and 6%, respectively. Furthermore, as compared to base bitumen, the ZycoTherm binder lowered the $J_{nr}$ value by 13.54%, showing that the bitumen binder reduced the susceptibility of bitumen to permanent deformation. Mirzababaei et al. (2017) stated that 0.1% ZycoTherm can improve binders' resistance to rutting by lowering non-recoverable values. However, the decrease in $J_{nr}$ for ZycoTherm binder was enhanced by 41.43%, 17.85%, and 50.92% when 2%, 4%, and 6% Nano-Silica were added, respectively, in comparison to base bitumen. The decrease in $J_{nr}$ indicated that Nano-Silica particles increased the ZycoTherm binder's rutting resistance. The decrease in $J_{nr}$ values with the addition of Nano-Silica might be explained by the greater stiffness of the modified bitumen binders.

### *4.2.3 Frequency Sweep Test*

One of the most frequent methods for testing the rheological properties of a bitumen binder is the frequency sweep test. The test was carried out to determine the viscoelastic characteristics at various temperatures and frequencies. The frequency sweep test findings were used to generate master curves for base and modified bitumen binders to determine material response across a broad range of frequencies and temperatures. Furthermore, an isochronal plot may be used to demonstrate the temperature dependency of complex modulus and phase angle.

**A. Master curves.** The TTSP is primarily used to describe the rheological characteristics of bitumen throughout a large frequency range. By using a shift factor, the viscoelastic behaviour of binders at a particular temperature throughout a specific range of frequencies can be equal to the behaviour evaluated at various temperatures at the same frequency. As a result, viscoelastic parameters such as $G^*$ and $\delta$ measured at various temperatures can be shifted to a reference temperature to form a continuous curve at a reduced frequency termed as a master curve, where 22 $^{\circ}$C was selected as the reference temperature.

The master curves for the $G^*$ and $\delta$ of the base binder and modified binders for the unaged samples is shown in Figure 43. The complex shear modulus, $G^*$, master curve for the base bitumen modified with Nano-Silica is displayed in Figure 43 a. As can be seen, the inclusion of Nano-Silica resulted in higher values of complex modulus over the whole range of frequency and temperature. Because of the presence of Nano-Silica, an increase in the complex modulus values indicates that the bitumen binder becomes stiffer and can considerably improve the bitumen's high-temperature performance. The degree of increase in complex modulus values for 6% NS is greater than for the other percentages. This tendency, however, is observed to be more evident at lower frequency ranges. Generally, lower frequencies indicate slower-moving vehicles, making the pavement more susceptible to rutting (Sukhija et al., 2021). Figure 43 b shows the complex shear modulus master curves for the base, ZycoTherm, and ZycoTherm/ Nano-Silica modified binders. The complex modulus of ZycoTherm binder was slightly higher than that of base bitumen, but not significantly; nevertheless, when Nano-Silica at any percentage was added to the ZycoTherm binder, the $G*$ greatly increased. This might contribute to the stiffening effect of Nano-Silica particles. The phase angle master curves of base and Nano-Silica modified binders in Figure 43 c exhibit a broad significant drop across all frequencies, as predicted, after the inclusion of the Nano-Silica in the base bitumen binder. In comparison, the phase angle of the base bitumen approaches 90˚, meaning that the bitumen will lose its elasticity and enter viscous flow phase. The Nano-Silica modified binders have a phase angle of no greater than 75˚, guaranteeing that the bitumen is resistant to rutting. Figure 43 d shows the phase angle master curves for the base, ZycoTherm, and ZycoTherm/ Nano-Silica modified binders. The base bitumen had the highest phase angle values; however, the phase angle values were reduced when 0.1% ZycoTherm was added; the drop-in phase angle values in the middle and low-frequency ranges is more noticeable. The phase angle values were significantly reduced when Nano-Silica was added to binders containing ZycoTherm.

A bitumen binder should be stiff and elastic to resist rutting. The higher the ($G*$) value, the stiffer the bitumen is, and the lower the ($\delta$) value, the higher the elastic part of ($G*$). Revealing that Nano-Silica stiffens bitumen binders, which is beneficial for resistance of rutting. Furthermore, the greatest specific area of Nano-Silica induced an increase in complex modulus values and a reduction in phase angle values for all Nano-Silica modified binders compared to base and ZycoTherm binders. When it comes to ZycoTherm binder without Nano-Silica, it is clear that 0.1% ZycoTherm has slightly better results in terms of complex modulus and phase angle values than base bitumen, and thus its performance against rutting is achievable but not significantly effective, and it requires more enhancement as the addition of Nano-Silica. Previous research studied the effect of ZycoTherm on rutting resistance on various types of bitumen and showed that it had a positive effect (Ibrahim & Mehan, 2015; Mirzababaei, et al., 2017; Raufi, et al., 2020).

**Figure 43 a**

*The Master Curves of (G*) for Base Bitumen Modified with Nano-Silica*

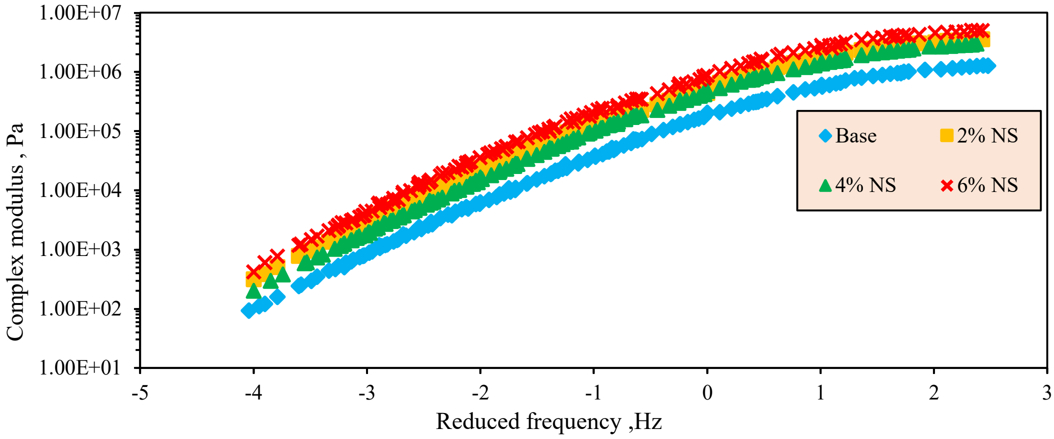



**Figure 43 b**

*The Master Curves of (G*) for Base, ZycoTherm, and ZycoTherm/Nano-Silica Binders*

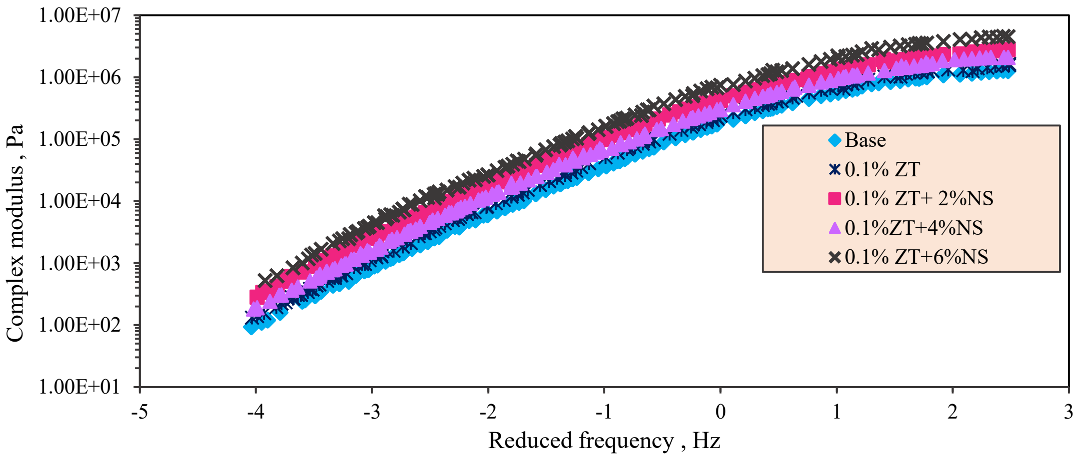



**Figure 43 c**

*The Master Curves of (δ) for Base Bitumen Modified with Nano-Silica*

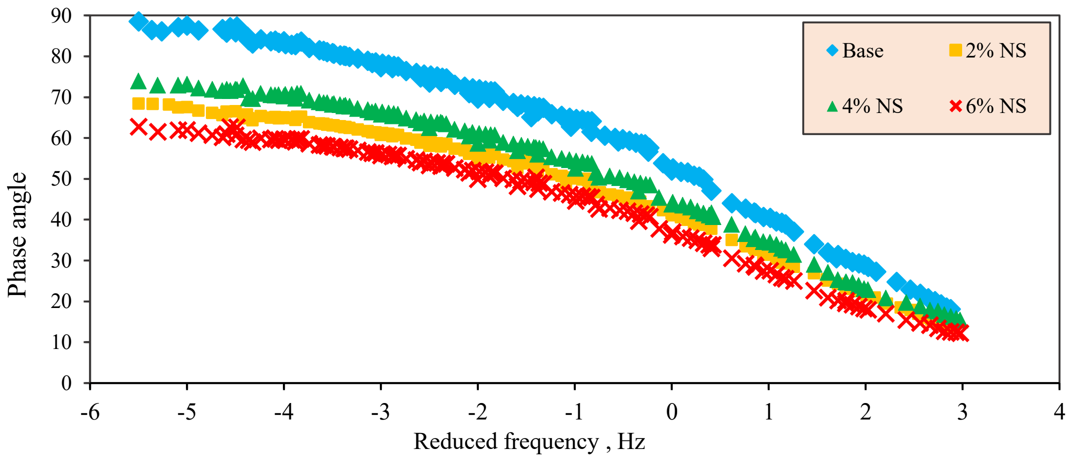
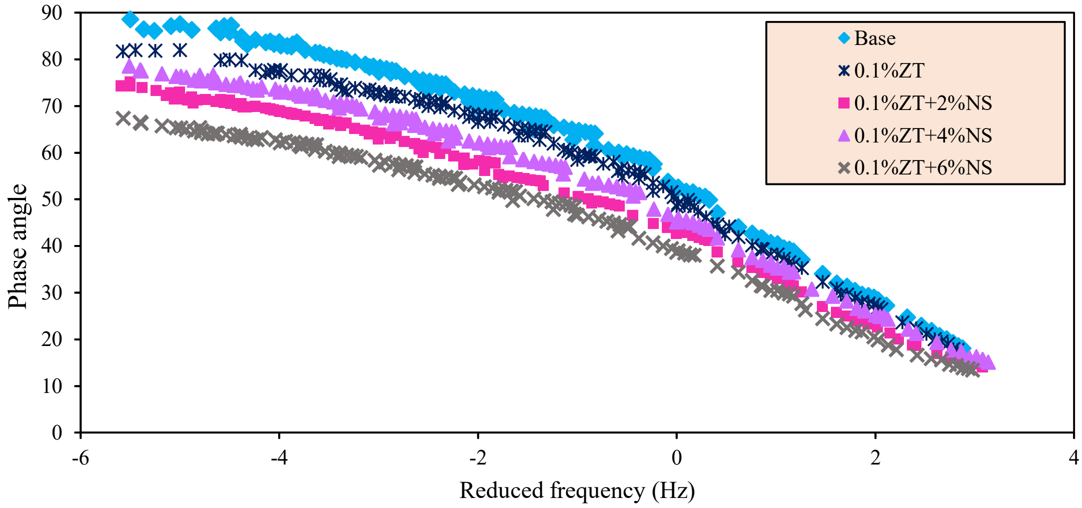


**Figure 43 d**

*The Master Curves of (δ) for Base, ZycoTherm, and ZycoTherm/Nano-Silica Binders*




**B. Isochronal plots.** The behaviour of a system at a constant frequency is shown as a curve on a chart and is known as an isochronal plot. The use of Isochronal plots allowed the viscoelastic attributes like the $G^*$ and $\delta$ of the base and modified binders against temperature to be illustrated at specific frequencies. The temperature ranges from 10 to 82 ˚C, with 6 degree increments for the unaged base and modified binders at 1 Hz and 15 Hz are displayed against the phase angle and complex modulus in Figures 44 (a-d). Figures 44 a and 44 b show the isochronal plots for the base bitumen modified with Nano-Silica at 1 and 15 Hz, respectively. According to the findings, base bitumen has the lowest $G^*$ at high and low temperatures, whereas 6% NS has the highest $G^*$ (stiffness) at both high and low temperatures. The mechanism contributing to the increase in complex modulus for Nano-Silica modified binders may be explained by the fact that the Nano-Silica particles stay evenly dispersed in the bitumen binder without dissolving in it. The homogeneous distribution of Nano-Silica particles in the bitumen binder improves the asphalt binder's strength (Bhat & Mir, 2021). The complex modulus of Nano-Silica modified binders has increased significantly, enhancing the binders' rutting resistance. This increase becomes more pronounced at higher frequencies. Moreover, when temperature increases, the phase angle of the binders increase, and the modified binders with Nano-Silica exhibit lower phase angles than the base bitumen. The phase angle of the 6% NS decreases significantly at both low and high temperatures. Furthermore, the phase angle of Nano-Silica modified binders decreases at low (1Hz) and high (15Hz) frequencies, showing that the modified binders seem to be elastic in nature (Nazari, et al., 2018; Bhat & Mir, 2021).

The isochronal plots for the base, ZycoTherm, and ZycoTherm/Nano-Silica binders at 1 and 15 Hz are shown in Figures 44 c and 44 d, respectively. It can be shown that the ZycoTherm binder without Nano-Silica has a comparable stiffness to the base bitumen at high temperatures, implying that its behavior at high temperatures is quite similar to that of base bitumen. The addition of Nano-Silica to ZycoTherm binders resulted in a considerable increase in complex modulus at both high and low temperatures, showing that the stiffness was increased of the modified binders. The 6% NS demonstrates the greatest $G^*$ values then followed by 2% NS. The variations found in the $G^*$ and $\delta$ values at 15 Hz for the base, ZycoTherm, and Nano-Silica modified binders were much more significant than those at 1 Hz but followed the same pattern, demonstrating that the effect of additives was more pronounced at higher frequencies. Because all modified binders with Nano-Silica show higher stiffness at both high and low temperatures, it can be stated that Nano-Silica modified binders with or without ZycoTherm were able to enhance the elevated temperatures performance (resistance to rutting) of base bitumen but were ineffective in enhancing the low – medium temperatures performance.

**Figure 44 a**

*Isochronal Plot of (G*) and (δ) for Base Bitumen Modified with Nano-Silica at 1 Hz*

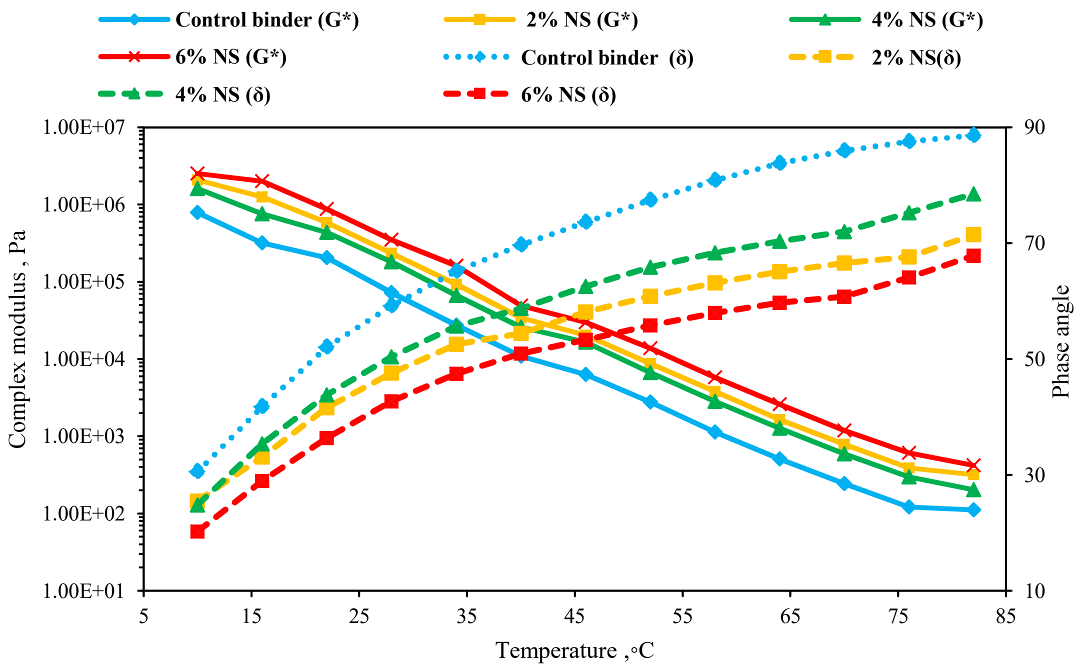


**Figure 44 b**

*Isochronal Plot of (G*) and (δ) of Base Bitumen Modified with Nano-Silica at 15 Hz*

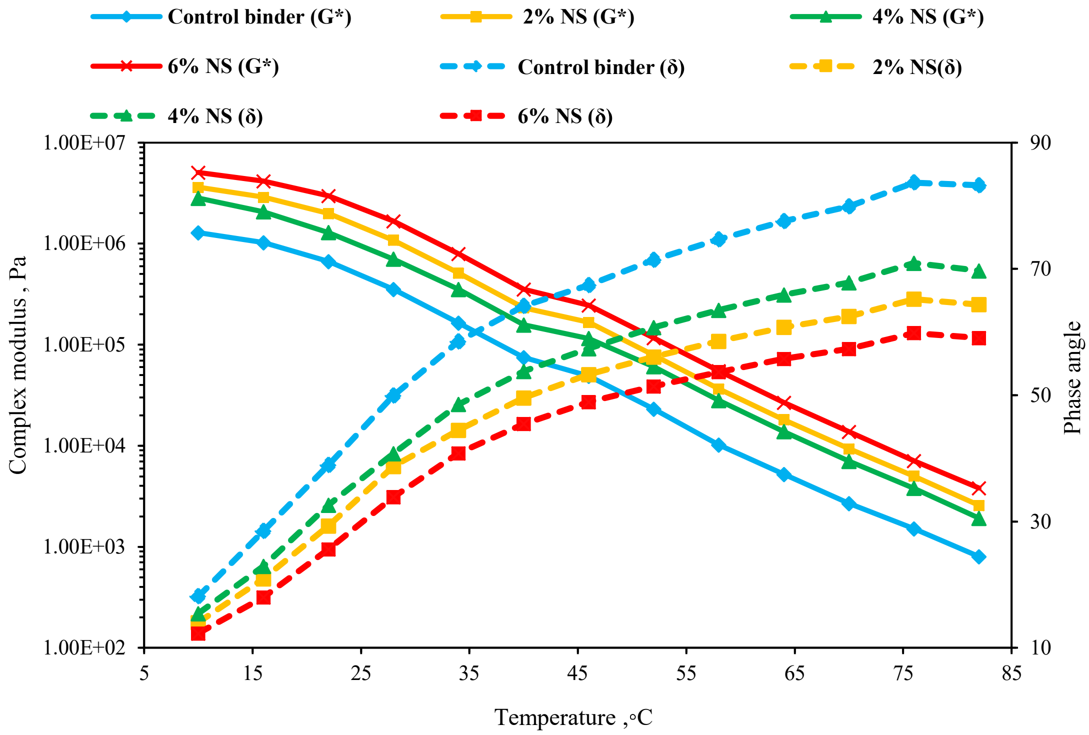



**Figure 44 c**

*Isochronal Plot of (G*) and (δ) for Base, ZycoTherm, and ZycoTherm/Nano-Silica Binders at 1 Hz*

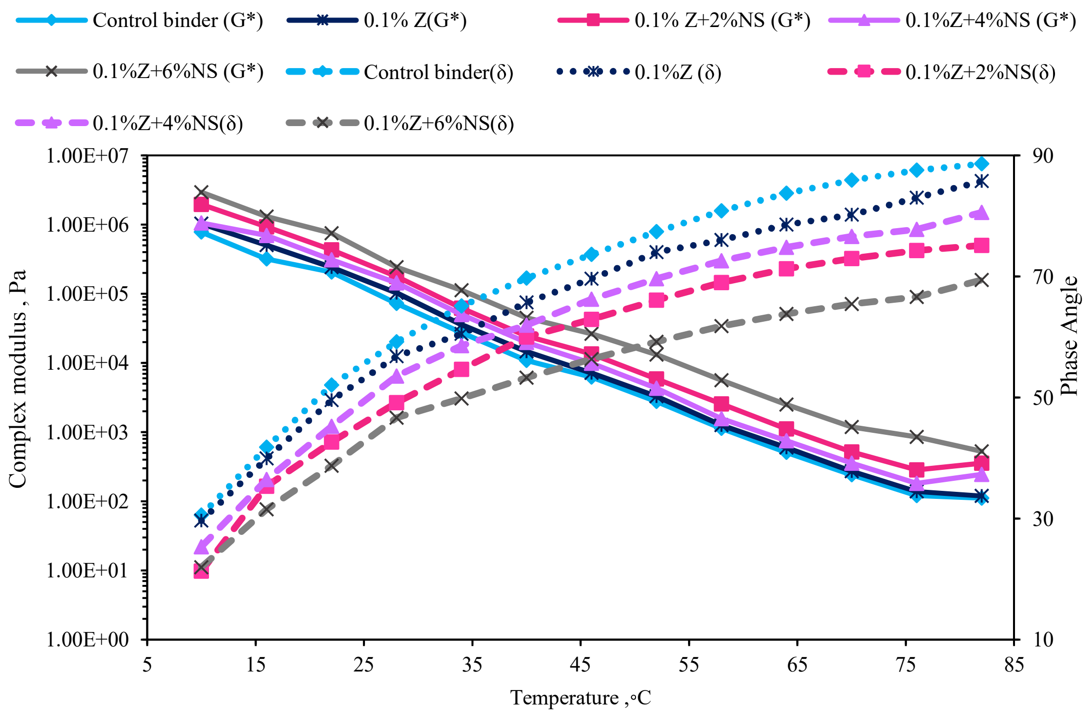
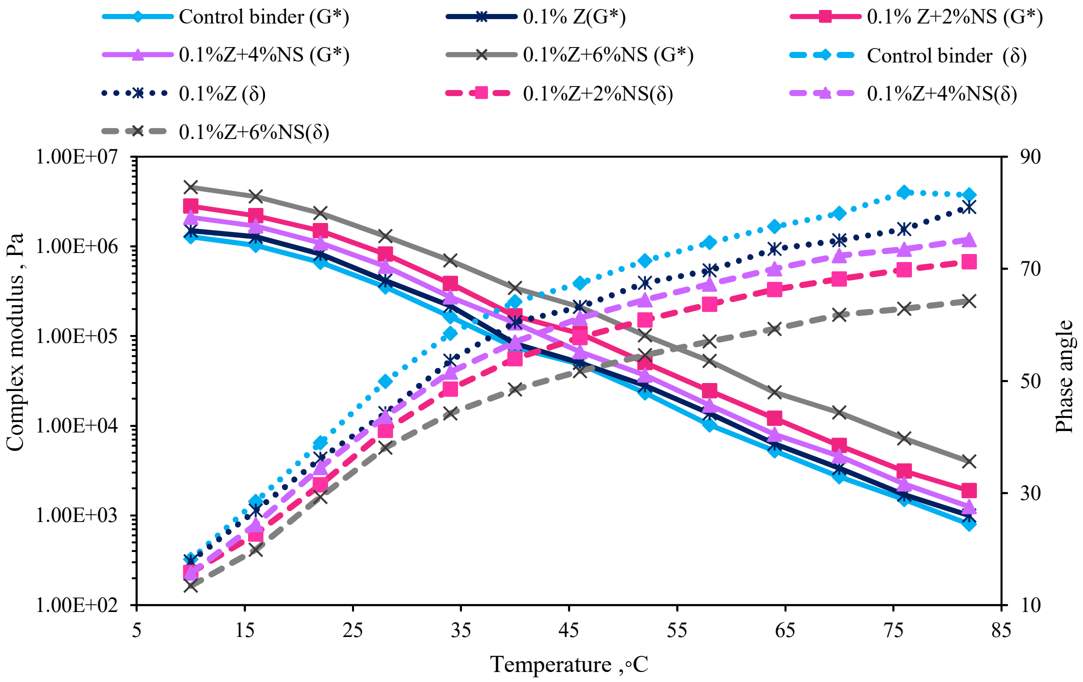


**Figure 44 d**

*Isochronal Plot of (G*) and (δ) for Base, ZycoTherm, and ZycoTherm/Nano-Silica Binders at 15 Hz*




### *4.2.4 Superpave Fatigue Resistance Parameter*

Because fatigue cracking is typically associated with aged binder, testing for this feature is typically performed on binder that has been exposed to a conditioning method utilizing PAV. There aren't many standardized test procedures for determining the fatigue characteristics of bitumen binders, and the methods that are available aren't widely accepted to correctly quantify this feature (Southern, 2015). Nonetheless, Superpave fatigue resistance using DSR has been created and is currently in use in standards. The DSR was used to measure $G^*$ and $\delta$ to evaluate the viscoelastic characteristics of bitumen in the intermediate temperature range. At temperatures ranging from 16 °C to 34 °C, specimens aged in RTFO and PAV were evaluated at a frequency of 10 rad/s (1.59 Hz). The Superpave fatigue resistance parameter ($G^*$. sin $\delta$) was utilized to assess the influence of Nano-particles modification on bitumen fatigue characteristics.

Figure 45 shows the values of the complex modulus and phase angle for base and modified binders. The drop-in phase angle values are insignificant for specimen containing 0.1% Nano-ZycoTherm, however the phase angle values of specimens containing Nano-Silica particles are lower when compared to base and ZycoTherm binders. As a result, the addition of Nano-Silica to base and ZycoTherm binders resulted in a more significant drop in phase angle, which is related with enhanced bitumen elastic properties. Figure 45 also displays the complex modulus values for all binders. The shear modulus of ZycoTherm binder was slightly higher than the base bitumen following RTFO and PAV conditioning procedures attributed to stiffening effects of the ZycoTherm, which is consistent with other observations (Mirzaaghaeian & Modarres, 2019). The addition of Nano-Silica, on the other hand, resulted in a considerable increase in complex modulus values. Higher stiffness of the Nano-Silica particles in contrast to the base and ZycoTherm binders result in higher complex modulus values.

**Figure 45**

*Complex Modulus and Phase Angle of Base and Modified Binders at Intermediate Temperatures after PAV*

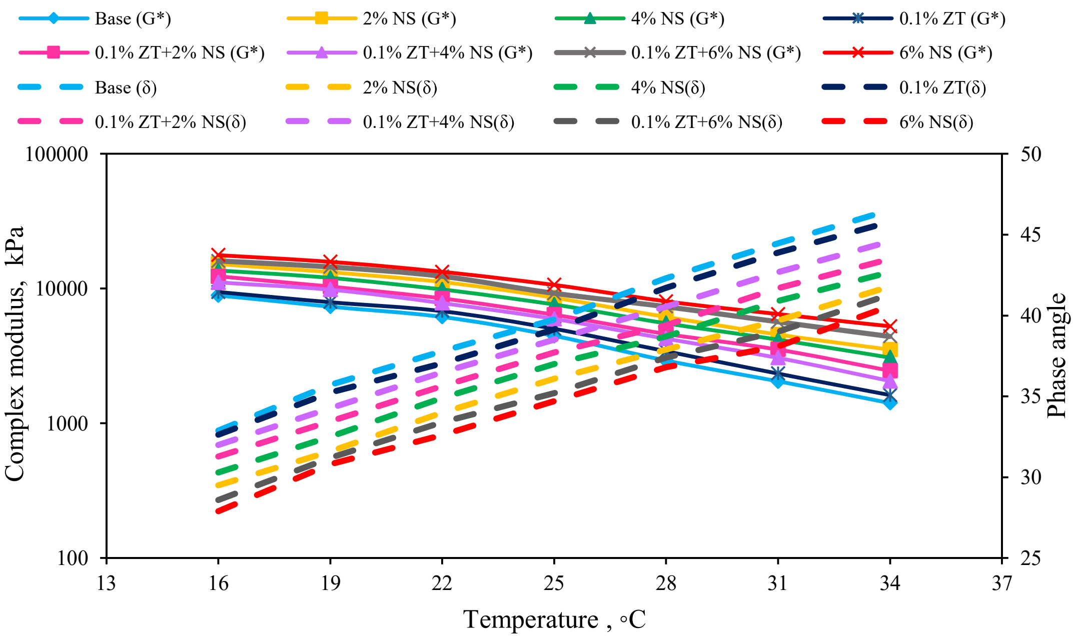
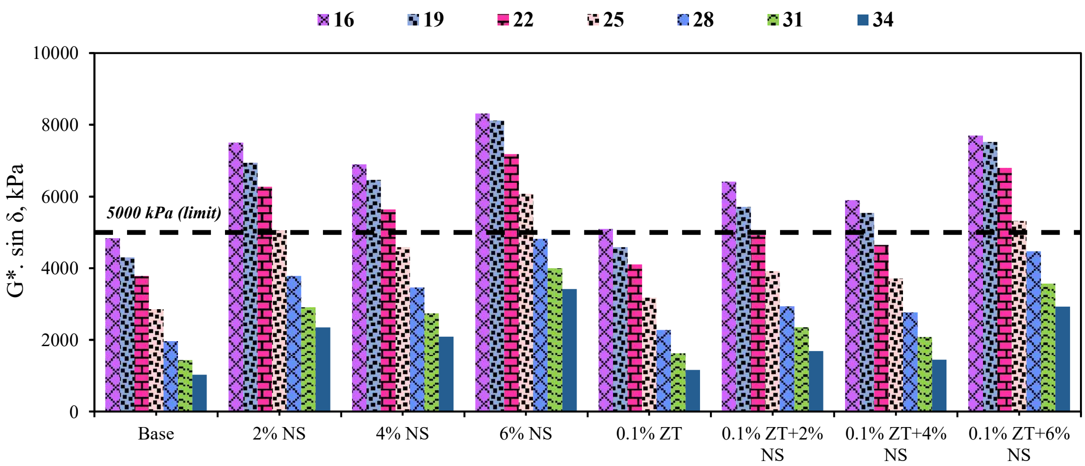



Figure 46 shows a fatigue parameter ($G^*.\sin\delta$) that used to evaluate the fatigue resistance of bitumen binder. At moderate temperatures, the Superpave restricts the fatigue parameter to 5000 kPa. For improved fatigue resistance, a lower value of $G^*.\sin\delta$ is preferred. As can be observed, adding Nano-Silica boosts the value of $G^*.\sin\delta$. The findings from $G^*.\sin\delta$ values also show that the fatigue life may be reduced owing to an increase in stiffness caused by the addition of Nano-Silica. It should be highlighted that the Superpave parameters, as previously defined, are dependent on the stiffness of the bitumen. The addition of Nano-Silica enhances the stiffness of bitumen, which is beneficial for rutting but not for fatigue resistance. The elastic performance of bitumen binder was enhanced by the addition of Nano-Silica, as evidenced by decreased phase angles. Increased in complex modulus resulted in greater $G^*. \sin\delta$. As a consequence, the Superpave fatigue parameter results show that the addition of Nano-Silica to bitumen binder resulted in a reduction in fatigue cracking resistance, which is similar to the previous findings (Nejad, et al., 2017; Moeini, et al., 2020; Sukhija, et al., 2021).

**Figure 46**

*Superpave Fatigue Parameters for Base and Modified Binders*




### *4.2.5 BBR Test*

The BBR test was used to evaluate the rheological properties of the binders at low temperatures. All binders' creep stiffness and m-values were evaluated at 60 seconds at varied low temperatures ranging from 0 to -18 ˚C, as shown in Figures 47 and 48, respectively. The stiffness of the binder increases as the testing temperature decreases. When the temperature was dropped from 0 ˚C to -18 ˚C for all bituminous binders, the creep stiffness increased and the m-value declined. Nano-Silica had a negative impact on binders' low-temperature performance, as evidenced by a drop-in m- values and an increase in stiffness values. Additionally, the addition of Nano-Silica to the base bitumen in the absence of ZycoTherm had no effect on the base binder's low-temperature performance grade, despite changing the m-values and creep stiffness values. The insignificant influence of Nano-Silica on the low-temperature PG of binders was also documented in previous studies (Yao, et al., 2013; Nejad, et al., 2017;

Saed, et al., 2022). Furthermore, when comparing ZycoTherm binder to base bitumen, it is evident that the stiffness is somewhat greater, and the stiffening effect in ZycoTherm binder is attributable to the impact of aging, as the binder reacts with oxygen (oxidized) and thus hardens. Furthermore, the ZycoTherm binder has a greater m-value as compared to base bitumen; a higher m-value indicates superior cracking resistance. The base bitumen met the low-temperature standards at -6 °C, whereas the ZycoTherm binder at -12 °C, as the m- value was more than 0.3. The addition of Nano-Silica to the ZycoTherm binder, on the other hand, resulted in increased creep stiffness and a lower m-value. As a result, adding Nano-Silica to the base bitumen has no effect on its low-temperature performance and is insufficient to change the low-temperature grade, whereas the ZycoTherm binder with or without Nano-Silica particles at low temperature is able of changing the performance grade for the base bitumen. In order to address the low performance grade, the stiffness must be less than 300 MPa and the m- value must be more than 0.300 of tested bitumen binder. As a consequence of the findings, all of the binders' stiffness were less than 300 MPa.

**Figure 47**

*Creep Stiffness for Base and Modified Binders*

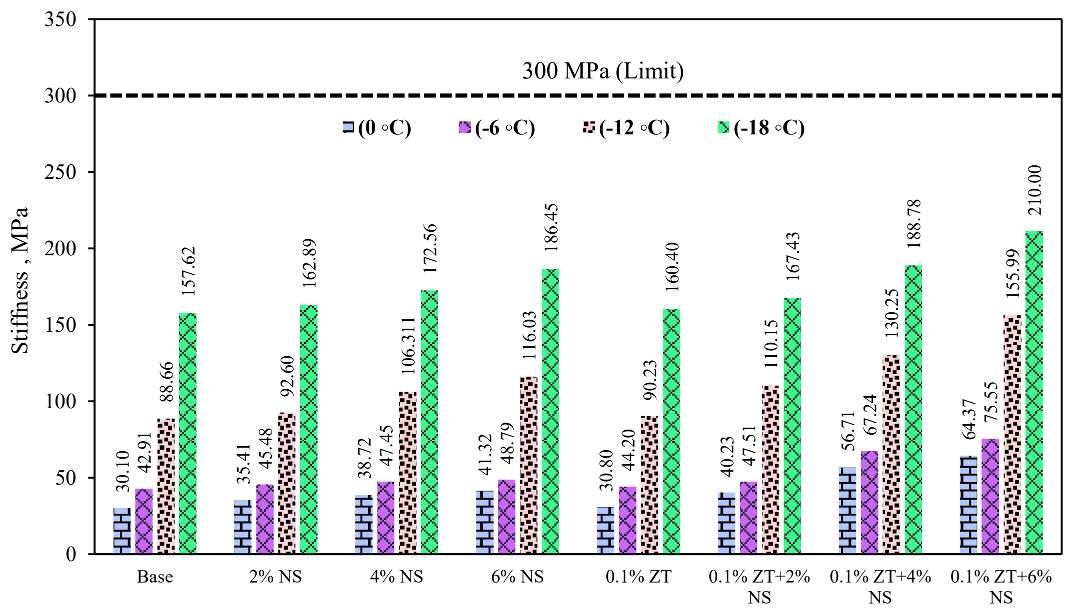
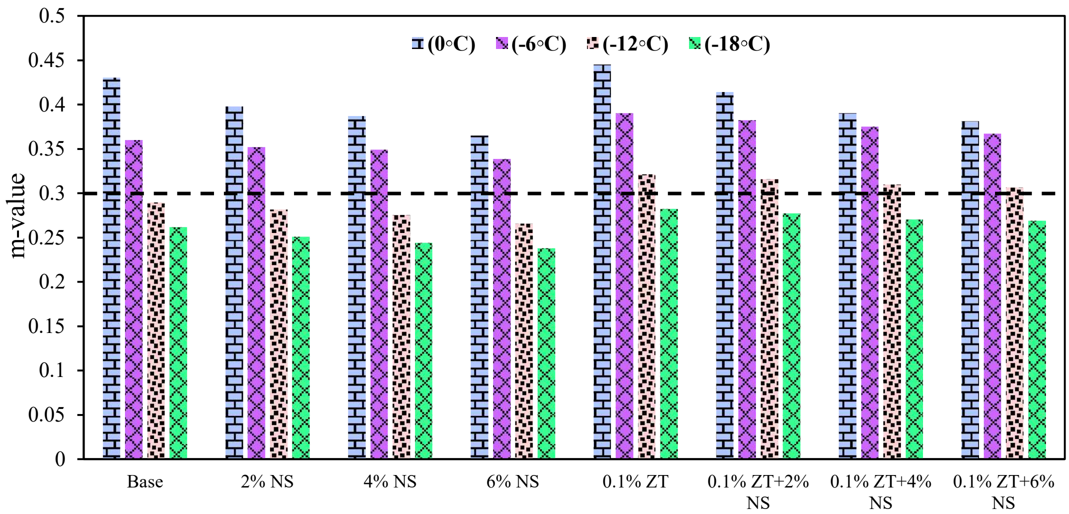


**Figure 48**

*m-Values for Base and Modified Binders*




### *4.2.6 Performance Grading (PG)*

To evaluate the performance grading of bitumen binder, the AASHTO M320 guideline was used. Generally, high-temperature performance grade is defined as the highest temperature at which the ($G^*/\sin\,\delta$) equal to or exceeds 1 kPa for unaged binder. $G^*/\sin\,\delta$ denotes a binder's resistance to rutting at elevated temperatures. The greater the $G^*/\sin\,\delta$ at a given temperature, the better the rutting resistance. The high-temperature grade of the base bitumen, either with or without ZycoTherm, was the same at 64 ˚C, as shown in Table 19. A one-grade increase was obtained after the base and ZycoTherm binders were modified with Nano- Silica at 2-6%. As previously stated, a BBR test was performed to determine the low temperature grade for bitumen. As a result, the addition of Nano-Silica to the base bitumen has no significant influence on the low-temperature PG of the base bitumen, implying that both the base bitumen and the Nano-silica modified binders have the same low-temperature grade. When it comes to ZycoTherm binder with or without Nano-Silica, as shown in Table 19, it is clear that the ZycoTherm has a positive effect on changing the grade from -16 ˚C to -22 ˚C, implying that the ZycoTherm/ Nano-Silica modification had a better effect on low-temperature grade due to the presence of ZycoTherm material.

**Table 19**

*Performance Grade Results for Base and Modified Binders*

| Test | Test Temp (˚C) | Binder Type | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Base | 2% NS | 4% NS | 6% NS | 0.1% ZT | 0.1% ZT+2% NS | 0.1% ZT+4% NS | 0.1% ZT+6 % NS |
| Unaged binder DSR, $G^*/\sin\delta$ Min.1.00 kPa | 64 | 1.84 | 2.69 | 2.55 | 3.40 | 2.15 | 2.45 | 2.28 | 3.05 |
| | 70 | 0.80 | 1.40 | 1.25 | 1.85 | 0.87 | 1.18 | 1.09 | 1.66 |
| RTFO-aged binder DSR, $G^*/\sin\delta$ Min. 2.20 kPa | 64 | 4.55 | 6.05 | 5.90 | 6.75 | 5.13 | 5.65 | 5.44 | 6.34 |
| | 70 | 2.21 | 2.95 | 2.65 | 3.75 | 2.24 | 2.45 | 2.37 | 3.25 |
| PAV- aged binder BBR Stiffness ≤ 300 MPa, m-value ≥ 0.300 | 0 | Pass | Pass | Pass | Pass | Pass | Pass | Pass | Pass |
| | -6 | Pass | Pass | Pass | Pass | Pass | Pass | Pass | Pass |
| | -12 | Fail | Fail | Fail | Fail | Pass | Pass | Pass | Pass |
| | -18 | Fail | Fail | Fail | Fail | Fail | Fail | Fail | Fail |
| Performance grade | - | 64-16 | 70-16 | 70-16 | 70-16 | 64-22 | 70-22 | 70-22 | 70-22 |

## 4.3 HMA and WMA Mixtures Tests Results

The Superpave mix design approach was used for both HMAs and WMAs to determine the optimum bitumen content and volumetric properties. The mixing and compaction temperatures for HMA and WMA were chosen based on the viscosity-temperature curve. After the determination of the OBC for HMA and WMA, all asphalt mixture samples were prepared to evaluate mechanical characteristics including moisture sensitivity and resilient modulus. Following that, the mixes were compacted with a Va of 7.0 ± 0.5 percent using the SGC and each mixture was tested with 9 samples.

### *4.3.1 Mixing and Compaction Temperatures*

Based on the viscosity-temperature curves represented in Figure 49, the mixing and compaction ranges were chosen for control HMA and Nano-Silica modified mixtures, Table 20 shows the mixing and compaction ranges. The mixing and compaction temperatures for control mix (HMA) were chosen to be 160 ˚C and 150 ˚C, respectively. The viscosity of Nano-Silica modified binder was increased when

Nano-Silica was added. The rise in stiffness is due to the increase in viscosity levels. The use of a high viscosity bitumen binder in the building of a road with a significant traffic load is recommended (Zghair, et al., 2019). As a result, hot asphalt mixes prepared with Nano-Silica need greater mixing and compaction temperatures than the HMA control mix.

**Figure 49**

*Mixing and Compaction Ranges for Control HMA and Nano-Silica Modified Mixtures*

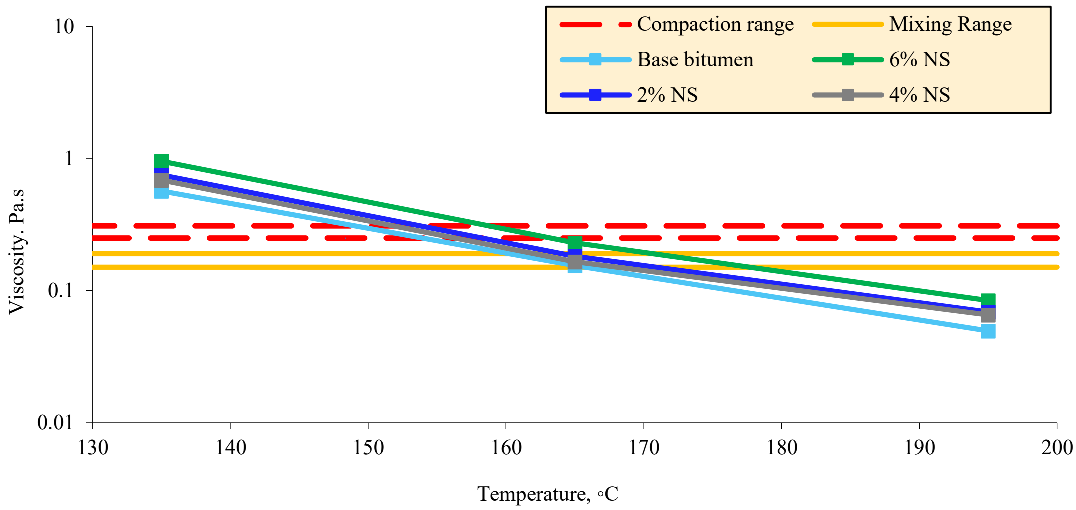



**Table 20**

*Mixing and Compaction Ranges for Control HMA and Nano-Silica Modified Mixtures*

| Sample | Mixing interval (˚C) | Compaction interval (˚C) |
|---|---|---|
| Base bitumen | 160-167 | 148-155 |
| 2% NS | 163-170 | 154-159 |
| 4% NS | 162-169 | 151-158 |
| 6% NS | 168-176 | 158-164 |

Temperatures for reduced mixing and compaction were found through trial and error. WMA samples containing 0.1% ZT were produced at mixing and compaction temperatures approximately to 30 ˚C lower than those derived from the viscosity-temperature curve in Figure 50 based on the producer's instructions and in line with other observations (Mirzaaghaeian & Modarres, 2019). The temperatures at which practically identical indices for WMA and control HMA were obtained and chosen as WMA production temperatures. Furthermore, after the aggregate is thoroughly coated, the mixing and compaction temperatures were chosen based on ease of mixing and compaction without excessive effort. The selected mixing and compaction temperatures for HMA and WMA are shown in Table 21. To keep the findings comparable, the HMA modified mixes with Nano-Silica were mixed at 168 ˚C and compacted at 158 ˚C, whereas the WMA modified mixtures with Nano-Silica were mixed at 145 ˚C and compacted at 136 ˚C.

**Figure 50**

*Mixing and Compaction Ranges for WMA Modified Mixtures with Nano-Silica*

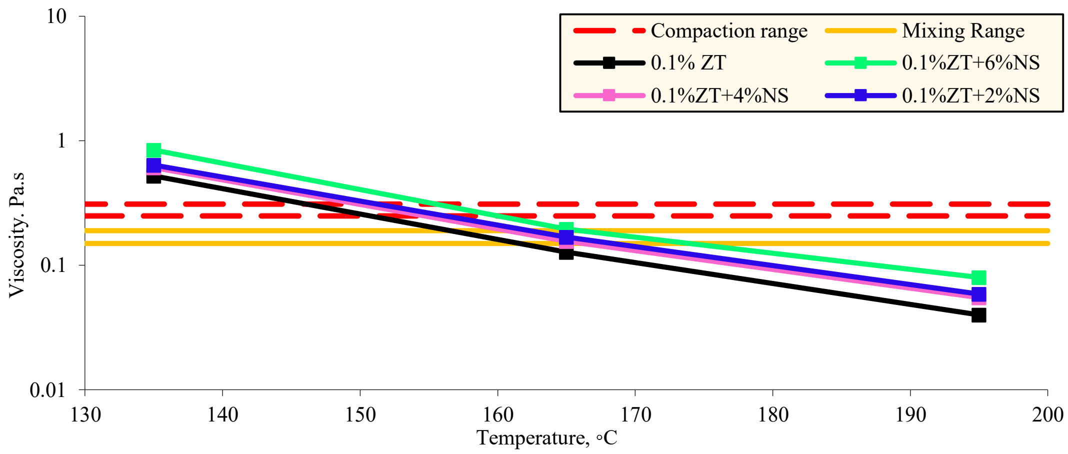



**Table 21**

*Mixing and Compaction Temperatures for HMA and WMA Mixtures*

| Asphalt mixtures | Mixing temperature (˚C) | Compaction temperature (˚C) |
|---|---|---|
| Control mix | 160 | 150 |
| 0.1%ZT | 130 | 125 |
| 2% NS | | |
| 4% NS | 168 | 158 |
| 6% NS | | |
| 0.1% ZT+2% NS | | |
| 0.1% ZT+4% NS | 145 | 136 |
| 0.1% ZT+6% NS | | |

### *4.3.2 Volumetric Properties of Asphalt Mixtures*

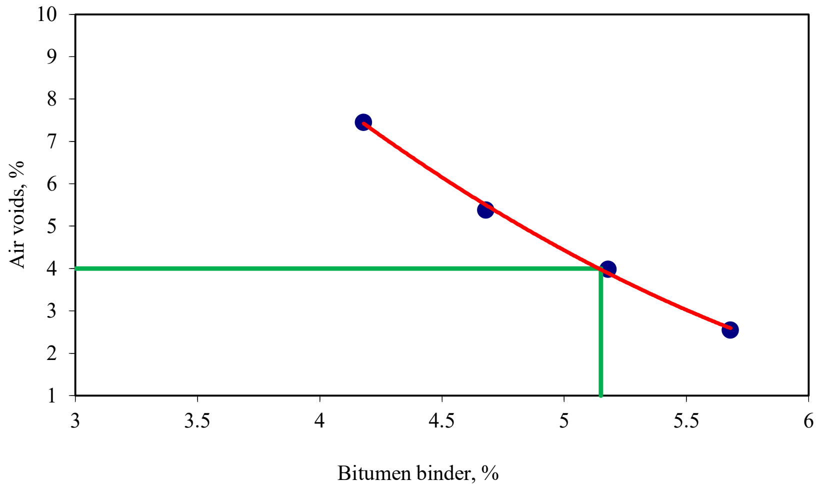
The aggregate gradation selection process, the process of determining volumetric properties and their restrictions, the mixing process, and the compaction process (determination of gyration numbers depending on traffic volume) were all comprehensively discussed in Chapter 3.

Superpave is used to determine the OBC. Superpave gyratory compactor (SGC) is utilized for compaction at 4% air void content with four different percentages: 4.2%, 4.7%, 5.2% and 5.7%. The optimal binder percentage for control HMA is 5.15% by weight while the optimum binder for WMA is 4.75%. Figures 51 and 52 show the optimal binder graphs for HMA and WMA, respectively. The optimal binder percentage guaranteed that both VMA and VFA met the standard limit. The binder content was maintained fixed for HMA and WMA mixes, the change in the characteristics of the compacted mixes is mostly due to the influence of the Nano-Silica concentrations.

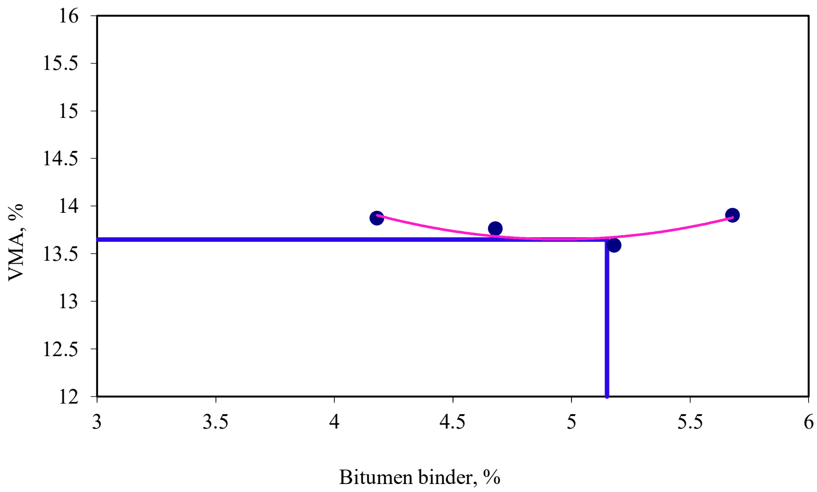
The volumetric parameters of compacted mixtures are critical factors for determining the quality of a bituminous mixture. Va, VMA, VFA, and DP are the major parameters of the mixture. The percentage of air voids reduces as asphalt content increases, eventually nearing a minimal void content. Superpave Volumetric Mix Design, AASHTO M 323, specifies 4.0% design air voids. As shown by the results, the mixture modified with 0.1% ZycoTherm has a lower optimum content when compared to the control HMA, which may be contributed to the ZycoTherm being in liquid type and lowering the viscosity of the bitumen binder, leading to lower mixing and compaction temperatures. Results are consistent with other observations (Raufi, et al., 2020). Furthermore, because ZycoTherm has the ability to thoroughly cover the aggregate even at very low temperatures, it may necessitate a lower bitumen binder percentage to get excellent results. The VMA normally falls to a minimal value due to improved compaction and subsequently rises with additional bitumen content, since the aggregate is being driven apart by excessive bitumen in the mixture, as seen in Figures 51 b and 52 b. Moreover, a greater VMA requires the use of additional bitumen to cover the aggregate particles. The VMA percent of HMA and WMA achieved the minimum standard limit of 13% for NMAS of 19 mm gradation prescribed by Superpave mixture requirements for the wearing course. If the VMA ratios were less than the standard, it indicated that there would be insufficient voids to the effective bitumen. Since the VMA is filled with bitumen, the VFA increases as the bitumen concentration increases. The VFA% of HMA and WMA met the Superpave standard which based on the selected anticipated project traffic level (10<30 million ESALs) in this research. Another essential parameter is DP, which addresses the workability of asphalt mixes in general. A low DP frequently produces a tender mix that lacks cohesiveness and is difficult to compact on the field due to its tendency to move beneath the roller. Following the determination of OBC, the volumetric parameters were double-checked, as shown in Table 22.

**Figure 51**

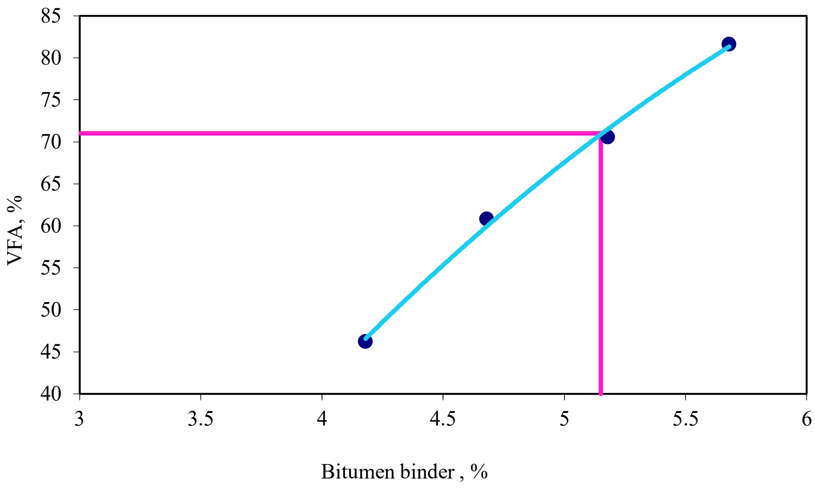
*Optimum Binder Content for Control HMA; (a) Determination of Bitumen Content @4% Air Voids, (b) Determination of VMA, (c) Determination of VFA*




*(a)*



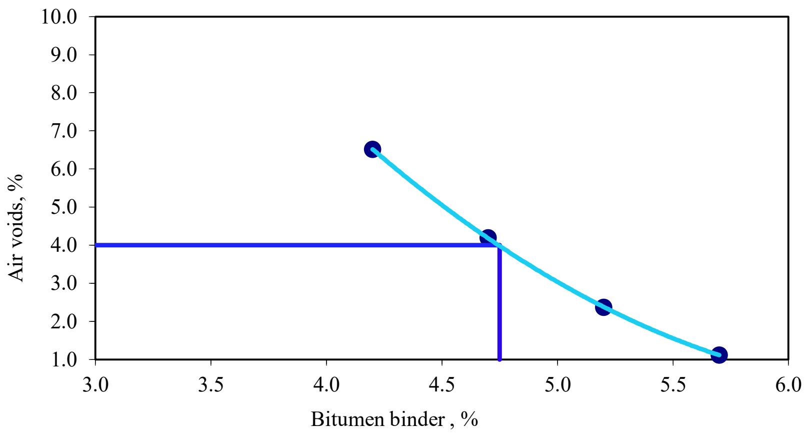

*(b)*



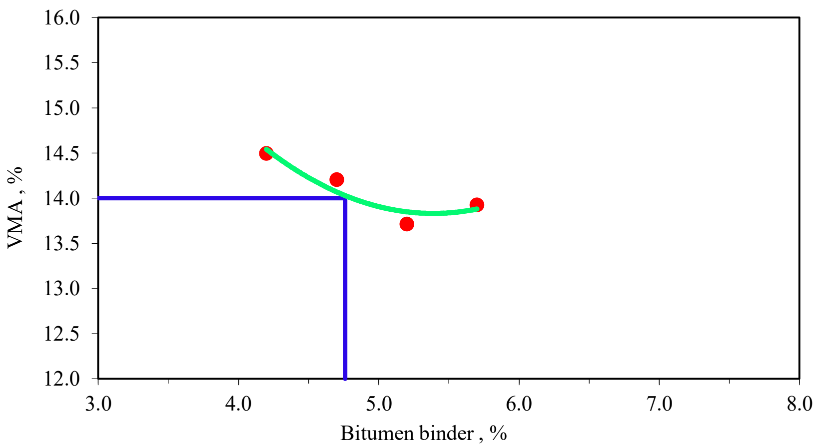

*(c)*

**Figure 52**

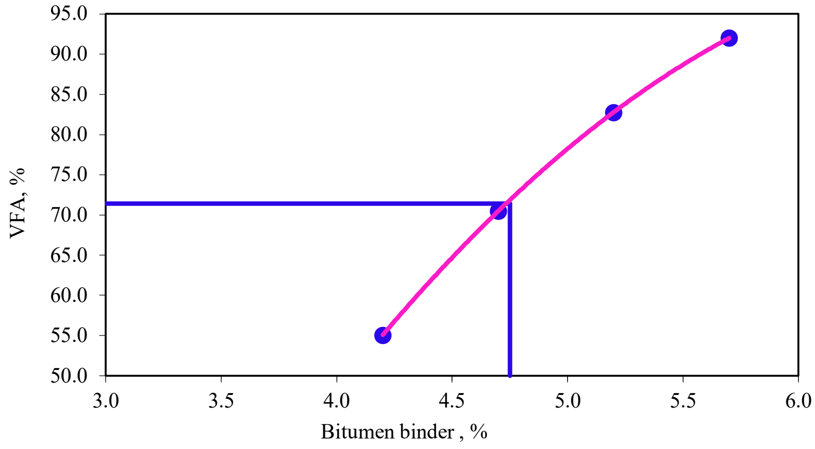
*Optimum Binder Content for WMA; (a) Determination of Bitumen Content @4% Air Voids, (b) Determination of VMA, (c) Determination of VFA*




*(a)*




*(b)*




*(c)*

**Table 22**

*Volumetric Properties for HMA and WMA*

| Property | HMA | WMA | Superpave criteria |
|---|---|---|---|
| Design bitumen content (%) | 5.15 | 4.75 | NA |
| Air voids (%) | 4.0 | 4.0 | 4.0 |
| VMA (%) | 14.2 | 14.0 | 13-15 |
| VFA (%) | 71.9 | 71.6 | 65-75 |
| DP ratio | 1.02 | 1.03 | 0.8-1.6 * |

* The permissible range may be raised to 0.8–1.6 for coarse-graded mixtures whose gradation plots beneath the Primary Control Sieve (PCS) on a 0.45 power graph.

### *4.3.3 Moisture Sensitivity*

Water sensitivity is also known as moisture susceptibility/damage. Moisture susceptibility is a result of moisture interacting with the bitumen-aggregate contact in bituminous mixture. As a consequence of this interaction, the adhesion between the bitumen and aggregate is reduced, which is referred to as stripping. The conditioning and preparation of modified Lottman testing samples is carried out in accordance with AASHTO T 283 as previously mentioned in section 3.5.1. Asphalt mixes should not degrade significantly owing to the presence or infiltration of moisture into the mix, which is an essential design criterion. To clearly differentiate the impact of Nano-modification on moisture sensitivity performance, the produced mixes (base and modified) were plotted versus their ITS values obtained for both dry and wet samples, as shown in Figure 53. According to Figure 53, the ITS values for dry samples are higher than those for wet samples. This is predicted behaviour since the presence of moisture in the conditioning phase reduces the bond between aggregate and bitumen, resulting in lower $ITS_{wet}$ values. Furthermore, the HMA modified mixes with Nano-Silica exhibited greater ITS values than the HMA control mix. The $ITS_{wet}$ for HMA modified mixes with Nano-Silica was greater than the $ITS_{dry}$ for HMA control mix, this acquired result is consistent with the observations in previous studies (Mirabdolazimi, et al., 2021; Saed, et al., 2022). The higher ITS values for Nano-Silica modified mixtures indicate that the inclusion of Nano-Silica enhanced the tensile strength of the mix due to bitumen stiffness and, as a result, improved the bond strength between the bitumen and aggregates (Mirabdolazimi, et al., 2021). Additionally, because the inclusion of Nano-Silica increases the stiffness of mixes, the debonding of aggregates from bitumen is reduced in stiffer mixes (Taherkhani & Tajdini, 2019). As a result of the inclusion of Nano-Silica, aggregates are more difficult to separate from the bitumen in the presence of moisture. Furthermore, the determination of the indirect tensile strength ratio (TSR) is regarded as one of the most significant pavement distresses because TSR is a significant indication for whether a mix can resist moisture susceptibility or is a susceptible to it. The TSR of the mixes was computed using the ITS findings provided in Figure 53 and Eq. 22.

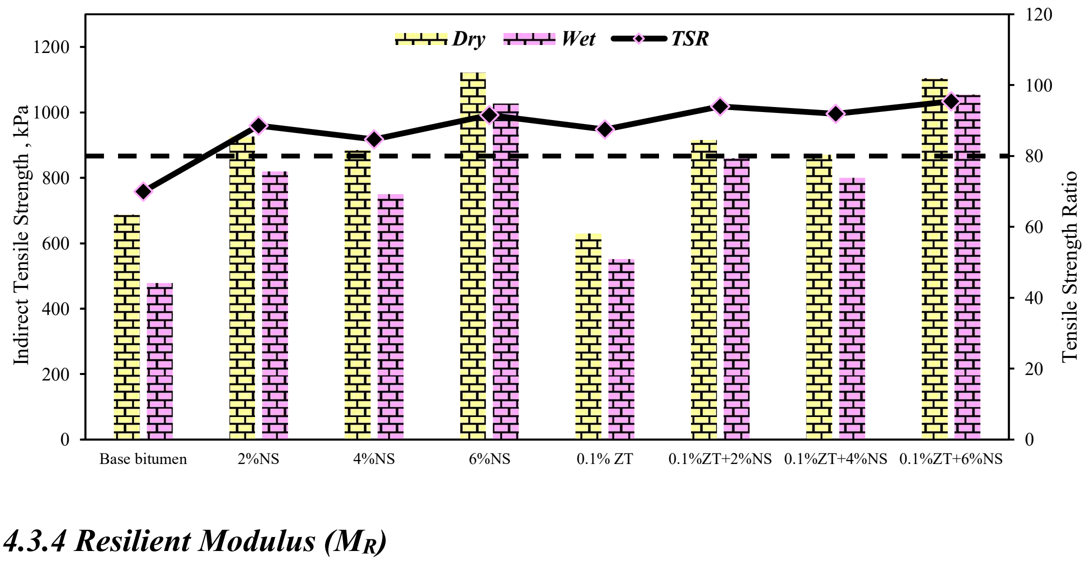
As shown in Figure 53, TSR values for mixtures should be more than 80% according to AASHTO T 283 to guarantee that the required resistance to moisture damage is achieved. The TSR values in the HMA mixes modified with Nano-Silica were increased. Since as previously reported, Nano-Silica enhances bitumen viscosity and hardness, which can restrict water penetration into the bitumen, improve bitumen cohesiveness, and make it more difficult to separate from the surface of the aggregate (Hamedi et al., 2015).

Figure 53 shows that the bitumen modified with 0.1% ZT for dry samples has a slightly lower ITS value than the base bitumen, indicating that ZycoTherm cannot improve the dry tensile strength, whereas the $ITS_{wet}$ for 0.1% ZT improved by 15%. Analyzing the outcomes for the TSR value of the mix including 0.1 % ZT, it is obvious that the modification of bitumen with it, although relatively lower doses, greatly improves the TSR value. The TSR improved to 24.3% as compared to the base bitumen. The improvement in $ITS_{wet}$ for ZycoTherm binder might be attributed to better adhesion between the bitumen and aggregate. Furthermore, ZycoTherm might produce a hydrophobic membrane with water-repellent qualities (Kataware & Singh, 2018). Additionally, because ZycoTherm is a nanoparticle, it gives a full covering of the surface of aggregate even extremely fine particles (Sanij, et al., 2019).

The results of $ITS_{wet}$ values for ZycoTherm bitumen modified with Nano-Silica were slightly better than the results of Nano-Silica modified bitumens without ZycoTherm, implying that the improvement in this case is due to the presence of both ZycoTherm and Nano-Silica, both of which have a positive effect on $ITS_{wet}$ values. Furthermore, it is notable that the TSR values were enhanced as a result of this combination, implying that the resistance to moisture damages was increased. This improvement is attributable to a variety of explanations: (1) by eliminating the air interface on the aggregate surface, ZycoTherm ensures chemical interaction between the bitumen and the aggregate, lowering the stripping possibility at the aggregate-bitumen interface (Raufi, et al., 2020); (2) Contact with bitumen transformed the surface of both ZycoTherm and Nano-Silica to hydrophobic; therefore, it turn water-repellent, which considerably reduces moisture damage (Kataware & Singh, 2018; Leiva-Villacorta & Vargas, 2019).

**Figure 53**

*ITS and TSR for Base and Modified Mixtures*




### *4.3.4 Resilient Modulus ($M_R$)*

The resilient modulus of bituminous mixes is a significant criterion in the mechanical design of pavements for measuring their response to traffic loads and determining pavement layer thickness (Ameri, et al., 2021). The lower the pavement thickness, the greater the resilience modulus. Figure 54 illustrates the average resilient modulus of 3 replicates of the base and modified mixes. The inclusion of Nano-Silica obviously has a positive influence on the resilient modulus of the bituminous mixtures. $M_R$ results, regardless of ZycoTherm presence or absence, showing substantially stiffer mixes than the control mix. The increase in $M_R$ values as a result of increased interaction between Nano-Silica and bitumen, owing to the large specific surface area of Nano-Silica particles (Taherkhani & Tajdini, 2019). Furthermore, it was revealed that the thickness required for pavement construction may be reduced by using stiffer bituminous, resulting in inexpensive costs and minimal air pollution (Behbahani, et al., 2015). When 0.1% ZycoTherm was added to the mixture, the $M_R$ value increased when compared to the control mixture. The enhanced adhesion between aggregates and bitumen in the mixes containing ZycoTherm explains the increase in the $M_R$. As a consequence, the stronger this bond is, the greater the sample's performance. The $M_R$ results for mixes including both ZycoTherm and Nano-Silica reveal that the influence of Nano-Silica on enhancing $M_R$ values was stronger in mixtures containing ZycoTherm material, for a variety of explanations, including (1) Because of the stiffening impact of Nano-Silica on asphalt mixes, it is anticipated that these mixtures will have more elasticity and hence be more resistant to rutting than control mixture ; (2) in accordance with other findings (Sanij, et al., 2019), the improvement in the $M_R$ of samples is related to the lower viscosity of bitumen owing to the utilization of ZycoTherm, which increases the coating of aggregates with bitumen.

**Figure 54**

*Resilient Modulus for Base and Modified Mixtures*

## 4.4 Cost Analysis

The cost analysis of ZycoTherm, Nano-Silica, and ZycoTherm/Nano-Silica composite modified bituminous mixtures in enhancing characteristics was performed compared to base bituminous mixture utilizing the price ratio ($P_R$) as well as the performance enhancement ratio (PER), which can be derived using Eqs. 24 and 25, respectively (Sun, et al., 2017).

$$P_R = P_m / P_{Base} \quad (24)$$

Where;

$P_m$ is the price of the modified mixture and $P_{Base}$ is the price of the base mixture

$$PER = PE_m / PE_{Base} \quad (25)$$

Where;

$PE_m$ is the performance of modified mixture and $PE_{Base}$ is the performance of base mixture

The prices for the used materials are shown in Table 23. The base bitumen price is based on the Jordanian Petroleum Refinery, the aggregate hot bins price is based on the Almanaseer quarry plant, and the Nano-Silica and ZycoTherm costs are based on the prices provided by the suppliers (Nouryon Chemicals, Bohus, Sweden and Zydex Industries, Gujarat, India, respectively). Table 24 presents the prices of various mixes; the wearing course layer has a varying price since it was modified with different modifiers, therefore the price of the material controls the price of this layer, but the other layers have a similar price for all mixes. The prices of flexible pavement layers in this study refer to the prices of the local Jordanian market. Table 25 summarizes the findings of the $P_R$ and PER ratios.

**Table 23**

*Unit Price (USD/Ton) of Aggregate, Base Bitumen, and Modifiers*

| Material | Aggregate | Base bitumen | ZycoTherm | Nano-Silica |
|---|---|---|---|---|
| Price | 15 | 423 | 14630 | 1660 |

**Table 24**

*Prices of Various Mixtures for Flexible Pavement Layers*

| Component | HMA | | | | WMA | | | |
|---|---|---|---|---|---|---|---|---|
| | Base | 2% NS | 4% NS | 6% NS | 0.1% ZT | 0.1% ZT/2% NS | 0.1% ZT/4% NS | 0.1% ZT/6% NS |
| OBC, % | 5.15 | | | | 4.75 | | | |
| Wearing Course 6cm (USD/Ton) | 36.01 | 37.26 | 38.46 | 39.62 | 35.05 | 36.19 | 37.29 | 38.34 |
| Binder Course 7cm (USD/Ton) | 10.58 | | | | | | | |
| Base Course 20cm (USD/Ton) | 8.81 | | | | | | | |
| Subbase Course 20cm (USD/Ton) | 7.05 | | | | | | | |
| Total (USD/Ton) | 62.45 | 63.70 | 64.90 | 66.06 | 61.49 | 62.63 | 63.72 | 64.78 |

Table 25 compares the cost-benefit analysis of different mixtures to the base mixture, when any percentage of Nano-Silica was added to the base mixture, the asphalt performance, including $ITS_{Dry}$ and $ITS_{Wet}$, TSR ratios, and resilient modulus, improved when compared to the base mixture, despite the fact that the price was not increase obviously. It can also be shown that the ZycoTherm/Nano-Silica composite asphalt mixes had greater qualities than the base and Nano-Silica modified bituminous mixtures, and their costs were also considerably cheaper.

According to the moisture susceptibility test results, all of the composite modified mixes ZycoTherm/Nano-Silica show the greatest TSR ratios and $ITS_{Wet}$ values, whereas the base and Nano-Silica mixes show the lowest. Furthermore, based on the findings of resilient modulus test, all of the composite modified mixes (ZycoTherm/Nano-Silica) may strengthen the adhesion between aggregates and bitumen. As a consequence, the stiffening effects of the composite modified asphalt mixtures are superior to that of the base mixture.

**Table 25**

*Analysis of the Economic Benefits of Modified Bituminous Mixes*

| Mixture Type | TSR | $ITS_{Dry}$ | $ITS_{Wet}$ | $M_R$ | Mix Price (USD/Ton) |
|---|---|---|---|---|---|
| Base | 70 | 687.7 | 478.4 | 3431 | 36.01 |
| 2% NS | 89 | 926.2 | 819.9 | 4689 | 37.26 |
| 4% NS | 85 | 885.2 | 750 | 4474 | 38.46 |
| 6% NS | 92 | 1121.7 | 1026.6 | 4854 | 39.62 |
| 0.1% ZT | 87 | 630.3 | 551.5 | 4047 | 35.05 |
| 0.1% ZT+2% NS | 94 | 915.2 | 860 | 4848 | 36.19 |
| 0.1% ZT+4% NS | 92 | 870.5 | 800.4 | 4596 | 37.29 |
| 0.1% ZT+6% NS | 95 | 1104.2 | 1053.9 | 5145 | 38.34 |
| $PER_2$ and $P_{R2}$ | 1.27 | 1.35 | 1.71 | 1.37 | 1.03 |
| $PER_4$ and $P_{R4}$ | 1.21 | 1.29 | 1.57 | 1.30 | 1.07 |
| $PER_6$ and $P_{R6}$ | 1.31 | 1.63 | 2.15 | 1.41 | 1.1 |
| $PER_{0.1}$ and $P_{R0.1}$ | 1.24 | 0.92 | 1.15 | 1.18 | 0.97 |
| $PER_{0.1/2}$ and $P_{R0.1/2}$ | 1.34 | 1.33 | 1.80 | 1.41 | 1.00 |
| $PER_{0.1/4}$ and $P_{R0.1/4}$ | 1.31 | 1.27 | 1.67 | 1.34 | 1.04 |
| $PER_{0.1/6}$ and $P_{R0.1/6}$ | 1.36 | 1.61 | 2.20 | 1.49 | 1.06 |

Based on the findings of the economic benefit analysis of modified bituminous mixes, it is obvious that the composite modified mixtures outperform in almost all mechanical properties. As a result, the cost-effectiveness analysis is limited to composite modified bituminous mixes. In addition, the cost-effectiveness analysis was conducted on the wearing course since this layer was only modified, the cost-effectiveness ratio (CER) was calculated using Eq. 26 and the results of CER are presented in Table 26.

$$\text{Cost effectiveness ratio} = \frac{\text{Effectiveness ( anticipated performance)}}{Cost\ per\ mix} \tag{26}$$

**Table 26**

*Cost-Effectiveness for Composite Modified Asphalt Mixtures*

| Mixture type | Mixture cost ($) | Moisture susceptibility test | | | | Resilient modulus test | |
|---|---|---|---|---|---|---|---|
| | | ITS $_{Dry}$ | CER | ITS $_{Wet}$ | CER | $M_R$ | CER |
| Base | 36.01 | 687.7 | 19.1 | 478.4 | 13.29 | 3431 | 95.28 |
| 0.1% ZT/2% NS | 36.19 | 915.2 | 25.29 | 860 | 23.76 | 4848 | 133.96 |
| 0.1% ZT/4% NS | 37.29 | 870.5 | 23.34 | 800.4 | 21.46 | 4596 | 123.25 |
| 0.1% ZT/6% NS | 38.34 | 1104.2 | 28.80 | 1053.9 | 27.49 | 5145 | 134.19 |

The measurement of effectiveness is given a monetary value in cost-effectiveness analysis. The CER is often represented as a ratio, with the numerator representing a performance gain from a measure of mechanical tests and the denominator representing the cost associated with the performance gain. Table 26 shows that the 0.1% ZT/6% NS mixture has the greatest CER values for moisture susceptibility and resilient modulus, followed by 0.1% ZT/2% NS, 0.1% ZT/4% NS, and base asphalt mix. For example, when 0.1% ZT/2% NS as a composite modifier was added to the base bituminous mix, the pavement gain performance for moisture damage was enhanced by 80% while the cost increased by 0.5%, indicating that the cost is negligible when compared to gain efficiency.

Based on the moisture damage and resilient modulus tests results, the $ITS_{Wet}$ value and $M_R$ for the base bituminous mixture were 478.4 and 3431, respectively. Meanwhile, the ITS $_{Wet}$ value and $M_R$ of the modified bituminous mixture with 0.1% ZT/6% NS were 1053.9 and 5145, respectively. These findings show that the modified bituminous mixture with 0.1% ZT/6% NS has a 2.203 and 1.5 times extended life than the base bituminous mixture for moisture damage and resilient modulus, respectively. Besides that, if the traditional construction life of a pavement utilizing base bituminous mixtures is 5 years, therefore the modified bituminous mixture with 0.1% ZT /6% NS will be 2.203 times more than base bituminous mixtures (2.203*5 = 11 years) for moisture damage resistance, while the resilient modulus will be around 1.5 times greater than base bituminous mixtures (1.5*5=7.5 years). It is worth noting that the composite mixture ZycoTherm/Nano-Silica raised the cost by nearly 6%, it also expanded the life by 183% and 125% for resistance to moisture damage and resilient modulus, respectively. Nonetheless, in order to keep moisture sensitivity within a tolerable range, the cost of production might be somewhat higher. Because of the improved performance of the bitumen with ZycoTherm/Nano-Silica, this expense may be well compensated throughout the lifetime of the pavement, implying that ZycoTherm/Nano-Silica as a composite bituminous mixture could be regarded monetarily effective and have a longer life span than base bituminous mix.

On the other hand, WMA technology reduces toxic emissions and gases from bitumen manufacturing plants, therefore enhancing working conditions for plant operators as well as laborers (Kumar, et al., 2017; Martin, et al., 2019). Aside from the benefits of lower temperatures, the use of WMA technology reduces energy usage, lowering manufacturing costs (Hasan, et al., 2017; Ranieri, et al., 2017). According to the World Bank, every 10 ˚C decreases in bitumen production temperature decreases fuel consumption by about 1 L and corresponding $CO_2$ emissions by 1 kg/Ton (Jamshidi, et al., 2013). According to the findings of this study, the application of ZycoTherm resulted in a 23 ˚C reduction in production temperature, resulting in a 2.3 L reduction in fuel usage and 2.3 Kg/ Ton $CO_2$ emissions.

### 4.5 Summary

Based on the findings of this investigation into the physical and rheological characteristics of the modified bitumen, the mechanical characteristics and the cost-effectiveness of the modified asphalt mixtures, Table 27 shows the improvement % in the bitumen properties after the modification and the following conclusions can be drawn:

1. In terms of physical properties (penetration and softening point), the ZycoTherm/ Nano-Silica binders outperformed.

2. In terms of ITS and TSR, the modification comprising both ZycoTherm and Nano-Silica as a composite performed better comparing to base and ZycoTherm and Nano-Silica as an individual modifier.

3. The resilient modulus of mixes containing both ZycoTherm and Nano-Silica as a composite increased significantly to 41.2% comparing to base and ZycoTherm and Nano-Silica as an individual modifier.

4. In terms of practical implementation, the use of both ZycoTherm and Nano-Silica as a composite led to reducing the mixing and compaction temperatures to 23 and 22 ˚C, respectively due to the presence of ZycoTherm compared to Nano-Silica modified mixtures without ZycoTherm. Therefore according to the literature, each 10 ˚C decrease led to a reduction in fuel consumption by about 1 L and corresponding $CO_2$ emissions by 1 kg/Ton. Furthermore, the reduction in $CO_2$ by 2.3 kg/Ton means a safer and healthy environment for labor and workers. Moreover, ZycoTherm mixture has a lower optimum content when compared to the control HMA, which may be contributed to the ZycoTherm being in liquid type and lowering the viscosity of the bitumen binder, leading to lower mixing and compaction temperatures which means more fuel cost reduction and lower the cost of using bitumen since the ZycoTherm can reduce the optimum content. Additionally, increasing the lifetime of the asphalt pavement due to minimizing the rutting and stripping distresses implying that ZycoTherm/Nano-Silica as a composite bituminous mixture could be regarded monetarily effective.

**Table 27**

*The Improvement % in the Properties of the Modified Bitumen*

| Test | 2%NS | 4%NS | 6%NS | 0.1%ZT | 0.1%ZT+2%NS | 0.1%ZT+4%NS | 0.1%ZT+6% NS |
|---|---|---|---|---|---|---|---|
| Penetration (Reduction %) | 21.3 | 26.3 | 30.6 | 18.0 | 24.6 | 27.0 | 32.4 |
| Softening point (Improvement %) | 10.6 | 15.8 | 18.8 | 5.6 | 13.0 | 17.6 | 21.2 |
| ***G*/sin δ*** **unaged (Improvement %)** | | | | | | | |
| 58 ˚C | 58.3 | 52.8 | 71.2 | 14.0 | 48.2 | 42.5 | 63.5 |
| 64 ˚C | 46.2 | 38.6 | 84.8 | 16.8 | 33.2 | 24.0 | 65.8 |
| 70 ˚C | 75.0 | 56.2 | 131.3 | 8.7 | 47.5 | 36.3 | 107.5 |
| ***G*/sin δ*** **RTFO- aged (Improvement %)** | | | | | | | |
| 58 ˚C | 28.8 | 25.1 | 43.3 | 6.3 | 17.2 | 11.6 | 35.8 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 64 °C | 33.0 | 29.7 | 48.4 | 12.7 | 24.2 | 19.6 | 39.3 |
| 70 °C | 33.5 | 20.0 | 69.7 | 1.4 | 10.9 | 7.2 | 47.1 |
| R% @ 0.1kPa (Improvement %) | 267.9 | 211.5 | 320.0 | 18.8 | 211.2 | 129.4 | 293.8 |
| Jnr @ 0.1kPa (Reduction %) | 46.4 | 42.5 | 56.9 | 13.7 | 41.5 | 18.0 | 51.0 |

# CHAPTER V

## Conclusion and Recommendations

The physical and rheological characteristics of base bitumen modified with ZycoTherm, Nano-Silica and their combinations with different percentages (i.e., 2%, 4%, and 6%) of Nano-Silica were investigated. The resilient modulus ($M_R$) test and the indirect tensile test (ITS) were carried out to evaluate the moisture susceptibility and stiffness of various mixtures at intermediate temperature. Based on the findings and discussion provided above, the following conclusions may be derived.

### 5.1 Conclusion

- According to the physical attributes (penetration and softening point) outcomes, adding Nano-Silica to the base bitumen reduces the penetration while increasing the softening point. The stiffness effect of the bitumen binder is indicated by the reduction in penetration, while the stiffness also improves as the Nano-Silica concentration rises. The bitumen binder's softening point increased, suggesting that its temperature susceptibility has improved. Furthermore, in comparison to the binders containing Nano-Silica without ZycoTherm, the ZycoTherm/ Nano-Silica binders showed superior enhancement.

- Adding Nano-Silica to the base bitumen increased its viscosity, which could be related to the stiffening of the Nano-Silica enhanced bitumen binder. However, the base binder modified with Nano-Silica had greater values than the ZycoTherm binders modified with Nano-Silica. As a result, adding ZycoTherm reduces viscosity. At 135 $^{\circ}$C, the modified binders' viscosity values were all within the Superpave standards.

- By adding Nano-Silica into base bitumen, the high-temperature susceptibility decreased while ZycoTherm binders modified with Nano-Silica showed a superior performance.
- By incorporating Nano-Silica into the base and ZycoTherm binders for storage stability, all modified binders containing Nano-Silica were shown to remain stable when maintained at elevated temperatures.

- According to the Superpave rutting resistance parameter, it can be inferred that adding Nano-Silica to the base and ZycoTherm binders increased ($G^*$) and decreased ($\delta$) values, resulting in a greater $G^*/\sin\delta$ values. An increment in $G^*/\sin\delta$ parameter reflects the influence of Nano-Silica in stiffening and improving the elasticity of the base and ZycoTherm binders, rendering them more rut-resistant. Furthermore, adding Nano-Silica to the base and ZycoTherm binders resulted in a one-grade improvement in the high-temperature PG.

- With the incorporation of Nano-Silica, the recovery of the base and ZycoTherm binders was enhanced, while the non-recoverable creep compliance was reduced, demonstrating that the Nano-Silica particles improved the rutting resistance.

- The frequency sweep analysis showed that adding Nano-Silica improved the stiffness of the base and ZycoTherm binders, leading in greater ($G^*$), lower ($\delta$), and improved rutting resistance.

- According to the Superpave fatigue resistance parameter, the inclusion of Nano-Silica to the base bitumen reduced phase angle values while increasing complex modulus values, as predicted because of Nano-Silica's increased stiffness and elasticity. Furthermore, following PAV aging, the complex modulus of ZycoTherm binder was greater than the base bitumen related to the stiffening impacts of the ZycoTherm mixing procedure with base bitumen. As a result, these modifications led to an increment in $G^*.\sin\delta$, indicating a worsening in fatigue resistance.

- The BBR test findings revealed that adding Nano-Silica to the base bitumen increased creep stiffness and decreased m-value at low temperatures. This exhibits the ineffectiveness of Nano-Silica at low temperatures, as well as its resistance to cracking. Furthermore, the inclusion of ZycoTherm material had a good influence on changing the grade from -16 ˚C to -22 ˚C, meaning that the ZycoTherm/ Nano-Silica modification had a greater effect on low-temp grade owing to the presence of ZycoTherm.

- The addition of ZycoTherm or Nano-Silica to the mixes raised the ITS and TSR values. The ITS findings of all samples revealed that bitumen modified with Nano-Silica without ZycoTherm considerably improves the ITS values of the samples in both dry and wet conditions. As a result, modified mixes with Nano-Silica in wet conditions have a greater ITS value than unmodified mixes in dry conditions. Furthermore, the addition of ZycoTherm increased the strength of the mixes against water. This beneficial impact may be related to the formation of strong bonding between asphalt binder and aggregate, which prevented water from passing through this binding. Additionally, the modification containing both ZycoTherm and Nano-Silica performed superior in terms of ITS and TSR.

- The addition of ZycoTherm and Nano-Silica to mixes resulted in a significant increase in the resilient modulus an average of 36.2% and 41.2% at 25°C for mixtures modified with Nano-Silica without ZycoTherm and mixtures containing both ZycoTherm /Nano-Silica, respectively. This indicated that the higher the resilient modulus values, the thinner the wearing course thickness.

- Generally, Nano-Silica, ZycoTherm, and ZycoTherm/ Nano-Silica modified binders enhanced physical and rheological characteristics at all percentages. The current research showed that adding Nano-Silica to the base and ZycoTherm binders could prevent bitumen from rutting at elevated temperatures. Furthermore, the combination of ZycoTherm and Nano-Silica as a WMA demonstrated superior performance in all rheological and mechanical performance tests, varying from lower mixing and compaction temperatures due to the presence of ZycoTherm to superior performance in resilient modulus tests and greater resistance to moisture damage. Additionally, ZycoTherm/Nano-Silica as composite bituminous mixtures were shown to be more cost effective and have a longer life duration than a base bituminous mixture.

### 5.2 Recommendations for Future Research

The following are some future prospective study fields and concerns that might be addressed:

- Overall, the findings showed that ZycoTherm/Nano-Silica may increase the performance of bitumen binder against rutting. The efficiency of this modification in rutting resistance may be investigated further by performing wheel-tracking test.

- To evaluate fatigue resistance of ZycoTherm/Nano-Silica bitumen binders, more advanced test methods like linear amplitude sweep is suggested. Moreover, additional performance testing for assessing rutting resistance and fatigue life are necessary to acquire a complete knowledge of the ZycoTherm/Nano-Silica impact on bituminous mixes.

- A comparable investigation on ZycoTherm/Nano-Silica modified bitumen binders to evaluate the chemical characteristics is suggested.

- A comparable investigation on ZycoTherm/Nano-Silica modified bituminous mixes including granite and limestone with varying aggregate gradations is suggested.

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# APPENDICES

## Appendix A

### MSCR Test Results @ 0.1kPa for Base, Nano-Silica, and ZycoTherm/Nano-Silica Composite

| | | Base | 2% NS | 4% NS | 6% NS | 0.1% ZT+2% NS | 0.1% ZT+4% NS | 0.1% ZT+6% NS |
|---|---|---|---|---|---|---|---|---|
| Stress level (kPa) | Time (s) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) |
| 0.1 | 0.1 | 0.04288 | 0.02610 | 0.02823 | 0.02317 | 0.02876 | 0.03941 | 0.02397 |
| 0.1 | 0.2 | 0.08256 | 0.04847 | 0.05326 | 0.04234 | 0.05326 | 0.07217 | 0.04527 |
| 0.1 | 0.3 | 0.11980 | 0.06977 | 0.07670 | 0.06045 | 0.07697 | 0.10440 | 0.06498 |
| 0.1 | 0.4 | 0.15630 | 0.09108 | 0.10010 | 0.07803 | 0.09987 | 0.13660 | 0.08416 |
| . | . | . | . | . | . | . | . | . |
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| . | . | . | . | . | . | . | . | . |
| 0.1 | 97.65 | 3.10254 | 1.70904 | 1.81316 | 1.35654 | 1.82659 | 2.57027 | 1.56557 |
| 0.1 | 98.10 | 3.10174 | 1.70874 | 1.80636 | 1.35237 | 1.82372 | 2.56947 | 1.56507 |
| 0.1 | 98.55 | 3.10094 | 1.70844 | 1.80136 | 1.34670 | 1.82120 | 2.56567 | 1.56427 |
| 0.1 | 99.00 | 3.10074 | 1.66880 | 1.78821 | 1.34220 | 1.81932 | 2.55157 | 1.52545 |

## Appendix A (Continued)

### MSCR Test Results @ 3.2 kPa for Base, Nano-Silica, and ZycoTherm/Nano-Silica Composite

| | | Base | 2% NS | 4% NS | 6% NS | 0.1% ZT+2% NS | 0.1% ZT+4% NS | 0.1% ZT+6% NS |
|---|---|---|---|---|---|---|---|---|
| Stress level (kPa) | Time (s) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) | Creep compliance (1/kPa) |
| 3.2 | 100.1 | 1.406 | 0.833 | 0.906 | 0.7345 | 0.9225 | 1.267 | 0.7896 |
| 3.2 | 100.2 | 2.704 | 1.561 | 1.707 | 1.327 | 1.744 | 2.369 | 1.457 |
| 3.2 | 100.3 | 4.101 | 2.256 | 2.486 | 1.911 | 2.558 | 3.441 | 2.113 |
| 3.2 | 100.4 | 5.307 | 2.953 | 3.262 | 2.472 | 3.389 | 4.508 | 2.773 |
| . | . | | | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| 3.2 | 97.65 | 3.10254 | 1.70904 | 1.81316 | 1.35654 | 1.82659 | 2.57027 | 1.56557 |
| 3.2 | 98.1 | 3.10174 | 1.70874 | 1.80636 | 1.35237 | 1.82372 | 2.56947 | 1.56507 |
| 3.2 | 98.55 | 3.10094 | 1.70844 | 1.80136 | 1.34670 | 1.8212 | 2.56567 | 1.56427 |
| 3.2 | 99 | 3.10074 | 1.66880 | 1.78821 | 1.34220 | 1.81932 | 2.55157 | 1.52545 |

# Appendix B

## Phase Angle for Base, Nano-Silica, and ZycoTherm/Nano-Silica Composite

| | | Base | 2% NS | 4% NS | 6% NS | 0.1% ZT+2% NS | 0.1% ZT+4% NS | 0.1% ZT+6% NS |
|---|---|---|---|---|---|---|---|---|
| **Temp (°C)** | **Freq (Hz)** | ***δ*** | ***δ*** | ***δ*** | ***δ*** | ***δ*** | ***δ*** | ***δ*** |
| 10 | 1 | 30.60 | 25.47 | 24.81 | 20.24 | 21.31 | 25.352 | 21.95 |
| 10 | 2.564 | 27.33 | 21.51 | 20.88 | 17.09 | 18.62 | 21.344 | 17.86 |
| 10 | 4.167 | 24.79 | 19.53 | 19.75 | 15.51 | 16.98 | 19.422 | 16.69 |
| 10 | 5.769 | 22.98 | 18.58 | 18.91 | 14.76 | 16.15 | 17.82 | 15.95 |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| 82 | 10.34 | 87.34 | 68.26 | 71.90 | 62.64 | 71.45 | 75.49 | 64.62 |
| 82 | 12 | 86.21 | 65.08 | 72.84 | 59.72 | 71.24 | 75.55 | 64.51 |
| 82 | 13.64 | 83.98 | 64.86 | 69.82 | 59.52 | 70.62 | 76.68 | 64.33 |
| 82 | 15 | 83.24 | 64.37 | 69.73 | 59.07 | 71.28 | 75.21 | 64.26 |

# Appendix C

## Complex modulus for base, Nano-Silica, and ZycoTherm/Nano-Silica composite

| | | Base | 2% NS | 4% NS | 6% NS | 0.1% ZT+2% NS | 0.1% ZT+4% NS | 0.1% ZT+6% NS |
|---|---|---|---|---|---|---|---|---|
| **Temp (°C)** | **Freq (Hz)** | ***G*(Pa)*** | ***G*(Pa)*** | ***G*(Pa)*** | ***G*(Pa)*** | ***G*(Pa)*** | ***G*(Pa)*** | ***G*(Pa)*** |
| 10 | 1 | 788800 | 2081200 | 1609920 | 2535100 | 1955772 | 1061450 | 2935940 |
| 10 | 2.564 | 964800 | 2835800 | 2340000 | 3946502 | 2119320 | 1541150 | 3556000 |
| 10 | 4.167 | 1094800 | 3075600 | 2508300 | 4374000 | 2359560 | 1729000 | 3827600 |
| 10 | 5.769 | 1118800 | 3390000 | 2726100 | 4606500 | 2419200 | 1866150 | 4128600 |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| 58 | 7.317 | 6024 | 20757 | 15669 | 30975 | 13935.6 | 7942.5 | 26910 |
| 58 | 8.824 | 6972 | 23991 | 18243 | 35925 | 16203.6 | 9247.5 | 31290 |
| 58 | 10.34 | 7828 | 27324 | 20664 | 40710 | 18379.2 | 10485 | 35355 |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| . | . | . | . | . | . | . | . | . |
| 82 | 12 | 681 | 2139 | 1674 | 3211 | 1389 | 1224 | 3281 |
| 82 | 13.64 | 750 | 2393 | 1755 | 3540 | 1546 | 1369 | 3677 |
| 82 | 15 | 801 | 2581 | 1927 | 3815 | 1898 | 1581 | 3998 |

# Appendix D

## Turnitin Similarity Report

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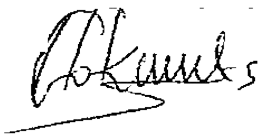

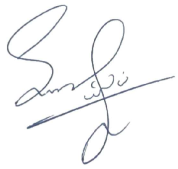

Prof. Dr. Hüseyin Gökçekuş

Assoc. Prof. Dr. Shaban Ismael Albrka

# Appendix E

## Curriculum Vitae

1. **Name –Last Name:** DANIA ATEF ALOTHMAN

2. **Date of Birth:** 31.08.1992

3. **Education:**

| Degree | Department | University | Year |
|---|---|---|---|
| Ph.D. | Civil & Environmental Engineering | Near East University | 2022 |
| M.Sc. | Civil Engineering | Isra University | 2018 |
| B.Sc. | Civil Engineering | Al-Ahliyya Amman University | 2014 |

4. **Academic Title:** M.Sc.

   **Date for Assist. Professor title: N/A**

   **Date for Associate Professor title: N/A**

   **Date for Professor title: N/A**

5. **Master and Ph.D Thesis Supervised:**

   **6.1. Master`s thesis: 0**

   **6.2. PhD thesis: 0**

6. **Publications**

   **6.1. Articles published in international peer-reviewed journals (SCI & SSCI & Arts and Humanities)**

   1. Rheological Properties of Hot and Warm Asphalt Binder Modified with Nanosilica. *MATERIALS SCIENCE (MEDŽIAGOTYRA)*. (2022).

   **6.2. Articles published in other international peer-reviewed journals**

   1. Impacts of Climate Change on Human Health. *International Journal of Innovative Technology and Exploring Engineering.* (2018). (Scopus).
   2. Evaluation of the Impact of Sustainable Transportation Alternatives on Environment Using Fuzzy PROMETHEE Method. *International Journal of Innovative Technology and Exploring Engineering. (*2019). (Scopus).
   3. Effect of Pavement Friction Factors on Skid Resistance of Highway Pavements using Prediction Models. *International Journal of Innovative Technology and Exploring Engineering*. (2019). (Scopus).
   4. AN INTRODUCTION TO ONSHORE STRUCTURES' CONSTRUCTION. *Academic Research International. (2019).*

   **6.3. Bulletins presented in international scientific meetings and published in proceedings**

### 6.4. International books or sections of books published

### 6.5. Articles published in national journals

### 6.6. Bulletins presented in national scientific meetings and published in proceedings

### 6.7. Other publications

### 6.8. International Citations

## 7. National & International Project

## 8. Scientific and Professional Memberships

Jordan Engineers Association

## 9. Awards

Ranked first in B.Sc among 150 students, Civil Engineering Departement, Al-Ahliyya Amman University, 2014

## 10. Undergraduate or Graduate courses taught:

| Academic Year | Semester | Course Name | Hours per Week | | Number of Students |
|---|---|---|---|---|---|
| | | | **Theory** | **Lab** | |
| 2014/2015 | **Fall** | Highway and Traffic | 0 | 3 | 60 |
| | | Soil Mechanics | 0 | 3 | 66 |
| | | Civil Engineering Drawing | 0 | 2 | 45 |
| 2014/2015 | **Spring** | Surveying | 0 | 3 | 67 |
| | | Civil Engineering Drawing | 0 | 2 | 56 |
| 2015/2016 | **Fall** | Environmental and Sanitary | 0 | 3 | 45 |
| | | Hydraulic | 0 | 3 | 58 |
| 2015/2016 | **Spring** | Soil Mechanics | 0 | 3 | 58 |
| | | Civil Engineering Drawing | 0 | 2 | 68 |
| 2016/2017 | **Fall** | Civil Engineering Drawing | 0 | 2 | 43 |
| 2016/2017 | **Spring** | Soil Mechanics | 0 | 3 | 50 |
| | | Highway and Traffic | 0 | 3 | 48 |
| | | Surveying | 0 | 3 | 61 |

## Appendix F

## Ethics Letter

***TO THE INSTITUTE OF GRADUATE STUDIES***

***REFERENCE: DANIA ATEF ALOTHMAN (20177819)***

We would like to inform you that the above candidate is one of our postgraduate students in Civil Engineering Department. She is taking thesis under our supervision on the thesis entailed: ***EVALUATION OF THE PERFORMANCE CHARACTERISTICS OF ASPHALT BINDERS AND MIXTURES CONTAINING WARM ADDITIVE MODIFIED WITH NANO-SILICA.*** The data used in her study was our own data obtained from experimental work conducted by the student in the Arab Center for Engineering Studies (ACES) -Jordan.

Please do not hesitate to contact us if you have any further queries or questions.

Thank you very much indeed.

*Best Regards,*

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