Influence of Modifiers, Anti-stripping Agents and Fillers on Rheological and Mechanical Performance of Asphalt Mastic and Asphalt Mixture

by

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A Thesis Submitted to the

School of Graduate Studies

In partial fulfillment of the requirements for the degree of

Master of Engineering

Faculty of Engineering and Applied Science

Memorial University of Newfoundland

May 2023

St. John's Newfoundland and Labrador Canada

Abstract

Filler, a fine powder used in asphalt mixture, plays a dual role as an inert filler to fill gaps between mineral aggregates and an active filler to mix with asphalt binder to generate a high-consistency asphalt mastic. Many studies have been conducted to develop a rheological parameter that can assess the deformation and creep characteristics of asphalt binders and asphalt mastics. This study investigated the creep recovery performance of asphalt binder and mastic. Mastic is prone to distresses in flexible pavement that worsen with aging, including cracking and moisture-induced damage. The study highlights the importance of fillers combined with modifiers and anti-stripping agents and compares the rheological and mechanical performance of aged asphalt mastics and asphalt mixtures. Multiple Stress Creep Recovery (MSCR) was utilized to understand the rutting performance of aged asphalt binder and mastic. The performance of asphalt mastic with different filler-binder ratios or proportions of different fillers combined with SBS or Gilsonite containing Zycotherm or AD-Here was utilized. Rolling Thin-Film Oven (RTFO), protocol was applied to simulate asphalt production time aging. The study utilized various parameters such as non-recoverable creep compliance, stress sensitivity analysis, percent recovery analysis, and polymer modification curve to compare the performance of the binders and mastics. Scanning Electron Microscope (SEM), and X-ray fluorescence spectroscopy test (XRF), were carried out to shed light on the physical and chemical properties of the fillers. The Marshall stability and flow test, Indirect Tensile Strength (ITS), and Retained Marshall Stability tests were performed to elucidate the mixtures' mechanical performance and moisture susceptibility. Finally, ANOVA analysis was conducted at the binder, mastic, and mixture level to determine the factors influencing the rutting performance of asphalt mastics and the mechanical performance of asphalt mixtures. According to the experimental data from binder level analysis, 0.1% Zycotherm as an anti-stripping agent modified with 4% SBS satisfied binder performance requirements. Mastic and mixture level analysis suggested that HL0.5 modified with 4% SBS containing 0.1% Zycotherm was predominant when only active or inert filler is used and 10% HL and 70% LS containing 4% SBS, and 0.1% Zycotherm was predominant when a combination of active and inert filler was used. These mastics satisfied all the requirements for rutting, moisture damage, and cracking resistance. However, the combination of active and inert filler (10% HL + 70% LS) performed slightly better than the mastic prepared with only active filler (HL). The findings highlight the importance of fillers, modifiers and anti-stripping agents in enhancing the rutting and moisture-induced damage resistance of asphalt mixtures and the usefulness of the MSCR test in evaluating the performance of the asphalt mastic.

Acknowledgments

"A dream does not become a reality through magic; it takes sweat, determination and hard work"-Colin Powell. From a small city in Bangladesh to study at one of the leading universities in North America need much motivation, determination, and hard work. I have always wanted to be a professional researcher and educator in pavement engineering. The Advanced Road & Transportation Engineering Lab (ARTEL) research group of the Memorial University of Newfoundland provided me with the necessary opportunity. I feel proud to be a part of the ARTEL pavement research group. Great ups and downs over the last two years have led to the creation of this enormous study. I wish to acknowledge numerous individuals for their contributions and support along the way to my master's degree because, without their help, it would not have been as easy to get where I am now.

Firstly, I want to thank my supervisor, Dr. Kamal Hossain, for his unwavering encouragement, inspiration, direction, and participation throughout my research program. His enthusiasm, commitment, and desire for cutting-edge knowledge and concepts have inspired me to do quality research and further my professional development. Being under the supervision of Dr. Hossain has been a blessing, and I have learned so much that I will keep in mind for the rest of my life. My co-supervisor Dr. Carlos Bazan always helped and guided me with any technical issue I faced during my lab activities and access to other labs at MUN. I am lucky to work with him.

I would like to acknowledge the financial support the City of St. John's and the School of Graduate Studies (SGS) provided. I wish to thank Gary Caul and Mark White from the City of St. John's. Appreciation is also extended to the Center for Research and Education in Advanced Transportation Engineering Systems (CREATES), Rowan University, USA, for their invaluable help in lab testing. Without the technical and laboratory support from CREATES member Ahmad Alfalah, Dr. Surya Teja Swarna Debzani Mitra, Dr. Sk Faisal Kabir, and CREATES director Dr. Yusuf Mehta, it was impossible for our new research group to conduct all laboratory works.

Also, I would like to acknowledge McAsphalt, Yellowline Asphalt Products Limited, Atlantic Minerals, Enjoy Stone, American Gilsonite Company, and Zydex Industries for providing asphalt, modifiers, fillers, and anti-stripping agents for the accomplishment of this study. Appreciation is also extended to Dylan Goudie from CREAIT Network, Matt Crocker from the Department of Earth Sciences crushing facility, Shawn Organ from Civil Engineering lab, Arman Mackey from the finance department, Ankan Mohajan and the Civil Engineering students of term 7 (Spring-2022) for their help during the sample preparation.

Thanks to all my ARTEL research group members, especially Dr. Surya Teja Swarna, and Towhidul Islam for their continuous suggestions, advice, research ideas, and feedback. Besides that, I want to thank the Teaching Assistants' Union of Memorial University of Newfoundland (TAUMUN), Graduate Students' Society (MUN) and Engineering Graduate Students' Society (EGSS) to give me some opportunity to work for them and help me to improve my leadership skills.

Last but not least, I want to honor the sacrifice made by my wife Samiha Prachi, my parents, my sister, and my in-laws for allowing me to follow my goal and leave them behind. I was inspired and fueled by their support and guidance from hundreds of miles away, which helped me stay mentally alert and committed to my studies.

Dedication

I would also like to dedicate this thesis to my loving mother, Shahena Hasnat.

Disclaimer

In collaboration with the City of St. John’s, this research was carried out. The opinions, results, and conclusions in this report are the result of the authors’ research, which is reflected in its contents. The opinions expressed in the materials may not necessarily represent those of Memorial University or the City of St. John’s. This thesis does not represent a rule, specification, or standard.

List of Acronyms

AASHTO: American Association of State Highway and Transportation Officials

ASTM: American Society for Testing and Materials

AI: Asphalt Institute

Ba: Basalt

BET: Brunauer Emmett Teller

CSCE: Conference of the Canadian Society for Civil Engineering

CTAA: Canadian Technical Asphalt Association

DOT: Department of Transportation

DSR: Dynamic Shear Rheometer

DM: Dolomite

FA: Fly Ash

F/B: Filler-Binder Ratio

FHWA: Federal Highway Administration

HL: Hydrated Lime

HMA: Hot Mix Asphalt

ITS: Indirect Tensile Strength

LAS: Liquid Anti-stripping Additives

LCA: Life Cycle Analysis

LS: Limestone

MD: Moisture Damage

MF: Marshall Flow

MS: Marshall Stability

MSCR: Multiple Stress Creep Recovery

MQ: Marshall Quotient

MUN: Memorial University of Newfoundland

NCHRP: National Cooperative Highway Research Program

NL: Newfoundland and Labrador

OBC: Optimum Binder Content

PAV: Pressure Aging Vessel

PG: Performance Graded

RAP: Reclaimed Asphalt Pavement

RMS: Retained Marshall Stability

RTFO: Rolling Thin Film Oven

SBS: Styrene-Butadiene-Styrene

SEM: Scanning Electron Microscope

SG: Specific Gravity

SHRP: Strategic Highway Research Program

SMA: Stone Mix Asphalt

SSA: Specific Surface Area

TAC: Transportation Association of Canada

TCH: Trans-Canada Highway

TSR: Tensile Strength Ratio

XRF: X-ray fluorescence spectroscopy

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Chapter 1: Research Background and Literature Review

1.1 Background Information and Motivation

The rapid development of asphalt pavement is credited with its successful global performance. Due to rising traffic volumes, axle weights, shifting weather patterns, and, in some cases, fewer maintenance operations, pavement structures have been degrading at an increased rate in recent years. Significant effort and resources are invested into preserving asphalt pavements to maintain conditions. As such, the annual demand for new asphalt binder is more than 110 million tonnes worldwide (Garciaa et al., 2010). Asphalt binder is a residue obtained from the crude oil refining process. As per the Canadian Energy Research Institute (CERI) crude oil forecasting model, there will be a drastic increase in binder production across Canada in the next two decades. A common problem encountered with many asphalt binders is that they can deteriorate rapidly from age-hardening (Lu et al., 1998). The sources of asphalt binder (non-renewable resource) are declining. Furthermore, a drastic increase in the cost of virgin asphalt binder has motivated many researchers to search for a durable and long-lasting pavement without any major distresses using additives.

Most pavement distresses are caused by wide temperature ranges, water infiltration in the subgrade, a change in traffic, or inadequate compaction during construction. The stress-strain characteristics of asphalt binder are both time and temperature-dependent because of the viscoelastic and thermoplastic characteristics of the material. Therefore, asphalt binders can exhibit significant deformation based on the wheel load and temperature change. A binder must be stiff for high-temperature performance, while the same material must be soft for low-temperature performance. When selecting a binder, asphalt manufacturers may emphasize high-temperature performance at the expense of low-temperature performance or vice versa (Anderson et al., 2010). The durability of pavements is greatly influenced by the materials used in asphalt pavement construction. Asphalt's structure, chemical composition, and surface tension characteristics are changed by aging of asphalt in the asphalt mix. As a result, the asphalt binder stiffens and is more susceptible to moisture-related problems (Hossain et al. 2018).

The City of St. John's (Newfoundland and Labrador, Canada) has a distinct climate with significant amounts of yearly precipitation (119.1 cm) and snowfall (322 cm), mild summers, and harsh winters. Over the past 80 years, the city's average temperature has varied from -9.3 to 15.5°C. Air humidity fluctuates between 79 to 83%. The typical wind speed is between 20 and 28 km/h (Ali et al., 2018). Due to the city's location on an island and its small population, its roadways see relatively little heavy traffic. Although St. John's roads have little traffic and have a solid base, they are often severely structurally and functionally distressed. The city frequently experiences pavement deterioration brought on by heavy traffic, tires, dampness, freezing and thawing, temperature variations, and other environmental conditions. In St John's, rutting, moisture-induced damage and cracking are the primary distress mechanism observed on the pavements (Ali et al., 2018). If not addressed immediately, these pavement problems necessitate extensive repair and maintenance work and cause overall irritation to users.

The Hot Mix Asphalt (HMA) used in St John’s should be resilient enough to endure these pavement distresses. Numerous studies have acknowledged that polymer modifiers, anti-stripping agents, and active and inert fillers in asphalt mixture can improve rutting resistance, moisture-induced damage resistance, and cracking resistance. The research goal of this thesis is to develop an asphalt mixture for the city of St John’s by using different modifiers, anti-stripping agents, and fillers that can withstand the pavement distresses.

1.2 Overview of the Materials

The aging process of asphalt causes it to become stiffer and gradually make it brittle (Lu & Isacsson, 2002). Although, age-related asphalt hardening improves rutting resistance performance, damage is caused by accumulated cracking and moisture-induced damage (Lesueur, 2009). To attain long-lasting and high-quality pavement, governmental agencies continue to spend a considerable amount of money into improving conditions. However, many of these facilities already exhibit early symptoms of distress (ITF Transport Outlook 2017).

Including modifiers improves the binder's performance and reduces pavement distresses (Yildirim, 2007). Polymer modifiers are the most effective strategy for preventing excessive plastic deformations at high temperatures (Airey, 2002). The addition of SBS polymer improves HMA's resistance to moisture-induced damage as well as its resistance to rutting and cracking (Alata & Ethem, 2013; Ahmed et al., 2021). This may result in a double boost in the pavement's service life (Iskender et al., 2012a). Gilsonite, which is brittle in its raw state, is a natural deposit of mineral binder. However, it has a good affinity for asphalt and shows identical performance to resist the rutting of binder (Liu & Li, 2009; Mirzaiyan et al., 2019). Additionally, it demonstrates that a higher Gilsonite concentration enhances rutting performance at high temperatures. A common downside of Gilsonite modification of binders is thermal cracking due to brittleness at low temperatures (Rajbongshi & Das, 2009). The shortfall in butadiene supplies might cause SBS prices to rise much more in the foreseeable future. Gilsonite can therefore be utilized as an alternative to polymers.

The two primary elements of asphalt pavement are asphalt binder and aggregate. The aggregate surface is more attracted to water than binder due to its surface energy characteristics

(Little & Jones, 2003). Therefore, anti-stripping agents are added to the asphalt binder to combat moisture damage. A good bond between the asphalt binder and aggregates ensures excellent performance. Should this bond be compromised, the asphalt surface may display stripping which can also eventually lead to rutting, raveling, cracking, and other problems that can cause the asphalt pavement to disintegrate completely (Airey et al., 2007; Baldi-Sevilla et al., 2017; Chen & Huang, 2007). Numerous studies demonstrate that applying anti-stripping compounds may reduce the stripping of asphalt pavement (Cheng et al., 2011; Xiao et al., 2010). For improving moisture resistance, the City of St. John's has traditionally employed anti-stripping agent AD-Here and Hydrated Lime (HL). However, several research and industry professionals advised using Zychotherm, Pave Bond, and Kling beta. It is possible to compare the use of AD-Here with Zychotherm, Pave Bond, and Kling beta in the experimental plan.

With the inclusion of fillers in the binder, the cohesion between components formed a mastic, where fillers influence the asphalt mixture by increasing the stiffness and altering the moisture resistance, workability, and compaction characteristics of asphalt mixtures (Rieksts et al., 2019; Huang et al., 2007). Most of this filler aggregate passes a 0.075 mm sieve (Kuity et al., 2014). A filler's role in an asphalt mixture may be divided into the following separate actions: 1) functioning as an inert filler material (Limestone (LS), Dolomite (DM), Basalt (Ba), etc.) to fill spaces between coarse aggregates, and 2) acting as an active filler material (Hydrated Lime (HL), Fly-ash (FA), diatomite) when it comes into contact with binder at the interface (Kim & Little, 2004). Fillers can alter the materials' chemical and physical features through surface interactions and their own physical qualities (Taylor & Airey, 2008; Wang et al., 2011).

In this study, HL and FA are used as active fillers. HL enhances the ductility of asphalt mastic, lowers aging and boosts rutting and moisture resistance (Bai et al., 2007). Several research efforts have looked at the positive impacts of FA on the asphalt mix's ability to resist moisture and rutting and maintain tensile strength (Asi & Assaad, 2005; Xiao et al., 2012). LS filler was selected for this study because it is a broadly used filler. Along with having a strong stiffening capacity, LS filler also helps the polymer phase function as effectively as possible (Rieksts et al., 2019). In a previous study 75% Ba with 5% HL showed better low-temperature cracking performance (Das & Shing, 2017), therefore, Ba was also selected for this study.

The ability of the asphalt mixture to withstand moisture-induced cracking, raveling, and rutting is a major factor in how well an asphalt pavement performs. The combination and interaction of various asphalt modifiers, anti-stripping agents, and fillers has sparked efforts to create a better asphalt mixture to lower the pavement distresses and cost of life-cycle pavement maintenance.

1.3 Overview of the Filler/Binder Ratio

A given amount of binder can be fixed by a particular filler in a filler-binder (F/B) combination. Thermal cracking can occur when filler usage reaches a predetermined threshold. This is due to the binder being cemented around by the filler particles in an excessive amount. Therefore, the right amount of filler should be utilized to give strong rutting resistance while also keeping good performance under low temperature (Antunes et at., 2014). For high-temperature performance, it is often advised that the filler-binder ratio not exceed 1.4 (Zhang et al., 2004), The ideal filler content for modified mastic is between 0.8 and 1.2. (Qiu, 2013). Due to the considerable rise in mastic consistency, HL and FA fillers must be introduced in small quantities. The F/B ratio was lowered to 0.3 after taking the absorption capacity test findings into account and a F/B range of 0.3-0.6 is advised to use (Antunes et al., 2014). Thus, the proportion of active filler was chosen to be 10% - 30% by the weight of the base binder while the proportion of inert filler was chosen between 50% - 70% by the weight of the base binder to prepare the mastics. In the case of combining active and inert filler, a F/B ratio of 0.8 is used in this study to prepare the mastics. Effects of F/B ratios on the high-temperature characteristics of various asphalt mineral filler mastics revealed nonlinear rheological behavior (Yi-qiu, 2010). On the other hand, unmodified mastics show 50% recovery at low temperature, whereas polymer-modified mastics offer more significant recovery (Rieksts, 2019). High-temperature recovery of modified asphalt mastic is yet to be evaluated. Use of high-quality modified asphalt is therefore important to prevent or minimize the negative impacts of asphalt ageing on the interaction between asphalt and filler (Wu et al., 2021).

1.4 Overview of the Experiment

SHRP's Superpave methods for binder characterization work well for neat binders but are inadequate for polymer-modified binders. Two important rheological parameters, complex modulus (G*) and phase angle (δ) of asphalt binder and mastic, can be measured from this test and under linear viscoelastic circumstances, master curves may be constructed (Underwood & Kim, 2015). The Superpave rutting parameter, often known as the index (G/sinδ), has also been used to measure the flow characteristics of asphalt mastic. On the contrary, the AASHTO T 315 test method cannot measure mechanical and viscoelastic characteristics of polymer-modified binders beyond their linear viscoelastic ranges. NCHRP Project 9-10, "Superpave Methods for Modified Binders," was initiated to evaluate whether the present Superpave binder test protocols are adequate for modified binders, and the study found that Superpave PG criteria cannot be employed to fully characterize binders modified with various polymers due to over simplification in assumptions (Bahia et al., 2000). Furthermore, the Superpave parameter can not measure the energy dissipation of most Polymer-Modified Binder (PMBs) due to delayed elasticity, so a non-reversible cycle loading is suggested (Delgadillo et al., 2006). Nonetheless, Soenen et al. demonstrated that this parameter well predicts the rutting susceptibility of modified binders (Soenen et al., 2006).

Additional tests such as Elastic Recovery (ER), tenacity, and forced ductility, have been developed by several state agencies to characterize polymer-modified binders. The Superpave "PG Plus" requirements refer to these tests and their parameters (D’Angelo et al., 2006). The "PG Plus" test findings, on the other hand, may not be accurate indications of field performance. Furthermore, they are expensive due to the need for special equipment and time. To define polymer-modified binders, another group of researchers proposed the Zero-Shear Viscosity (ZSV) concept (Tabatabaee et al., 2013; Sybilski, 2010). However, in the case of polymer-modified binders, the coherency and reliability of the ZSV test methods are not always guaranteed. The concept of Low Shear Viscosity (LSV) was developed when evaluating the effects of modified binder on laboratory mixing and compaction to solve this problem (Morea et al., 2010; Desmazes et al., 2000). According to Zoorob et al. ZSV and LSV test procedures were not adequate for assessing the high-temperature creep behaviour of polymer-modified binders and he suggested the Multiple Stress Creep & Recovery (MSCR) test technique for polymer-modified (Zoorob et al., 2012). Bahia et al. proposed the Repeated Creep Recovery Test (RCRT) approach to determine the rate of accumulation of permanent strain in the binder (Bahia et al., 2000). Low-stress levels are used in the RCRT test procedure, which may not accurately reflect the real field situation. By performing creep and recovery tests on binder samples at several stress levels (0.1 kPa and 3.2 kPa), D'Angelo et al. enhanced the RCRT test technique. Non-recoverable compliance (J_{nr}), a parameter developed by these researchers, may distinguish a polymer-modified binder from a plain binder. To capture the influence of rejuvenation and inclusion of SBS to rejuvenated binders, MSCR parameters are more efficient than other parameters (Ahmad et al., 2021).

The MSCR test technique involves loading a binder sample for 1 second at constant creep stress and then allowing it to recover for 9 seconds at zero stress. The test is performed at two different levels of stress: 0.1 kPa and 3.2 kPa. at 64°C (Hossain, 2016). For a total of 20 cycles, ten cycles are done at each of the two stress levels. There are no rest intervals between the creep and recovery cycles or when the creep stress is adjusted. In the DSR test, usually there is a 1.0 mm gap between the two parallel plates with a 25 mm plate diameter. However, there is always a debate if the existing test procedures are workable for mastic samples as the filler particles are mixed with the binder. Li et al. suggested to use the 1.0 mm gap between the two parallel plates (Li et al., 2019). The non-recoverable creep compliance (J_{nr}), and MSCR % Recovery (R) values are computed from the test results at both stress levels, as stated in the applicable AASHTO and ASTM standards (AASHTO TP 70, 2009; ASTM D 7405, 2010). Instead of using the rutting parameter ($G^*/\sin\delta$), these two findings from the MSCR test can be utilized to assess rutting potential (D'angelo, 2011). J_{nr} is recommended to represent the binder contribution to an asphalt mixed permanent deformation. J_{nr} diff values may also be generated to analyze a binder's stress sensitivity. Stemphihar et al. proposed a new parameter (J_{nr} slope) for analyzing stress sensitivity (Stemphihar et al., 2018). The MSCR % Recovery value is connected to the delayed elasticity, i.e., the elastomeric response of the polymer in a binder. The % Recovery is used to construct the polymer method MSCR curve (polymer medication curve) to interpret the elastomeric performance of the modified binders and ensure whether the binders are modified with an acceptable range with elastomeric polymer (ASTM D 7405, 2010). According to the Asphalt Institute (AI) guideline, % Recovery value can be used in the quadrant analysis, which will guide the agencies to enhance their operation based on customer satisfaction (AASHTO M 320 Specification, 2010).

The Marshall mix design is the most frequent technique employed for asphalt mix design (Asphalt Institute, MS-2, 2014; Stephen B, 2015). Even though the Marshall technique is empirical in nature, it might be beneficial in specific situations to compare mixes (Diab& Enieb, 2018). For laboratory mix design and assessment of asphalt mixes, Marshall Stability and Flow values combined with density, air voids in the entire mix, voids in the mineral aggregate, or voids filled with asphalt, or both, filled with asphalt are utilized. Additionally, asphalt mixture production at the facility may be observed using Marshall Stability and Flow. Additionally, Marshall Stability and Flow may be used to compare and assess various mixtures and conditioning results, such as with water. Another critical parameter that can be calculated from the Marshall test is the Marshall quotient (MQ), a well-known indicator of a material's resistance to shear loads, permanent deformation, and therefore rutting (Zoorob et al., 2000). Because of the issues related to cracking, the tensile characteristics of bituminous mixes are of interest to pavement engineers. SMA's tensile strength is significant in pavement applications even though it is not nearly as strong in tension as in compression. The tensile characteristics of the bituminous mixture, which are also connected to the cracking characteristics of the pavement, are found using the indirect tensile strength test (ITS). To determine the tensile characteristics of the asphalt mixtures, which are also connected to the pavement's cracking behavior, the indirect tensile strength test (ITS) is utilized (Islam et al., 2015). Moreover, moisture damage is a critical problem affecting asphalt pavements' durability and must be checked during the mix design (Ekblad et al., 2015). The Tensile Strength Ratio (TSR) is the most important factor to consider when assessing the moisture damage of asphalt mixes. The TSR represents the ratio of ITS in wet conditions and ITS in dry conditions. However, occasionally it lacks credibility because it is only a ratio of two numbers (Diab, 2016). Therefore, another parameter, Retained Marshall stability (RMS) (Defense works functional standards, 2005), will be used to explain the mixes' vulnerability to moisture.

Scanning Electron Microscope (SEM), X-ray fluorescence spectroscopy test (XRF), specific gravity (SG) with the pycnometer method, specific surface area (SSA) with Blaine's air permeability test were carried out to shed light on the physical and chemical properties of the fillers. To analyze the effect of different active and inert fillers on the stability, ITS and TSR, an experimental analysis using Design of Experiments (DOE) is performed based on analysis of variance (ANOVA). A multilevel factorial design is widely used in experiments that involve several factors where it is necessary to study the combined effect of the factors on a response (Smucker, 2019). Design-Expert 13 software package generates the treatment combinations, processes the data, and plots the result.

1.5 Objectives

The objectives of this thesis are as follows:

- Develop a comparative analysis of the binders containing different modifiers and different dosages of different liquid anti-stripping agents based on MSCR analysis, Polymer Modification Curve and Quadrant Plot as per the Asphalt Institute (AI) guideline.
- Investigate the performance of different mastics modified with SBS and Gilsonite modifiers in terms of creep recovery performance.
- Understand the effect of filler-binder (F/B) ratio and different proportions of active and inert filler in mastic scales by comparing their rheological performance at high temperature and propose an optimum dose.

- Compare the creep recovery and mechanical performance of different active, inert, and a combination of active and inert fillers in aged asphalt mastic and asphalt mixture containing modifier and liquid anti-stripping agent.
- Gain a basic understanding of the impacts of the combination of active and inert filler in asphalt mixture and compare the mechanical properties of the mixtures.
- Conduct ANOVA analysis at the binder level, mastic level, and mixture level to evaluate the effect of the modifiers, liquid anti-stripping agents and F/B ratios and find an optimum combination.

1.6 Thesis Framework

This thesis has been written in manuscript form. The study's findings are provided in 5 chapters.

Chapter 1 presents the background, motivation, overview of the materials, dose rate, and experiments, objectives, and the contribution of the present study.

Chapter 2 presents the creep recovery performance of aged binder containing modifiers and anti-stripping agents. This chapter was presented as a technical paper at the Canadian Technical Asphalt Association (**CTAA**), 67th Annual Conference 2022, Kelowna, BC, Canada, November 6-9.

Chapter 3 presents the effect of fillers, modifiers and anti-stripping agents in aged asphalt mastic and asphalt mixture. This chapter submitted as a technical paper in Construction and Building Materials by Elsevier. Also, a portion of this chapter submitted as a technical paper to the Canadian Society for Civil Engineering (**CSCE**), Annual Conference 2023, Moncton, NB, Canada.

Chapter 4 investigates the combined effect of active and inert filler on rheological and mechanical performance of asphalt mastic and asphalt mixture. This chapter will be submitted to a journal as a technical paper. A portion of this chapter submitted to Transportation Association of Canada (**TAC**), Annual Conference 2023, Ottawa, ON, Canada and some portion submitted to Canadian Technical Asphalt Association (**CTAA**), 68th Annual Conference 2023, Charlottetown, PEI, Canada.

Chapter 5 summarizes the general conclusions of this study and recommendations and suggestions for future works.

1.7 Significant Contributions

1.7.1 Journal Articles

- **Feroz S I,** Alfalah A, Swarna S T, Hossain K., & Mehta Y. (2023). Investigating the Effect of Fillers, Modifiers and Anti-stripping Agents in Aged Asphalt Mastic and Asphalt Mixture. Journal of Construction and Building Materials by Elsevier. (Under review)
- **Feroz S I,** Mitra D, Kabir S K, Hossain K & Mehta Y (2023). Combined effect of active and inert filler on rheological and mechanical performance of asphalt mastic and asphalt mixture. Road Materials and Pavement Design by Taylor and Francis. (Planning to Submit)

1.7.2 Conference Papers

- **Feroz S I**, Islam T, Hossain K, Ahmed R B & Bazan C (2022). Effect of Rejuvenators and Anti-Stripping Agents on Creep Recovery Property of Modified Aged Binder (Presented at Canadian Technical Asphalt Association (**CTAA**), 67th Annual Conference 2022, Kelowna, BC, Canada, November 6-9. This is the most reputable conference in Pavement Engineering field in Canada.

- **Feroz S I**, Alfalah A, Mitra D, Hossain K & Mehta Y (2022). Creep Recovery Performance of Hydrated Lime and Limestone in Asphalt Mastic. Canadian Society for Civil Engineering (**CSCE**), Annual Conference 2023, Moncton, NB, Canada. (Paper accepted)
- **Feroz S I**, Mitra D, Kabir S K, Hossain K & Mehta Y (2023). Performance of aged asphalt mastic combining active and inert filler materials in terms of creep recovery. Transportation Association of Canada (**TAC**), Annual Conference 2023, Ottawa, ON, Canada. (Abstract accepted and paper under review)
- **Feroz S I**, Mitra D, Mohajan A, Hossain K & Mehta Y (2022). Investigating the combined effect of active and inert filler on rheological and mechanical performance of asphalt mastic and asphalt mixture. (Submitted in Canadian Technical Asphalt Association (**CTAA**), 68th Annual Conference 2023, Charlottetown, PEI, Canada. (Abstract accepted and paper under review)

1.7.3 Technical Report

- **Feroz S I**, Islam T, Hossain K, Bazan C & Aurilio M (2023). Development of Improved Asphalt Mixture for the City of St. John's: Study on Rutting and Moisture Damage Characteristics of Modified Asphalt Binder. City of St. John's, St. John's, Newfoundland, and Labrador.

1.7.4 Poster Presentation

- **Feroz S I**, Alfalah A, Swarna S, Hossain K, Islam T, Mehta Y, & Caul G (2022). Influence of Fillers on Creep Recovery Performance of Aged Asphalt Mastic Containing Modifiers and Liquid Anti-Stripping. (Presented in CTAA, 66th Annual Conference 2022, Kelowna, BC, Canada, November 6-9)

1.8 Co-Authorships

All the research presented in the technical reports, journals, and conference papers in chapters 2-4 has been conducted by the author of this thesis Shahrul Ibney Feroz, under the supervision of Dr. Kamal Hossain and Dr. Carlos Bazan. Shahrul Ibney Feroz also prepared the draft manuscript. The other co-authors supervised the research and reviewed the manuscript.

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Chapter 2: Effect of Modifiers and Anti-Stripping Agents on Creep Recovery Performance of Aged Binder

This chapter has been presented in the 67th Annual Conference of Canadian Technical Asphalt Association (CTAA) as a technical paper as: Feroz, S. I., Islam, T., Hossain, K., Ahmad, R. B., & Bazan, C. (2022), "Effect of Rejuvenators and Anti-Stripping Agents on Creep Recovery Performance of Modified Aged Binder."

2.1 Abstract

Many studies have been conducted to develop a rheological parameter that can assess deformation and creep characteristics of modified asphalt binders. However, very few studies show the effect of modifiers and anti-stripping agents on creep recovery performance of binders. This paper employs the Multiple Stress Creep Recovery (MSCR) test as per AASHTO T 350 to understand creep recovery properties of aged binders. A PG 58-28 binder was blended with two modifiers and varying doses of four different anti-stripping agents. Performance of these binders was compared using non-recoverable creep compliance, stress sensitivity analysis, and % Recovery analysis. AASHTO M 332 specifications have been used to classify all the 24 binders based on the J_{nr} value at 3.2 kPa and stress sensitivity. In addition, Polymer and Quadrant procedures specified by the Asphalt Institute (AI) were employed to interpret the test results. A statistical analysis was conducted at the binder level to identify the significant factors and their comparative effect on the creep recovery performance of binders. According to the experimental data, 0.1 percent Zycotherm as an anti-stripping agent modified with Styrene-Butadiene-Styrene (SBS) satisfied binder performance requirements and showed the best creep recovery performance. Furthermore, SBS-modified binders outperformed Gilsonite-modified binders in terms of creep recovery performance.

2.2 Introduction

Significant effort and resources are invested in preserving bituminous pavements. However, the annual demand for new binder is more than 110 million tonnes worldwide (Garciaa et al., 2010). The cost of virgin binder is high, and is supply is limited. Besides, some other problems associated with binder are that it can deteriorate rapidly from age-hardening (Lu et al., 1998). Stress-strain characteristics of binder or asphalt are both time and temperature-dependent because of the viscoelastic and thermoplastic characteristics of this material. Therefore, asphalt binders can exhibit significant deformation based on the change in wheel load and temperature. For high-temperature performance, a binder must be stiff, while for low-temperature performance, the same material must be soft. When selecting a binder, asphalt manufacturers may emphasize high-temperature performance at the expense of low-temperature performance or vice versa (Anderson et al., 2010).

SHRP's Superpave methods for binder characterization work well for neat binders but are inadequate for polymer-modified binders. For example, the AASHTO T 315 test method cannot measure mechanical and viscoelastic characteristics of polymer-modified binders beyond their linear viscoelastic ranges. NCHRP Project 9-10, "Superpave Methods for Modified Binders," was initiated to evaluate whether the present Superpave binder test protocols are adequate for modified binders, and the study found that Superpave PG criteria cannot be employed to fully characterize binders modified with various polymers due to overly simplification in assumptions (Bahia et al., 2000). Also, the Superpave parameter can not measure the energy dissipation of most Polymer-Modified Binder (PMBs) due to delayed elasticity, so a non-reversible cycle loading is suggested (Delgadillo et al., 2006). Nonetheless, Soenen et al. demonstrated that this parameter well predicts the rutting susceptibility of modified binders (Soenen et al., 2006).

Additional tests such as Elastic Recovery (ER), tenacity, and forced ductility, have been developed by several state agencies to characterize polymer-modified binders. The Superpave "PG Plus" requirements refer to these tests and their parameters (D'Angelo et al., 2006). The "PG Plus" test findings, on the other hand, may not be accurate indications of field performance. Furthermore, they are expensive due to the need for special equipment and time. To define polymer-modified binders, another group of researchers proposed the Zero-Shear Viscosity (ZSV) concept (Tabatabaee et al., 2013; Sybilski, 2010). However, in the case of polymer-modified binders, the coherency and reliability of the ZSV test methods are not always guaranteed. The concept of Low Shear Viscosity (LSV) was developed when evaluating the effects of modified binder on laboratory mixing and compaction to solve this problem (Morea et al., 2010; Desmazes et al., 2000). ZSV and LSV test procedures were not adequate for assessing the high-temperature creep behaviour of polymer-modified binders, according to Zoorob et al. and he suggested the Multiple Stress Creep & Recovery (MSCR) test technique for polymer-modified (Zoorob et al., 2012). Bahia et al. proposed the Repeated Creep Recovery Test (RCRT) approach to determine the rate of accumulation of permanent strain in the binder (Bahia et al., 2000). Low-stress levels are used in the RCRT test procedure, which may not accurately reflect the real field situation. By performing creep and recovery tests on binder samples at several stress levels (0.1 kPa and 3.2 kPa), D'Angelo et al. enhanced the RCRT test technique. Non-recoverable compliance (J_{nr}), a parameter developed by these researchers, may distinguish a polymer-modified binder from a plain binder. To capture the influence of rejuvenation and inclusion of SBS to rejuvenated binders, MSCR parameters are more efficient than other parameters (Ahmad et al., 2021).

The conventional MSCR test technique involves loading a binder sample for 1 second at constant creep stress and then allowing it to recover for 9 seconds at zero stress. The test is performed at two different levels of stress: 0.1 kPa and 3.2 kPa. For a total of 20 cycles, ten cycles are done at each of the two stress levels. There are no rest intervals between the creep and recovery cycles or when the creep stress is adjusted. The non-recoverable creep compliance (J_{nr}), and % Recovery value is computed from the test results at both stress levels, as stated in the applicable AASHTO and ASTM standards (AASHTO TP 70, 2009; ASTM D 7405, 2010). J_{nr} is recommended to represent the binder contribution to an asphalt mixed permanent deformation. J_{nr} diff values may also be generated to analyze a binder's stress sensitivity. Stemphihar et al. proposed a new parameter (J_{nr} slope) for analyzing stress sensitivity (Stemphihar et al., 2018). The % Recovery value is connected to the delayed elasticity, i.e., the elastomeric response of the polymer in a binder. The % Recovery is used to construct the polymer medication curve to interpret the elastomeric performance of the modified binders and ensure whether the binders are modified with an acceptable range with elastomeric polymer (ASTM D 7405, 2010). According to the Asphalt Institute (AI) guideline, % Recovery value can be used in the quadrant analysis, which will guide the agencies to enhance their operation based on customer satisfaction (AASHTO M 320 Specification, 2010).

2.3 Objectives

The major objectives of this experimental study include:

- To evaluate the creep recovery performance of modified binders containing different dosages of liquid anti-stripping agents based on non-recoverable creep compliance, % Recovery, and stress sensitivity.
- To compare the performance of different bituminous binders modified with SBS and Gilsonite modifiers in terms of creep recovery.

- To develop s Polymer Modification Curve to interpret the binders' elastomeric performance and to check if the modifications are within the acceptable range.
- To construct the Quadrant Plot as per the Asphalt Institute (AI) guideline to guide agencies to serve their customers better.

2.4 Materials and Methodology

2.4.1 Materials

2.4.1.1 Asphalt Binder

Following the ever-increasing use of modifiers and additives in asphalt mixture, the research on the characterization of asphalt binders has increased significantly in the past few years. Previously, different traditional tests, i.e., viscosity, penetration, ductility, softening, and flashing point tests, have been extensively used to determine the rutting performance of asphalt binders (Domingos & Faxina, 2015; Loizos et al., 2009). Due to several limitations of these tests, such as loading condition, test temperature frequency, lack of interrelation with properties, and undesirable performance for modified binders, researchers have tried to develop rutting parameters for asphalt

(a) (b)

€ (d) (e) (f)

Figure 2.1 Binder modifiers (a) Gilsonite, and (b) SBS and anti-stripping agents (c) Pave bond lite (d) Zyycotherm e) Kling beta and f) AD-Here

binders which can be used to evaluate rutting behaviour at high temperatures. The American Strategic Highway Research Program (SHRP) developed an approach to assess the rutting potential of asphalt binder by using Dynamic Shear Rheometer (DSR), known as the Superpave rutting parameter ($G^*/\sin\delta$) (AASHTO T 315-12, 2012). According to the Superpave rutting factor criteria, ($G^*/\sin\delta$) must be a minimum value of 2200 Pa for aged binders.

2.4.1.2 Modifiers and Anti-stripping Agents

Two different modifiers were used in this study (**Figure 2.1**) to compare the effect of modifiers on creep recovery performance. Gilsonite was obtained from American Gilsonite Company, whereas SBS modified PG 58-28 was obtained from Yellowline Asphalt Products Limited. Four different liquid anti-stripping agents (**Figure** 2.1**)** with different dosages were used to evaluate the creep recovery performance of liquid anti-stripping agents. Pave Bond Lite® was obtained from Yellowline Asphalt Products Limited, AD-Here was obtained from Valero Energy Inc. ZycoTherm was obtained from Zydex Industries and Kling Beta 2914 (Redicote C-2914) was obtained from Nouryon. Details of the anti-stripping agents and modifiers are shown in **Table 2.1**.

Table 2.1 Anti-stripping agents and dosages parameters

Name of Anti-stripping Agent	Dosage	Modifier
Pave Bond Lite	0.5%, 0.75%, 1%	Styrene-Butadiene-Styrene (SBS) and Gilsonite
AD-Here	0.5%, 0.75%, 1%	
Zycotherm	0.05%, 0.075%, 0.1%	
Kling Beta 2914	0.5%, 0.75%, 1%	

2.4.2 Methodology

2.4.2.1 Sample Preparation and Testing of Binders Containing Modifiers and Liquid Anti-Stripping Agents

To prepare the samples, neat PG 58-28 asphalt was preheated at 160°C for 1 hr to make it fluid. The SBS (4 percent of weight) modified asphalt was provided by Yellowline Asphalt Products Limited, whereas Gilsonite (10% of weight) was blended for 30 minutes at 180°C to prepare the Gilsonite modified binder. Different anti-stripping agents with different dosages were added with the modified binders. A magnetic stirrer was used for 30 minutes at 180°C to prepare the samples containing liquid anti-stripping agents and modifiers. Finally, to simulate short-term laboratory aging of the base binders and modified binders, The Rolling Thin-Film Oven Test (RTFO) in accordance with AASHTO T 240 was employed. To prepare the aged samples, a continuous heat of 163°C was applied to the binders for 75 minutes. Finally, the MSCR test protocol was employed using the DSR equipment. There were 24 binders containing different dosages of anti-stripping agents. All the samples including the control binder tested only once due to time constraint. The experimental plan of the binder level study is shown in **Figure 2.2**.

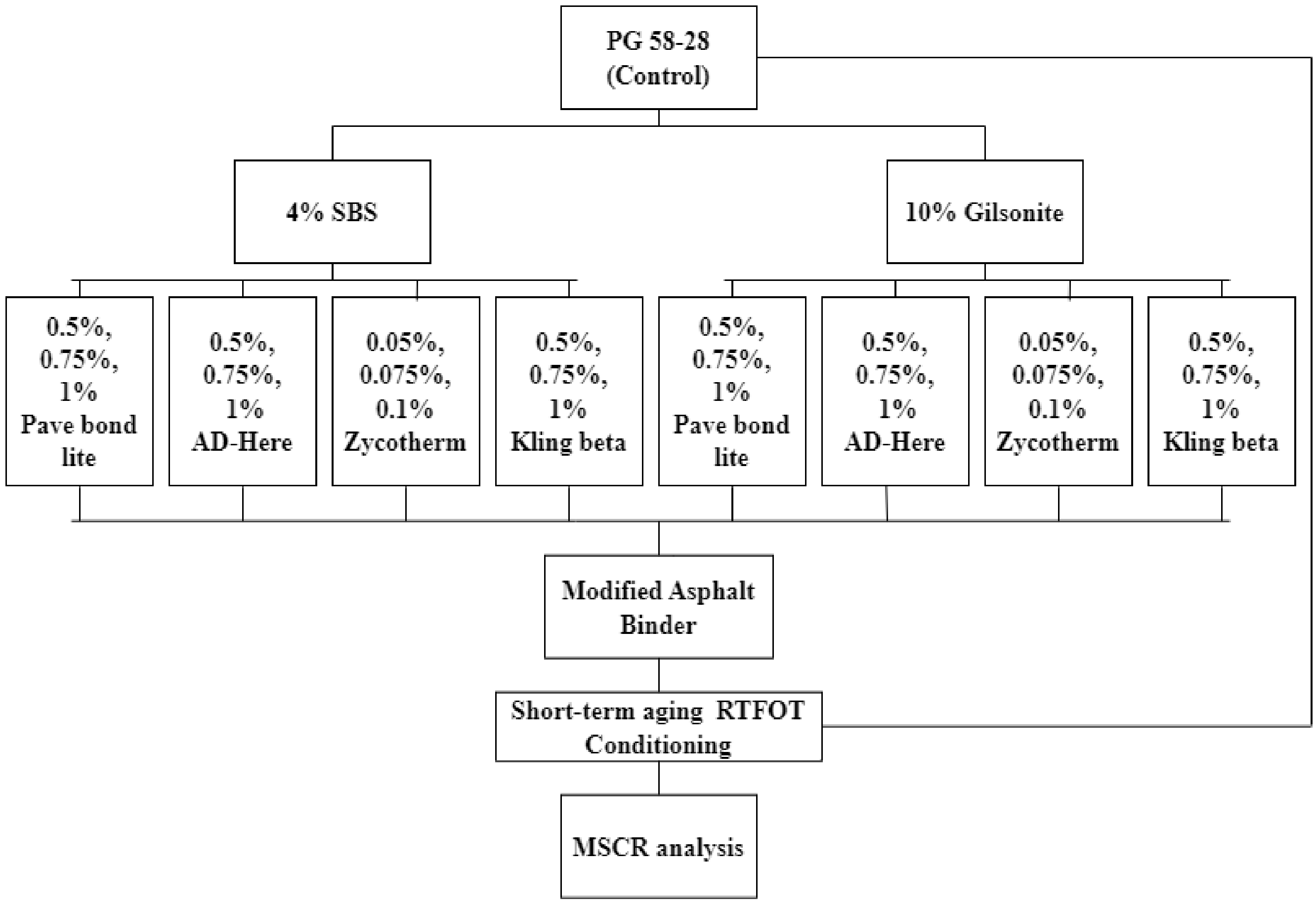


Figure 2.2 Experimental design matrix for binders

2.4.3 Method of Analysis

The MSCR protocol using DSR equipment has been widely accepted to evaluate the binder's permanent deformation behaviour using the creep-recovery concept (Singh & Kataware, 2016). Three significant parameters like non-recoverable compliance (J_{nr}), stress sensitivity, and % Recovery are used to identify the creep recovery performance of the asphalt binder obtained from the MSCR test (Golalipour et al., 2016). AASHTO M 332 specifications classify the binders as E, V, H, or S, as shown in **Table 2.2**, based on the J_{nr} value at 3.2 kPa.

Table 2.2 AASHTO M 332 Specification

AASHTO M 332 Specification	Binder Classification	Meaning	J_{nr} value at 3.2 kPa(1/kPa)
Greater than 30 million ESALs and < 20 km/h	E	Extreme	0.0–0.5
Greater than 30 million ESALs or < 20 km/h	V	Very Heavy	0.5–1.0
Between 10 and 30 million ESALs or 20–70 km/h	H	Heavy	1.0–2.0
<10 million ESALs and ＞70 km/h	S	Standard	2.0–4.5

2.4.3.1 Non-recoverable Creep Compliance at 3.2 kPa

The non-recoverable creep compliance (J_{nr}) is calculated to evaluate the deformation as per the AASTHO M 332. The non-recoverable creep compliance, which is evaluated at 3.2 kPa, is used to assess an asphalt binder's resistance to permanent deformation under repeated loading. A lower value of J_{nr} implies a lower rate of deformation, which implies good elasticity and higher rutting resistance (Wasage et al., 2011). The test temperature was selected at 58°C for MSCR analysis.

2.4.3.2 Stress Sensitivity

The MSCR test allows the assessment of the nonlinearity of asphalt binder response and identifies the excessive stress sensitivity of asphalt binders in the nonlinear range. J_{nr} diff. is the difference between the J_{nr} value at stress levels of 3.2 and 0.1 kPa, as defined in **Equation 2.1** (AASHTO T 350–14, 2014), is utilized as an indicator of stress sensitivity of asphalt binders. According to AASHTO TP 70, J_{nr} diff. should not exceed 75 percent. If it crosses this limit, then the asphalt binder may fail when experiencing higher stress or higher temperature in the real world, which is different from the consideration in the laboratory (AASHTO M 332-14, 2014).

$$J_{nr,diff.} = \frac{J_{nr,3.2kPa} - J_{nr,0.1kPa}}{J_{nr,0.1kPa}} \times 100\% \quad (2.1)$$

Where:

$J_{nr,diff.}$ is the % change of the J_{nr} values at stress levels of 3.2 kPa and 0.1 kPa;

$J_{nr,3.2kPa}$ is the J_{nr} value at stress levels of 3.2 kPa; and;

$J_{nr,0.1kPa}$ is the J_{nr} value at stress levels of 0.1 kPa;

2.4.3.3 Modified Method of Stress Sensitivity

Initially, as an indicator of the stress sensitivity of asphalt binders, the percent difference in non-recoverable creep compliance (J_{nr} diff.) obtained from the MSCR test is used. It is simply calculated as **Equation 2.1**. However, there is no correlation between the percent difference and field performance (Gaspar et al., 2019). The MSCR test is widely used, however many researchers have been concerned about the applicability of this 75 percent limit (Laukkanen et al., 2015, Behnood & Olek, 2017). The percent difference value of a wax-modified asphalt binder is more than 75 percent (Laukkanen et al., 2015). As a result of the previous approach of stress sensitivity study, this binder should be avoided in road construction since it is very stress sensitive. However, the investigation revealed that the Jnr value for a wax-modified asphalt binder was very low at 3.2 kPa, implying that this binder was exceptionally rut resistant. As a result, non-recoverable creep compliance and percent difference are incompatible, which is why the previous approach of stress sensitivity was ineffective for modified asphalt binders.

Stemphihar et al. provided a promising approach for analyzing stress sensitivity (Stemphihar et al., 2018). The proposed parameter is denoted as the Jnr slope. **Equation 2.2** is used to calculate the modified stress sensitivity. This new method does not unfairly penalize modified asphalt binders that have a low Jnr value at 3.2 kPa and provides a comparable assessment of stress sensitivity.

$$J_{nr,slope} = \frac{dJ_{nr}}{d\sigma} = \frac{J_{nr,3.2kPa} - J_{nr,0.1kPa}}{3.1\ kPa} \times 100\% \tag{2.2}$$

Where:

$J_{nr,slope}$ is the proposed parameter for stress sensitivity;

$J_{nr,3.2kPa}$ is the J_{nr} value at stress levels of 3.2 kPa; and

$J_{nr,0.1kPa}$ is the J_{nr} value at stress levels of 0.1 kPa;

2.4.3.4 Percent Recovery at 3.2 kPa

One of the critical parameters influencing the creep recovery performance of binder in the MSCR test is % Recovery. It is indicative of the ability of an asphalt binder to restore its deformation after the removal of the creep load. For any asphalt binder, the value of % Recovery should be non-negative. In other words, the residual strain at the end of the recovery portion should be no more than the accumulated strain at the end of the creep portion for a given creep and recovery cycle. However, many studies have reported negative % Recovery, which is against the physical significance (Soenen et al., 2013). Negative % Recovery is more common for unmodified asphalt binders at 3.2 kPa (Saboo & Kumar, 2015). However, sometimes due to a combination of high temperature, high stress, and low modification level, it can also be observed for modified asphalt binders (Jafari & Babazadeh, 2016). Instrument inertia may be one of the possible reasons for negative % Recovery. During the DSR test, the instruments are sometimes unable to unload the creep stress as quickly as is requested by the test protocol (Visscher et al., 2016). Therefore, there can be a delay between the theoretical end of the creep loading and the actual time at which the stress comes back to zero (Visscher et al., 2016). So, the strain recorded at the theoretical end of the creep portion is likely to be smaller than the strain at the end of the recovery portion, yielding a negative value of % Recovery. Another possible explanation for negative % Recovery is the tertiary creep behaviour that results from extremely large strain, and that makes the binder flow even at zero stress (Singh et al., 2017, Jafari et al., 2015). At high temperatures and high stress, the tertiary flow is more dominant (Singh et al., 2017). To avoid a negative % Recovery, lower temperature and/or lower stress should be applied (Jafari et al., 2016). ASTM D7405–15 provided a solution to negative % Recovery. According to ASTM, if the % Recovery turns out to be negative, then the actual % Recovery can be regarded to be zero. AASHTO M 332 proposed a method to detect polymer in asphalt binders, as shown in **Equation 2.3**. There is no requirement for Jnr values larger than 2 kPa^{-1} to have a minimum percent recovery value (Anderson, 2007,2014).

$$\%R = \begin{cases} 29.37(J_{nr,3.2kPa})^{-0.2633}, & J_{nr,3.2kPa} \geq 0.1 \\ 55 \quad , & J_{nr,3.2kPa} < 0.1 \end{cases} \tag{2.3}$$

Where:

% R is the % Recovery; and

$J_{nr,3.2kPa}$ is the Jnr value at stress levels of 3.2 kPa.

2.4.3.5 Quadrant Plot

Customer satisfaction data may be organized using quadrant analysis. An agency might use this plot to determine how and where it can enhance its operations (AASHTO M 320,2010). **Figure 2.3** depicts a typical quadrant plot with Phase Angle (Degree) on the X-axis and MSCR % Recovery at 3.2 kPa on the Y-axis.

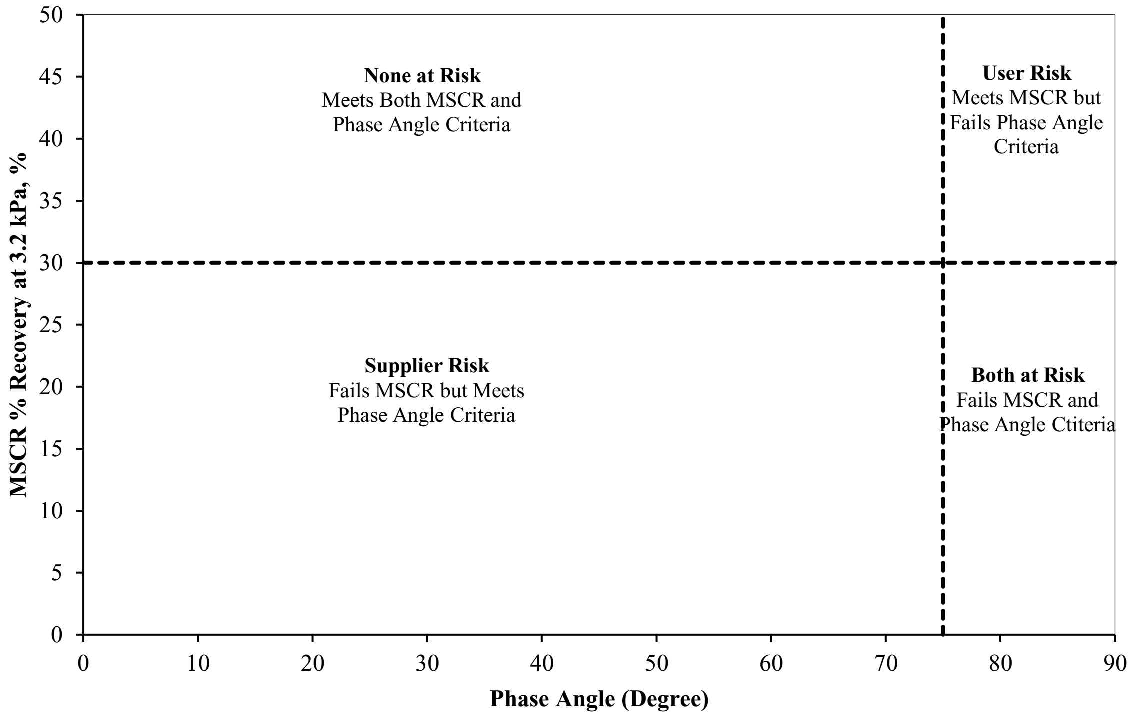


Figure 2.3 Quadrant plot of multiple stress creep & recovery (MSCR) % Recovery

The four quadrants in this graph are labeled 1st (User Risk), 2nd (None at Risk), 3rd (Supplier Risk), and 4th (Both at Risk). The term "user risk" refers to a circumstance where the MSCR % Recovery number matches the stated standards but not the phase angle or Elastic Recovery condition. The 'Supplier Risk,' on the other hand, is used to describe a circumstance in which the existing phase angle or Elastic Recovery criterion is reached, but the MSCR % Recovery number does not satisfy the suggested standards. Both the supplier and the user are at risk if neither the Phase Angle nor the % Recovery criteria are met (Both at Risk). Neither the supplier nor the user is at risk if both the % Recovery and the Phase Angle conditions are satisfied (None at Risk). The maximum phase angle allowed for polymer-modified binders is represented by the phase angle limit. When employing the DSR Phase Angle of the asphalt binder, a maximum phase angle of 75 degree (Anderson, M, 2012) is recommended. Recommended minimum MSCR % Recovery at a

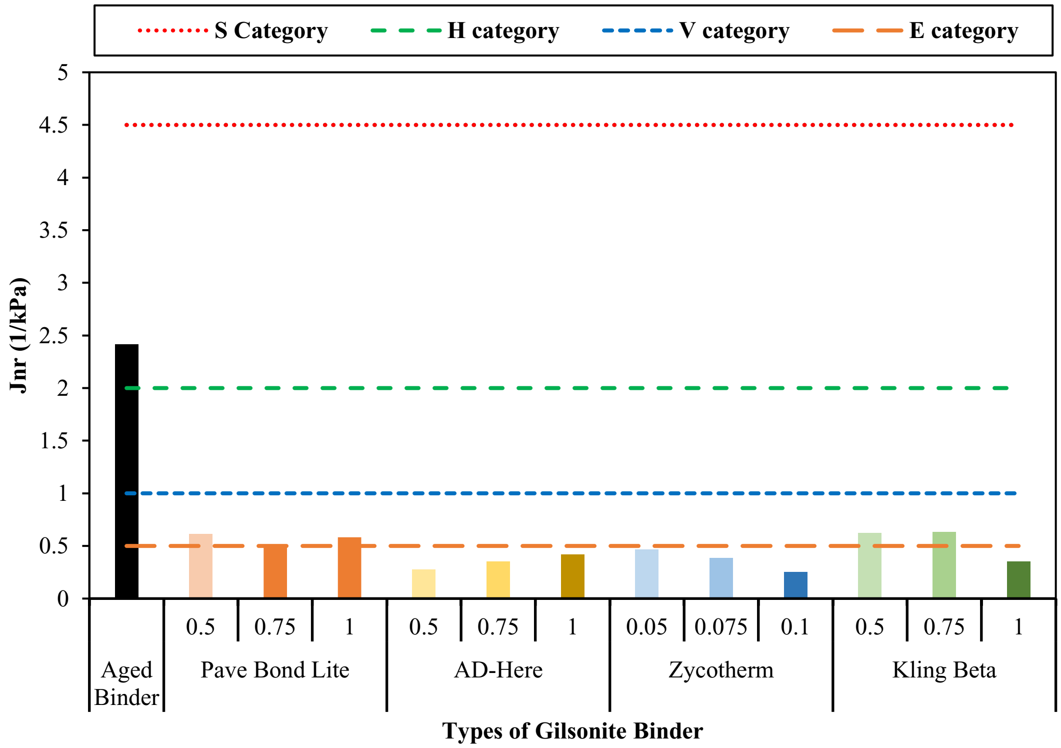
3.2 kPa value is 60 percent for PG 76-28, 50 percent for PG 70-28, and 40 percent for PG 64-28 (AASHTO M 320,2010). To be conservative, the minimum MSCR % Recovery at a 3.2 kPa value is assumed to be 30 percent for this study.

2.5 Result and Discussion

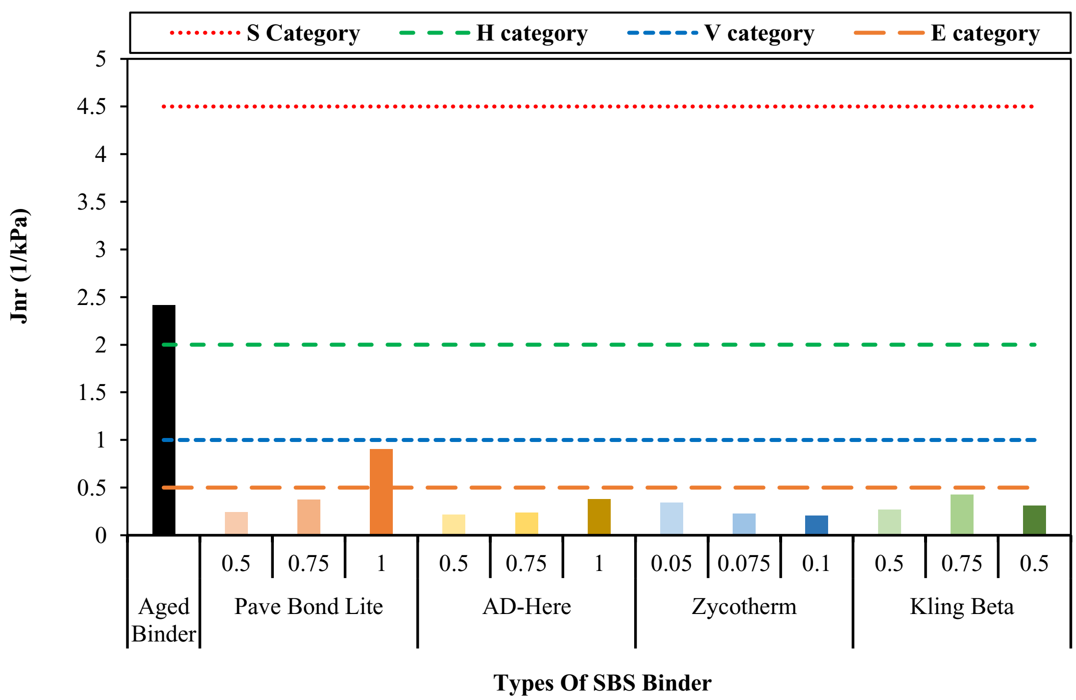
2.5.1 MSCR Analysis

2.5.2.1 Analysis of Jnr for Modified Binder containing Anti-Stripping Agents

Figure 2.4 captures the comparison of the non recoverable creep compliance of Gilsonite and SBS-modified binders containing different dosages of liquid anti-stripping agents. The aged binder's J_{nr} value was measured to be 2.415 kPa^{-1}. So, the aged binder passed the standard traffic loading criteria "S." From **Figure 2.4 (a),** J_{nr} value varied considerably when various dosages of anti-stripping agent were added. All in all, the modified aged binder containing anti-stripping agents outperforms the aged binder, with J_{nr} values less than 2.415 kPa^{-1}. According to AASHTO M 332 specification all the Gilsonite modified binders containing liquid anti-stripping agents passed the Standard traffic loading criteria "S," Heavy traffic loading criteria "H," and Very heavy traffic loading criteria "V." When the percentage was 0.5 percent, the highest J_{nr} value was measured for the Pave Bond Lite, indicated lower rut resistance and maximum deformation in comparison with other dosages When the proportion of Pave Bond Lite was 0.75 percent, the J_{nr} value was the lowest, indicating high rutting performance and passed the Extremely heavy traffic criteria "E." The recovery performance of the 1 percent Pave Bond Lite was better than 0.5 percent but not as excellent as 0.75 percent. In the case of AD-Here, for all the dosages J_{nr} value was less than 0.5 kPa^{-1}, thus passed all the traffic loading criteria.


S Category
H category
V category
E category
Jnr (1/kPa)
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0.5
0.75
1
0.5
0.75
1
0.05
0.075
0.1
0.5
0.75
1
Aged Binder
Pave Bond Lite
AD-Here
Zycotherm
Kling Beta
Types of Gilsonite Binder

(a)


S Category
H category
V category
E category
Jnr (1/kPa)
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
0.5
0.75
1
0.5
0.75
1
0.05
0.075
0.1
0.5
0.75
0.5
Aged Binder
Pave Bond Lite
AD-Here
Zycotherm
Kling Beta
Types Of SBS Binder

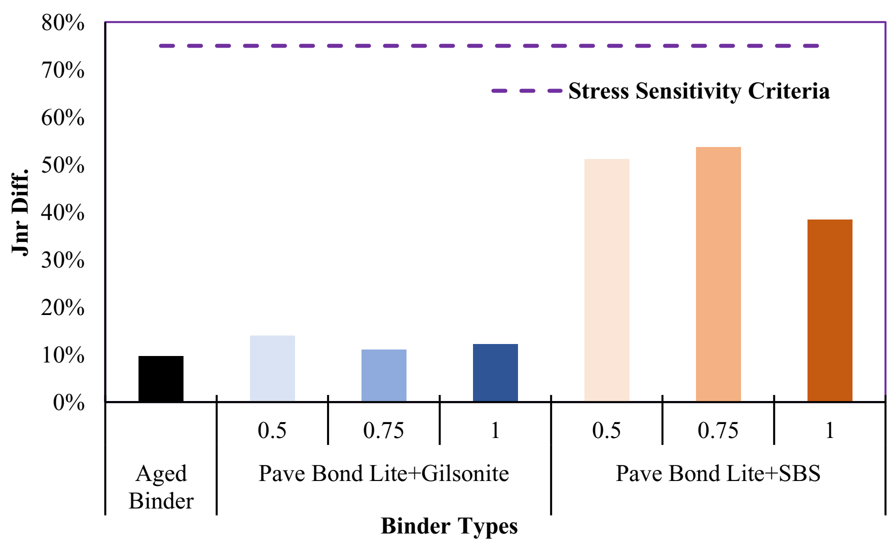
(b)

Figure 2.4 Comparison of Jnr of a) Gilosonite b) SBS-modified binders containing Pave Bond Lite, AD-Here, Zycotherm, Kling Beta anti-stripping agents

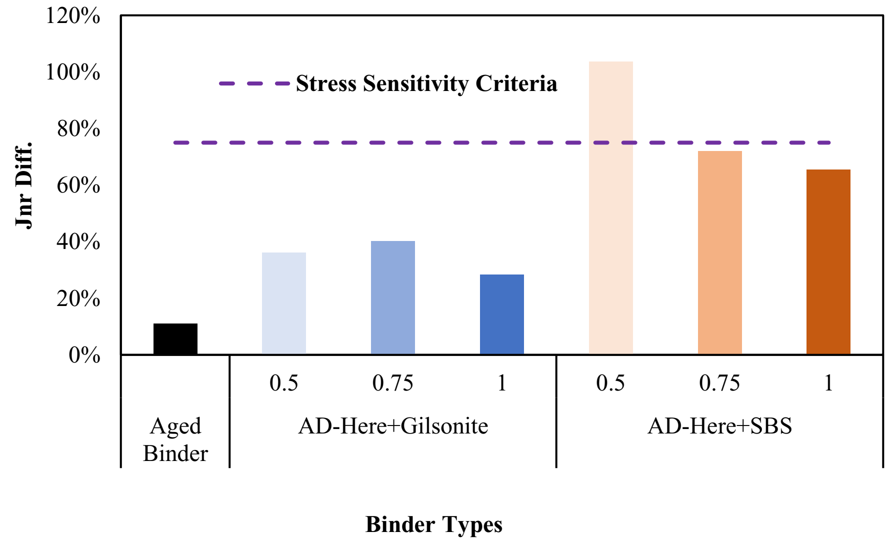
It was observed that 0.5 percent AD-Here had the lowest J_{nr} value indicating the best rutting performance. With the increase in dosage, the J_{nr} value increased, and rutting performance decreased. Zycotherm, on the other hand, had the opposite performance. For 0.01 percent dosage, high recovery performance could be noticed, but when the percentage was 0.05 percent, poor performance was detected. But all the dosages of Zycotherm passed all the traffic loading criteria. The J_{nr} value for Kling Beta anti-stripping agent began to rise but then dropped abruptly for the 1 percent, and it passed all the traffic loading criteria. Other two dosages of Kling Beta failed to pass the Extremely heavy traffic loading criteria "E." Overall, all the Gilsonite modified binders showed good performance, but Zycotherm 0.1 percent showed the best the rutting performance with Gilsonite. From **Figure 2.4 (b)**, with the modification with SBS, it was expected that there would be a reduction of the J_{nr} value of the aged binder containing anti-stripping agents. For the Pave Bond Lite, J_{nr} value doubled with each increasing concentration, which indicates more deformation and less recovery. Maximum J_{nr} value could be seen for a 1 percent concentration of Pave Bond Lite which indicated the worst recovery performance. The same pattern could be seen for AD-Here. A dosage of 0.5 percent AD-Here had the minimum J_{nr} value with maximum rutting performance. On the contrary, a different pattern could be seen for Zycotherm. With the increase of dosage, the J_{nr} value was decreasing. A dosage of 0.1 percent Zycotherm with SBS-modified binder showed the best rutting performance as the value of J_{nr} was minimum. All the dosages of AD-Here, Zycotherm, and Kling Beta modified with SBS passed all traffic loading criteria. Overall, Zycotherm with SBS-modified binder showed better rutting performance in comparison with binders with Gilsonite.

2.5.2.2 Analysis of Stress Sensitivity for Modified Binder containing Anti-Stripping Agents

Figure 2.5 explains the stress sensitivity analysis of a modified binder containing different dosages of liquid anti-stripping agents. Twenty-one (21) out of 24 binders passed the stress sensitivity criteria (J_{nr} diff. < 75 percent), which suggested that unexpectedly high temperatures or heavy loads did not excessively stress these binders. **Figure 2.5 (a)** shows that all Pave Bond Lite dosages modified with Gilsonite were less stress sensitive than the SBS. The material with 0.75 percent Pave Bond Lite modified with Gilsonite showed the least stress sensitivity, whereas 0.75 percent Pave Bond Lite modified with SBS showed the highest stress sensitivity. **Figure 2.5 (b)** captures that, 0.5 percent AD-Here modified with SBS failed to meet the stress sensitivity criteria. But other dosages of AD-Here modified with SBS and Gilsonite passed the stress sensitivity criteria. **Figure 2.5 (c)** shows that, 0.075 and 0.1 percent Zycotherm-modified with SBS failed the stress sensitivity criteria, but 0.05 percent passed the criteria. All the other dosages of Gilsonite-modified binders with the inclusion of AD-Here passed the stress sensitivity criteria. **Figure 2.5 (d)** shows that all the Gilsonite-modified binders had less stress sensitivity than the SBS modified binders.

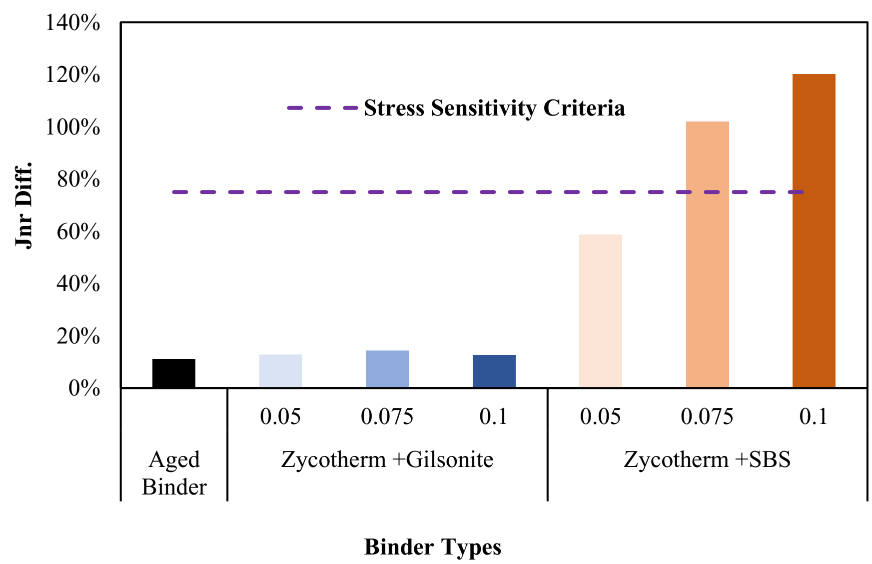


(a)



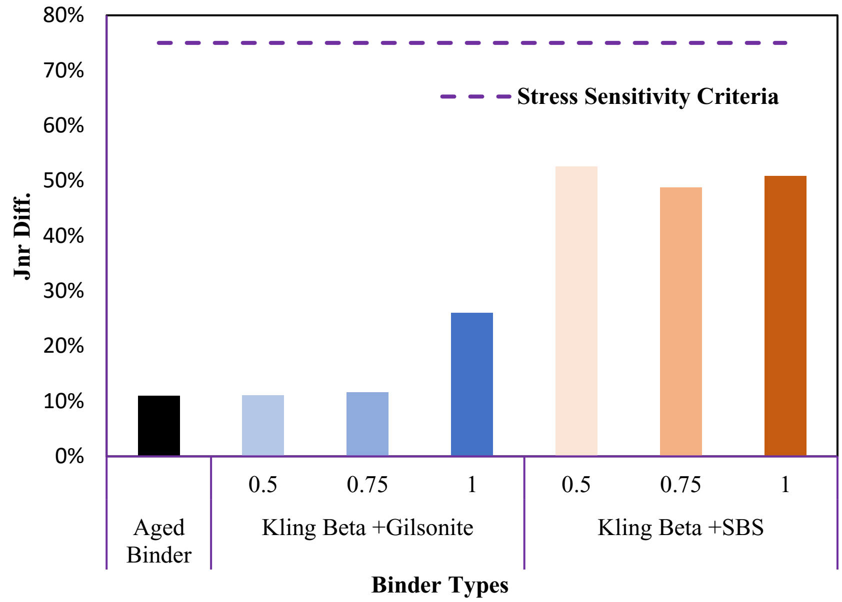
120%
100%
80%
60%
40%
20%
0%
Jnr Diff.
Stress Sensitivity Criteria
0.5
0.75
1
0.5
0.75
1
Aged Binder
AD-Here+Gilsonite
AD-Here+SBS
Binder Types

(b)



140%
120%
100%
80%
60%
40%
20%
0%
Jnr Diff.
Stress Sensitivity Criteria
0.05
0.075
0.1
0.05
0.075
0.1
Aged Binder
Zycotherm +Gilsonite
Zycotherm +SBS
Binder Types

(c)



(d)

Figure 2.5 Stress Sensitivity Analysis of modified binder containing (a) Pave Bond Lite (b) AD-Here (c) Zycotherm (d) Kling Beta.

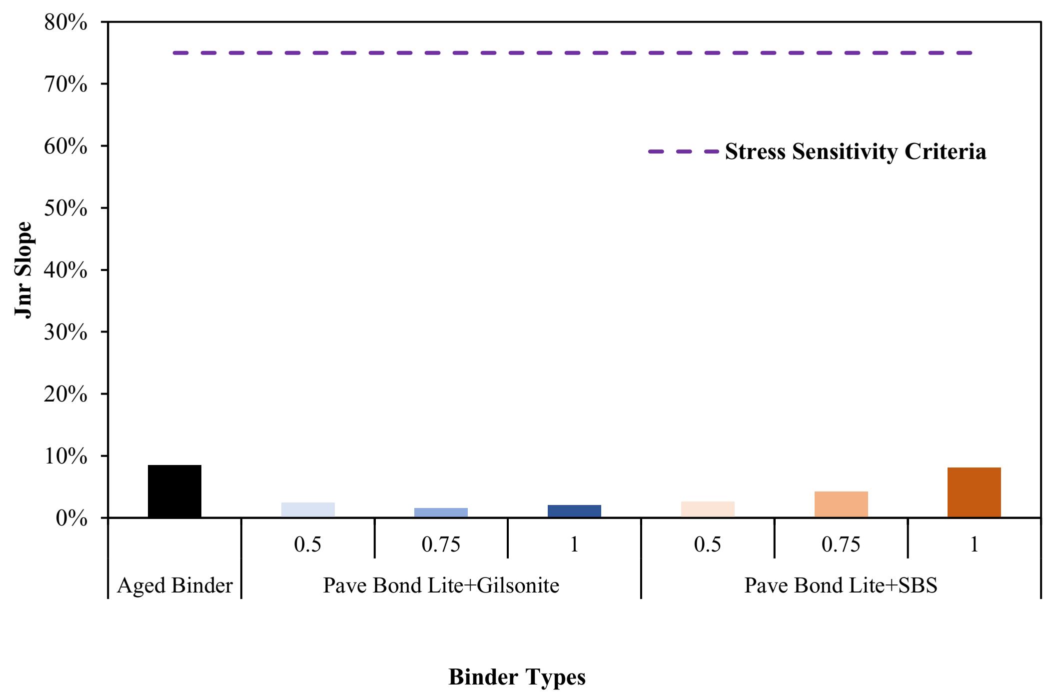
2.5.2.3 Analysis of Modified Stress Sensitivity for Modified Binder containing Anti-Stripping Agents

Figure 2.6 shows the modified stress sensitivity criteria for modified binders containing different dosages of liquid anti-stripping agents. All the 24 binders passed the modified stress sensitivity criteria, whereas 21 binders passed the previously mentioned stress sensitivity criteria. According to the modified method of stress sensitivity, all the binders had a stress sensitivity less than the aged binder. In contrast, according to the previous method of stress sensitivity, almost all the binders had stress sensitivity more than the aged binder.

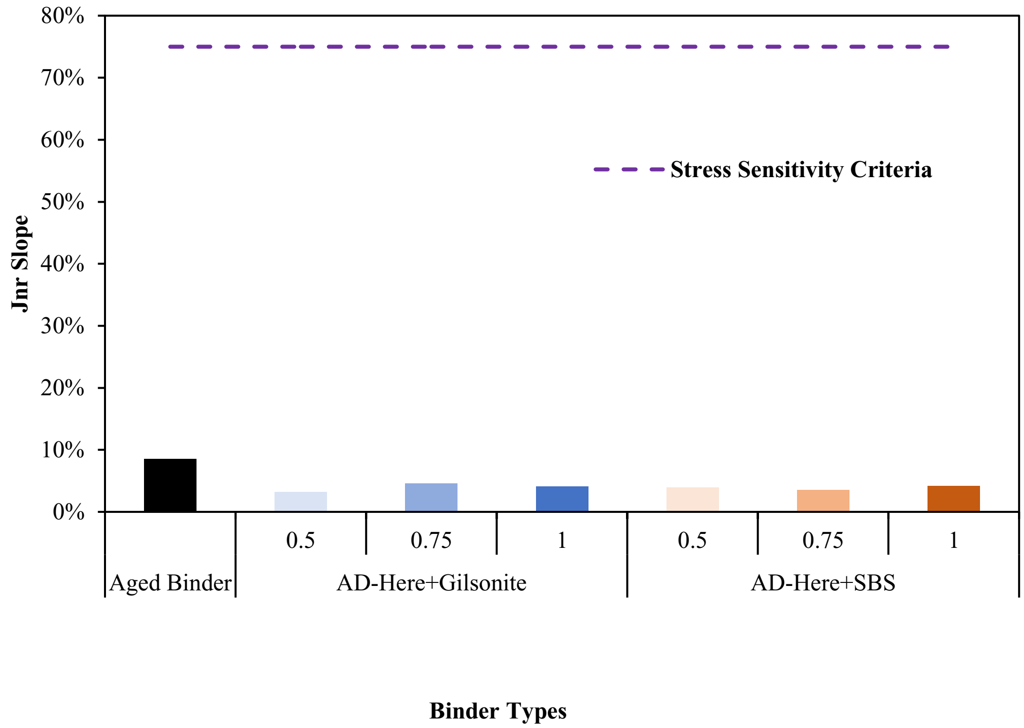
Figure 2.6 (a) shows that all Pave Bond Lite dosages modified with Gilsonite were less stress sensitive than the SBS. The binder with 0.75 percent Pave Bond Lite modified with Gilsonite showed the least stress sensitivity, whereas 1 percent Pave Bond Lite modified with SBS showed the highest stress sensitivity.

Figure 2.6 (b) shows that all AD-Here dosages modified with Gilsonite and SBS had almost the same stress sensitivity. Previously, SBS modified 0.5 percent Ad-Here failed to meet the stress sensitivity criteria, but with the modified method, it passed the stress sensitivity criteria.

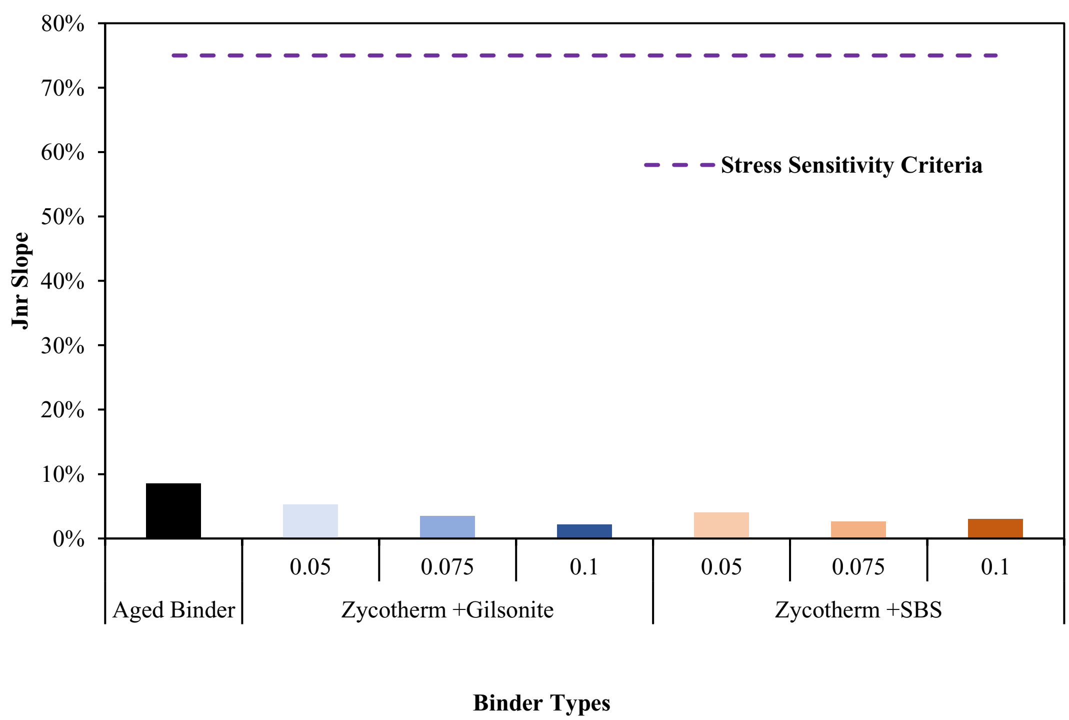
All the dosages of Zycotherm and Kling beta in **Figures 2.6 (c)** and **2.6 (d)**, respectively, meet the stress sensitivity criteria. Binders with 0.075 and 0.1 percent Zycotherm modified with SBS failed the previous stress sensitivity criteria but passed the modified stress sensitivity criteria.



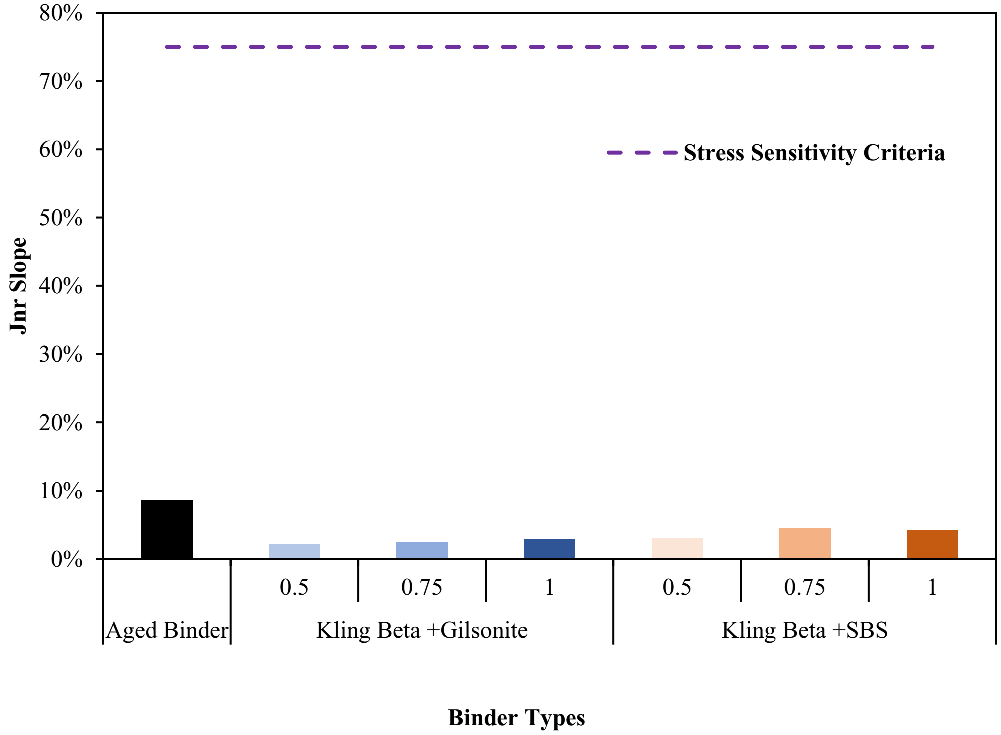
(a)


80%
70%
60%
50%
40%
30%
20%
10%
0%
Jnr Slope
Stress Sensitivity Criteria
0.5
0.75
1
0.5
0.75
1
Aged Binder
AD-Here+Gilsonite
AD-Here+SBS
Binder Types

(b)


80%
70%
60%
50%
40%
30%
20%
10%
0%
Jnr Slope
Stress Sensitivity Criteria
0.05
0.075
0.1
0.05
0.075
0.1
Aged Binder
Zycotherm +Gilsonite
Zycotherm +SBS
Binder Types

(c)



(d)

Figure 2.6 Modified method of stress sensitivity analysis of modified binder containing (a) Pave Bond Lite (b) AD-Here (c) Zycotherm (d) Kling Beta.

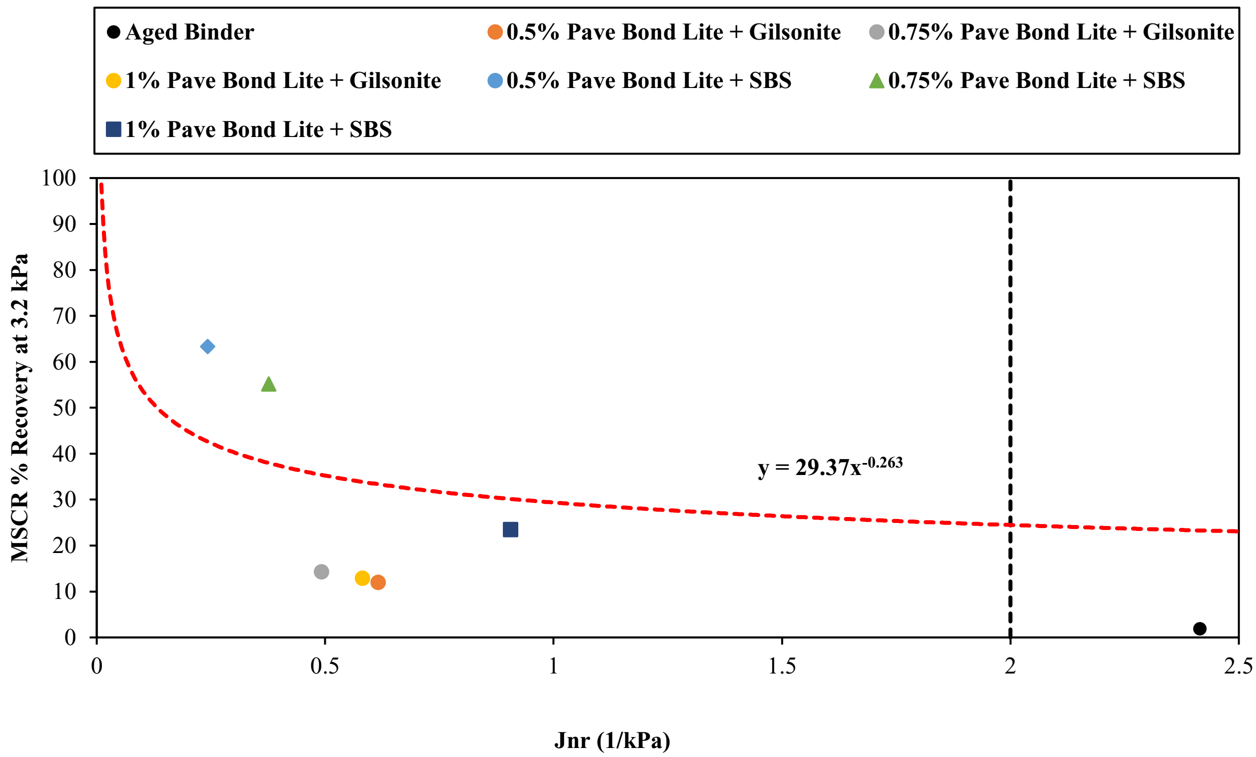
2.5.2.4 Analysis of Polymer Method for Modified Binder containing Anti-Stripping Agents

Polymer modification curves for modified binders containing different dosages of liquid anti-stripping agents are shown in **Figure 2.7**. From **Figure 2.7 (a)**, only 0.5 percent Pave Bond Lite and 0.75 percent Pave Bond Lite modified with SBS had a percent recovery above the standard line. So, these two binders showed an excellent recovery performance, whereas 1 percent Pave Bond Lite modified with SBS showed poor recovery performance. On the other hand, all dosages of Pave Bond Lite modified with Gilsonite clustered under the line and showed poor recovery performance. The modified binders containing Pave Bond Lite could be graded as the J_{nr} value is less than 4.5 kPa^{-1}.

As shown in **Figure 2.7 (b)**, all the dosages of AD-Here modified with SBS clustered above the standard polymer modification curve. The 0.5 percent AD-Here modified with SBS had the highest % Recovery, thus excelling in elastic recovery. On the other hand, all the dosages of AD-Here modified with Gilsonite failed to pass the polymer modification criteria. All the dosages of modified AD-Here could be graded.

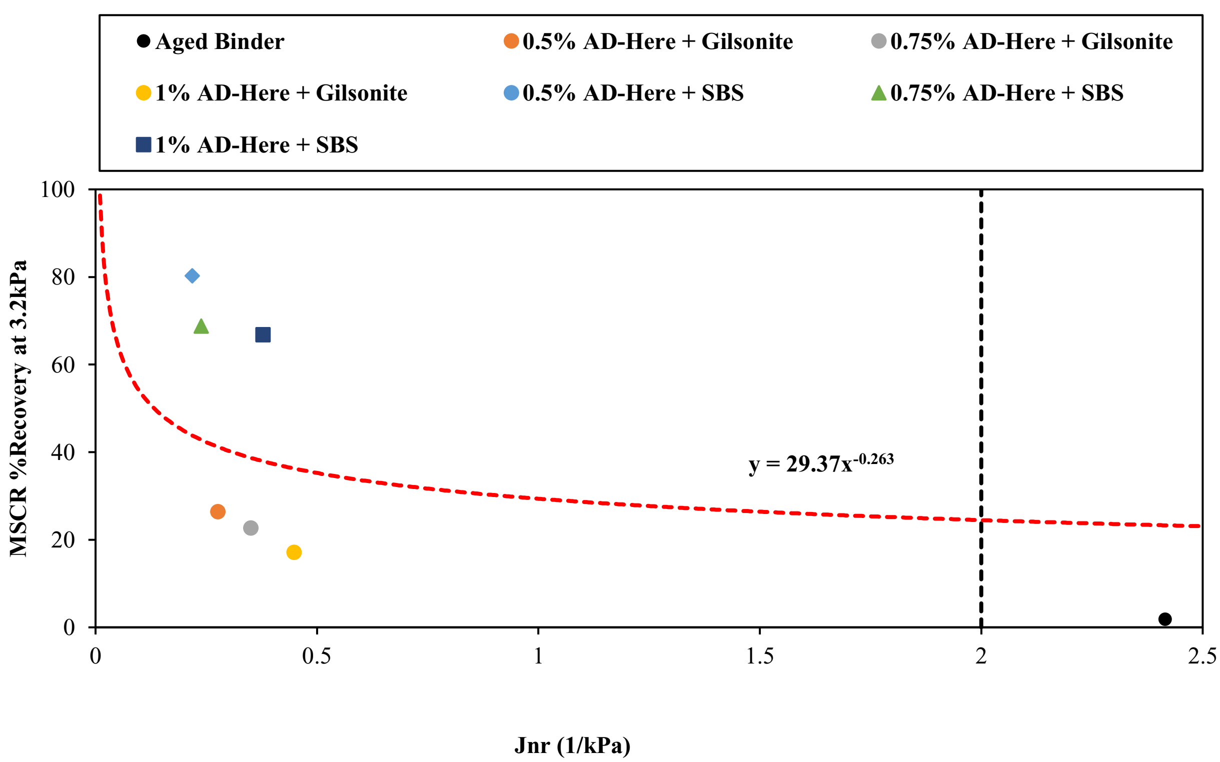
Figure 2.7 (c) shows the polymer modification curve of Zycotherm where all the dosages of Zycotherm modified with SBS passed the polymer modification criteria and clustered above the line, whereas all the dosages of Zycotherm modified with Gilsonite failed to pass the polymer modification criteria.

As shown in **Figure 2.7 (d)**, Kling Beta shows the same behaviour as Ad-Here and Zycotherm. All the dosages of Kling Beta modified with SBS passed the polymer method, whereas all the dosages of Kling Beta modified with Gilsonite failed to pass the criteria. Almost all the binders modified with SBS showed good % Recovery performance and clustered above the polymer modification curve. All the binders modified with Gilsonite failed to show a good recovery performance. Both the SBS and Gilsonite modified binders could be graded according to MSCR grading as the J_{nr} value was less than 4.5 kPa^{-1} for all the modified binders containing different dosages of liquid anti-stripping agents.



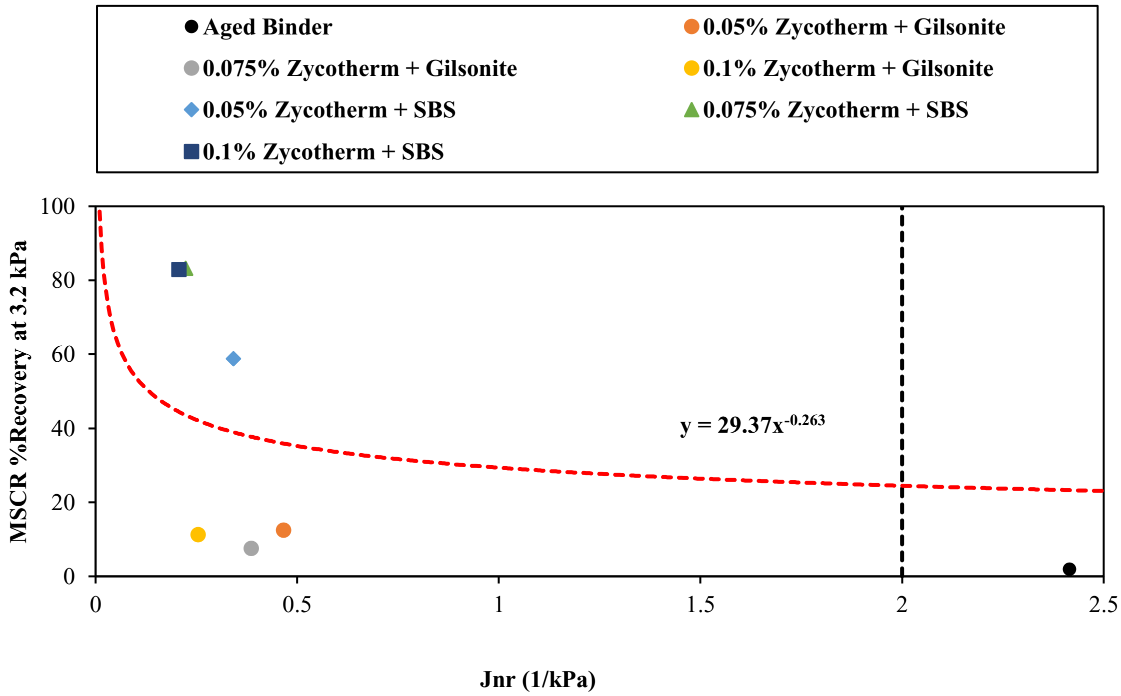
Aged Binder
0.5% Pave Bond Lite + Gilsonite
0.75% Pave Bond Lite + Gilsonite
1% Pave Bond Lite + Gilsonite
0.5% Pave Bond Lite + SBS
0.75% Pave Bond Lite + SBS
1% Pave Bond Lite + SBS
MSCR % Recovery at 3.2 kPa
Jnr (1/kPa)
y = 29.37x^-0.263

(a)

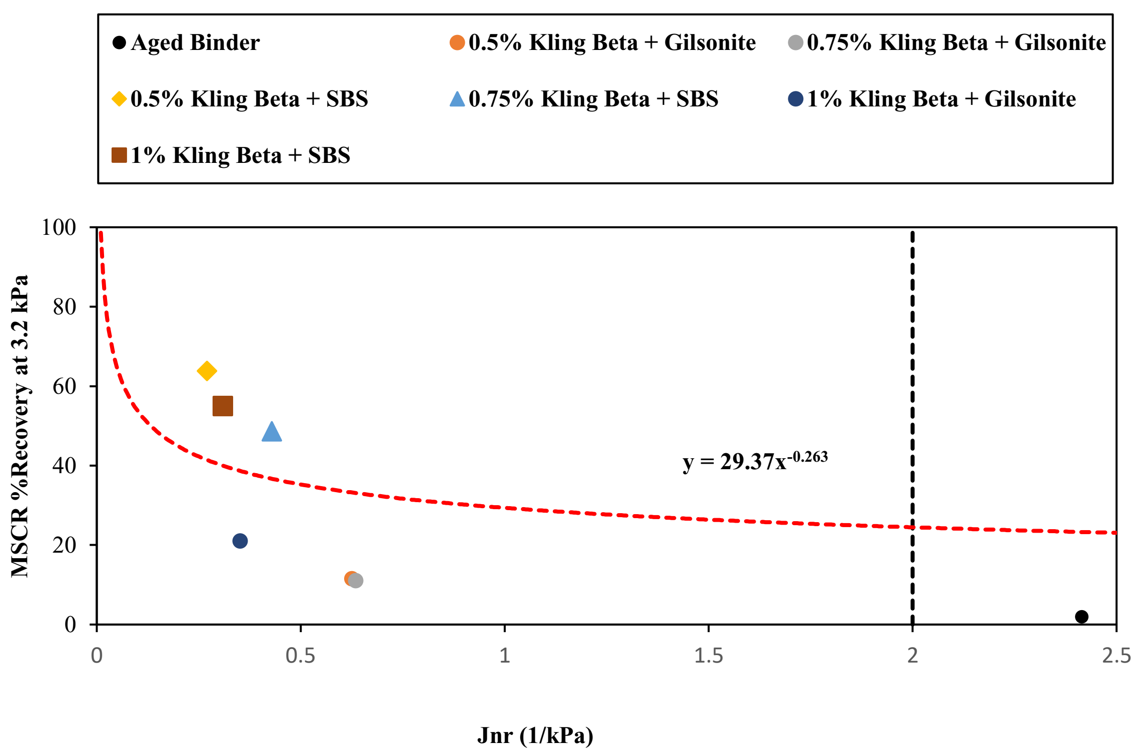


Aged Binder
0.5% AD-Here + Gilsonite
0.75% AD-Here + Gilsonite
1% AD-Here + Gilsonite
0.5% AD-Here + SBS
0.75% AD-Here + SBS
1% AD-Here + SBS
MSCR %Recovery at 3.2kPa
Jnr (1/kPa)
y = 29.37x^-0.263

(b)


Aged Binder
0.05% Zycotherm + Gilsonite
0.075% Zycotherm + Gilsonite
0.1% Zycotherm + Gilsonite
0.05% Zycotherm + SBS
0.075% Zycotherm + SBS
0.1% Zycotherm + SBS
y = 29.37x^-0.263
MSCR %Recovery at 3.2 kPa
Jnr (1/kPa)

(c)

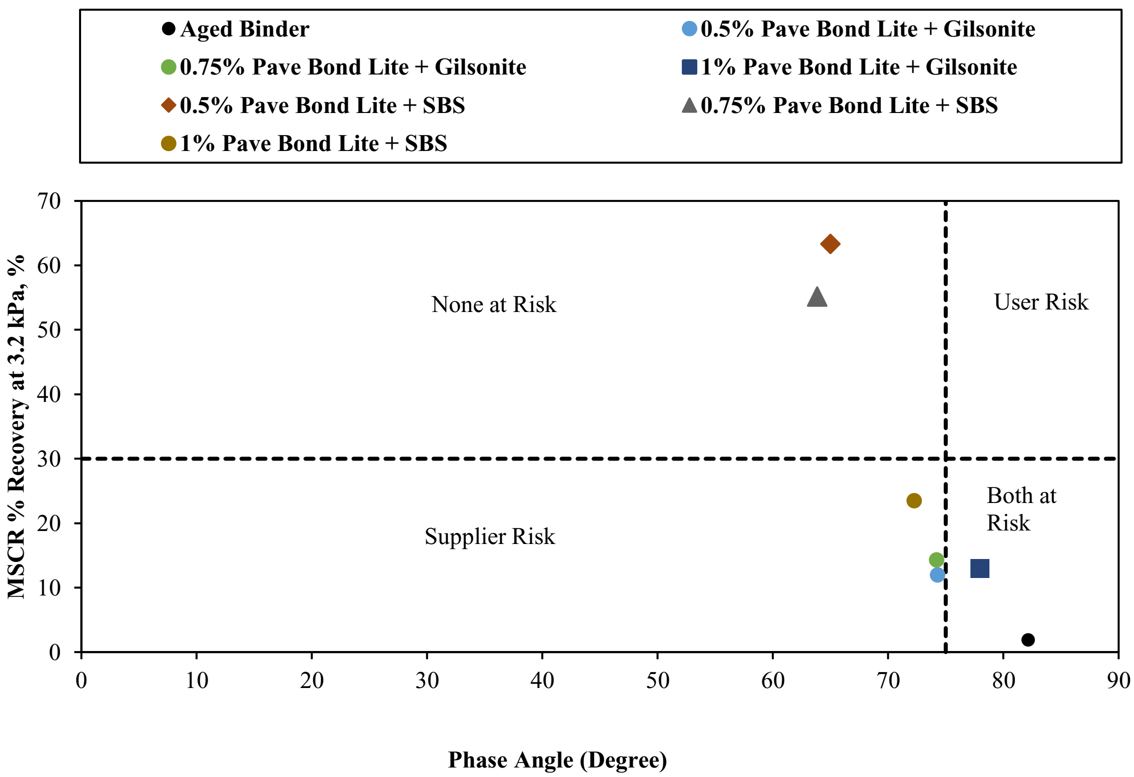

Aged Binder
0.5% Kling Beta + Gilsonite
0.75% Kling Beta + Gilsonite
0.5% Kling Beta + SBS
0.75% Kling Beta + SBS
1% Kling Beta + Gilsonite
1% Kling Beta + SBS
y = 29.37x^-0.263
MSCR %Recovery at 3.2 kPa
Jnr (1/kPa)

(d)

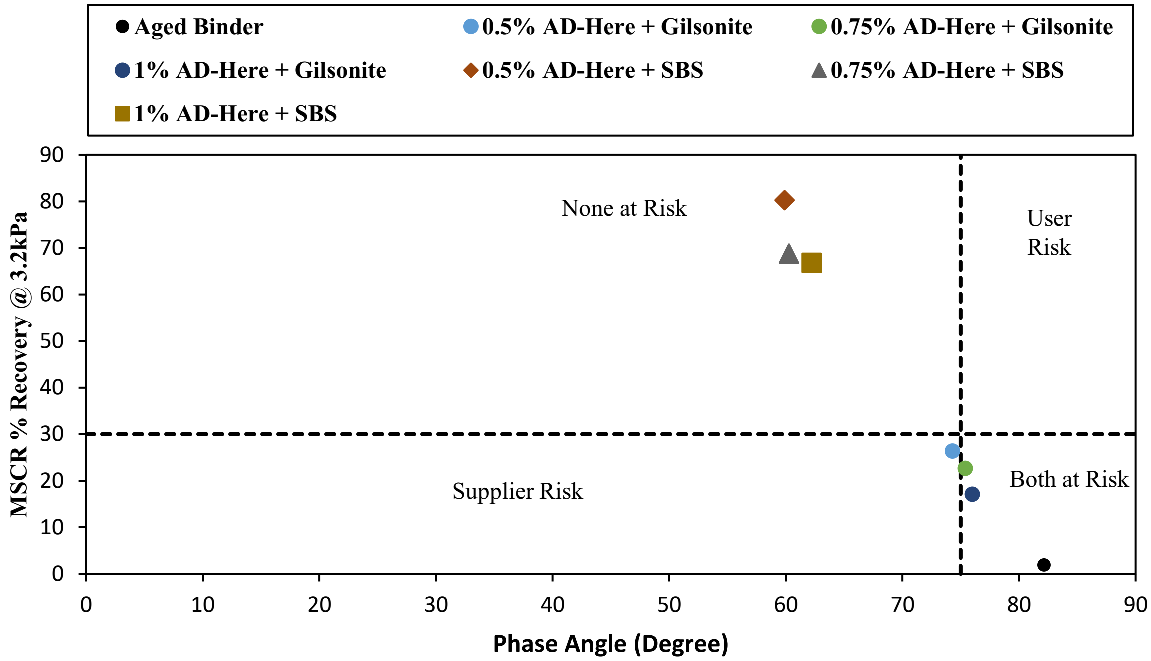
Figure 2.7 Polymer modification curve of modified binder containing (a) Pave Bond Lite (b) AD-Here (c) Zycotherm (d) Kling Beta.

2.5.2.5 Quadrant Plot for Modified Binder containing Anti-Stripping Agents

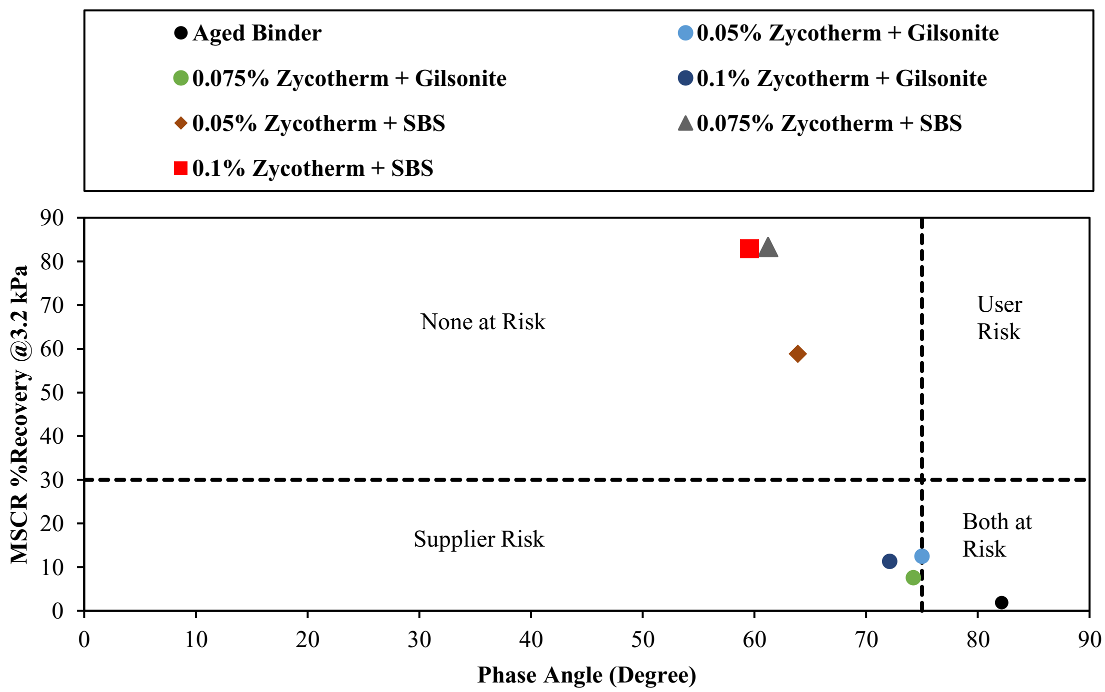
Figure 2.8 shows the Quadrant Plot for the modified binder containing different dosages of liquid anti-stripping agents.



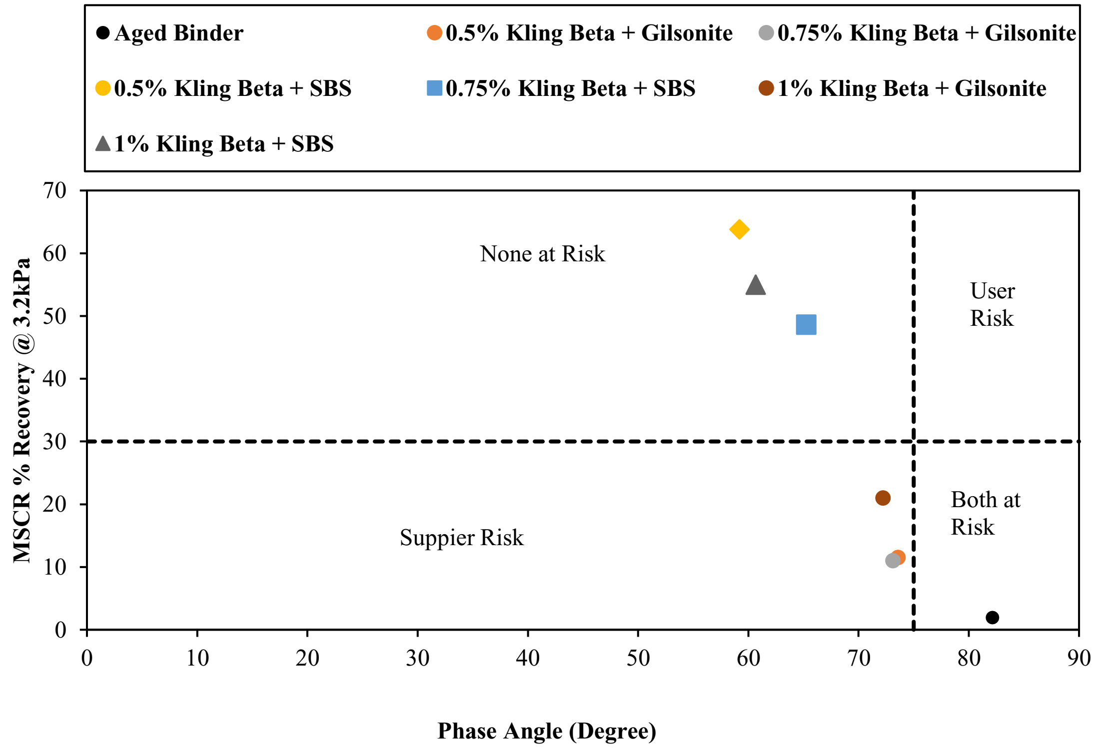
(a)


Aged Binder
0.5% AD-Here + Gilsonite
0.75% AD-Here + Gilsonite
1% AD-Here + Gilsonite
0.5% AD-Here + SBS
0.75% AD-Here + SBS
1% AD-Here + SBS
None at Risk
User Risk
Supplier Risk
Both at Risk
MSCR % Recovery @ 3.2kPa
Phase Angle (Degree)
0
10
20
30
40
50
60
70
80
90

(b)


Aged Binder
0.05% Zycotherm + Gilsonite
0.075% Zycotherm + Gilsonite
0.1% Zycotherm + Gilsonite
0.05% Zycotherm + SBS
0.075% Zycotherm + SBS
0.1% Zycotherm + SBS
None at Risk
User Risk
Supplier Risk
Both at Risk
MSCR %Recovery @3.2 kPa
Phase Angle (Degree)
0
10
20
30
40
50
60
70
80
90

(c)



(d)

Figure 2.8 Quadrant plot for modified binder containing (a) Pave Bond Lite (b) AD-Here (c) Zycotherm (d) Kling Beta.

From **Figure 2.8 (a),** only 0.5 percent Pave Bond Lite and 0.75 percent Pave Bond Lite modified with SBS passed both Phase Angle and % Recovery criteria. These two binders fell in the 'None at Risk' quadrant. On the other hand, 1 percent Pave Bond Lite modified with SBS, 0.5 percent Pave Bond Lite and 0.75 percent Pave Bond Lite modified with Gilsonite were in the 'Supplier Risk' quadrant, implying they met the Phase Angle criteria but failed % Recovery criteria. Both the aged binder and 1 percent Pave Bond Lite modified with Gilsonite failed to meet both Phase Angle and % Recovery criteria, termed as a 'Both at Risk' according to the Quadrant method.

As shown in **Figure 2.8 (b)**, all the dosages of AD-Here modified with SBS passed both the criteria, whereas 0.5 percent AD-Here modified with Gilsonite only passed the Phase Angle criteria. Other dosages of AD-Here modified with Gilsonite fell in the 'Both at Risk' quadrant.

Figure 2.8 (c) shows the Quadrant plot of Zycotherm where all the dosages of Zycotherm are modified with SBS in the 'None at Risk' quadrant. On the contrary, all the dosage of Zycotherm modified with Gilsonite was in the 'Supplier Risk' quadrant.

Kling Beta, as shown in **Figure 2.8 (d)**, showing the same behaviour as Zycotherm. All the dosages of Kling Beta modified with SBS passed both the criteria, whereas all the dosages of Kling Beta modified with Gilsonite only passed the Phase Angle criteria. Out of 24 modified binders containing different dosages of liquid anti-stripping agents, only 11 met Phase Angle and % Recovery criteria. Almost all the binders modified with SBS showed good % Recovery performance. All the binders modified with Gilsonite failed to meet the % Recovery criteria. A summary of all the performance parameters considered for this study is given below in **(Table 2.3).**

Table 2.3 Summary of MSCR test parameters

No	Anti-stripping Agent	Dose Rate (%)	Modifier	Meet Stress Sensitivity Criteria	Meet Modified Stress Sensitivity	%Recovery (Meets AASTHO T 350)	Quadrant Plot Criteria	MSCR GRADE AASHTO M 332
1	Pave bond lite	0.5	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28
2	Pave bond lite	0.75	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28
3	Pave bond lite	1	SBS 4%	Yes	Yes	No	Supplier Risk	PG 58V-28

4	Pave bond lite	0.5	Gilsonite 10%	Yes	Yes	No	Supplier Risk	PG 58V-28
5	Pave bond lite	0.75	Gilsonite 10%	Yes	Yes	No	Supplier Risk	PG 58E-28
6	Pave bond lite	1	Gilsonite 10%	Yes	Yes	No	Both at Risk	PG 58V-28
7	AD-Here	0.5	SBS 4%	No	Yes	Yes	None at Risk	PG 58E-28
8	AD-Here	0.75	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28
9	AD-Here	1	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28
10	AD-Here	0.5	Gilsonite 10%	Yes	Yes	No	Supplier Risk	PG 58E-28
11	AD-Here	0.75	Gilsonite 10%	Yes	Yes	No	Both at Risk	PG 58E-28
12	AD-Here	1	Gilsonite 10%	Yes	Yes	No	Both at Risk	PG 58E-28
13	Zycotherm	0.05	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28
14	Zycotherm	0.075	SBS 4%	No	Yes	Yes	None at Risk	PG 58E-28
15	Zycotherm	0.1	SBS 4%	No	Yes	Yes	None at Risk	PG 58E-28
16	Zycotherm	0.05	Gilsonite 10%	Yes	Yes	No	Supplier Risk	PG 58E-28
17	Zycotherm	0.075	Gilsonite 10%	Yes	Yes	No	Supplier Risk	PG 58E-28
18	Zycotherm	0.1	Gilsonite 10%	No	Yes	No	Supplier Risk	PG 58E-28
19	Kling beta	0.5	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28
20	Kling beta	0.75	SBS 4%	Yes	Yes	Yes	None at Risk	PG 58E-28

21	Kling beta	1	SBS 4%	No	Yes	Yes	None at Risk	PG 58E-28
22	Kling beta	0.5	Gilsonite 10%	Yes	Yes	No	Supplier Risk	PG 58V-28
23	Kling beta	0.75	Gilsonite 10%	No	Yes	No	Supplier Risk	PG 58V-28
24	Kling beta	1	Gilsonite 10%	No	Yes	No	Supplier Risk	PG 58E-28

2.5.2 Statistical Analysis

To determine the relative effects of modifiers, anti-stripping agents, and dosage rate, an ANOVA analysis was carried out. Three distinct quantities are present in each of the anti-stripping agents employed in this investigation. These ratios are regarded as being high, medium, and low. **Table 2.4 for** the Design Expert Software's ANOVA analysis may be obtained from the experimental plan.

Table 2.4 Parameters for binder level ANOVA analysis

Factor	Name	Unit	Type	Number of Levels	Levels
A [Categoric]	Modifier	N/A	Nominal	2	SBS, Gilsonite
B [Categoric]	Anti-stripping agent	N/A	Nominal	4	Pave bond lite, AD-Here, Zycotherm, Kling beta
C [Categoric]	Dose rate	N/A	Nominal	3	Low, Medium, High
Response			**Number of Replication**		
J_{nr}	Non-recoverable	1/kPa		1	

	creep compliance		
%R	Percent Recovery	N/A	1

2.5.2.1 Analyzed result

The results of the analysis of variance (ANOVA) for the J_{nr} and %R data gathered from MSCR are shown in **Table 2.5**. The model F-value for both J_{nr} and %R was statistically significant with a p-value of less than 0.0001, where the p-value assessed the significance of each regression coefficient. Hence, J_{nr} and %R may be used to characterize how well asphalt binders containing different modifiers and anti-stripping agents perform in terms of creep recovery.

Table 2.5 Results obtained from ANOVA analysis

Source	**For response J_{nr}**			**For response %R**		
	Sum of Squares	**F-value**	**p-value**	**Sum of Squares**	**F-value**	**p-value**
Model	0.2478	4.07	0.0114	13.35	21.18	< 0.0001
A-Modifier	0.1283	12.69	0.0026	12.46	118.63	< 0.0001
B- Anti-stripping agent	0.0978	3.22	0.0407	0.8319	27.78	< 0.0001
C-Dose rate	0.0083	0.40	0.0641	0.0521	2.64	0.0459
Residual	0.1619			1.79		
R^2	0.8042			0.8820		
Adjusted R^2	0.7558			0.8403		
Predicted R^2	0.7143			0.7648		
Adeq. Precision	6.0384			11.7220		

Variation of modifiers (A) and variation of liquid anti-stripping agents (B) had a significant effect on both J_{nr} and %R. However, the variation of dose rate (C) significantly impacted only %R. Factor A modifiers had the highest value of F for both the models, which indicated the addition of SBS or Gilsonite with the binder had the maximum effect on J_{nr} and %R. SBS-modified binders and Gilsonite-modified binders performed differently in terms of creep recovery. Factor B, liquid anti-stripping agents had a lower F value than modifiers. Variation of anti-stripping agents had a significant effect on J_{nr} on %R. On the other hand, factor C, the dose rate, had the lowest effect on the %R and no effect on J_{nr}.

For $J_{nr,}$ the Predicted R^2 of 0.8042 was in reasonable agreement with the adjusted R^2 of 0.7558. The lower difference between R^2 and adjusted-R^2 implied that all significant terms were involved in the model [Nayak et al. 2019]. For %R, the difference between R^2 and adjusted- R^2 was also less than 0.2, which implied that the data was not overfitting. Adequate precision measures the signal-to-noise ratio. A ratio greater than 4 was desirable. In these models, the ratio of 6.0384 and 11.7220 respectively indicated an adequate signal. All samples should be independent of one another, residuals should have a constant variance, and residuals should be normally distributed, as these are assumptions for picking a model (Raylov & Marcoulides,2013). All the assumptions were checked so these models could navigate the design space.

The developed model was optimized to get the parameters to minimize the J_{nr} value and maximize the %R. **Figure 2.9** shows the constraints for the optimization process. All the factors studied were categorical, and optimized results were found after eight solutions. SBS modifier, Zycotherm liquid anti-stripping agent, and a high dose rate of 0.1% results in minimizing J_{nr} and maximizing the %R. The minimum J_{nr} value is 0.211 kPa^{-1}, and the maximum %R is 57.35% with high desirability of 0.902.

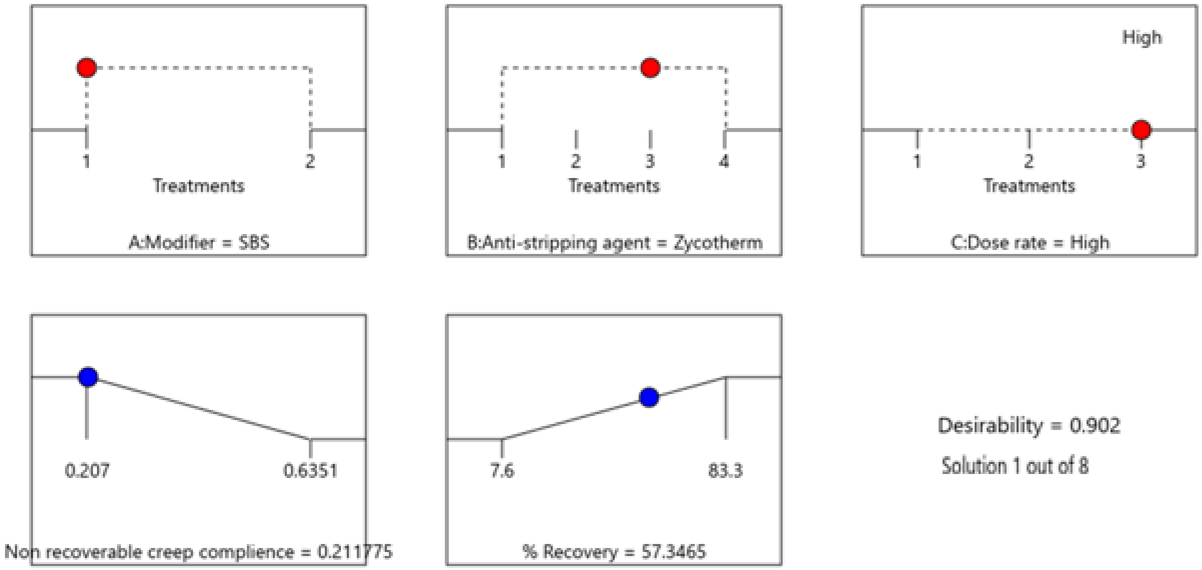


Figure 2.9 Optimization of Jnr and %R

2.6 Conclusions

Based on the experimental results collected from the MSCR test and analysis of different parameters, the following conclusions can be drawn.

- All the Gilsonite-modified and SBS-modified binders containing different dosages of liquid anti-stripping agents showed good rutting performance and passed all the traffic loading criteria based on AASHTO M 332 specification. But SBS-modified binders outperformed the Gilsonite modified binders in case of recovery performance. The 0.1 percent Zycotherm modified with SBS had the lowest value of J_{nr} and passed all the traffic loading conditions with the least deformation.
- All the dosages of Gilsonite and SBS modified binders containing different dosages of liquid anti-stripping agents passed the modified method of stress sensitivity. Although SBS modified

0.5 percent Ad-Here, 0.075 and 0.1 percent Zycotherm modified with SBS binders failed to meet the aforementioned stress sensitivity criteria.

- All the SBS and Gilsonite-modified binders containing different dosages of liquid anti-stripping agents could be graded according to MSCR grading as the J_{nr} value is less than 4.5 kPa^{-1} for all the binders. In case of elastomeric performance, all the SBS modified binders clustered above the polymer modification standard curve and passed the criteria except 1 percent Pave Bond Lite. But all the Gilsonite-modified binders clustered below the standard line and failed to pass the criteria.

- Almost all the SBS-modified binders containing different dosages of liquid anti-stripping agents passed the % Recovery and Phase Angle criteria and fell in the "None at Risk" quadrant in the quadrant plot except 1 percent Pave Bond Lite. All these binders showed good % Recovery performance, whereas none of the Gilsonite-modified binders passed the % Recovery and Phase Angle criteria.

- Modifiers, anti-stripping agents, and dose rate substantially impacted J_{nr} and %R, according to the ANOVA study. The creep recovery performance of these binders varied greatly depending on these parameters. Modifiers had the most effects for J_{nr} and %R. A combination of the SBS modifier with 0.1% Zycotherm minimized the J_{nr} value rate and maximized the %R.

2.7 Application of the Research

In this study, four different anti-stripping agents with different dosages were mixed with SBS and Gilsonite modified binder. RTFO test was employed to evaluate the creep recovery performance of these short-term aged binders. Gilsonite modified binders were outperformed by SBS modified binders. Almost all the SBS binders containing different dosages of liquid anti-stripping agents passed Extremely Heavy traffic loading criteria, modified stress sensitivity criteria, Polymer modification curve, % Recovery and Phase Angle criteria and fell on the "None at Risk" quadrant. 0.1 % Zycotherm modified with SBS had the lowest value of J_{nr} and highest % Recovery. This dosage of SBS modified Zycotherm is suggested to use with a binder to reduce the moisture susceptibility and to strengthen the adhesion of binder with aggregate. The creep recovery performance of liquid anti-stripping in asphalt mastic and asphalt mix can be studied in the future. Long-term aging can also be considered for future analysis.

2.8 Acknowledgments

The Authors gratefully acknowledge the financial support provided by the City of St. John's and are thankful to Gary Caul and Mark white for their support. Appreciation is also extended to Mike Aurilio for his invaluable help in laboratory testing. The Authors are also grateful to, Bitumer Inc., Yellowline Asphalt Products Limited, Valero Energy Inc., American Gilsonite Company, Nouryon, Valero Energy Inc., and Zydex Industries for providing modifiers, and anti-stripping agents for the accomplishment of this study.

2.9 Author Contributions

The authors confirm contribution to the paper as follows: study conception and experimental design: Shahrul Ibney Feroz, Towhidul Islam, Dr. Kamal Hossain; data collection: Shahrul Ibney Feroz, Towhidul Islam; analysis and interpretation of results: Shahrul Ibney Feroz, Towhidul Islam Dr. Kamal Hossain; draft manuscript preparation: Shahrul Ibney Feroz. Dr. Carlos Bazan reviewed and improved the manuscript All authors reviewed the results and approved the final version of the manuscript.

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Chapter 3: Effect of Fillers, Modifiers and Anti-stripping Agents in Aged Asphalt Mastic and Asphalt Mixture

This chapter has been submitted as a technical paper in Construction and Building Materials by Elsevier as: Feroz S I**,** Alfalah A, Swarna S T, Hossain K., & Mehta Y. (2023). "Investigating the Effect of Fillers, Modifiers and Anti-stripping Agents in Aged Asphalt Mastic and Asphalt Mixture." Also, a portion of this chapter submitted as a technical paper to the Canadian Society for Civil Engineering (CSCE), as a technical paper as: Feroz S I, Alfalah A, Mitra D, Hossain K & Mehta Y (2022). "Creep Recovery Performance of Hydrated Lime and Limestone in Asphalt Mastic."

3.1 Abstract

This paper investigated the rutting performance of asphalt mastic, which is prone to distresses in flexible pavement that worsen with aging, including cracking and moisture-induced damage. The paper highlights the importance of fillers combined with modifiers and anti-stripping agents to enhance the creep recovery performance of asphalt mastic. The Multiple Stress Creep Recovery (MSCR) was utilized to understand the rutting performance of aged asphalt mastic. The performance of asphalt mastic with different filler-binder ratios of Hydrated Lime (HL) and Limestone (LS) combined with SBS or Gilsonite-modified binder containing Zycotherm or AD-Here was utilized. The study utilized various parameters such as non-recoverable creep compliance, stress sensitivity analysis, percent recovery analysis, and polymer modification curve to compare the performance of the mastics. Scanning Electron Microscope (SEM), and X-ray fluorescence spectroscopy test (XRF), were carried out to shed light on the physical and chemical properties of the fillers. The Marshall stability and flow test, Indirect Tensile Strength (ITS), and

Retained Marshall Stability tests were performed to elucidate the mixtures' mechanical performance and moisture susceptibility. Finally, an ANOVA analysis was conducted at the mastic level to determine the factors influencing the rutting performance of asphalt mastics. The results suggest that HL0.5 modified with SBS containing Zycotherm is predominant and satisfies all the rutting, moisture damage, and cracking resistance requirements. The findings highlight the importance of fillers and modifiers in enhancing the rutting resistance of asphalt mixtures, and the usefulness of MSCR test and other testing methods in evaluating the performance of the asphalt mastic.

3.2 Introduction

The construction of flexible pavement involves an aging process of the asphalt due to installation and maintenance. The aging process of asphalt causes it to become stiffer and gradually makes asphalt brittle (Lu & Isacsson, 2002). Although age-related asphalt hardening improves rutting resistance, damage is caused by accumulated cracking and moisture-induced damage (Lesueur, 2009). One effective solution is to add modifiers to the binder, with polymer modifiers being a particularly effective strategy for preventing excessive plastic deformations at high temperatures (Yildirim, 2007; Airey, 2002). Furthermore, the surface of aggregate is more attracted to water than binder due to its surface energy characteristics (Little & Jones, 2003). To prevent moisture damage, fillers and anti-stripping agents are added to the asphalt binder.

The addition of fillers to asphalt binder leads to the formation of mastic, which enhances the cohesive bonds between its components. The fillers influence the asphalt binder by increasing the stiffness and altering the moisture resistance, workability, and compaction characteristics of asphalt mixtures (Rieksts et al.,2019; Huang et al.,2007). Mineral filler, coarse, and fine aggregates are mixed in predetermined weight proportions defined by the mix design process with the asphalt binder to create a hot mix asphalt (HMA) mixture. The component of the asphalt mixture known as the mastic deforms when stress is applied (Taylor & Airey, 2008).

Asphalt mastic is a combination of asphalt binder and specific ratios of mineral filler used to manage its mixture's mechanical behavior (Roman et al., 2016; Yi-qiu et al., 2010). Most of this mineral aggregate passes a 0.075 mm sieve (Kuity et al., 2014). The HMA can be thought of as a system where the aggregates are covered with mastic rather than the asphalt binder (Wang et al., 2011). Mastic testing and research on the optimum filler-binder combination are subjects that are gradually garnering attention in this area and have demonstrated more potential than traditional binder testing (Moraes & Bahia. 2015). It is important to evaluate the performance of the combination of modifiers, anti-stripping agents, and fillers in asphalt mastic and mixture. However, few previous studies describe the rheology-like creep recovery performance of mineral fillers in modified asphalt mastic containing liquid anti-stripping agent. Therefore, this study will focus on evaluating the performance of the combination of modifiers, anti-stripping agents, and fillers in asphalt mastic and mixture, specifically focusing on the rheology-like creep recovery performance of mineral fillers in modified asphalt mastic containing liquid anti-stripping agent.

When the quantity of filler used exceeds a certain limit, thermal cracking may develop. This is because the filler particles are cementing the binder too strongly. Thus, effective rutting resistance can only be achieved by using the proper quantity of filler (Antunes et at., 2014). This investigation aims to understand the function of adding different types and F/B ratios of mineral fillers to prepare the mastic and compare their high temperature rutting and recovery performance. It is often advised that the F/B ratio does not exceed 1.4 (Zhang et al., 2004). The ideal filler content for modified mastic is between 0.8 and 1.2, while the F/B ratio for asphalt mastics with

4% SBS is 1.0, and mastic with 3.5% SBS is 1.2 (Qiu, 2013; Li et al., 2019). In this study, active filler HL is used, which enhances the ductility of asphalt mastic, lowers aging, and boosts rutting and moisture resistance (Bai et al., 2014). However, due to the considerable rise in mastic consistency, HL fillers must be introduced in small quantities. The F/B ratio was lowered to 0.3 after taking the absorption capacity test findings into account and a F/B range of 0.3-0.6 is advised to use (Antunes et al., 2014). It is important to note that the performance of an asphalt mixture may be decreased by using too much HL (Zou & Sun, 2019). Thus, an F/B ratio of 0.3-0.5 will be used to prepare the HL mastics, while 0.8-1.0 will be used to prepare the LS mastics. LS filler was selected for this study because it is a broadly used filler that has a strong stiffening capacity and helps the polymer phase function as effectively as possible (Rieksts et al., 2019).

The Effects of F/B ratios on the high-temperature characteristics of various asphalt mineral filler mastics have been found to reveal nonlinear rheological behavior (Taylor & Airey, 2008). On the other hand, unmodified mastics show less % recovery, whereas polymer-modified mastics offer more significant recovery. It is important to note that high-temperature recovery of modified asphalt mastic is yet to be evaluated. SBS and Gilsonite are two commonly used modifiers for binder modification. While sources of SBS are limited, Gilsonite is more economical. Therefore, comparing these two modifiers is essential to determine whether Gilsonite can be a viable alternative to SBS. It is crucial to use high-quality modified asphalt to prevent or minimize the negative impacts of aging on the interaction between asphalt and filler (Wu et al., 2021).

Several studies have been conducted to evaluate the rheological characteristics of asphalt mastic. The Dynamic Shear Rheometer (DSR) being the most commonly used test method following AASHTO T 315. The DSR can work in two modes: controlled stress or controlled strain (Hafeez et al., 2014; Shenoy, 2008). Two important rheological parameters, the complex modulus

(G*) and phase angle (δ), of asphalt binder and mastic, can be measured from this test and master curves can be constructed under linear viscoelastic circumstances (Underwood & Kim, 2015). The Superpave rutting parameter, often known as the index (G/sinδ), has also been used to measure the flow characteristics of asphalt mastic. However, this test method cannot measure mechanical and viscoelastic characteristics of polymer-modified binders and mastics beyond their linear viscoelastic ranges. Also, the Superpave parameter cannot measure the energy dissipation of most polymer-modified binder (PMBs) due to delayed elasticity, so a non-reversible cycle loading is suggested (Delgadillo et al., 2006).

The multiple stress creep recovery test (MSCR) method is used during this study to evaluate the creep recovery performance of mastics by applying a constant stress condition to the sample. The MSCR test technique involves loading a sample for one second at constant creep stress and then allowing it to recover for nine seconds at zero stress. The test is performed at two different levels of stress: 0.1 kPa and 3.2 kPa at 64°C (Hossain, 2016). The non-recoverable creep compliance (J_{nr}) and percent recovery (%R) values are computed from the test results at both stress levels, as stated in the applicable AASHTO and ASTM standards (AASHTO TP 70, 2009; ASTM D 7405, 2010). Instead of using the rutting parameter (G*/sinδ), the Jnr and %R can be utilized to assess rutting potential (Smucker, 2019). The %R is used to construct the polymer modification curve to interpret the elastomeric performance of the aged asphalt mastic and ensure whether the samples are modified within an acceptable range with elastomeric polymer (ASTM D 7405, 2010).

To analyze the effect of different factors like modifiers, liquid anti-stripping agents, and different F/B ratios of fillers on the J_{nr} and %R, an experimental analysis using Design of Experiments (DOE) is performed based on analysis of variance (ANOVA). Li et al. suggested using multiple regression analysis and concluded that %R was a better index to estimate the rheological properties of asphalt mastic. A multilevel factorial design is widely used in experiments that involve several factors where it is necessary to study the combined effect of the factors on a response (Smucker, 2019). The Design-Expert 13 software package generates the treatment combinations, processes the data, and plots the result.

The Marshall mix design is the most frequent technique employed for asphalt mix design (Asphalt Institute, MS-2, 2014; Stephen B, 2015). Even though the Marshall technique is empirical in nature, it might be beneficial in specific situations to compare mixes (Diab& Enieb, 2018). The Marshall mix design includes features like Marshall stability (MS) and Marshall flow (MF). Another critical parameter that can be calculated from the Marshall test is the Marshall quotient (MQ), a well-known indicator of a material's resistance to shear loads, permanent deformation, and therefore rutting (Zoorob et al., 2000). To determine the tensile characteristics of the asphalt mixtures, which are also connected to the pavement's cracking behavior, the indirect tensile strength test (ITS) is utilized (Islam et al., 2015). Moreover, moisture damage is a critical problem affecting asphalt pavements' durability and must be checked during the mix design (Ekblad et al., 2015). The Tensile strength ratio (TSR) is the most important factor to consider when assessing the moisture damage to asphalt mixes which is the ratio of ITS in wet conditions and ITS in dry conditions. However, occasionally it lacks credibility because it is only a ratio of two numbers (Diab, 2016). Therefore, besides TSR, another parameter, Retained Marshall stability (RMS) (Defense works functional standards,2005), will be used to explain the mixes' vulnerability to moisture.

3.3 Objectives

The major objectives of this experimental study include:

- Evaluate the creep recovery performance of HL and LS fillers in aged asphalt mastic containing modifiers and liquid anti-stripping agents based on non-recoverable creep compliance, percent recovery, stress sensitivity and polymer modification curve.
- Investigate the effect of filler-binder (F/B) ratio on the creep recovery performance of different aged asphalt mastic modified with SBS and Gilsonite modifiers at high temperature.
- Conduct ANOVA analysis to determine the effects of modifiers, liquid anti-stripping agents, and F/B ratios of fillers on asphalt mastic and develop an index that accurately characterizes the creep recovery performance of asphalt mastics.
- Evaluate the mix design and compare the mechanical characteristics of the mixtures to gain a fundamental understanding of the effects of HL and LS with modifiers and anti-stripping agents on asphalt mastic.

3.4 Materials and Methodology

3.4.1 Materials

3.4.1.1 Asphalt Binder

The selection of binder is crucial in preventing common pavement distresses such as rutting. For this study, PG 58-28 binder was chosen as the control binder due to its suitability for the specific environmental conditions of the southern region of Newfoundland and Labrador, Canada. PG 58-28 was selected based on its ability to perform well in extremely hot and cold climates, ensuring that the binder has the right qualities for the field's particular environmental conditions. The PG 58-28 binder was chosen to accommodate changes in traffic volume and speed as well as anticipated climate conditions.

3.4.1.2 Modifiers and Anti-stripping Agents

Two different modifiers were used in this study to improve the stiffness of the mastic: SBS (4% by the weight of base binder) and Gilsonite (10% by the weight of base binder), obtained from local sources, as shown in **Figure 3.1**. The addition of SBS polymer improves HMA's resistance to moisture-induced damage as well as its resistance to rutting and cracking (Ahmed et al.,2021). Gilsonite, a natural mineral binder, has a good affinity for asphalt (Liu & Li, 2008) and performs similarly to resist the rutting of binder (Mirzaiyan, et al.,2007). To prevent stripping between the binder and the aggregate bond, liquid anti-stripping agents Zycotherm (0.1% by the weight of base binder) or AD-here (0.5% by the weight of base binder), shown in **Figure 3.1**, were added to the modified asphalt. Xiao et al. demonstrated that applying anti-stripping agents can reduce asphalt pavement stripping (Xiao et al.,2010). The modified asphalt with 4% SBS or 10% Gilsonite and 0.1% Zycotherm or 0.5% AD-Here showed good rutting performance (Islam et al., 2022). Therefore, these ratios of modifiers and anti-stripping agents were selected for this study.

(a) (b) (c) (d)

Figure 3.1 Binder modifiers (a) Gilsonite, and (b) SBS and anti-stripping agents c) Zycotherm and d) AD-Here

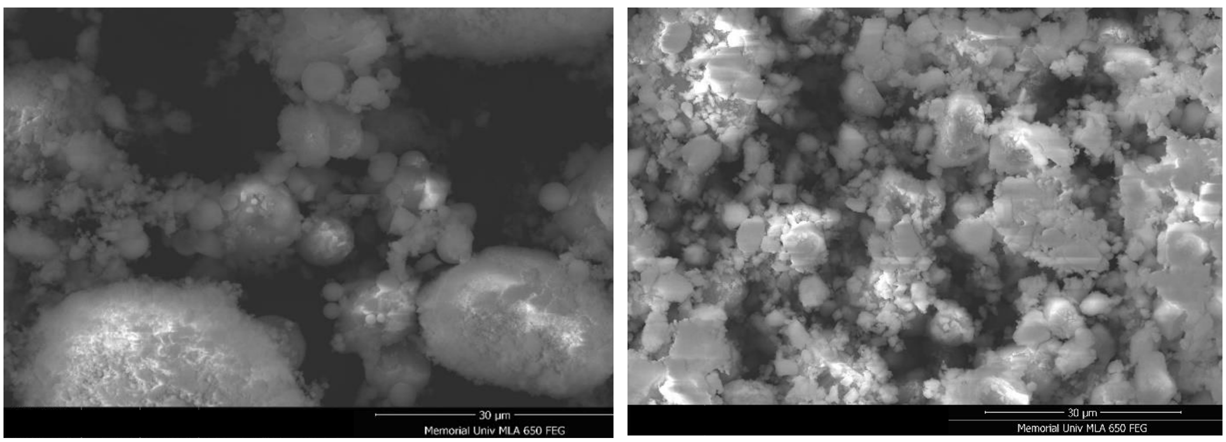
3.4.1.3 Fillers and Aggregates

Two types of fillers were selected for this study: Hydrated lime (HL) and limestone powder (LS). HL, obtained in powder form, passing sieve No. 200, and was used at three different F/B (0.3,0.4 and 0.5%). LS was obtained from local quarries, and ground using planetary ball mill equipment. The fine particles of LS were sieved with sieve No. 200, and the LS powder passing the sieve was collected for use as a filler. The fillers are presented in **Figure 3.2**. While preparing the mastic with LS, three different F/B ratios (0.8, 0.9, and 1.0) by the weight of the base binder were used to evaluate the creep recovery performance of asphalt mastic.

(a) (b) (c)

Figure 3.2 a) HL Filler, b) Limestone, and c) Limestone Filler

As part of this investigation, the physical and chemical characteristics of the fillers were examined to better understand their properties. Specific gravity tests with the 71mmett71ter method, brunauer– 71mmett–teller (BET) tests for specific surface area (SSA), scanning electron microscopy (SEM) imaging, and X-ray fluorescence spectrometry were used for this characterization. According to SSA and SG tests, HL filler had lower density while LS filler had higher density (As shown in **Table 3.1**). Additionally, the SSA of the HL filler was 2.2 times greater than the LS filler. **Table 3.1** also shows XRF oxide profiles for chemical analysis, indicating HL filler had a comparable oxide composition to LS filler, with more than 84% CaO. CaO dominated HL and LS fillers with 84.4% and 91.2%, respectively. HL had 6.4% MgO, whereas LS filler had 2.3%. **Figure 3.3** displays SEM and physical photographs of the fillers. In the case of LS filler, most particles had a small grain size and tended to agglomerate. In contrast, HL particles (**Figure 3.3(b)**) were coarser, irregular, and porous.



(a) (b)

Figure 3.3 SEM images of a) HL Filler and b) Limestone Filler

The course and fine aggregates used in this study were obtained from nearby quarries and have previously been used in several local pavement projects in Canada. Crushed stone with a maximum size of 19 mm was utilized to create a Stone Matrix Asphalt (SMA) mix. SMA mixes have shown strong resistance to rutting, owing the distinct gradation and particular strength of the coarse aggregate' (National Asphalt Pavement Association, 2002, Ahmadinia et al., 2011). Moreover, the use of SMA mixes is cost-effective in maintaining pavement and lowers noise pollution. The gap-graded structure of this combination leads to a high binder concentration, making it necessary to use modifiers to restrict the drain down of the binder (Brown et al., 1997). **Figure 3.4** depicts the gradation of the aggregate mixtures. **Table 3.1** Oxide composition and physical properties of fillers

Table 3.1 Oxide composition and physical properties of filler

	Oxide Composition							**Physical Properties**	
Fillers	MgO	Al_2O_3	SiO_2	CaO	Fe_2O_3	K_2O	Others	SSA (m^2/g)	SG
HL	6.38	0.19	7.42	84.43	0.18	0.67	0.73	10.957	2.23
LS	2.32	1.09	1.75	91.19	1.82	0.97	0.86	4.021	2.74

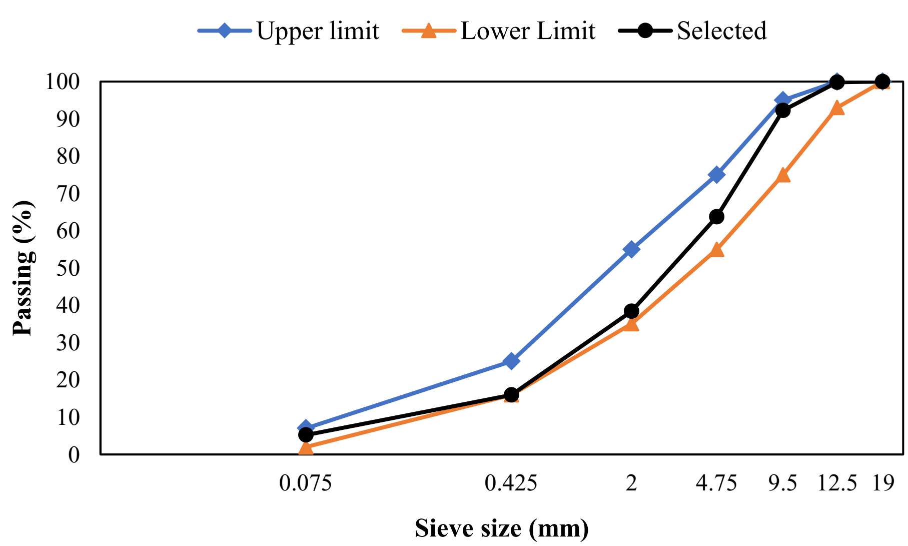


Figure 3.4 Aggregate gradation curve for HMA sample preparation

3.4.2 Methodology

3.4.2.1 Sample Preparation and Testing of Binders Containing Modifiers and Liquid Anti-Stripping Agents

To modify the binder, the neat PG 58-28 binder was preheated at 160°C until fluid. The SBS modified binder (4% by weight of base binder) was collected from a local pavement company, whereas the Gilsonite modified binder (10% by weight of base binder) was prepared by blending it with the base binder at 160°C for 1 hour to ensure homogeneity without any agglomeration. The liquid anti-stripping agents (i.e., Zycotherm and AD-here) were added separately to the modified binders. The modified PG 58-28 binder with additives was prepared by stirring with a magnetic stirrer for 45 minutes at 160°C, after several trials to determine the optimal mixing time and temperature.

3.4.2.2 Preparation of Asphalt Mastic

Prior to blending the fillers with the modified binder, each filler was carefully cleaned and dried for 24 hours at 105°C in the oven. To ensure the homogeneity of the mixture of binder with fillers, the mixing conditions were adjusted for different F/B ratios. To achieve this, each type of filler with different F/B ratios was gradually added into the heated binder and mixed using a magnetic stirrer at 160, 170, and 180°C for 60, 120, and 180 minutes, respectively. The mixing temperature and time was adjusted to avoid filler sedimentation, and to ensure the homogeneity of the asphalt mastic mixture.

3.4.2.3 Aging, Testing and Statistical Analysis

All the binders were heated at 160°C until they became pourable to prepare the sample. To simulate short-term oxidative conditioning of the base binders and modified mastics to simulate pavement aging during construction, the Rolling Thin-Film Oven (RTFO) conditioning was employed in accordance with AASHTO T 240.Continuous oxidative conditioning was applied to the binders for 85 minutes at 163°C to prepare the asphalt mastic. The DSR equipment was used for the MSCR test protocol, with 25mm plate used to prepare the mastics testing samples. In total, 24 asphalt mastics were prepared with varying F/B ratios as per the experimental plan illustrated in **Figure 3.5**. All the mastics, including the control binder, were tested twice to ensure the reliability of the data. The statistical analysis of the data was carried out using ANOVA to evaluate the effect of modifiers, liquid anti-stripping agents, F/B ratios, and fillers on the asphalt mastic.

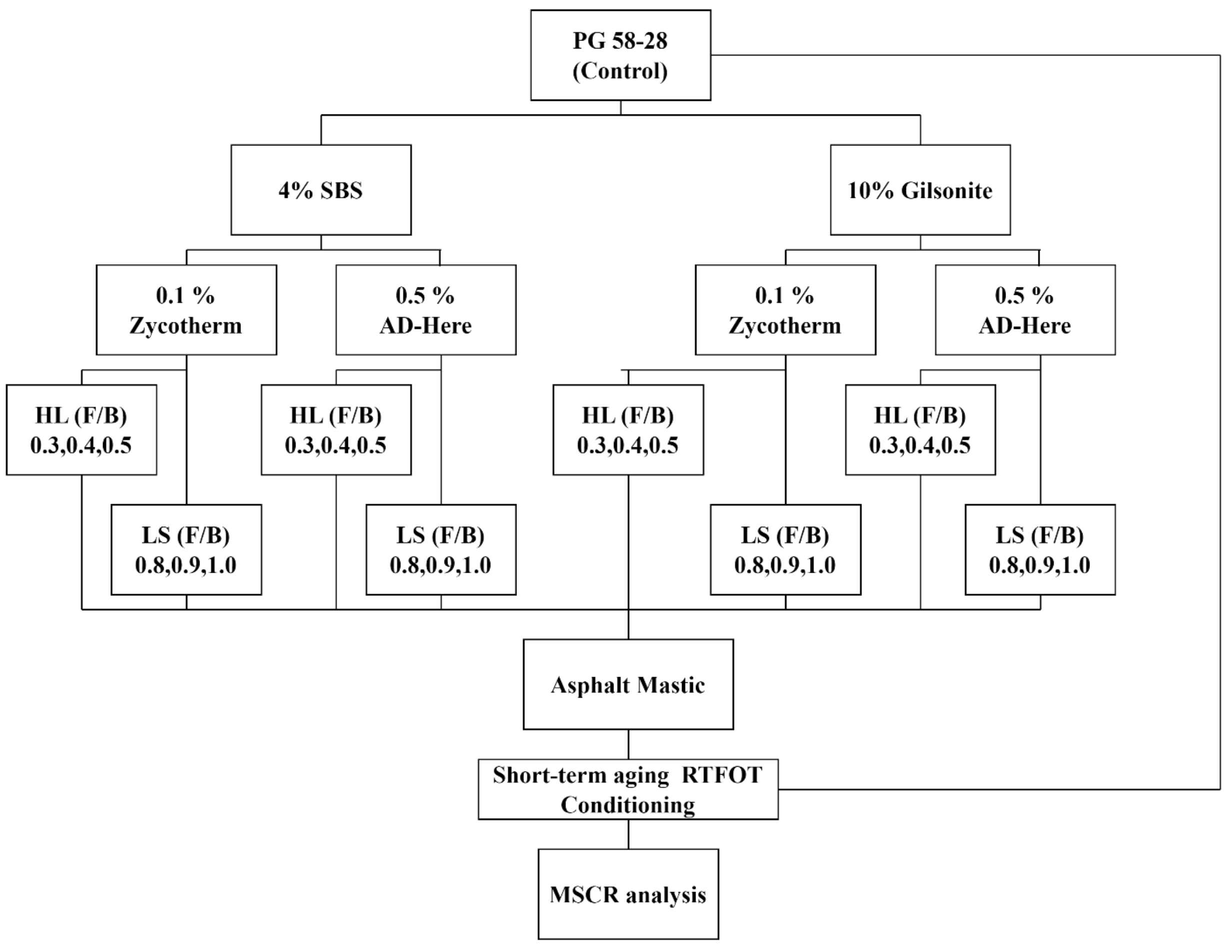


Figure 3.5 Experiment design matrix for mastic

3.4.2.4 Preparation of HMA Mixture

The HMA mixture was prepared using the Marshall method considering several modifiers, anti-stripping agents, and filler types. For fillers, the F/B ratios were chosen based on the performance at the mastic level. Marshall samples were prepared in accordance with ASTM D 6926. Initially, the aggregates used to make HMA were dried by heating them to 105°C overnight. The dry coarse and fine aggregates were then weighed and heated in a pan at 180°C for 10 minutes before being combined with the mastic, which had been heated to 130°C. Cylindrical specimens with 101.6 mm in diameter and 63.5 mm in height were prepared for the Marshall design technique by being subjected to 75 blows on each side with a standard hammer. After compacting the specimens at 145°C, the materials were combined for 5 minutes at a temperature of 155°C (ASTM D 6926). Following this, various volumetric parameters were determined, and all samples were put through tests for Marshall stability (MS) and flow values (ASTM D 6926). The modifiers, anti-stripping agents, fillers, and percentages of binder content used in the Marshall samples are listed in **Table 3.2**. Ten different mixtures were prepared, with binder contents varying between 5-7% with 0.5% increments. The mix without modifiers and anti-stripping agents, with HL filler and LS filler, is referred to as the control mixture in this study. Thus, one control mixture was prepared for HL filler and another for LS filler.

Table 3.2: Details of filler type, modifiers, anti-stripping agents and binder content to prepare the HMA mixture

Filler Type	HL					LS				
Modifier	NA	SBS		Gilsonite		NA	SBS		Gilsonite	
Anti-stripping agents	NA	Zycotherm	AD-Here	Zycotherm	AD-Here	NA	Zycotherm	AD-Here	Zycotherm	AD-Here
Binder Content*	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5
	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5
	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0

*Binder content by total mixing weight

3.4.3 Method of Analysis for Asphalt Mastic

The MSCR protocol using DSR equipment has been widely accepted to evaluate the permanent deformation behavior of asphalt mastic using the creep-recovery concept (Singh & Kataware, 2016). Three significant parameters like non-recoverable compliance (J_{nr}), stress sensitivity, and percent recovery are used to identify the creep recovery performance of the asphalt mastic obtained from the MSCR test (Golalipour et al., 2016). AASHTO M 332 specifications classify the binders as E (Extreme), V (Very heavy), H (Heavy), or S (Standard), as based on the J_{nr} value at 3.2 kPa.

3.4.3.1 Non-recoverable Creep Compliance at 3.2 kPa

The non-recoverable creep compliance (J_{nr}) is calculated to evaluate the deformation as per the AASTHO M 332. The non-recoverable creep compliance, which is evaluated at 3.2 kPa, is used to assess the sample's (i.e., mastic in this study) resistance to permanent deformation under repeated loading. A lower value of J_{nr} implies a lower rate of deformation, which implies good elasticity and higher rutting resistance (Wasage et al., 2011). The test temperature was selected at 64° C for MSCR analysis.

3.4.3.2 Stress Sensitivity

The MSCR test allows the assessment of the nonlinearity of asphalt mastic response and identifies the excessive stress sensitivity of samples in the nonlinear range. The J_{nr} difference is the difference between the J_{nr} value at stress levels of 3.2 kPa and 0.1 kPa, as defined in **Equation 3.1** (AASHTO) T 350–14, 2014), is utilized as an indicator of stress sensitivity of asphalt mastics. According to AASHTO TP 70, the J_{nr} difference should not exceed 75%. If it crosses this limit, then the samples may fail when experiencing higher stress or higher temperature in the real world, which is different from the consideration in the laboratory (AASHTO M 332, 2014).

$$J_{nr,diff.} = \frac{J_{nr,3.2kPa} - J_{nr,0.1kPa}}{J_{nr,0.1kPa}} \times 100\% \tag{3.1}$$

Where:

$J_{nr,diff.}$ is the % change of the J_{nr} value at stress levels of 3.2 kPa and 0.1 kPa;

$J_{nr,3.2kPa}$ is the J_{nr} value at stress levels of 3.2 kPa; and,

$J_{nr,0.1kPa}$ is the J_{nr} value at stress levels of 0.1 kPa.

3.4.3.3 Modified Method of Stress Sensitivity

Initially, as an indicator of the stress sensitivity of binder's, the percent difference in non-recoverable creep compliance (J_{nr} difference) obtained from the MSCR test is used. It is simply calculated as **Equation 3.1**. However, there is no correlation between the percent difference and field performance (Gaspar et al., 2019). MSCR test is widely used, and many researchers have been concerned about the applicability of its 75% limit (Laukkanen et al., 2015, Behnood & Olek, 2017). The percent difference value of a wax-modified binder is more than 75% (Laukkanen et al., 2015). As a result of the previous approach of stress sensitivity study, this binder should be avoided in road construction since it is very stress sensitive. However, the investigation revealed that the J_{nr} value for a wax-modified binder was very low at 3.2 kPa, implying that this binder was exceptionally rut resistant. As a result, non-recoverable creep compliance and percent difference are incompatible, which is why the previous approach of stress sensitivity was ineffective for modified binders. Stemphihar et al. provided a promising approach for analyzing stress sensitivity. This proposed parameter is denoted as the J_{nr} slope. **Equation 3.2** is used to calculate stress sensitivity. This new method does not unfairly penalize modified binder's which have a low J_{nr} value at 3.2 kPa and provides a comparable assessment of stress sensitivity.

$$J_{nr,slope} = \frac{dJ_{nr}}{d\sigma} = \frac{J_{nr,3.2kPa} - J_{nr,0.1kPa}}{3.1\ kPa} \times 100\% \tag{3.2}$$

Where:

$J_{nr,slope}$ is the proposed parameter for stress sensitivity;

$J_{nr,3.2kPa}$ is the J_{nr} value at stress levels of 3.2 kPa; and,

$J_{nr,0.1kPa}$ is the J_{nr} value at stress levels of 0.1 kPa.

3.4.3.4 Percent Recovery at 3.2 kPa

Percent recovery at 3.2 kPa is a critical parameter that influences the creep recovery performance of mastics in the MSCR test. It is an indication of the ability of an asphalt mastic to restore its deformation after the removal of the creep load. For any asphalt mastic, the value of %R should be non-negative meaning the residual strain at the end of the recovery portion should be no more than the accumulated strain at the end of the creep portion for a given creep and recovery cycle.

However, Soenen et al. (Soenen et al., 2013) have reported negative %R values, which are physically contradictory. The negative %R values are more common for unmodified samples at 3.2 kPa (Saboo & Kumar, 2015), but sometimes they can be observed for modified samples due to a combination of high temperature, high stress, and low modification level (Jafari et al., 2016). AASHTO M 332 proposed **Equation 3.3** as a method to detect the polymer in the samples. There is no requirement for J_{nr} values larger than 2 kPa^{-1} to have a minimum %R value (Anderson,2007,2014).

$$\%R = \begin{cases} 29.37)(J_{nr,3.2kPa})^{-0.2633}, & J_{nr,3.2kPa} \geq 0.1 \\ 55 \qquad , & J_{nr,3.2kPa} < 0.1 \end{cases} \tag{3.3}$$

Where:

$\%R$ is the Percent Recovery; and,

$J_{nr,3.2kPa}$ is the J_{nr} value at stress level of 3.2 kPa.

3.4.4 ANOVA Analysis

Analysis of variance (ANOVA) is a common statistical technique used to determine whether the mean values of two or more independent groups are the same. The ANOVA analysis helps evaluate the susceptibility of the various variables. As seen in **Equation 3.4**, if the number of treatments or levels is a and mean of different treatment is $\mu_1, \mu_2 \dots, \mu_a$, then the null hypothesis (H_0) is:

$$H_0: \mu_1 = \mu_2 = \cdots = \mu_a \tag{3.4}$$

Where:

$H_1: At\ least\ one\ mean\ is\ different$

If the null hypothesis is rejected, and an alternative hypothesis H_1 asserts that at least two groups have different mean values, then H_1 is the negation of H_0. Some fundamental indices, such as variance, sum of squares, and degree of freedom, are determined initially in the ANOVA technique (Bukat et al., 2008). **Equations 3.5-3.9** shows the sum of squares between treatments, the corrected total sum of squares, and the mean squares due to error.

$$SS_{Treatments} = \sum_{i=1}^{a} \frac{y_{i.}^2}{n} - \frac{y_{..}^2}{an} \tag{3.5}$$

$$SS_T = SS_{Treatments} + SS_E \tag{3.6}$$

$$SS_T = \sum_{i=1}^{a} \sum_{j=1}^{n} \frac{y_{ij}^2}{n} - \frac{y_{..}^2}{an} \tag{3.7}$$

Where:

$SS_{Treatments}$ is the sum of squares due to treatments (i.e., between treatments);

SS_E is the sum of squares due to error (i.e., within treatment);

SS_T is the corrected total sum of squares;

$y_{i.}$ The total of the observations for the i^{th} factor level;

$y_{..}$ grand total of all the observations;

y_{ij} observation on experimental unit in j^{th} block receiving treatment *I*; and,

n is the number of replications.

$$MS_{Treatments} = \frac{SS_{Treatments}}{a-1} \tag{3.8}$$

$$MS_E = \frac{SS_E}{a(n-1)} \tag{3.9}$$

Where:

$MS_{Treatments}$ is the mean squares due to treatments (i.e., between treatments);

MS_E is the mean squares due to error (i.e., within treatment);

The significant model term (ANOVA) was verified through Fisher's test (F-test) as seen in **Equation 3.10**.

$$F = \frac{MS_{Treatments}}{MS_E} \tag{3.10}$$

The analysis of residuals confirms the adequacy of the selected model, and the assumptions for selecting a model are that residuals are normally distributed; residuals should have constant variance, and all samples are independent of one another (Raykov & Marcoulide, 2013).

3.4.5 Method of Analysis for Asphalt Mixture

3.4.5.1 Marshall Properties and Quotient

The objective of asphalt mixture design is to determine the optimum binder content (OBC) for different combinations of hot mix asphalt (HMA) materials. The OBC is determined for each combination against 4% air voids. At the OBC, the stability and flow of all mixtures are evaluated using the same loading rate. The Marshall quotient (MQ) is then calculated as the ratio of stability to flow (kN to mm), which serves as a measure of the stiffness of mixtures. A higher MQ indicates a more rigid material that is better able to distribute the applied load and is less susceptible to permanent deformation and rutting. Therefore, as seen in **Equation 3.11**, the MQ is commonly used as an indicator of the material's resistance to shear stresses and deformation (Zoorob & Suparma, 2000).

$$MQ = \frac{Stability(kN)}{Flow(mm)} \tag{3.11}$$

3.4.5.2 Retained Marshall Stability

The ratio of stability to flow was measured for all the mixtures with different combinations at OBC to determine the MQ. These MQ values were initially considered as unconditioned MQ ($MQ_{uncond.}$). To prepare the conditioned specimens, all the mixtures were placed in a water bath at 60°C for 24 hours. The MQ values were then measured for these wet samples and referred to as MQ ($MQ_{cond.}$). To calculate the retained Marshall stability (RMS), the following formula was used as shown in **Equation 3.12**:

$$RMS = \frac{MQ_{cond.}}{MQ_{uncond.}} \times 100 \tag{3.12}$$

3.4.5.3 Indirect tensile strength (ITS) and tensile strength ratio (TSR)

Pavement engineers are concerned about the cracking issue in asphalt mixtures and therefore, they are interested in evaluating the tensile strengths of asphalt mixtures. A higher tensile strength is associated with better cracking resistance, whereas asphalt mixtures that can withstand higher stresses before failing are more likely to resist cracking. To measure the prepared specimens' moisture resistance, the loss of ITS after 24 hours of immersion in water at 60°C was used as an indicator (ASTM D4867, 2009). The TSR test was used in this investigation to determine how sensitive asphalt mixtures were to moisture. For each group of mixes, three unconditioned (dry) and three conditioned (wet) specimens were evaluated. The wet samples were then soaked in a 60°C for 24 hours, while the dry samples were kept under standard laboratory conditions. The ITS was measured on both dry and wet specimens, with each sample being tested at a constant temperature of 25°C. The thickness of each specimen was measured, and steel loading strips were inserted between the specimen and the bearing plates. Using the Marshall loading apparatus, a load was applied to the specimen by compressing the bearing plates together. The following **Equation 3.13** is used to get the ITS in kPa based on the maximum load at failure, and **Equation 3.14** shows the calculation for TSR:

$$ITS = \frac{2000 \times P_{max}}{\pi \times t \times D} \quad (3.13)$$

Where:

ITS = Indirect tensile strength (ITS), kPa

P_{max} = maximum load, N

t = specimen height immediately before test, mm

D = specimen diameter, m

$$TSR = \frac{ITS_{wet.}}{ITS_{dry.}} \times 100\% \quad (3.14)$$

Where:

$ITS_{wet.}$t = average ITS of all wet specimens in the set

$ITS_{dry.}$= average ITS of all dry specimens in the set

3.5 Result and Discussion

3.5.1 Non-Recoverable Creep Compliance, J_{nr}

3.5.1.1 Analysis of J_{nr} of SBS-Modified Asphalt Mastic

The results from the analysis of J_{nr} of SBS-Modified Asphalt Mastic are presented in **Figure 3.6**. It can be seen from **Figure 3.6** that the inclusion of filler (HL and LS) with SBS had considerably reduced the J_{nr} value compared to the neat aged binder. The J_{nr} value for the neat aged PG 58-28 was 2.15 kPa^{-1}, while all the SBS- modified mastics had a J_{nr} value less than 0.07 kPa^{-1}. In contrast, in the binder level analysis, without the inclusion of any filler J_{nr} value of SBS-modified 0.1% Zycotherm was found to be 0.2 kPa^{-1} and SBS-modified 0.5% AD-Here had a J_{nr} value of 0.22 kPa^{-1} (Feroz et al.,2022). Further analysis of mastics treated with polymers revealed that J_{nr} absolute values were substantially lower (Rieksts et al.,2019). SBS-modified HL0.5 contains 0.1% Zycotherm had the lowest value of J_{nr} and thus showed better resistance to permanent deformation. All SBS-modified HL asphalt mastic containing 0.1 % Zycotherm offered better rutting performance compared to other SBS-modified mastics, with J_{nr} value ranged from 0.01-0.02 kPa^{-1}. The J_{nr} value was decreased with the increase in HL, and the same pattern could be seen for the LS mastics containing 0.1% Zycotherm. This observation could be explained by the mastics increasing density with rising F/B ratios. Mastics with greater mineral filler concentrations generally performed better than mastics with lower mineral filler concentrations (Diab & Enieb, 2018). J_{nr} value ranged from 0.02-0.04 kPa^{-1} for LS mastics containing 0.1% Zycotherm. The mastic produced with HL filler contains 0.1% Zycotherm had somewhat lower J_{nr} values than LS filler and was hence more resistant to permanent deformations.

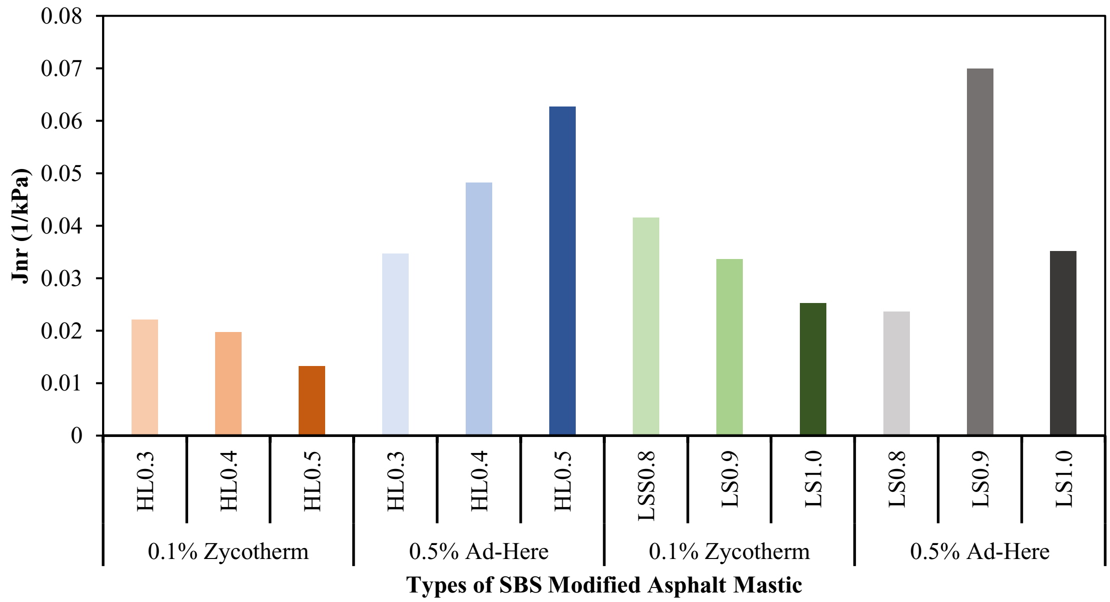


Figure 3.6 Comparison of J_{nr} at 3.2 kPa^{-1} of HL and LS SBS-modified asphalt mastic containing Zycotherm or AD-Here

Surprisingly, HL filler mastics contain 0.5% AD-Here had an increasing pattern with the increase of the F/B ratios. This may be due to the higher dose rate of AD-Here compared to Zycotherm. Among the HL filler mastic containing AD-Here, HL0.3 had the lowest J_{nr} value,. Conversely, no specific pattern for LS filler mastics contains 0.5% AD-Here was observed. The maximum J_{nr} value could be seen when the F/B ratio was 0.9, indicating lower rutting performance than other F/B ratios. On the other hand, the SBS-modified asphalt mastic with LS filler and an F/B ratio of 0.8 had the minimum J_{nr} value. Overall, SBS-modified HL and LS filler containing 0.1% Zycotherm showed better rutting performance than mastics with 0.5% AD-Here. Adding Zycotherm to the binder enhances the binding between the binder and aggregate. Zycotherm makes the aggregate surfaces more hydrophobic, eventually improving the contact between the aggregate and binder, thus increasing the performance (Mirzaiyan et al.,2019).

3.5.1.2 Analysis of J_{nr} of Gilsonite-Modified Asphalt Mastic

Figure 3.7 shows the results for Gilsonite-Modified Asphalt Mastic. **Figure 3.7** shows that the J_{nr} values ranging from 0.04-0.21 kPa^{-1} for Gilsonite-modified asphalt mastic, whereas the J_{nr} values for SBS modified mastics ranging from 0.013-0.06 kPa^{-1}. As expected SBS-modified mastics had lower J_{nr} values which implied higher rutting resistance. However, all the SBS and Gilsonite-modified mastics met the extremely heavy traffic criteria based on AASHTO M 332 specification. Gilsonite-modified HL filler and LS filler mastics contain Zycotherm had a decreasing pattern like the SBS-modified mastics although the J_{nr} values were less. HL0.5 contain 0.1% Zycotherm had the lowest value of J_{nr}. Gilsonite modified HL mastic contain 0.1% Zycotherm had the lowest J_{nr} value compared to other mastics.

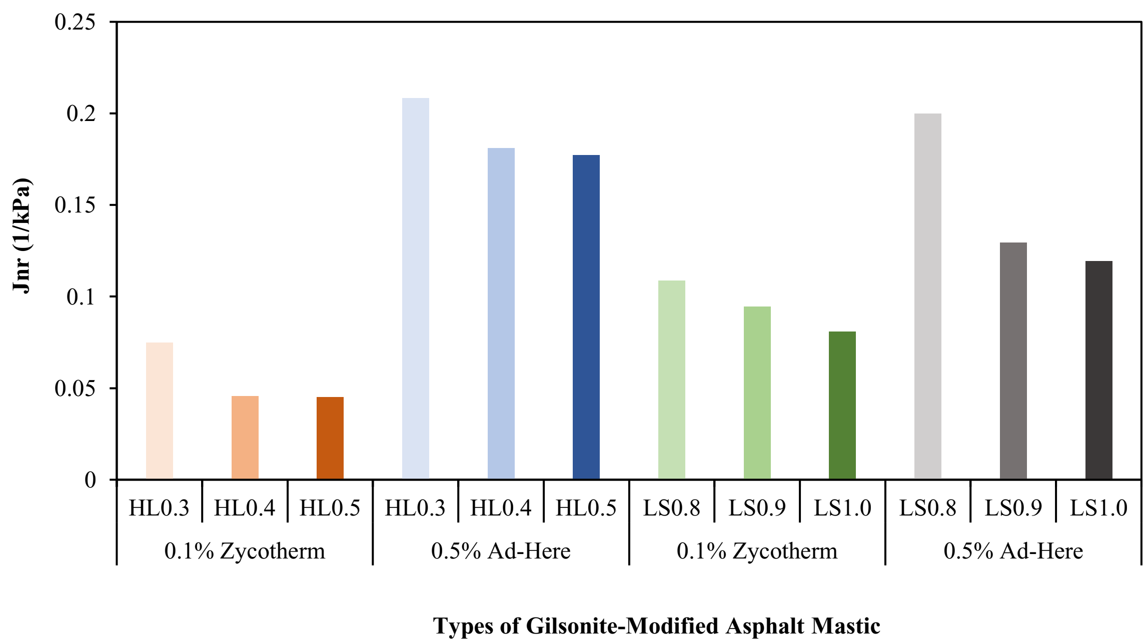


Figure 3.7 Comparison of J_{nr} at 3.2 kPa^{-1} of HL and LS Gilsonite-modified asphalt mastic containing Zycotherm or AD-Here

Gilsonite modified HL filler and LS filler mastics containing AD-Here also had a similar decreasing pattern although the J_{nr} values were higher than Zycotherm contained mastics. The greatest J_{nr} value was recorded as HL0.3 containing 0.5% AD-Here which indicated minimal rut resistance and maximum deformation in compared to other mastics. Gilsonite modified HL and LS filler containing 0.1% Zycotherm showed better resistance to permanent deformation compared to mastics with 0.5% AD-Here. Overall, SBS modified HL filler contains 0.1% Zycotherm had a lower range of J_{nr} value which implied greater recovery performance.

3.5.2 Stress Sensitivity

3.5.2.1 Analysis of Stress Sensitivity of SBS-Modified Asphalt Mastic

Figure 3.8 compares the stress sensitivity of SBS-modified asphalt mastic with different F/B ratios of HL and LS. All the mastics of HL contain Zycotherm that passed the stress sensitivity criteria and less stress sensitive than the neat aged binder. Conversely, all the mastics of LS containing Zycotherm failed the stress-sensitive criteria, which suggested that high temperatures or heavy loads excessively stress these mastics. Some mastics like HL0.4. HL0.5 and LS1.0 containing 0.5% AD-Here failed the stress-sensitive criteria.

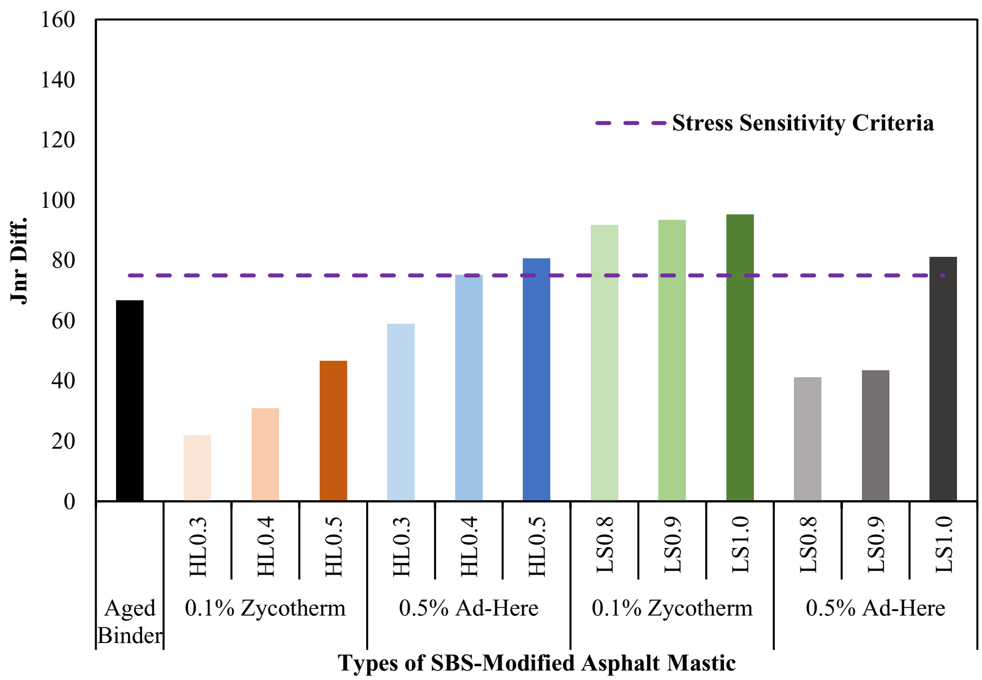


Figure 3.8 Comparison of Stress Sensitivity of HL and LS SBS-modified asphalt mastic containing Zycotherm or AD-Here

3.5.2.2 Analysis of Stress Sensitivity of Gilsonite-modified Asphalt Mastic

Figure 3.9 compares the stress sensitivity of Gilsonite-modified asphalt mastic with various F/B ratios of HL and LS. Gilsonite-modified mastics were more stress sensitive than SBS-modified mastics. The addition of Gilsonite made the asphalt mastic generally more sensitive to stress (Ameri, et al.,2011). This can also be explained by the higher J_{nr} value of Gilsonite-modified mastics compared to SBS-modified mastics. HL0.5 contain Zycotherm or AD-Here, revealed that high temperatures or large loads severely stressed these binders since they failed the stress sensitivity criteria. The stress sensitivity criteria were met by all other mastics containing HL. Most LS mastics failed the stress sensitivity criteria, except LS0.8 contains 0.5% AD-Here.

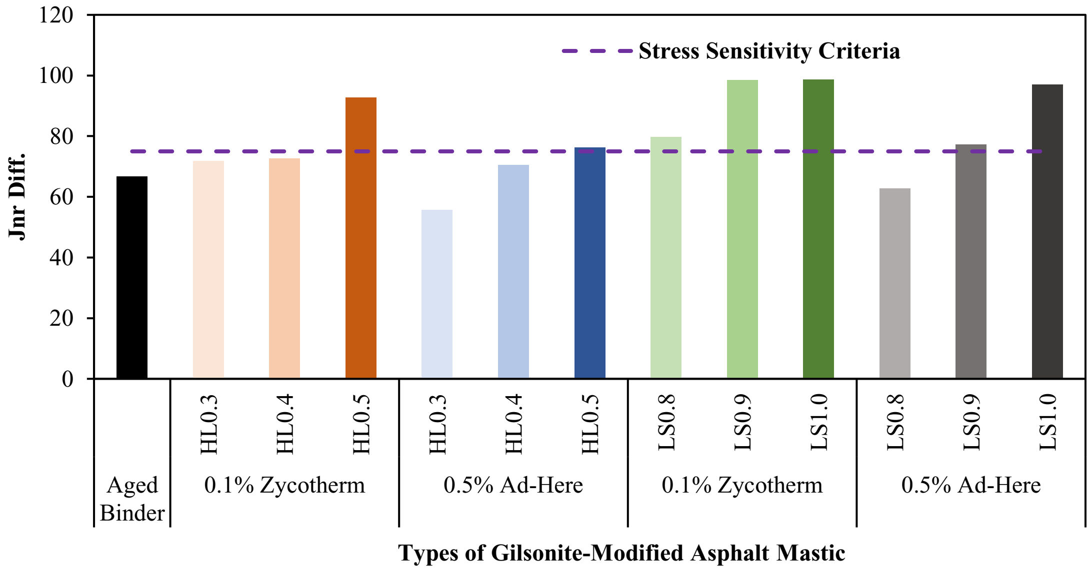


Figure 3.9 Comparison of stress sensitivity of HL and LS Gilsonite-modified asphalt mastic containing Zycotherm or AD-Here.

3.5.3 Modified Stress Sensitivity

3.5.3.1 Analysis of Modified Stress Sensitivity of SBS-Modified Asphalt Mastic

The comparison of the modified stress sensitivity of SBS-modified asphalt mastic with varying F/B ratios of HL and LS is shown in **Figure 3.10**. All the mastics passed the modified stress sensitivity criteria, whereas HL0.5 and LS1.0 contain AD-Here, and all the LS mastics containing Zycotherm failed the previous stress sensitivity criteria. The newly proposed stress sensitivity technique indicated that all mastics had a lower stress sensitivity than the neat aged binder. All HL mastics containing Zycotherm were less stress sensitive than other mastics.

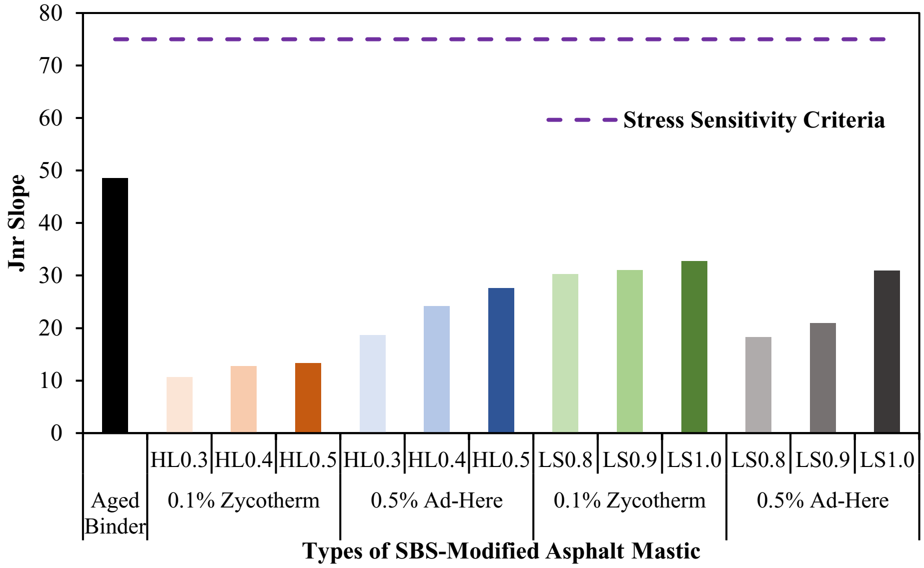


Figure 3.10 Comparison of modified stress sensitivity of HL and LS SBS-modified asphalt mastic containing Zycotherm or AD-Here

3.5.3.2 Analysis of Modified Stress Sensitivity of Gilsonite-Modified Asphalt Mastic

Figure 3.11 illustrates a comparison of the Gilsonite-modified asphalt mastic's modified stress sensitivity with various F/B ratios of HL and LS. All the mastics passed the modified stress sensitivity criteria, whereas most mastics failed the previous stress sensitivity criteria. According to the modified method of stress sensitivity analysis, all mastics had lower stress sensitivity than the aged binder.

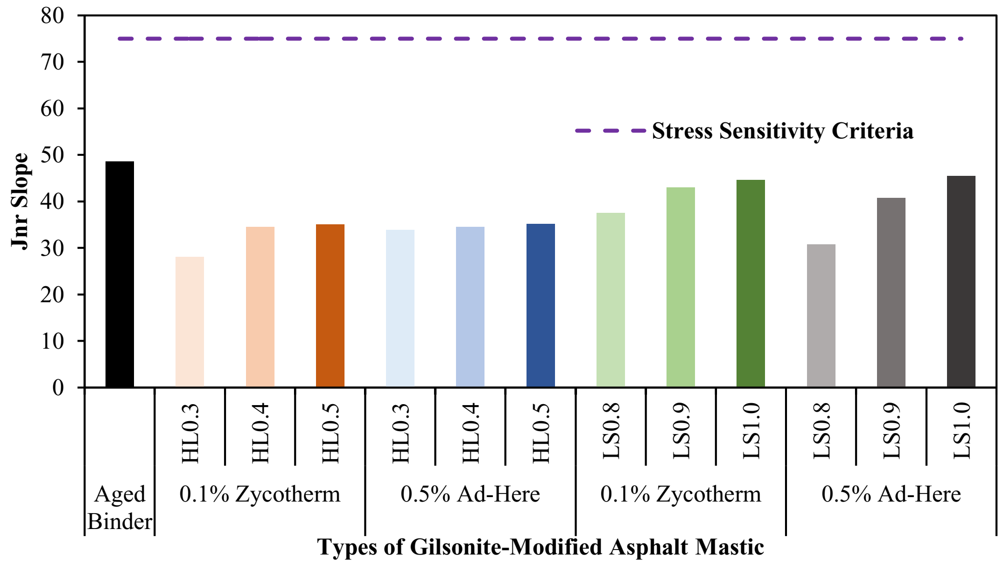


Figure 3.11 Comparison of Modified Stress Sensitivity of HL and LS Gilsonite-Modified asphalt mastic containing Zycotherm or AD-Here

3.5.4 Polymer Method and MSCR Grade

3.5.4.1 Analysis of Polymer Method of SBS-Modified Asphalt Mastic

Polymer modification curves for SBS modified mastics containing liquid anti-stripping agents 0.1% Zycotherm and 0.5%AD-Here with different F/B ratios of HL and LS are shown in **Figure 3.12**.

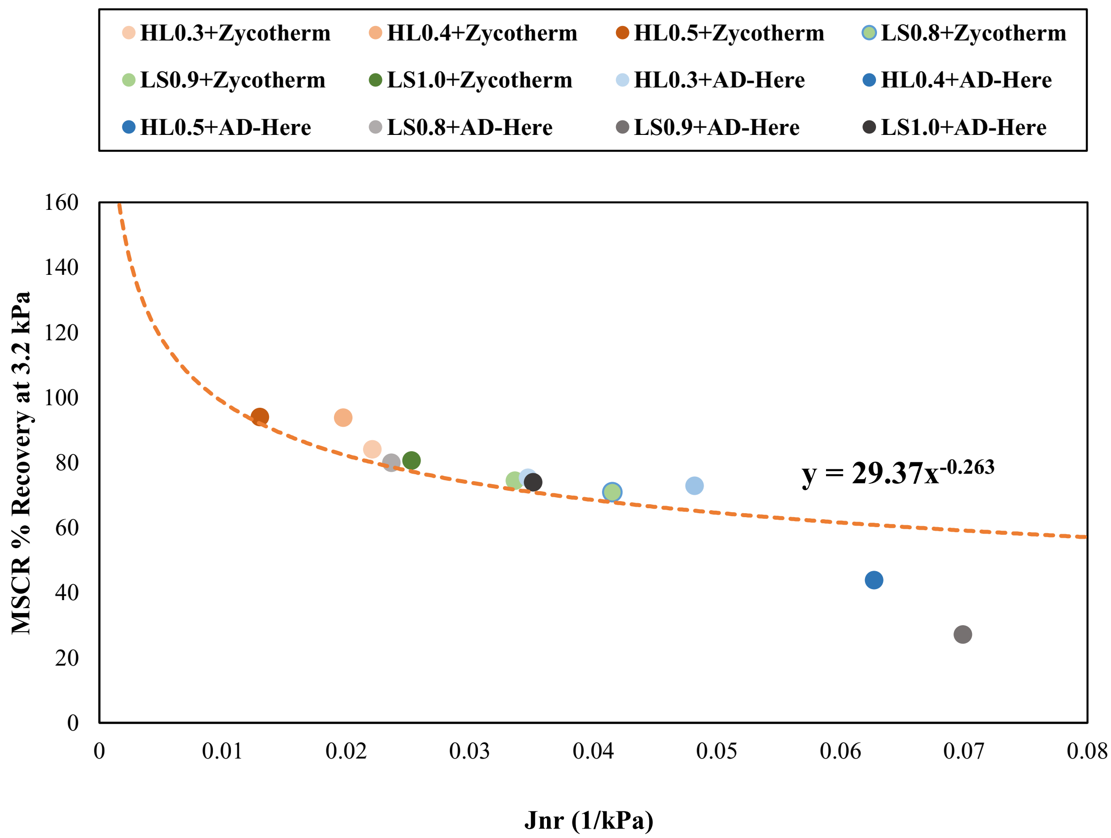


Figure 3.12 Polymer Modification curve of HL and LS SBS-modified asphalt mastic containing Zycotherm or AD-Here

The data analysis presented in **Figure 12** shows that SBS was effective in reducing the permanent deformation of asphalt mastics due to its elastomeric properties. **Figure 3.12** shows that most of the asphalt mastics (10 out of 12) contain SBS that passed the polymer modification criteria and clustered above the line. According to asphalt institute guidelines, the modification was done within an acceptable range for these mastics, and these mastics showed an excellent recovery performance. The use of Zycotherm as a modifier in every F/B ratio of HL and LS also showed promising results for recovery performance. All mastics containing Zycotherm exhibited excellent recovery compared to those containing AD-Here, as shown by their clustering above the line in **Figure 3.12**. However, HL0.5 and LS0.9 containing AD-Here modified with SBS failed to pass the polymer modification criteria and the %R value is less than 55%, indicated poor recovery performance. This implied that the modification was not done within an acceptable range. HL0.5 containing Zycotherm modified with SBS had the highest %R, indicating superior elastic recovery compared to other mastics in the study. Furthermore, all the mastics containing SBS could be graded according to MSCR grading, as their J_{nr} values were less than 4.5 kPa^{-1}. Overall, the data analysis supported the use of SBS as a modifier in asphalt mastics to enhance their elastic behavior and reduce permanent deformation. The addition of Zycotherm also showed promising results for recovery performance. However, it is important to ensure that the polymer modification was done within an acceptable range to avoid poor recovery performance.

3.5.4.2Analysis of Polymer method of Gilsonite modified Asphalt Mastic

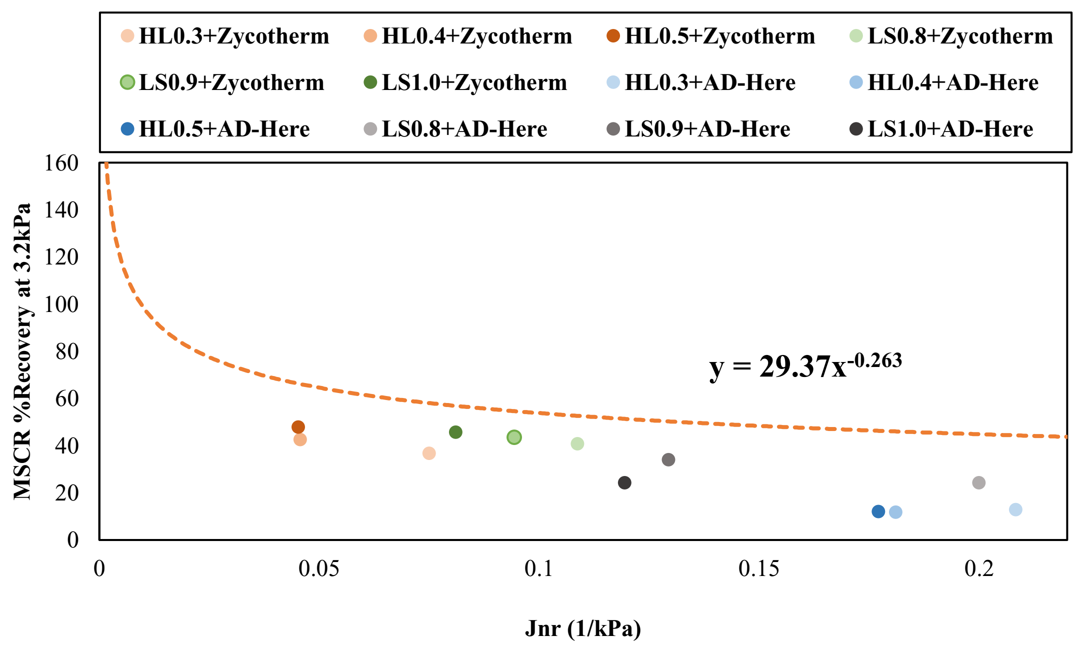


Figure 3.13 Polymer modification curve of HL and LS Gilsonite-modified asphalt mastic containing Zycotherm or AD-Here

Figure 3.13 displays the polymer modification curves for Gilsonite-modified mastics with various amounts of HL and LS containing liquid anti-stripping agent Zycotherm and AD-Here. It could be seen that all the proportions of HL and LS containing Zycotherm and AD-Here failed the polymer modification criterion and clustered below the line. Therefore, the modification was not carried out within a range suitable for these mastics, and they exhibited performed poorly after recovery. From **Figure 3.12** and **Figure 3.13**, it could be seen that SBS- modified mastics outperformed the Gilsonite-modified mastics. SBS mastics passed the polymer modification criterion, the MSCR% Recovery of the passed mastics varied between 70-94%, but for Gilsonite-modified mastics, the range was 12-48%. All the mastics containing Gilsonite could be graded according to MSCR grading**.** A summary of all the performance parameters considered for this study is given below in **(Table 3.3).**

Table 3.3 Summary of MSCR test parameters

No	Anti-stripping Agent	Filler	F/B Ratio	Modifier	Meet Stress Sensitivity Criteria	Meet Modified Stress Sensitivity	%Recovery	MSCR Grade
1	0%	0%	0%	0%	Yes	Yes	No	PG 58S-28
2	Zycotherm 0.1%	HL	0.3	SBS 4%	Yes	Yes	Yes	PG 58E-28
3	Zycotherm 0.1%	HL	0.4	SBS 4%	Yes	Yes	Yes	PG 58E-28
4	Zycotherm 0.1%	HL	0.5	SBS 4%	Yes	Yes	Yes	PG 58E-28
5	Zycotherm 0.1%	HL	0.3	Gilsonite 10%	Yes	Yes	No	PG 58E-28
6	Zycotherm 0.1%	HL	0.4	Gilsonite 10%	Yes	Yes	No	PG 58E-28

7	Zycotherm 0.1%	HL	0.5	Gilsonite 10%	No	Yes	No	PG 58E-28
8	Zycotherm 0.1%	LS	0.8	SBS 4%	No	Yes	Yes	PG 58E-28
9	Zycotherm 0.1%	LS	0.9	SBS 4%	No	Yes	Yes	PG 58E-28
10	Zycotherm 0.1%	LS	1	SBS 4%	No	Yes	Yes	PG 58E-28
11	Zycotherm 0.1%	LS	0.8	Gilsonite 10%	No	Yes	No	PG 58E-28
12	Zycotherm 0.1%	LS	0.9	Gilsonite 10%	No	Yes	No	PG 58E-28
13	Zycotherm 0.1%	LS	1	Gilsonite 10%	No	Yes	No	PG 58E-28
14	AD-Here 0.5%	HL	0.3	SBS 4%	Yes	Yes	Yes	PG 58E-28
15	AD-Here 0.5%	HL	0.4	SBS 4%	No	Yes	Yes	PG 58E-28
16	AD-Here 0.5%	HL	0.5	SBS 4%	No	Yes	No	PG 58E-28
17	AD-Here 0.5%	HL	0.3	Gilsonite 10%	Yes	Yes	No	PG 58E-28
18	AD-Here 0.5%	HL	0.4	Gilsonite 10%	Yes	Yes	No	PG 58E-28
19	AD-Here 0.5%	HL	0.5	Gilsonite 10%	No	Yes	No	PG 58E-28
20	AD-Here 0.5%	LS	0.8	SBS 4%	Yes	Yes	Yes	PG 58E-28
21	AD-Here 0.5%	LS	0.9	SBS 4%	Yes	Yes	No	PG 58E-28
22	AD-Here 0.5%	LS	1	SBS 4%	No	Yes	Yes	PG 58E-28
23	AD-Here 0.5%	LS	0.8	Gilsonite 10%	Yes	Yes	No	PG 58E-28

24	AD-Here 0.5%	LS	0.9	Gilsonite 10%	No	Yes	No	PG 58E-28
25	AD-Here 0.5%	LS	1	Gilsonite 10%	No	Yes	No	PG 58E-28

3.5.5 ANOVA Analysis

From the experimental plan, **Table 3.4** can be derived for ANOVA analysis in Design Expert Software.

Table 3.4 Parameters for multilevel factorial design

Factor	Name	Unit	Type	Number of Levels	Levels
A [Categoric]	Modifier	N/A	Nominal	2	SBS, Gilsonite
B [Categoric]	Anti-stripping agent	N/A	Nominal	2	Zycotherm, AD-Here
C [Categoric]	Filler	N/A	Nominal	6	HL0.3, HL0.4, HL0.5, LS0.8, LS0.9, LS1.0
Response			**Number of Replication**		
J_{nr}	Non-recoverable creep compliance	1/kPa		2	
%R	Percent Recovery	%		2	

3.5.5.1 Analyzed results

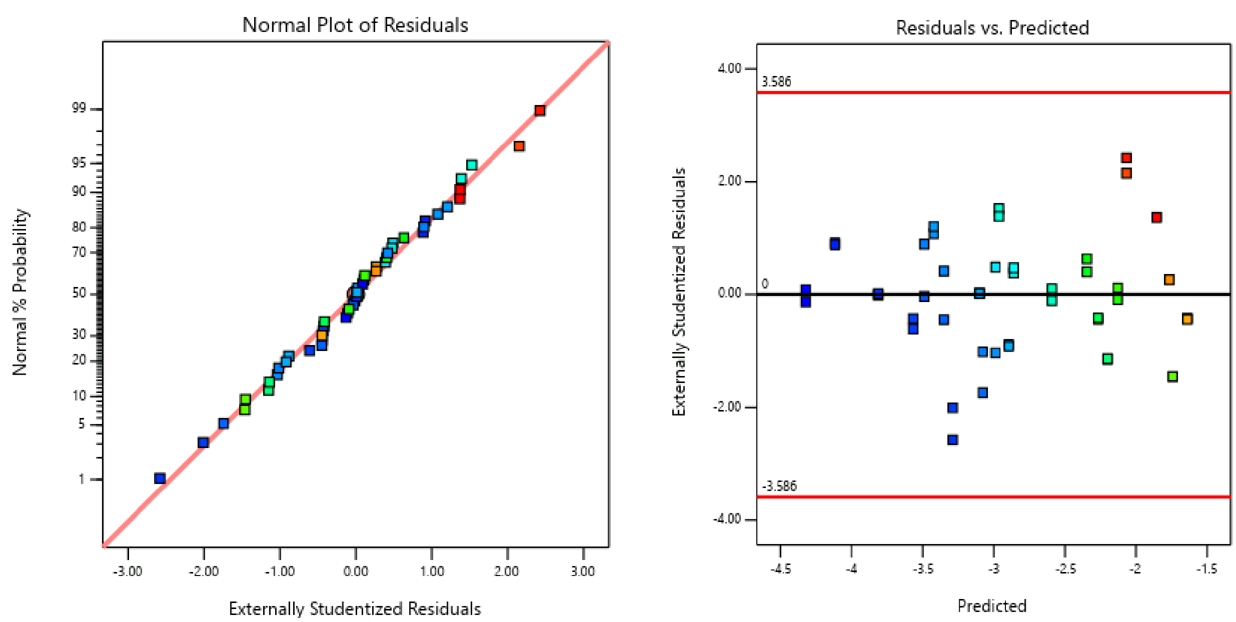
The analysis of variance (ANOVA) was conducted to analyze the collected data for both J_{nr} and %R collected from MSCR, and the results are presented in **Table 3.5**. For both J_{nr} and %R, the model F-value was significant with a p-value less than 0.0001, where the p-value measured the statistical significance of each regression coefficient. Hence, J_{nr} and %R can be used to describe the creep recovery performance of asphalt mastics. In addition, the modifiers (A), liquid anti-stripping agents (B), and fillers (C) had a significant effect on both J_{nr} and %R. Furthermore, two significant interactions were observed: between modifiers and anti-stripping agents (AB), and between anti-stripping agents and fillers (BC). A p-value less than 0.05 indicated that model terms were statistically significant, and only the statistically significant terms were included in the model. The results showed that Factor A (modifiers) had the highest F value, indicating the maximum effect on J_{nr}. Factor B (liquid anti-stripping agents) had a greater effect on J_{nr} compared to the types of fillers. On the other hand, factor B had the highest effect on the %R, while the effect of fillers was less than the effect of modifiers and liquid anti-stripping agents. All the two-way interactions were significant in the case of %R.

Table 3.5 ANOVA for both models

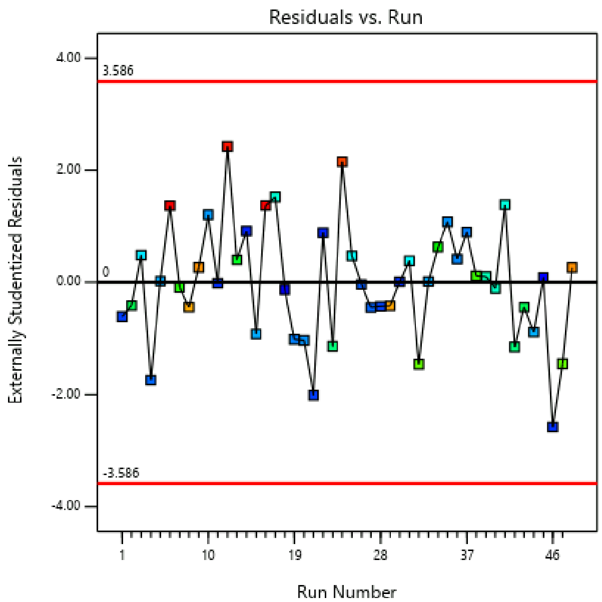
Source	**For response J_{nr}**			**For response %R**		
	Sum of Squares	**F-value**	**p-value**	**Sum of Squares**	**F-value**	**p-value**
Model	0.5259	40.92	< 0.0001	1.200E+09	23.19	< 0.0001
A-Modifier	0.3234	327.18	< 0.0001	1.559E+08	54.21	< 0.0001
B- Anti-stripping agent	0.1263	127.78	< 0.0001	6.054E+08	210.57	< 0.0001
C-Filler	0.0133	2.69	0.0372	8.019E+07	5.58	0.0010
AB	0.0146	14.79	0.0005	7.368E+07	25.63	< 0.0001
BC	0.0482	9.76	< 0.0001	7.927E+07	5.51	0.0011
AC	-	-	-	2.057E+08	14.31	< 0.0001
Residual	0.0336			8.337E+07		
R^2	0.9399			0.9350		

Adjusted R^2	0.9170	0.8947
Predicted R^2	0.8803	0.8220
Adeq. Precision	21.7884	15.9630

For J_{nr}, the predicted R^2 of 0.8803 is in reasonable agreement with the adjusted R^2 of 0.9170. The low difference between R^2 and adjusted R^2 implied that all significant terms were involved in the model (Nayak et al.,2019). Additionally, the difference between R^2 and adjustedR^2 for %R was also less than 0.2, which implied that the data was not over fitting. Adeq. Precision measures the signal-to-noise ratio, and a ratio greater than 4 is desirable. The models showed an adequate signal, with a ratio of 21.788 and 15.963 for J_{nr} and %R, respectively, indicated an adequate signal. These models could be used to navigate the design space.



(a) (b)



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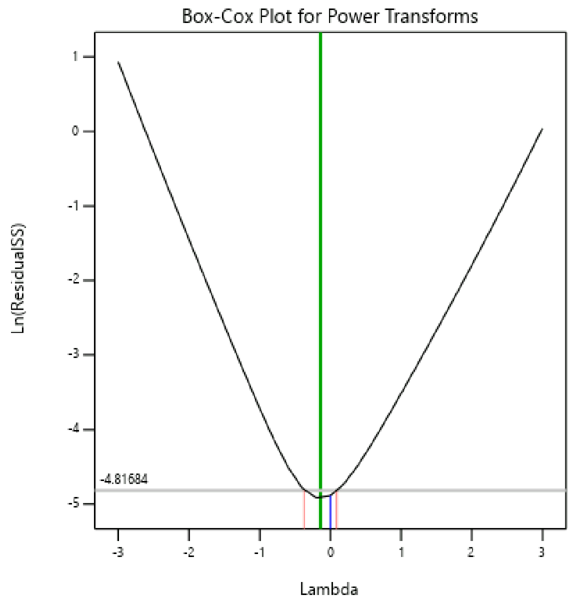
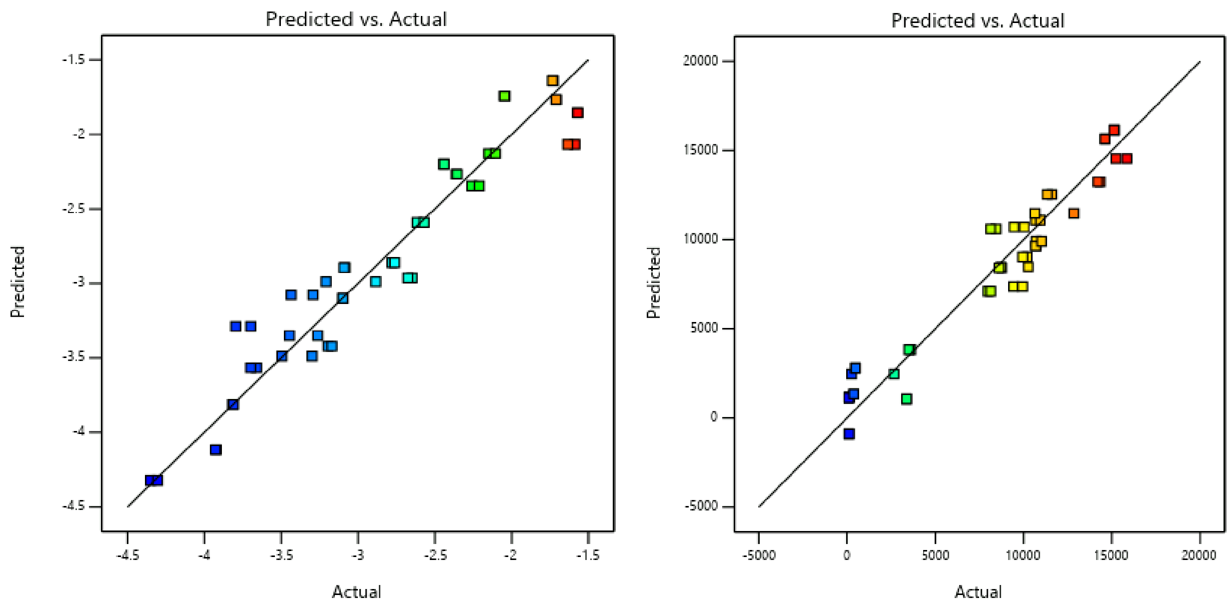


(d)

Figure 3.14 (a) Normal Plot of Residuals of J_{nr} (b) Residual vs. Predicted of J_{nr} (c) Residual vs Run of J_{nr} (d) Box-Cox plot of J_{nr}

The analysis included diagnostic tests to check the validity of the statistical assumptions. The Normal plot of the residuals in **Figure 3.14 (a)** showed that most of the points followed a straight line, with some scattered points around the straight line, which means that the residuals were normally distributed. From **Figure 3.14 (b)**, the residuals were randomly scattered, around zero, for the entire range of fitted values. Furthermore, the residuals were randomly scattered around zero for the entire range of fitted values, indicating constant variance. **Figure 3.14 (c)** shows the residuals vs. run numbers for the ANOVA test. All the residual points were scattered within upper and lower limits and did not follow a specific pattern, confirming the assumptions of independent observations. From the box-cox plot in **Figure 3.14 (d)**, the current Lambda line fell between the orange lines, and square root transformation was recommended. Therefore, the required transformation was done for this design. All the assumptions were also checked for %R and found to be satisfactory. The power transformation was recommended and applied accordingly. Overall, the diagnostic tests confirmed that the assumptions of normality, constant variance, independence of observations, and transformation were valid for the study.



(a) **(b)**

Figure 3.15 Predicted vs Actual plot for (a) J_{nr} (b) %R

The Predicted vs Actual plot data from **Figure 3.15 (a)** shows that the data was evenly split by the 45° line, with all points close to the straight line. This indicated that the model fit well and the predicted vs. actual plot was satisfactory. **Figure 3.15 (b)** shows that the expected vs. actual plot was good and that the model matches the data adequately. It could be concluded that both the models of J_{nr} and %R met all the assumptions, where almost the same R^2 value. Therefore, both J_{nr} and %R can be used to describe the creep recovery performance of asphalt mastics.

The developed model was optimized to obtain the parameters that would minimize the J_{nr} value and maximize the %R. **Figure 3.16** shows the constraints for the optimization process. All the factors' modifiers, anti-stripping agents, and fillers have significant influences on the J_{nr} and %R. All the factors studied were categorical, and optimized results were found after twenty-three solutions. SBS modifier, Zycotherm liquid anti-stripping agent, and HL filler with F/B ratio of 0.5 resulted in minimized J_{nr} and maximized %R. The minimum J_{nr} value was 0.013 kPa^{-1}, and the maximum %R was 94% with high desirability of 0.994.

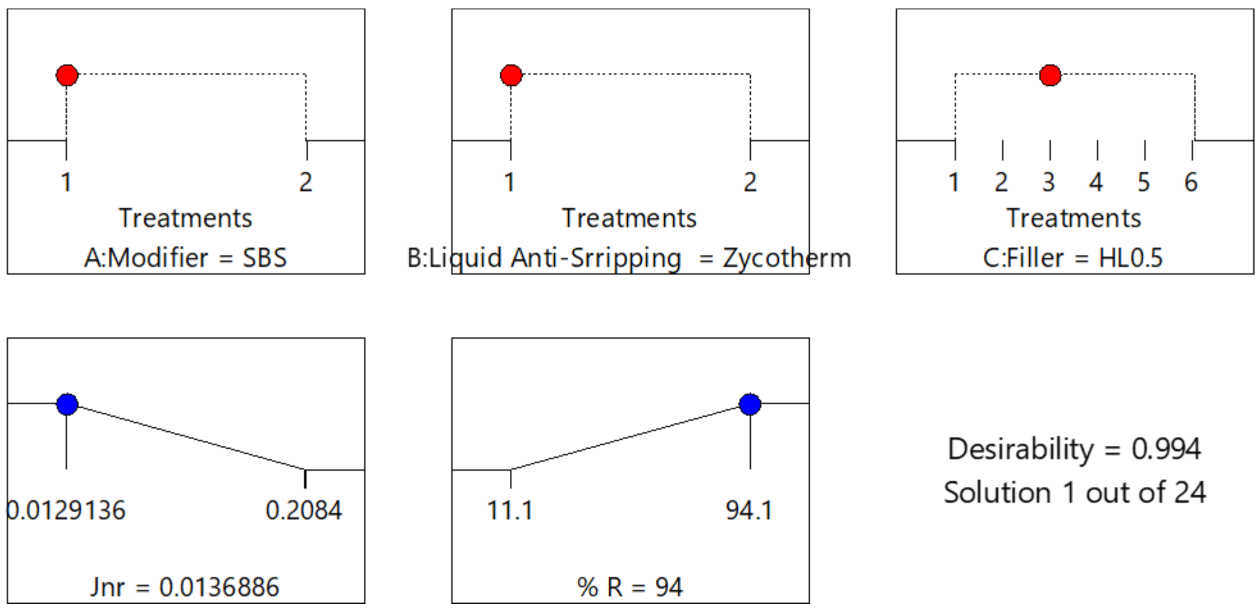


Figure 3.16 Optimization of J_{nr} and %R

3.5.6 Marshall properties of Asphalt Mixture

Figure 3.17 displays the OBC of all the mixes, which range between 6-6.5%. The mixtures' gap-graded structure explains their high OBC. HL required a higher OBC for all the combinations than LS due to its porous structure and propensity to absorb binder, resulting in a ticker film on the aggregates and a reduction of air voids in the mix (Arabani & Tahami, 2017). For both HL and LS, mixtures modified with Gilsonite had higher OBC requirement than the control mixtures and SBS modified mixtures. This is due to the affinity of Gilsonite to the asphalt binder and high binder absorption capacity. However, AD-Here had higher OBC requirements compared to Zycotherm for both modifiers.

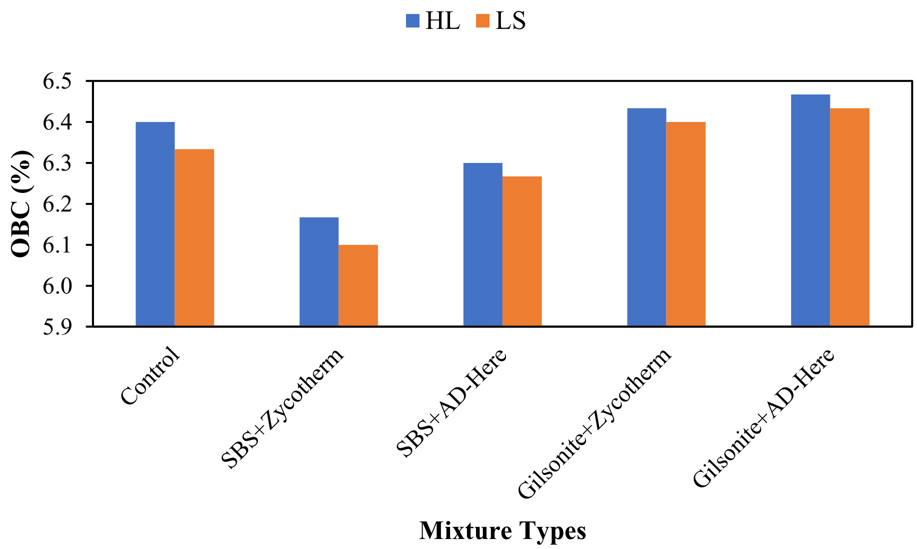


Figure 3.17 OBC of mixtures

Figure 3.18 shows the stability test outcomes at OBC for each mixture. According to the findings, the mixtures' stability increased with modifiers and anti-stripping agents. Mixtures containing HL were more stable than that containing LS due to improved adhesion between HL and binder. The mixture with SBS and Zycotherm was predominant for HL and LS, although there was no excessive change in the stability value for other combinations. Mixtures containing HL and LS with the combination of SBS and Zycotherm had the highest stability values of 13.2 kN and 12.4 kN, respectively. All combinations met the Asphalt Institute guideline for the heavy traffic of not less than 8.01 kN.

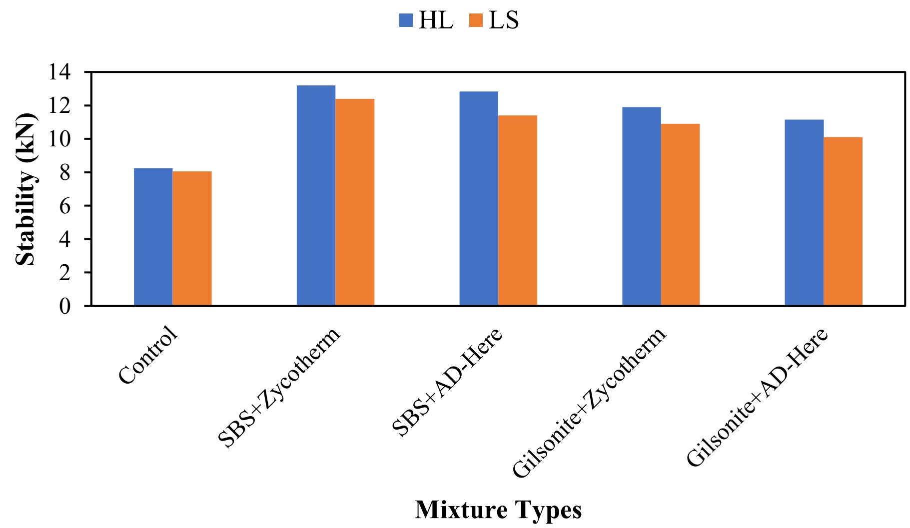


Figure 3.18 Stability of mixtures at OBC

Figure 3.19 displays the impact of all the combinations, as mentioned earlier, on MQ values. The MQ values for the control mix of HL and LS were 2.84 kN/mm and 2.88 kN/mm, respectively. LS had higher MQ values than HL for all modifier combinations and anti-stripping agents. The higher MQ values for LS may be attributed to the fact that LS had a decreased OBC, which improved binder's viscosity and stiffness. This improved the HMA's overall capacity to bear stresses and strengthens the bond between mastic and aggregates (Hamedi & Tahami, 2017). Moreover, Gilsonite-modified mixtures had higher MQ values than SBS-modified mixtures. Gilsonite improved the stiffness of asphalt binders (Anderson M, 2010). The highest MQ values of 5.05 kN/mm were obtained for Gilsonite with AD-Here and LS, which slightly exceeds the prescribed maximum limit of 5 kN/mm (AASHTO T 283, 2014). However, all the SBS-modified mixtures showed MQ values within the prescribed maximum limit of 5 kN/mm.

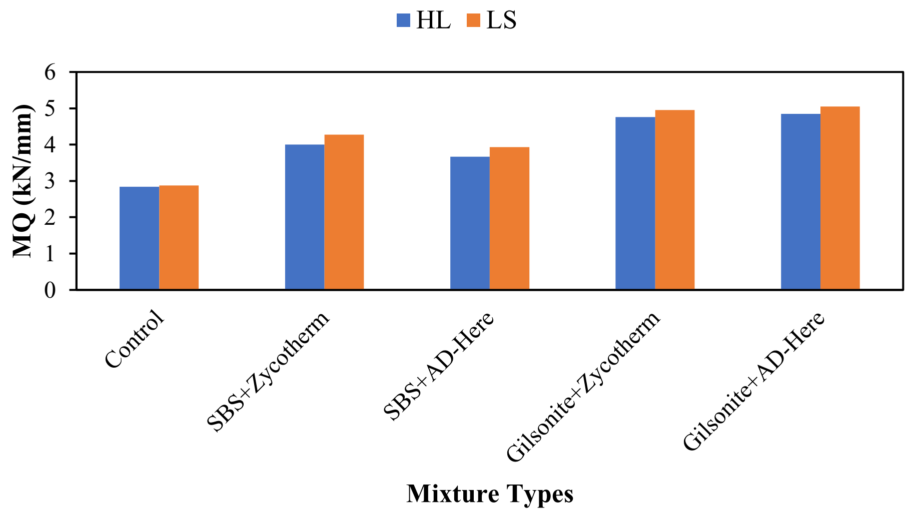


Figure 3.19 MQ values of mixtures at OBC

3.5.7 ITS and TSR of asphalt mixtures

The impact of various fillers, modifiers, and anti-stripping agents on the ITS of HMA are shown in **Figure 3.20**. To fully evaluate the resistance of asphalt mixes to moisture damage, it is necessary to consider both the dry and wet ITS values. The HL filler exhibited higher dry and wet ITS values, indicating higher resistance to cracking. This can be attributed to the adhesive properties of HL filler, which work well with asphalt mixes. HL in combination with SBS and Zycotherm showed the maximum ITS value of 1328 kPa, which is 31% more than the mixture without modifiers and anti-stripping agents. The findings also reveal that Gilsonite-modified mixtures exhibit lower ITS values compared to SBS-modified mixtures, although the former had higher stiffness. This suggests that HMAs with high stiffness are more likely to crack easily and have lower tensile strength bearing capacity. Moreover, Zycotherm showed better performance compared to AD-Here as an anti-stripping agent.

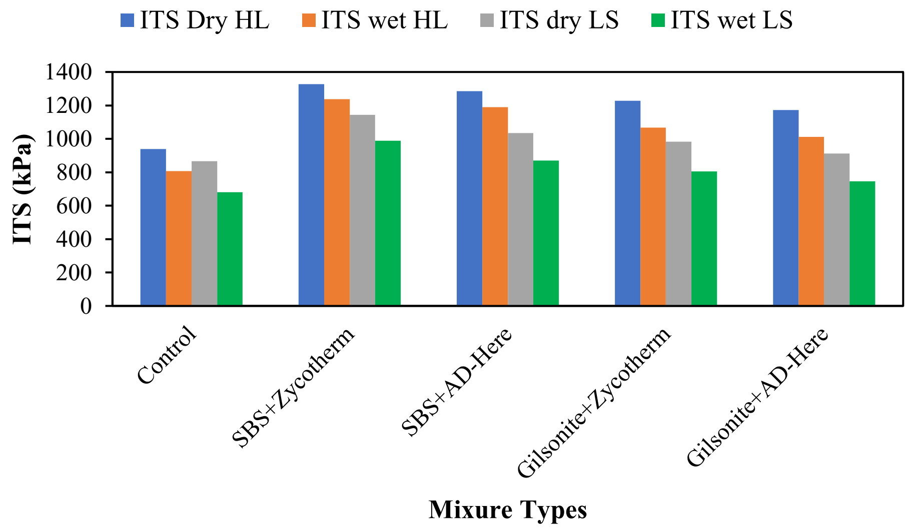


Figure 3.20 ITS values of mixtures at OBC

The TSR values for the investigated mixes are shown in **Figure 3.21**. Compared to the LS-containing mixtures, the HL asphalt mixtures showed greater TSR values, indicating better moisture resistance. The use of HL as a multifunctional filler significantly impacted the moisture resistance of the HMA mixture, which is consistent with other research findings (Diab,2016). Moreover, HL particles dispersed well in the HMA mixture due to their large SSA and porous structure, improving the adhesion between binder and aggregates. All mixtures, except for the control LS, met the minimum 80% TSR value required by AASHTO T 283 specifications. The mineral filler tends to fill the spaces within the mixture, increasing density andpotentially reducing moisture ingress and thus reducing moisture damage. The highest TSR value can be seen for HL contains SBS and Zycotherm. In comparison, Gilsonite-modified mixtures had a lower TSR value than SBS modified mixtures. Furthermore, Zycotherm performed well as an anti-stripping agent, compared to AD-Here.

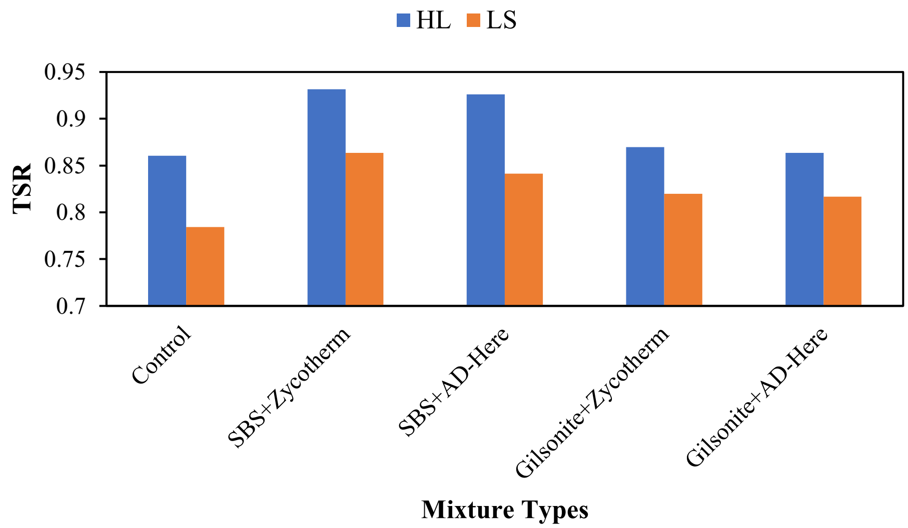


Figure 3.21 TSR values of mixtures at OBC

3.5.8 Retained Marshall Stability

Figure 3.22 shows RMS values for the different mixtures of HL and LS. RMS is an important factor to consider when assessing the moisture damage to asphalt mixes because it provides a measure of the mixture's long-term performance. The control mixtures had lower RMS than other mixtures. LS fillers contained mixture had lower RMS values than HL-contained mixtures, which explains the dominant performance of HL in water resistance. Similar performance can be seen when the TSR was considered.

In the case of HL filler, SBS with Zycotherm had the highest RMS value and the highest water resistance. Whereas, for LS filler, SBS with AD-Here had the highest value of RMS value. However, the Gilsonite-modified mixtures showed good stiffness performance but failed to achieve a dominant performance in water resistance compared to SBS-modified mixtures. Gilsonite-modified mixtures have an RMS value similar to control mixtures.

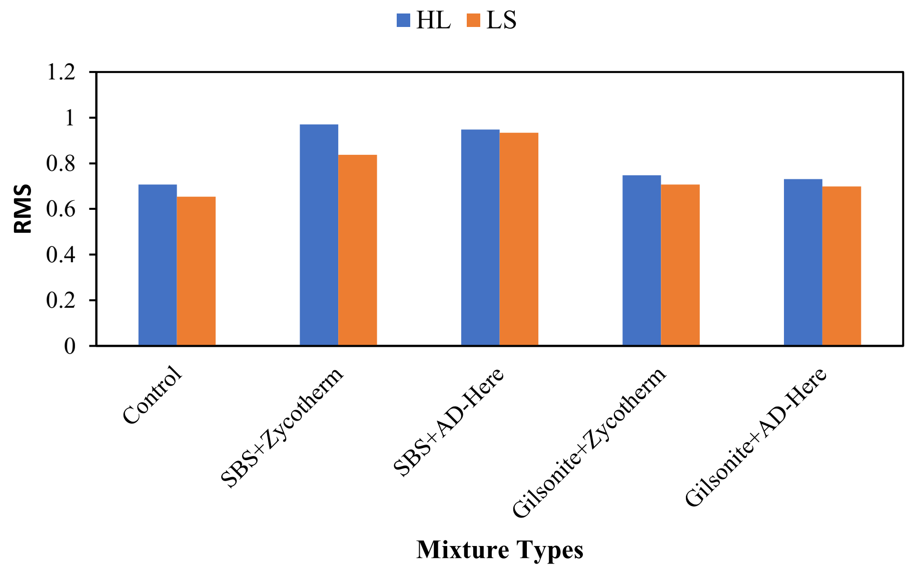


Figure 3.22 RMS values of mixtures at OBC

3.6 Summary of Findings

The research aimed to evaluate the creep recovery performance and rutting resistance of the combination of fillers, modifiers, and anti-stripping agents at the mastic scales and to evaluate the moisture-induced damage resistance and cracking resistance of these mastics at the mixture level. This investigation used HL and LS as fillers, SBS and Gilsonite as modifiers, and Zycotherm and AD-Here as anti-stripping agents. Three different F/B ratios of each filler were used to prepare the mastics to investigate the effect of varying F/B proportions. The following findings can be drawn based on the experimental results collected from different parameters' mastic and mixture level analysis.

- The inclusion of HL and LS filler material reduced the asphalt mastic's J_{nr} value compared to the neat aged binder and other binders considered in the binder level study (i.e., SBS modified 0.1% Zycotherm and 0.5% AD-Here, Gilsonite modified 0.1% Zycotherm and 0.5% AD-

Here). SBS-modified mastics showed a J_{nr} value range of 0.01-0.07 kPa^{-1}, whereas Gilsonite-modified had 0.04-0.21 kPa^{-1}. All mastics passed modified stress sensitivity and had less stress sensitivity than the neat aged binder.

- From the polymer modification curve, all the SBS and Gilsonite modified mastic containing different amounts of HL and LS could be graded according to MSCR grading as the J_{nr} value was less than 4.5 kPa^{-1} for all the mastics. The MSCR % Recovery for SBS-modified mastics passed the polymer modification curve fluctuated between 70-94%, whereas, for Gilsonite, all mastics clustered below the curve and failed the criteria, the range was 60-78%.

- From the ANOVA analysis, modifiers, anti-stripping agents, and filler significantly affected J_{nr} and %R. For J_{nr}, modifiers had the highest effect, whereas, for %R, the anti-stripping agent had the highest impact. Both J_{nr} and %R can be used to describe the creep recovery performance of asphalt mastics.

- OBC ranged between 6-6.5% for all the mixtures. OBC requirements varied with the combination of different fillers, modifiers, and anti-stripping agents. The addition of SBS and Zycotherm demonstrated a 4% reduction in OBC compared to the control mix, suggesting a considerable reduction in the cost of producing HMA.

- The Marshall stability values for all combinations can be observed to meet the Asphalt Institute guideline for heavy traffic of not less than 8.01 kN. HL and LS mastics containing SBS had a higher stability value than Gilsonite.

- ITS increased significantly for all the other mixtures compared to the control mix. A mixture with HL filler, SBS, and Zycotherm has the highest ITS value of 1328 kPa, 31% more than the mixture without modifiers and anti-stripping agents, implying better-cracking resistance. In the case of TSR value, HL mastics had a better TSR value range of 0.86-0.93; for LS mastics, this range was 0.78-0.86. Mixture's RMS value showed the same results as TSR.

3.7 Conclusions

The study's findings allow to draw the following conclusions:

- HL0.5 with Zycotherm had the lowest J_{nr} compared to the other mastics, suggesting higher resistance to persistent deformation and rutting. This mastic passed the stress sensitivity criteria.
- In the case of elastomeric performance, SBS had better impact than Gilsonite. SBS modified HL0.5 with Zycotherm cluster above the polymer modification line and had the highest delayed elastic response.
- From the optimization analysis of the mastics, SBS modified HL0.5 mastic contains 0.1% Zycotherm was found to be minimized the J_{nr} value and maximized the %R.
- HL mastics had higher OBC requirement compared to LS mastics. The combination of HL0.5, SBS and Zycotherm reduced the OBC requirement compared to the control mix.
- A mixture with HL filler, SBS, and Zycotherm had the highest ITS and TSR value, implied better cracking, and moisture-induced damage resistance.

3.8 Limitations and Recommendations

The above-mentioned results were obtained after completing a limited number of laboratory tests. The performance of the mastics depended on the mixing procedure. The binder, modifiers, and anti-stripping agents were collected from only one source, where they can perform differently depending on the source. Long-term aging, the low-temperature performance of the mixtures, and the chemical characterization of these mastics can also be considered for future analysis. A combination of active and inert filler in mastic scales and mixture level can also be evaluated. Superpave mix design can be considered for future study. Additional lab studies are needed to understand other rheological properties and field tests are needed to obtain more practical results of these alternative fillers, additives and modifiers.

3.9 Application of the Research

In this study, different proportions of two different fillers (HL and LS) were mixed with SBS or Gilsonite modified binder containing anti-stripping agent Zycotherm or AD-Here to fabricate the asphalt mastic. RTFO conditioning was employed to evaluate the creep recovery performance of these short-term aged asphalt mastics. SBS-modified HL mastics outperformed the other SBS-modified and Gilsonite-modified mastics. All the SBS modified HL mastics containing Zycotherm passed Extremely Heavy traffic loading criteria based on AASHTO M 332, modified stress sensitivity criteria, and polymer modification curve. These mastics are suggested to use in the asphalt mixture to reduce the moisture susceptibility and to strengthen the adhesion of binder with aggregate. The mixture level analysis showed these mastics' good cracking and moisture damage-resistant performance.

3.10 Acknowledgements

The authors gratefully acknowledge the financial support provided by the City of St. John's and are thankful to Mark White and Gary Caul for their support. Appreciation is also extended to the Center for Research and Education in Advanced Transportation Engineering Systems (CREATES), Rowan University, USA, for their invaluable helps in lab testing. The authors are also grateful to McAsphalt, Yellowline Asphalt Products Limited, Atlantic Minerals, American

Gilsonite Company, and Zydex Industries for providing asphalt, modifiers, and anti-stripping agents for the accomplishment of this study.

3.11 Author Contributions

The authors confirm contribution to the paper as follows: study conception and experimental design: Shahrul Ibney Feroz, Dr. Kamal Hossain; data collection: Shahrul Ibney Feroz, Ahmad Alfalah, Dr Surya Teja Swarna; analysis and interpretation of results: Shahrul Ibney Feroz, Dr. Kamal Hossain; draft manuscript preparation: Shahrul Ibney Feroz. Dr. Yusuf Mehta reviewed and improved the manuscript All authors reviewed the results and approved the final version of the manuscript.

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Chapter 4: Combined effect of active and inert filler on rheological and mechanical performance of asphalt mastic and asphalt mixture

A portion of this chapter has been submitted to the Transportation Association of Canada (TAC*)* Conference 2023, as a technical paper as: Feroz S I, Mitra D, Kabir SK, Hossain K & Mehta Y (2023), "Performance of aged asphalt mastic combining active and inert filler materials in terms of creep recovery." Some other portion of this chapter has been submitted to the Canadian Technical Asphalt Association (CTAA) Conference 2023, as a technical paper as: Feroz S I, Mitra D, Mohajan A, Hossain K & Mehta Y (2023), "Investigating the combined effect of active and inert filler on rheological and mechanical performance of asphalt mastic and asphalt mixture."

4.1 Abstract

Filler, a fine powder used in asphalt mixture, plays a dual role as an inert filler to fill gaps between mineral aggregates and an active filler to mix with asphalt binder to generate a high-consistency asphalt mastic. This mastic is the main component of an asphalt structure that deforms when stress is applied and may significantly alter the mixture's physical and mechanical properties. Substantial research focused on using either active or inert fillers, whereas only a few research has examined the combined impact of active and inert fillers. This study compares the rheological and mechanical performance of aged asphalt mastics and asphalt mixtures fabricated by combining active and inert fillers containing modifier and anti-stripping agent. This paper employed the Multiple Stress Creep Recovery (MSCR) test to understand the rutting performance of aged asphalt mastic. This investigation used Styrene-Butadiene-Styrene (SBS) as modifiers to modify the neat PG 58-28 binder and Zycotherm as liquid anti-stripping agents. For fabricating the mastics, different proportions (10%,20%,30% by the weight of base binder) of Hydrated lime (HL) and Fly

ash (FA) were selected as active fillers. In contrast, different proportions (70%,60%,50%) of Limestone (LS), Dolomite (DM), and Basalt (BS) were selected as inert filler materials. The active and inert fillers were added so that the Filler Binder (F/B) ratio remains 0.8. Rolling Thin-Film Oven (RTFO) protocol was applied to simulate asphalt production time aging. The performance of these mastics was compared using non-recoverable creep compliance, stress sensitivity analysis, percent recovery analysis, and polymer modification curve. Scanning Electron Microscope (SEM), and X-ray fluorescence spectroscopy test (XRF), were carried out to shed light on the physical and chemical properties of the fillers. The Marshall stability and flow test, Indirect Tensile Strength (ITS), and Retained Marshall Stability tests were performed to elucidate the mixtures' mechanical performance and moisture susceptibility. Finally, an ANOVA analysis was conducted at the mixture level to determine the significant factors to describe the mechanical performance of asphalt mixtures. Mastic scale investigation finds the combination of 10% HL and 70% LS containing SBS, and Zycotherm is predominant with higher rutting resistance and recovery. In the mixture level, 10% HL and 70% LS exhibit a stronger affinity for the binder, responsible for their significant stiffening effect and acceptable resistance to rutting and moisture damage.

4.2 Introduction

The aging process of asphalt causes it to become stiffer and gradually makes asphalt brittle (Lu & Isacsson, 2002). Although age-related asphalt hardening improves rutting resistance, damage is caused by accumulated cracking and moisture-induced damage (Lesueur, 2009). Including modifiers improves the binder's performance and reduces pavement distress (Yildirim, 2007). Using polymer modifiers is the most effective strategy for preventing excessive plastic deformations at high temperatures (Airey, 2002). The aggregate surface is more attracted to water than binder due to its surface energy characteristics (Little & Jones, 2003). Therefore, anti-stripping agents are added to the asphalt binder to combat moisture damage. With the inclusion of fillers with the binder, the cohesion between components forms mastic, where fillers influence the asphalt mixture by increasing the stiffness and changing the moisture resistance performance, workability, and compaction characteristics of asphalt mixtures (Rieksts et al., 2019); Huang et al.,2007). Mineral filler, coarse, and fine aggregates are mixed in predetermined weight proportions defined by the mix design process with the asphalt binder to create a hot mix asphalt (HMA) mixture. A component of the asphalt mixture is the mastic that deforms when stress is applied (Taylor & Airey, 2008). Asphalt mastic is a combination of asphalt binder and specific ratios of mineral filler used to manage its mixture's mechanical behavior (Roman et al., 2016; Yi-qiu et al., 2010). Most of this mineral aggregate passes a 0.075mm sieve (Kuity et al., 2014). The hot mix asphalt (HMA) mixture may be thought of as a system where the aggregates are covered with mastic rather than the asphalt binder (Wang et al., 2011). Mastic testing and research on the optimum filler-binder combination are subjects that are gradually garnering attention in this area and have demonstrated more potential than traditional binder testing (Moraes & Bahia. 2015).

A filler's function in an asphalt mixture can be divided into the following separate actions: 1) functioning as an inert filler material (Limestone, dolomite, basalt, etc.) to fill spaces between coarse aggregates, and 2) acting as an active filler material (Hydrated Lime, fly-ash, diatomite) when it comes into contact with binder at the interface (Kim & Little. 2004). Very few literatures describe the combined effect of active and inert filler in modified asphalt mastic containing a liquid anti-stripping agent. The performance of the combination of active and inert filler in asphalt mastic and mixture will be evaluated in this study. When the proportion of active and inert filler is used above a specific limit, thermal cracking may develop. This is because the filler particles are cementing the binder too strongly. Thus, effective rutting resistance can only be achieved by using the proper quantity of filler (Antunes et at., 2014). This investigation aims to understand the function of adding a different proportion of active and inert filler to prepare the mastic and compare their high-temperature rutting and recovery performance. The optimized proportion of active and inert filler will be used to prepare the mixture. For high-temperature performance, it is often advised that the F/B ratio not exceed 1.4 (Zhang et al., 2004). The ideal filler content for modified mastic is between 0.8 and 1.2. (Qiu, 2013). A F/B ratio of 0.8 is used in this study to prepare all the mastics.

In this study, HL and FA are used as active fillers. HL is used, which enhances the ductility of asphalt mastic, lowers aging and boosts rutting and moisture resistance (Bai et al., 2014). Several research efforts have looked at the positive impact of FA on the asphalt mix's ability to resist moisture and rutting and maintain tensile strength (Asi & Assaad. 2004; Xioa et al., 2012). Due to the considerable rise in mastic consistency, HL and FA fillers must be introduced in small quantities. The performance of an asphalt mixture may be decreased by using too much HL (Zou & Sun, 2019). Thus, the proportion of active filler was chosen to be 10% - 30% by the weight of the base binder to prepare the mastics. LS filler was selected for this study because it is a broadly used filler. Along with having a strong stiffening capacity, LS filler also helps the polymer phase function as effectively as possible (Rieksts et al., 2019). In a previous study 75% Ba (Basolt) with 5% HL showed better low-temperature cracking performance (Das & Shing, 2017), so, Ba was also selected for this study. DM is available locally and has almost a similar chemical composition as LS. The proportion of inert filler was chosen 50% - 70% by the weight of the base binder to prepare the mastics. Various asphalt mineral filler mastics revealed nonlinear rheological behavior at high temperatures (Taylor & Airey, 2008). On the other hand, unmodified mastics show a low %

recovery, whereas polymer-modified mastics offer a high significant recovery. High-temperature recovery of modified asphalt mastic is yet to be evaluated. SBS is used to modify the binder.

Some studies have been conducted to evaluate the rheological characteristics of asphalt mastic. Dynamic shear rheometer (DSR) was used in these studies following AASHTO T 315 test method. Two important rheological parameters like complex modulus (G*) and phase angle (δ) of asphalt binder and mastic can be measured from this test and under linear viscoelastic circumstances, master curves may be constructed (Underwood & Kim, 2015*)*. The superpave rutting parameter (G/sinδ), has also been used to measure the flow characteristics of asphalt mastic. But this test method cannot measure mechanical and viscoelastic characteristics of polymer-modified binders and mastics beyond their linear viscoelastic ranges. Also, the Superpave parameter can not measure the energy dissipation of most polymer-modified binder (PMBs) due to delayed elasticity, so a non-reversible cycle loading is suggested ((Delgadillo et al., 2006). Direct measurements of the damage resistance of mastic may be obtained using the MSCR test. Therefore, it is advised that the MSCR test rather than the elastic recovery test be used to assess the recovery property of binders and mastics (Clopotel & Bahia, 2012). Typically, rheological parameters like creep recovery performance of viscous materials are measured and evaluated using dynamic shear rheometers (DSRs) by applying a shear force to specimens following AASHTO T 315 test method. DSR can work in two modes – controlled stress or controlled strain (Hafeez et al., 2014, Shenoy, 2008). The multiple stress creep recovery test (MSCRT) method is used during this study, applying a constant stress condition to the sample. To capture the influence of modifiers, MSCR parameters are more efficient than other parameters (Ahmed et al., 2021). The conventional MSCR test technique involves loading a sample for one second at constant creep stress and then allowing it to recover for nine seconds at zero stress. The test is performed at two different levels of stress: 0.1 kPa and 3.2 kPa at 64°C (Hossain, 2016). The non-recoverable creep compliance (J_{nr}) and percent recovery (%R) values are computed from the test results at both stress levels, as stated in the applicable AASHTO and ASTM standards (AASHTO TP 70, 2009; ASTM D 7405, 2010). Instead of using the rutting parameter (G*/sinδ), these two findings from the MSCR test can be utilized to assess rutting potential (Smucker, 2019). The %R is used to construct the polymer modification curve to interpret the elastomeric performance of the aged asphalt mastic and ensure whether the samples are modified with an acceptable range with elastomeric polymer (ASTM D 7405, 2010).

The Marshall mix design is the most frequent technique employed for asphalt mix design (Asphalt Institute, MS-2, 2014; Stephen B, 2015). Even though the Marshall technique is empirical in nature, it might be beneficial in specific situations to compare mixes (Diab & Enieb, 2018). The Marshall mix design includes features like Marshall stability (MS) and Marshall flow (MF). Another critical parameter that can be calculated from the Marshall test is the Marshall quotient (MQ), a well-known indicator of a material's resistance to shear loads, permanent deformation, and therefore rutting (Zoorob et al., 2000). To determine the tensile characteristics of the asphalt mixtures, which are also connected to the pavement's cracking behavior, the indirect tensile strength test (ITS) is utilized (Islam et al., 2015). Moreover, moisture damage is a critical problem affecting asphalt pavements' durability and must be checked during the mix design (Ekblad et al., 2015). The Tensile strength ratio (TSR) is the most important factor to consider when assessing the moisture damage to asphalt mixes which is the ratio of ITS in wet conditions and ITS in dry conditions. However, occasionally it lacks credibility because it is only a ratio of two numbers (Diab, 2016). Therefore, besides TSR, another parameter, Retained Marshall stability (RMS)

(Defense works functional standards,2005), will be used to explain the mixes' vulnerability to moisture.

4.3 Objectives

The major objectives of this experimental study include:

- Compare the creep recovery performance of different active, inert, and a combination of active and inert fillers in aged asphalt mastic containing modifier and liquid anti-stripping agent based on non-recoverable creep compliance, percent recovery, stress sensitivity, and polymer modification curve.
- Understand the effect of different proportions of active and inert filler in mastic scales by comparing their rheological performance at high temperature and propose an optimum dose.
- Gain a basic understanding of the impacts of the combination of active and inert fillers in asphalt mixture and compare the mechanical properties of the mixtures.
- Conduct ANOVA analysis at the mixture level to evaluate the effect of the active and inert filler combination and compare the performance with the mixtures containing only active filler.

4.4 Materials and Methodology

4.4.1 Materials

4.4.1.1 Asphalt Binder

Rutting is a common distress that may be mitigated by carefully choosing the binder. Performance Grade PG 58-28 binder is employed in several regions of Newfoundland and Labrador, Canada, particularly in the southern region. Binders are rated according to their performance in lowest and highest temperatures (Performance Grade, or PG). Making sure the binder has the appropriate characteristics for the unique climatic conditions of the field is the main objective of using the PG. The PG binder was selected to adapt to shifts in traffic flow and speed as well as predicted climatic conditions. The control binder in this investigation was PG 58-28.

4.4.1.2 Modifiers and Anti-stripping Agent

Mastics perform much better in terms of stiffness when polymer is added (Rieksts et al., 2019). SBS (4% by the weight of base binder) was used in this study as shown in **Figure 4.1**. SBS was obtained from local sources. Stripping between the binder and the aggregate bond can fasten pavement distresses. Xiao et al. demonstrated that applying anti-stripping agents may reduce asphalt pavement stripping (Xiao et al.,2010). Adding Zycotherm to the binder enhances the binding between the binder and aggregate. Zycotherm makes the aggregate surfaces more hydrophobic (Mirzaiyan, et al.,2019), eventually improving the contact between the aggregate and binder, thus increasing the performance. Liquid anti-stripping agent Zycotherm shown in **Figure 4.1** was added with the modified asphalt to evaluate the creep recovery performance of liquid anti-stripping agents. 0.1% Zycotherm with 4% SBS showed good rutting performance (Islam et al., 2022). So, 4% SBS and 0.1% Zycotherm have been chosen in this study.

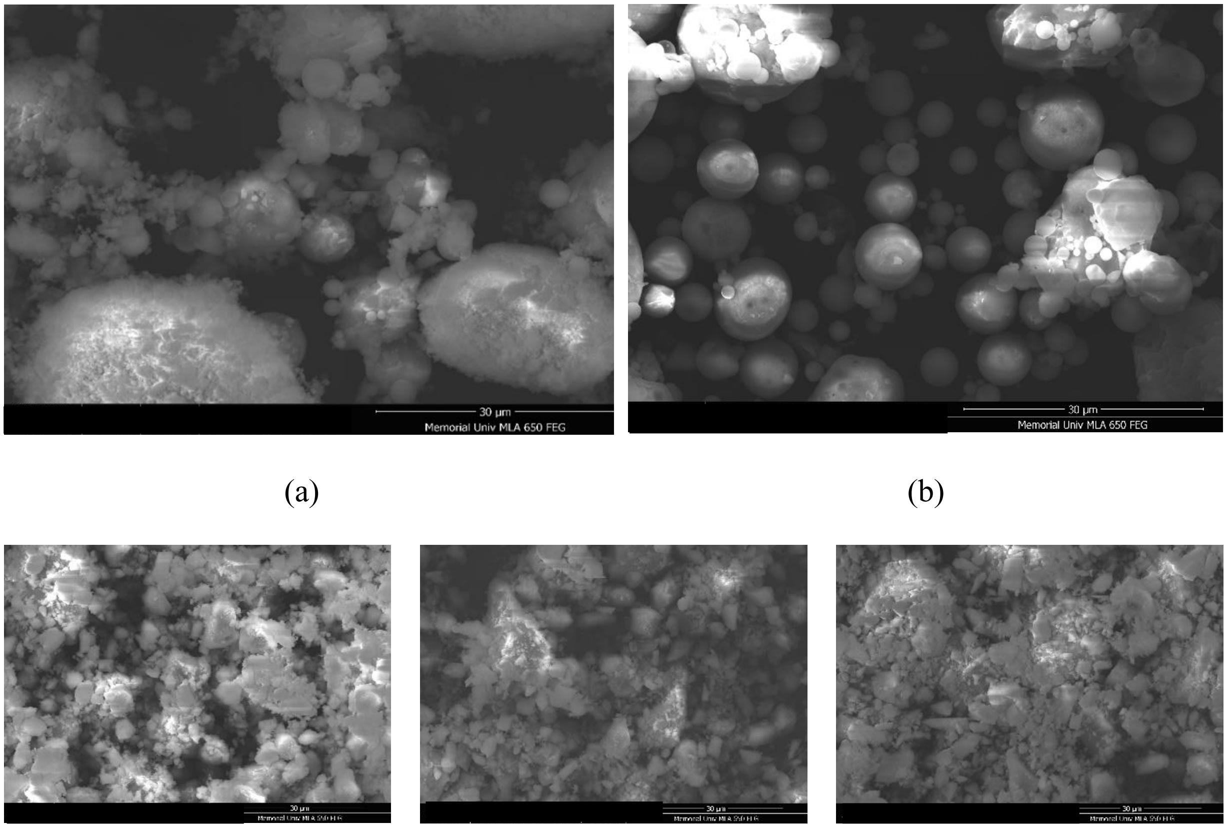
(a) (b)

Figure 4.1 Binder Modifier (a) SBS and b) Anti-stripping agent Zycotherm

(a) (b)

(c) (d) (e)

Figure 4.2 active fillers: a) HL, b) FA, and inert fillers: c) LS d) Ba e) DM



(a) (b)

(c) (d) (e)

Figure 4.3 SEM images of active fillers: a) HL, b) FA, and inert fillers c) LS d) DM e) Ba

4.4.1.3 Fillers and Aggregates

HL and FA were obtained in powder form, passing sieve No. 200. Conversely, LS, Ba, and DM were obtained from local quarries. Initially, the average size of the collected fillers was between 2-10 mm. To grind the materials, planetary ball mill equipment was used. After grinding the materials, the fine particles of LS, Ba, and DM were sieved with sieve No. 200. The fillers passing sieve No. 200 were collected to use as a filler. The fillers are presented in **Figure 4.2**. While preparing, the mastic F/B ratios of 0.8 (80% by the weight of the base binder) were used. To evaluate the creep recovery performance of the asphalt mastic and the mechanical performance of the asphalt mixture, active and inert fillers were mixed so that the F/B ratios remained 0.8. For fabricating the mastics, different proportions (10%,20%,30% by the weight of base binder) of active fillers and different proportions (70%,60%,50% by the weight of base binder) of inert filler materials were mixed, i.e., HL0.1+LS0.7, HL0.2+LS0.6, HL0.3+LS0.5, etc. Some mastics were prepared without mixing any active and inert fillers (80% by the weight of the base binder) to compare the performance of the combination of active and inert fillers, i.e., HL0.8, FA0.8, etc.

As part of this investigation, the physical and chemical characteristics of the fillers were examined to better understand their properties. Specific gravity tests with the pycnometer method, brunauer– emmett–teller (BET) tests for specific surface area (SSA), scanning electron microscopy (SEM) imaging, and X-ray fluorescence spectrometry were used for the characterization.

According to SSA and SG tests, active filler HL has a lower density while Fa has a higher density (**Table 4.1**). The SSA of the HL filler was 1.4 times greater than the FA filler. Dolomite filler has the highest density of all the active and inert fillers. Ba has a higher SSA compared to other inert fillers. **Table 4.1** shows XRF oxide profiles for chemical analysis. For HL, LS, and DM dominating oxide is CaO, while FA and Ba dominating oxide are SiO_2.

Figure 4.3 displays SEM and physical photographs of all the fillers. In the case of HL, most particles are coarser, irregular, and have a porous structure, whereas most FA particles are rounded. For LS filler, most particles have a small grain size while particles of Ba and DM tend to agglomerate. Usually, the particle of Ba is flaky but due to the use of plenary ball mill equipment for grinding, the particle size cannot be properly defined.

The course and fine aggregates were obtained from nearby quarries and has already been used to several local pavement projects in Canada. In order to create a Stone Matrix Asphalt (SMA), crushed stone with a maximum size of 19 mm was used. The resistance of SMA mixes against rutting is very strong. The distinct gradation and particular strength of the coarse aggregate increase this mixture's resistance to rutting (National Asphalt Pavement Association, 2002; Ahmadinia et al., 2011). Moreover, the use of SMA mixes lowers the cost of maintaining pavement and lowers noise pollution. The gap graded structure of this combination accounts for the high binder concentration. Thus, it is best to employ the modifiers to restrict the drain down of the binder (Brown et al., 1997). **Figure 4.4** depicts the gradation of the aggregate mixtures.

Table 4.1 Oxide composition and physical properties of fillers

	Oxide Composition							Physical Properties	
Fillers	MgO	Al_2O_3	SiO_2	CaO	Fe_2O_3	K_2O	Others	SSA (m^2/g)	SG
HL	6.38	0.19	7.42	84.43	0.18	0.67	0.73	10.95	2.23
FA	1.72	26.95	50.69	11.57	5.23	2.77	1.07	7.93	2.35
LS	2.32	1.09	1.75	91.19	1.82	0.97	0.86	4.02	2.74
Ba	8.31	13.61	48.57	12.78	14.76	1.21	0.76	9.31	2.75
DM	14.89	1.78	2.12	80.52	0.11	0.07	0.51	3.89	2.83

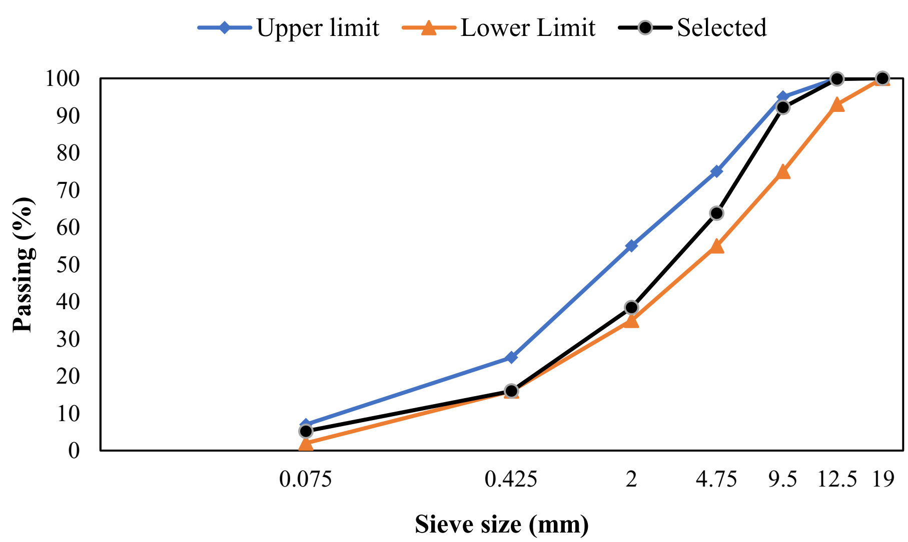


Figure 4.4 Aggregate gradation curve for HMA sample preparation

4.4.2 Methodology

4.4.2.1 Sample Preparation and Testing of Binders Containing Modifiers and Liquid Anti-Stripping Agents

To modify the binder, the neat PG 58-28 binder was preheated at 160°C. The SBS modified binder (4% by weight of base binder) was collected from a local pavement company, whereas the Gilsonite modified binder (10% by weight of base binder) was prepared by blending it with the base binder at 160°C for 1 hour to ensure homogeneity without any agglomeration. The liquid anti-stripping agents (i.e., Zycotherm and AD-here) were added separately to the modified binders. The modified PG 58-28 binder with additives was prepared by stirring with a magnetic stirrer for 45 minutes at 160°C, after several trials to determine the optimal mixing time and temperature.

4.4.2.2 Preparation of Asphalt Mastic

Prior to blending the fillers with the modified binder, each filler was cleaned and dried for 24 hours at 105°C in the oven. To ensure the homogeneity of the mixture of binder with fillers, the mixing conditions were adjusted for different F/B ratios. To achieve this, each type of filler with different F/B ratios was gradually included into the heated binder, mixing at 160, 170, and 180°C for 60, 120, and 180 minutes, respectively using a magnetic stirrer. The mixing temperature and time was adjusted to avoid the filler sedimentation.

4.4.2.3 Aging, Testing and Statistical Analysis

All the binders were heated at 160°C until they became pourable to prepare the sample. Finally, to simulate short-term laboratory aging of the base binders and modified mastics, The Rolling Thin-Film Oven (RTFO) conditioning in accordance with AASHTO T 240 was employed. To prepare the aged mastics, continuous oxidative conditioning was applied at 163°C to the binders for 85 minutes. Finally, the MSCR test protocol was employed using the DSR equipment. A 25mm plate was used to prepare the mastics. In this investigation, 23 asphalt mastics with varying F/B ratios were prepared. All the mastics, including the control binder, were tested twice to ensure the reliability of the data. The experimental plan of the mastic level study is illustrated in **Figure 4.5.**

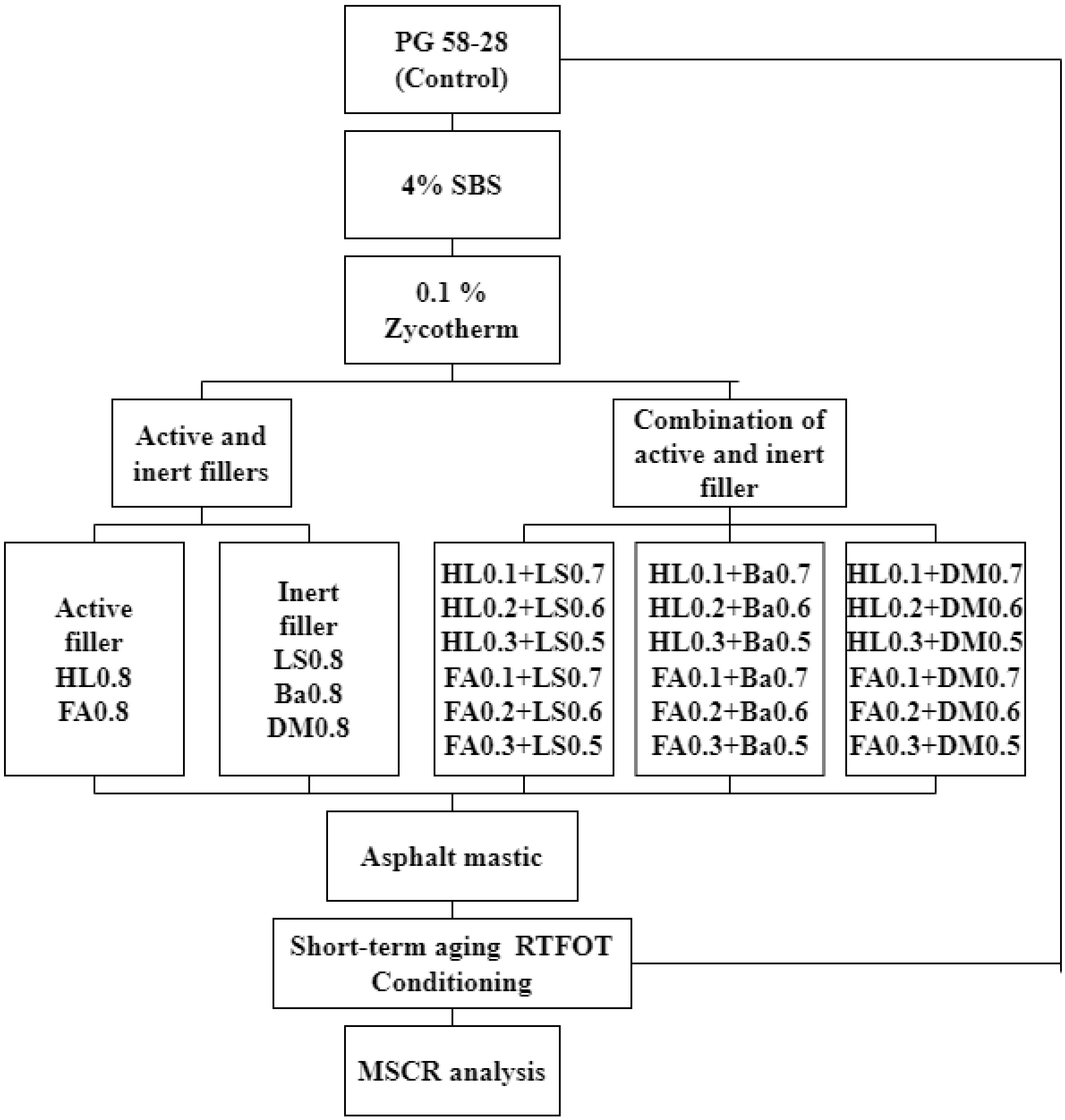


Figure 4.5 Experiment design matrix for mastic

4.4.2.4 Preparation of HMA Mixture

The HMA mixture was developed using the Marshall method accommodating several modifiers, anti-stripping agents, and filler types. For fillers, the F/B ratios were chosen based on the performance at the mastic level. Marshall samples were prepared in accordance with ASTM D 6926-04. The aggregates used to make HMA were dried out by heating them to 105°C for an entire night. The dry coarse aggregate and fine aggregate were weighed and then cooked in a pan at 180°C for 10 minutes before being combined with the mastic, which had been heated to 130°C. Cylindrical specimens, 101.6 mm in diameter and around 63.5 mm in height are prepared for the Marshall design technique by being subjected to 75 blows on each side with a standard hammer. After compacting the specimens at a temperature of 145°C, the materials were combined for 5 minutes at a temperature of 155°C (ASTM D 6926-04). After determining the various volumetric parameters, all the samples were put through tests for Marshall stability (MS) and flow values (ASTM D 6926-04). **Table 4.2** shows the various modifiers, anti-stripping agents, and fillers used in the Marshall Samples and the percentages of binder content. Two mixtures were prepared without including inert fillers, and six different mixtures were prepared with the combination of active and inert fillers with binder content varying between 5-7%.

Table 4.2: Details of active and inert filler type and binder content to prepare the HMA mixture.

Active Filler	**HL**				**FA**			
Inert Filler	N/A	LS	Ba	DM	N/A	LS	Ba	DM
Binder Content*	5.0 5.5	5.0 5.5	5.0 5.5	5.0 5.5	5.0 5.5	5.0 5.5	5.0 5.5	5.0 5.5

6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5
7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0

*Binder content by total mixing weight

4.4.3 Method of Analysis for Asphalt Mastic

Same as section **3.4.3 Method of Analysis for Asphalt Mastic** described in Chapter 3.

4.4.4 ANOVA Analysis

Same as **section 3.4.4 ANOVA Analysis** described in Chapter 3.

4.4.5 Method of Analysis for Asphalt Mixture

Same as **section 3.4.5 Method of Analysis for Asphalt Mixture** described in Chapter 3

4.5 Result and Discussion

4.5.1 Non-Recoverable Creep Compliance, J_{nr}

4.5.1.1 Analysis of Jnr of Asphalt Mastics Contain HL and other Inert Fillers

From **Figure 4.6**, all the mastics contained different filler or combination of active and inert filler had a lower J_{nr} value compared to the neat, aged binder. J_{nr} value for neat aged PG 58-28 is 2.15 kPa^{-1}. All the mastics had a J_{nr} value less than 0.14 kPa^{-1}. From the binder level analysis, without the inclusion of any filler J_{nr} value of SBS-modified 0.1% Zycotherm was found to be 0.2 kPa^{-1} (Feroz et al.,2022). Inclusion of filler thus lower the J_{nr} value and improve the rutting performance. Combination of active filler HL and inert filler LS had a lower value of J_{nr} compared to other mastics. This observation could be explained by the combined well graded particle size distribution of HL and LS. Mastic prepared with 10% (by the weight of base binder) HL and 70 % (by the weight of base binder) LS had the lowest value of J_{nr} and thus showed better resistance to permanent deformation. With the increase in HL and decrees of LS, the J_{nr} value was increasing. This might be due to the higher absorption capacity of HL. The same pattern could be seen for the HL with Ba and HL with DM.

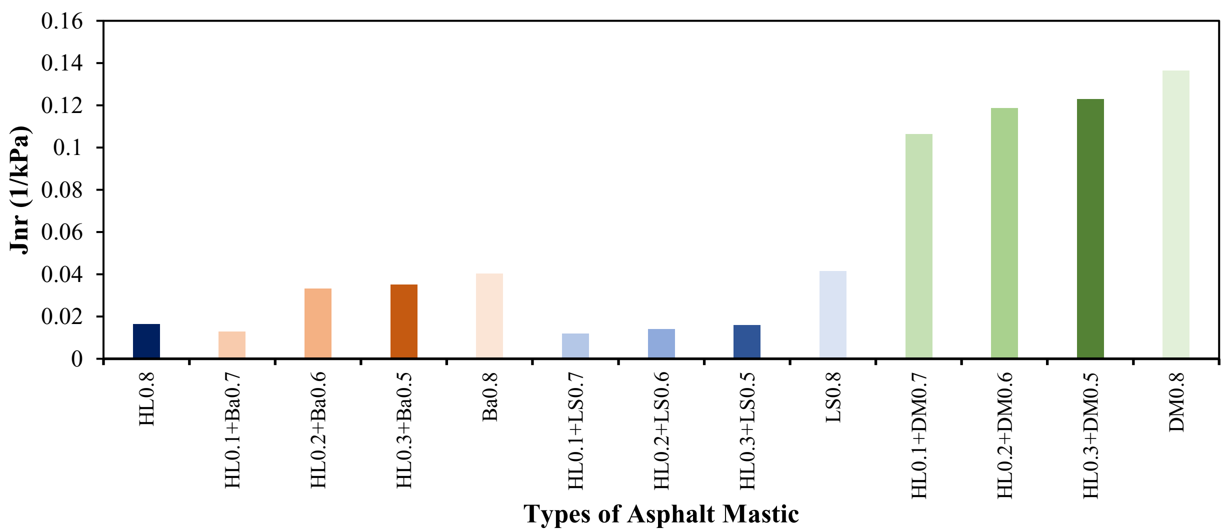


Figure 4.6 Comparison of J_{nr} at 3.2 kPa^{-1} of asphalt mastics contain HL and other inert fillers

10% (by the weight of base binder) HLand 70 % (by the weight of base binder) Ba had lower J_{nr} value than HL0.8 whereas other dosage of HL and Ba had a higher J_{nr} value then HL 0.8. All the mastics of HL+DM had higher J_{nr} value compared to other mastics. Thus, the combination of HL and DM showed poor rutting performance.

4.5.1.2 Analysis of Jnr of Asphalt Mastics Contain FA and other Inert Fillers

According to **Figure 4.7**, the J_{nr} values were ranging from 0.04-0.14 kPa^{-1} for FA asphalt mastic whereas the J_{nr} values for HL mastics ranged from 0.012-0.13 kPa^{-1}. So, as expected inclusion of fillers with binder had lower J_{nr} values then the neat aged binder which implied better rutting resistance. Most of the mastics prepared with the combination of active filler (FA) and other inert filler (LS, Ba, DM) had a J_{nr} value lower than the mastic prepared with only inert filler LS and Ba. Mastic prepared with 30% (by the weight of base binder) FA and 50 % (by the weight of base binder) LS had the lowest value of J_{nr} compared to other mastics. With the increase in FA and decrees of LS, the J_{nr} value was decreasing. The same pattern could be seen for mastics with FA+Ba and FA+DM. This was due to the addition of FA improved the rutting performance of mastics (Asi & Assaad*,* 2005*)*.

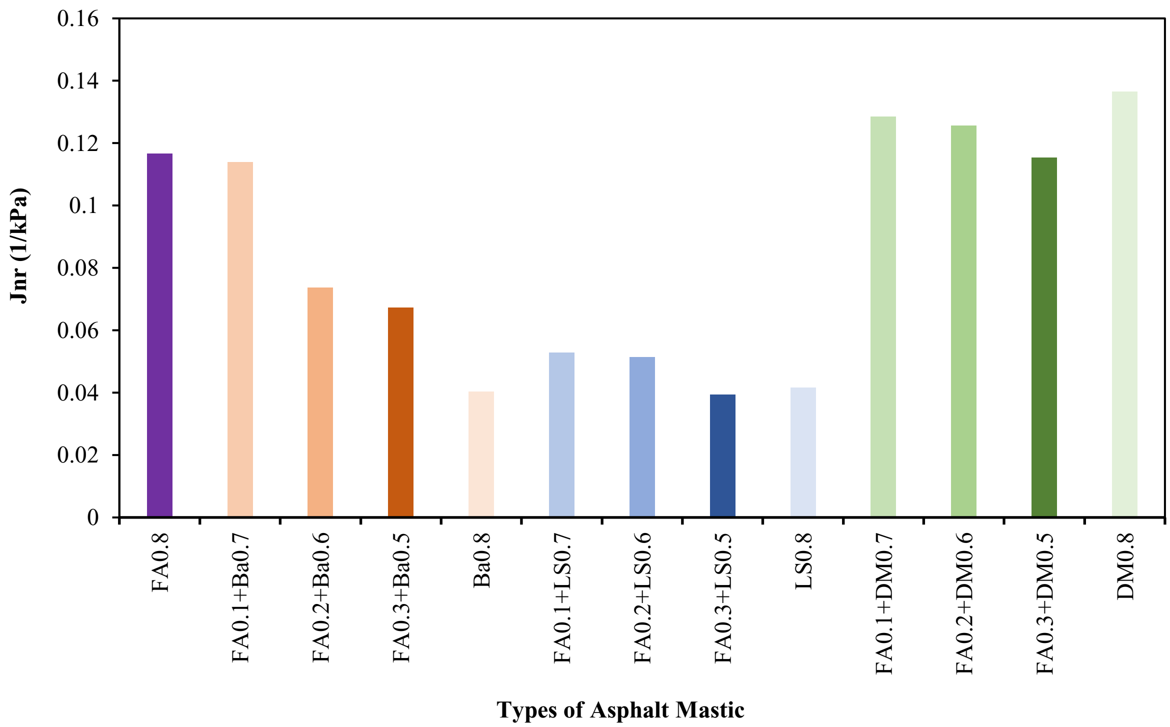


Figure 4.7 Comparison of J_{nr} at 3.2 kPa^{-1} of asphalt mastics contain HL and other inert fillers

All the mastics met the extremely heavy traffic criteria based on AASHTO M 332 specification. Overall, SBS and 0.1% Zycotherm modified mastics prepared with active filler (HL, FA) and inert filler LS had a lower range of J_{nr} value which implied better rutting performance.

4.5.2 Stress Sensitivity

4.5.2.1 Analysis of Stress Sensitivity of Asphalt Mastics Contain HL and other Inert Fillers

Figure 4.8 compares the stress sensitivity of all the asphalt mastic with different proportion of HL and other inert filler. All the mastics were more stress sensitive than the neat aged binder according to the previous method of stress sensitivity. All the HL+Ba and HL+LS mastics passed the stress sensitivity criteria. However, all the HL+DM failed the stress-sensitive criteria, which suggested that high temperatures or heavy loads excessively stress these mastics. Some mastics contain only active (HL) or inert filler (Ba) passed the stress sensitivity criteria. Combination of 10% HL and 70 % LS had the lowest stress sensitivity compared to the other mastics.

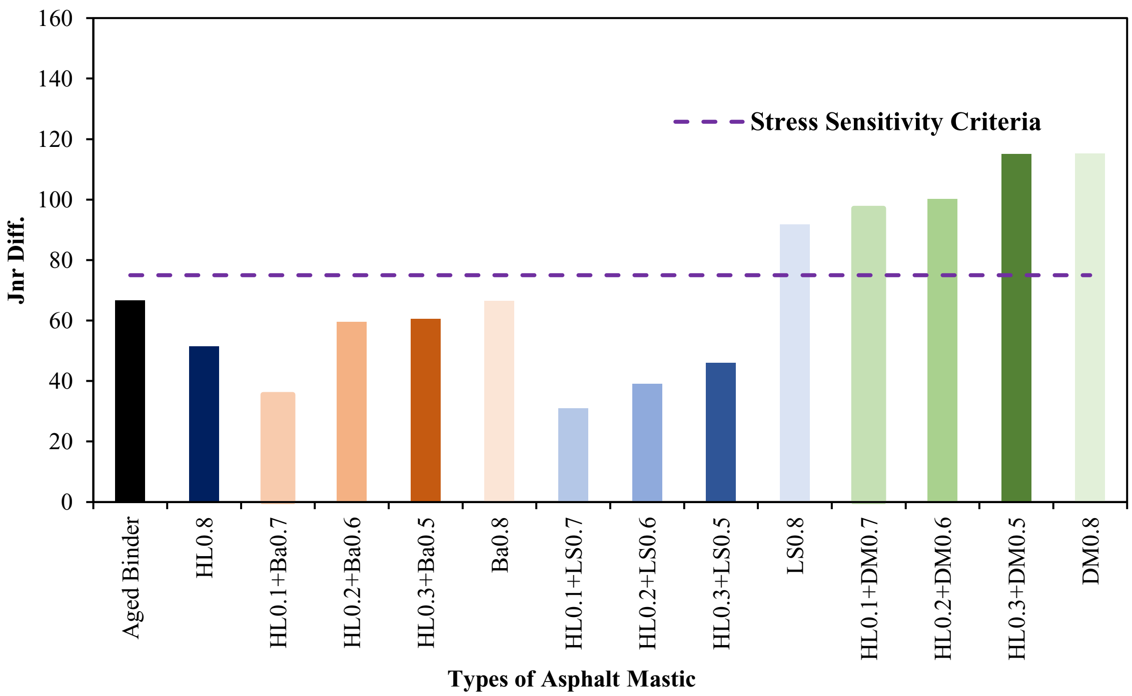


Figure 4.8 Comparison of stress sensitivity of asphalt mastics contain HL and other inert fillers

4.5.2.2 Analysis of Stress Sensitivity of Asphalt Mastics Contain FA and other Inert Fillers

Figure 4.9 compares the stress sensitivity of all the asphalt mastic with different proportion of FA and other inert filler. FA mastics were more stress sensitive than HL mastics. This could be explained by the higher J_{nr} value of FA mastics compared to HL mastics. All the FA+Ba mastics failed the stress sensitivity criteria whereas all HL+Ba passed the stress sensitivity criteria. all the FA+DM, revealing that high temperatures or large loads severely stress these binders since they failed the stress sensitivity criteria. The stress sensitivity criteria were met by all other mastics containing FA+LS. Combination of 30% FA and 50 % LS had the lowest stress sensitivity compared to the other mastics.

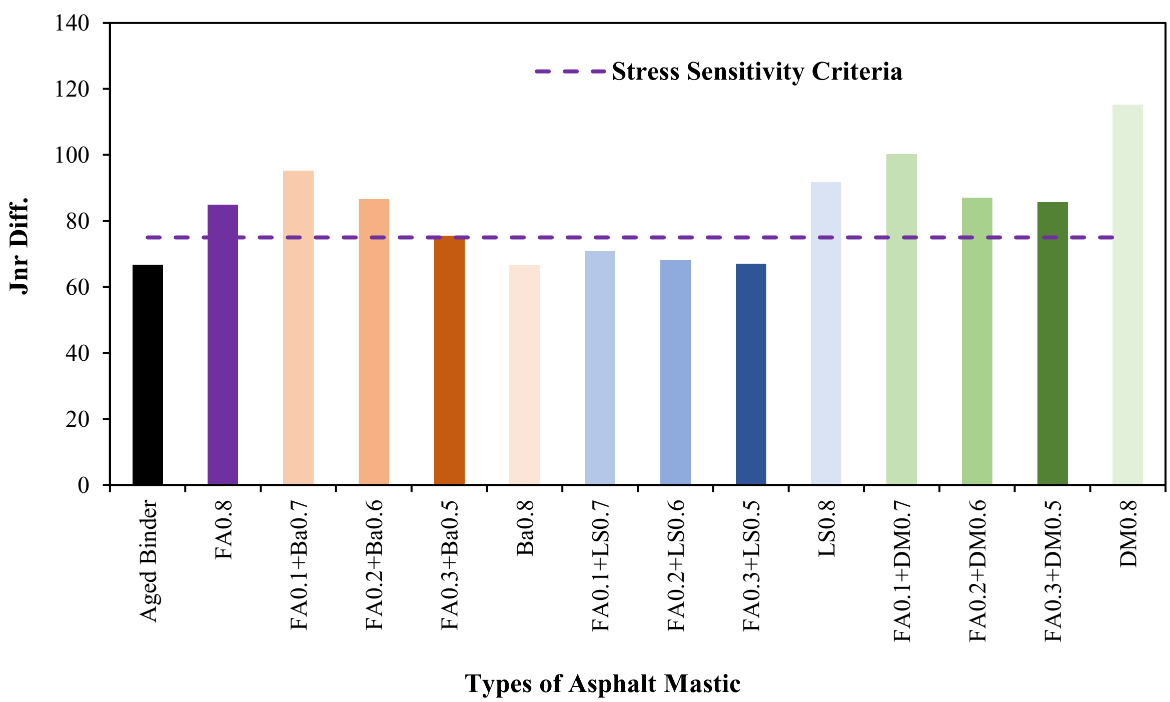


Figure 4.9 Comparison of stress sensitivity of asphalt mastics contain HL and other inert fillers

4.5.3 Modified Stress Sensitivity

4.5.3.1 Analysis of Modified Stress Sensitivity of Asphalt Mastics Contain HL and other Inert Fillers

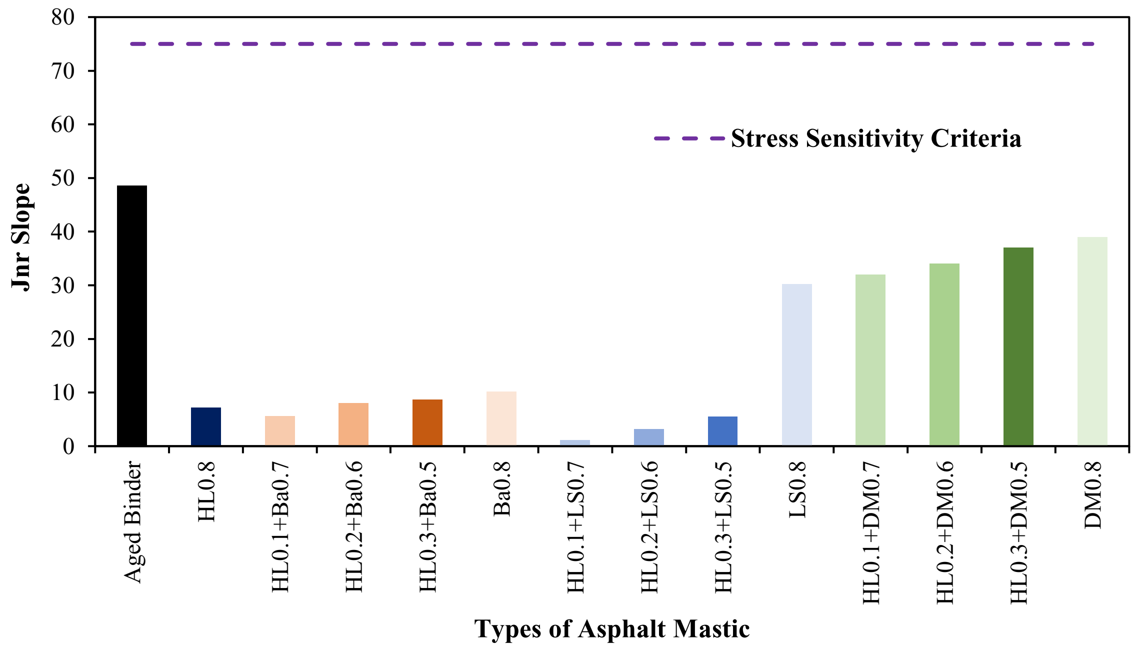


Figure 4.10 Comparison of modified stress sensitivity of asphalt mastics contain HL and other inert fillers

The comparison of the modified stress sensitivity asphalt mastic with varying proportions of HL other inert filler is shown in **Figure 4.10**. All the mastics passed the modified stress sensitivity criteria, whereas LS0.8, DM0.8 and all the HL+DM failed to pass the previous stress sensitivity criteria. The newly proposed stress sensitivity technique indicated that the mastics, had a lower stress sensitivity than the neat aged binder. All HL mastics containing Zycotherm were less stress sensitive than other mastics. Like the previous method, combination of 10% HL and 70 %) LS had the lowest stress sensitivity compared to the other mastics.

4.5.3.2 Analysis of Modified Stress Sensitivity of Asphalt Mastics Contain FA and other Inert Fillers

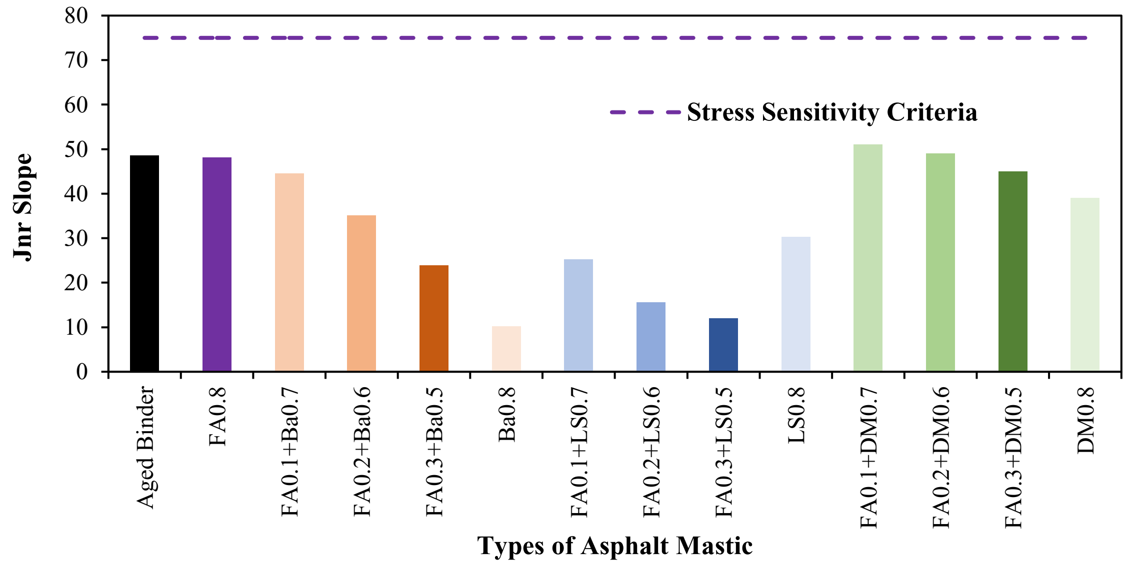


Figure 4.11 Comparison of modified stress sensitivity of asphalt mastics contain FA and other inert fillers

Figure 4.11 illustrates a comparison of the asphalt mastic with different proportion of FA and other inert filler. All the mastics passed the modified stress sensitivity criteria, whereas most mastics failed to pass the previous stress sensitivity criteria. All mastics had lower stress sensitivity than the aged binder, except 10% FA+ 70% DM and 20% FA+ 60% DM.

4.5.4 Polymer Method and MSCR Grade

4.5.4.1 Analysis of Polymer Method of Asphalt Mastics Contain HL and other Inert Fillers

Polymer modification curves of asphalt mastic with different proportion of HL and other inert filler are shown in **Figure 4.12**.

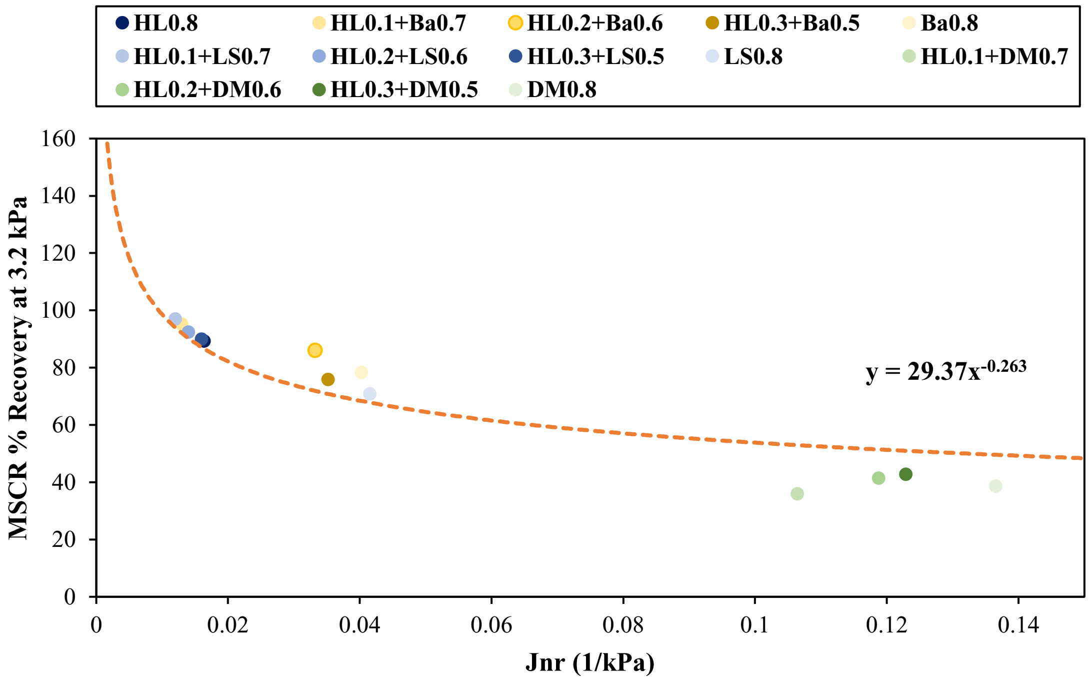


Figure 4.12 Polymer modification curve of asphalt mastic contain HL and other inert fillers

Addition of fillers enhanced the elastic behavior of the mastics. Most of the asphalt mastics (9 out of 13) passed the polymer modification criteria and clustered above the line. According to asphalt institute guidelines, the modification was done within an acceptable range for these mastics, and these mastics showed an excellent recovery performance. All the HL+LS and HL+Ba showed an excellent recovery. All the HL+DM failed to pass the polymer modification criteria and clustered under the line and showed poor recovery performance. This implied that the modification was not done within an acceptable range. Combination of 10% HL and 70 % LS had the highest %R compared to the other mastics, thus excelling in elastic recovery. All the mastics could be graded according to MSCR grading as the J_{nr} value was less than 4.5 kPa^{-1}.

4.5.4.2 Analysis of Polymer method of Asphalt Mastics Contain FA and other Inert Fillers

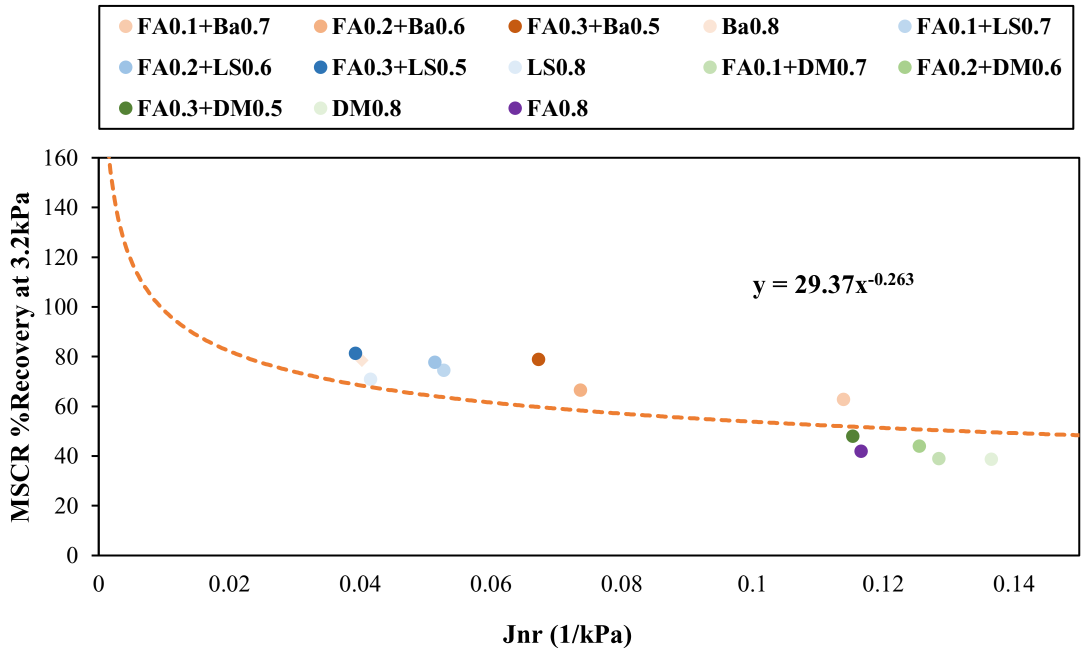


Figure 4.13 Polymer modification curve of asphalt mastics contain FA and other inert fillers

Figure 4.13 displays the polymer modification curves for asphalt mastic with different proportion of FA and other inert filler. All the proportions of FA+LS and FA+BA passed the polymer modification criterion and clustered over the line. Therefore, the modification was carried out within a range suitable for these mastics, and they exhibited outstanding recovery capabilities. However, all the FA+DM congregated beneath the line and performed poorly after recovery. This suggested that the modification was not made within an acceptable range. Given that the J_{nr} value was less than 4.5 kPa^{-1}, all the mastics of FA and other inert filler may be graded according to MSCR grading**.** The combination of 30% FA and 50 % LS had the highest %R compared to the other mastics**.** A summary of all the performance parameters considered for this study is given below in **(Table 4.3).**

Table 4.3 Summary of MSCR test parameters

No	Anti-stripping Agent	Active Filler	Inert Filler	Modifier	Meet Stress Sensitivity Criteria	Meet Modified Stress Sensitivity	%Recovery (Meets AASTHO T 350)	MSCR GRADE AASHTO M 332
1	Zycotherm 0.1%	HL0.8	N/A	SBS 4%	Yes	Yes	Yes	PG 58E-28
2	Zycotherm 0.1%	HL0.1	LS0.7	SBS 4%	Yes	Yes	Yes	PG 58E-28
3	Zycotherm 0.1%	HL0.2	LS0.6	SBS 4%	Yes	Yes	Yes	PG 58E-28
4	Zycotherm 0.1%	HL0.3	LS0.5	SBS 4%	Yes	Yes	Yes	PG 58E-28
5	Zycotherm 0.1%	N/A	LS0.8	SBS 4%	No	Yes	Yes	PG 58E-28
6	Zycotherm 0.1%	HL0.1	Ba0.7	SBS 4%	Yes	Yes	Yes	PG 58E-28
7	Zycotherm 0.1%	HL0.2	Ba0.6	SBS 4%	Yes	Yes	Yes	PG 58E-28
8	Zycotherm 0.1%	HL0.3	Ba0.5	SBS 4%	Yes	Yes	Yes	PG 58E-28
9	Zycotherm 0.1%	N/A	Ba0.8	SBS 4%	Yes	Yes	Yes	PG 58E-28
10	Zycotherm 0.1%	HL0.1	DM0.7	SBS 4%	No	Yes	No	PG 58E-28
11	Zycotherm 0.1%	HL0.2	DM0.6	SBS 4%	No	Yes	No	PG 58E-28
12	Zycotherm 0.1%	HL0.3	DM0.5	SBS 4%	No	Yes	No	PG 58E-28
13	Zycotherm 0.1%	N/A	DM0.8	SBS 4%	No	Yes	No	PG 58E-28
14	Zycotherm 0.1%	FA0.8	N/A	SBS 4%	No	Yes	No	PG 58E-28

15	Zycotherm 0.1%	FA0.1	LS0.7	SBS 4%	Yes	Yes	Yes	PG 58E-28
16	Zycotherm 0.1%	FA0.2	LS0.6	SBS 4%	Yes	Yes	Yes	PG 58E-28
17	Zycotherm 0.1%	FA0.3	LS0.5	SBS 4%	Yes	Yes	Yes	PG 58E-28
18	Zycotherm 0.1%	FA0.1	Ba0.7	SBS 4%	No	Yes	Yes	PG 58E-28
19	Zycotherm 0.1%	FA0.2	Ba0.6	SBS 4%	No	Yes	Yes	PG 58E-28
20	Zycotherm 0.1%	FA0.3	Ba0.5	SBS 4%	No	Yes	Yes	PG 58E-28
21	Zycotherm 0.1%	FA0.1	DM0.7	SBS 4%	No	Yes	No	PG 58E-28
22	Zycotherm 0.1%	FA0.2	DM0.6	SBS 4%	No	Yes	No	PG 58E-28
23	Zycotherm 0.1%	FA0.3	DM0.5	SBS 4%	No	Yes	No	PG 58E-28

4.5.5 Marshall properties of Asphalt Mixture

The OBC for each blend is shown in **Figure 4.14**. All the mixtures have an OBC that ranges from 5.9 to 6.5%. The gap-graded structure of these mixtures' accounts for their high OBC**.** HL had a higher OBC requirement for all the combinations than FA. HL's propensity to absorb binder because of its high SSA and porous structure may be responsible for the higher OBC requirement and eventually need a ticker film on the aggregates. This thickening of the binder film on aggregate reduces the amount of air voids in the mix (Arabani & Tahami, 2017). The addition of LS or Ba with HL reduced the OBC requirement. The combined well-graded particle size distribution of HL+LS and HL+Ba could explain this observation. Contrarily, FA had shown less OBC compared to the HL mixes, despite having a lower SSA. It is feasible to say that the FA, also known as binder extender (Sobolev et al., 2017), can partially replace asphalt binder, which decreases the OBC by reducing the air void of HMA. Adding inert fillers with FA required higher OBC than the mix with only active filler FA.

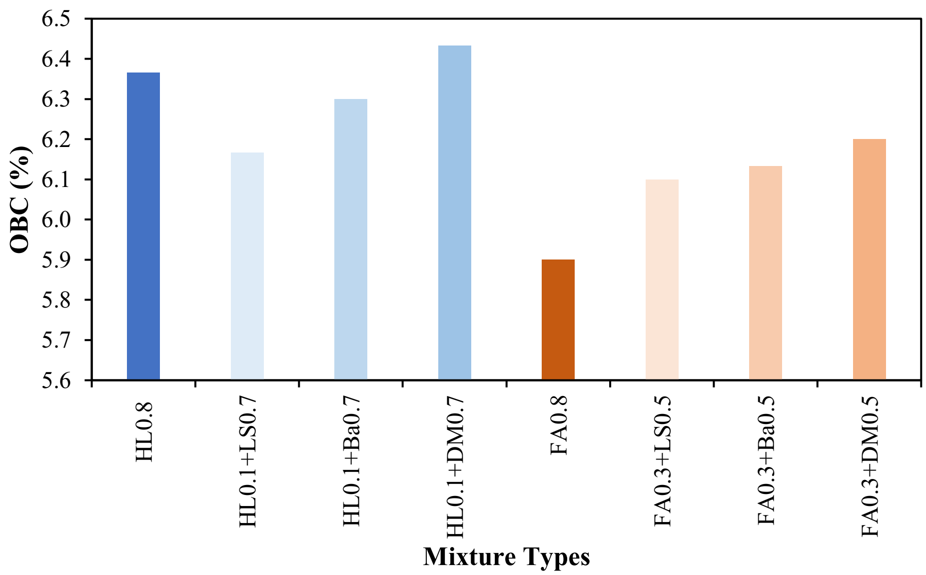


Figure 4.14 OBC of mixtures

Figure 4.15 shows the stability test outcomes at OBC for each mixture. The findings suggested that the asphalt mixture containing HL (13.7-11 kN) had a higher stability range than that containing FA (12.5-10.9 kN). The improved adhesion between HL and binder is to blame for the increase in stability. Combination of 10% HL and 70 % LS had the highest stability which is 6.2% more stable than mixtures prepared with only HL, HL+Ba had almost similar stability as HL where addition of DM with HL reduced the stability. The addition of LS with FA improved the stability by 4%. However, addition of Ba and DM lowered the stability. The stability values for all combinations could be observed to meet the Asphalt Institute guideline for the heavy traffic of not less than 8.01 kN.

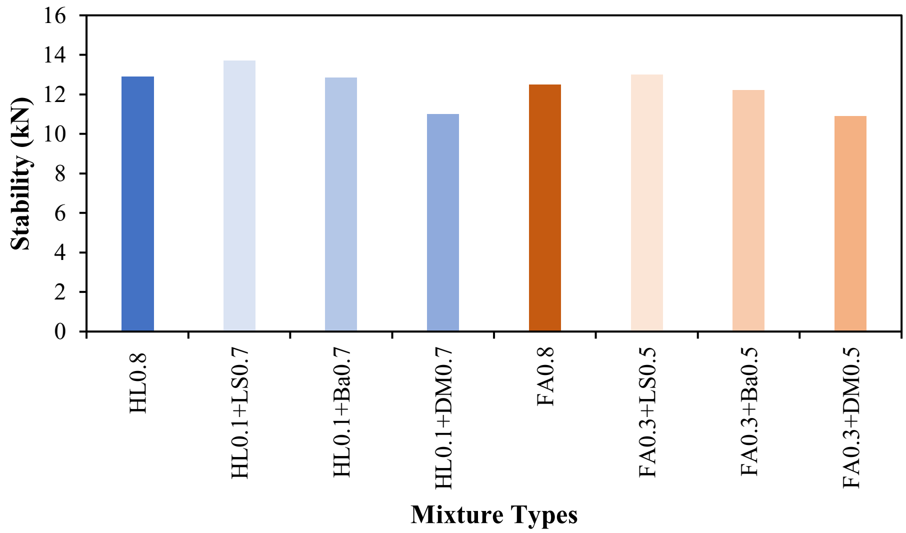


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Figure 4.15 Stability of mixtures at OBC

Figure 4.16 displays the impact of all the combinations, as mentioned earlier, on MQ values. The MQ values are 4.16 and 4.46 kN/mm for HL0.8 and FA0.8, respectively. FA has higher MQ values than HL for all active and inert filler combinations. The higher MQ values for FA may be attributed to the fact that the binder's viscosity and stiffness have improved due to decreased OBC of FA. This improves the HMA's capacity to bear stresses and strengthens the bond between mastic and aggregates (Hamedi & Tahami, 2017).

The MQ is a well-known indicator of a material's resistance to shear loads, persistent deformation, and therefore rutting (Zoorab et al.,2018). 10% HL and 70 % LS had higher MQ values than the HL0.8 mixture, which implies better rutting performance. Adding Ba and DM with HL didn't improve the MQ value. For the mixtures with FA, the addition of inert filler had a lower MQ value than the mixture prepared with only FA. All the mixtures had a value less than this limiting value of 5 kN/mm (Ministry of Road Transport, and Highways, 2013).

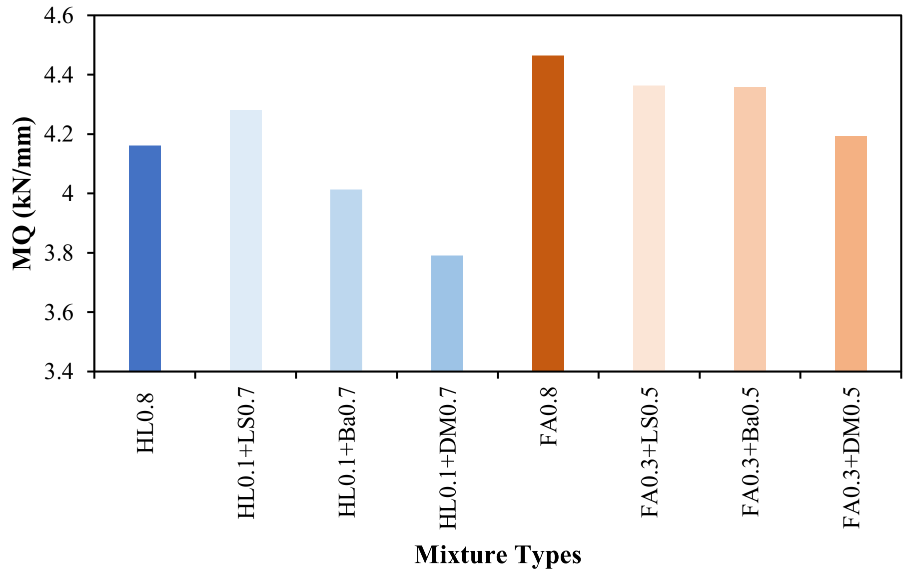

Figure 4.16 MQ values of mixtures at OBC

4.5.6 ITS and TSR of asphalt mixtures

The outcomes of the ITS of HMA with various active and inert fillers are shown in **Figure 4.17**. It is necessary to discuss both the dry and wet ITS to have a fair debate on how resistant the asphalt mixes are to moisture damage. Higher values of dry and wet ITS for all combinations could be seen for HL filler, which implied higher cracking resistance. This is due to the fact that HL filler works well as an adhesive for asphalt mixes. 10% HL and 70% LS with SBS and Zycotherm had the maximum ITS value of 1358 kPa, 4.62% more than the mixture with only HL. HL+Ba had a similar ITS to the HL mixture, whereas HL+DM had a lower ITS value compared to the HL mixture. However, the FA mixtures had higher stiffness but lower ITS value. HMAs with high stiffness were more likely to break easily and had lower tensile strength bearing capacity. 30% FA and 50% LS had higher ITS value than a mixture prepared with only HL or only FA.

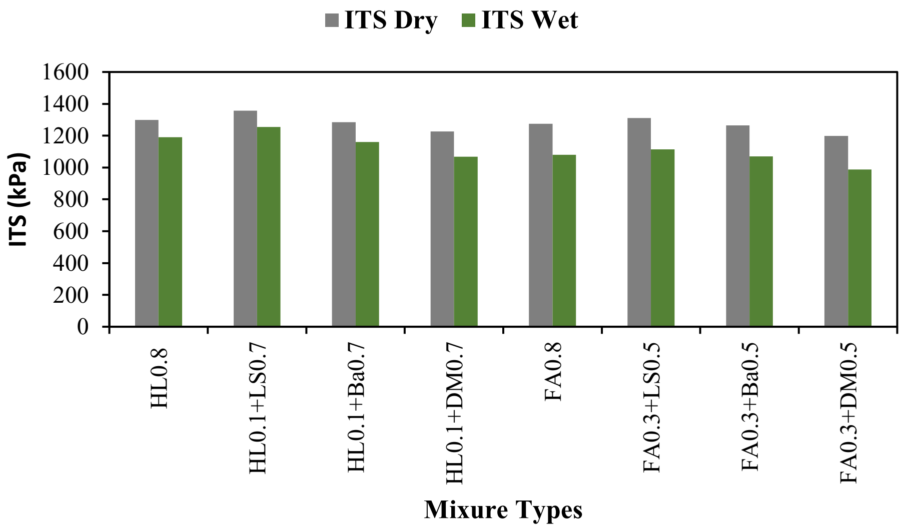

Figure 4.17 ITS values of mixtures at OBC

Moisture damage is a critical problem affecting asphalt pavements' durability and must be checked during the mix design (Ekblad et al., 2015). The standard method to check the moisture susceptibility of the asphalt mixes, including different mineral fillers, is evaluating the TSR value. The TSRs for the investigated mixes are shown in **Figure 4.18**. Compared to the FA-containing mixtures, the TSR values of the HL asphalt mixtures were greater. The HL significantly impacted the moisture resistance of the HMA mixture as a multifunctional filler, consistent with other research findings (Diab, 2016). Moreover, HL particles dispersed well in the HMA mixture due to its large SSA and porous structure, improving the adhesion between binder and aggregates. All the mixtures meet the minimum 80% TSR value required by standards. (AASHTO T 283). The mineral filler tends to fill the spaces within the mixture, increasing density and, as a result, perhaps reducing moisture ingress and, thus, reducing moisture damage. The highest TSR value can be seen for the HL and LS mixture combination. Nevertheless, compared to the combination made with only HL, the inclusion of Ba and DM with HL has a lower TSR value. In contrast, a mixture with solely FA has a lower RMS value than one with 30% FA and 50% LS.

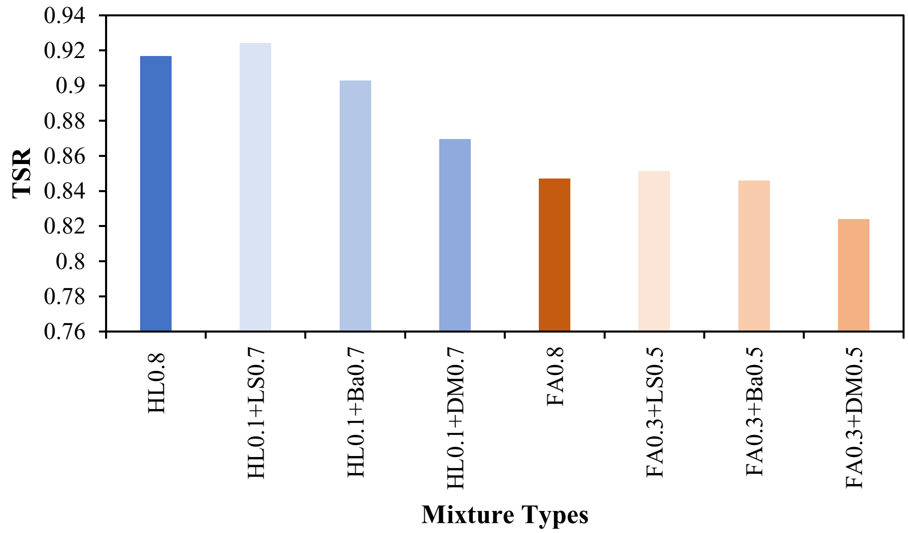

Figure 4.18 TSR values of mixtures at OBC

4.5.7 Retained Marshall Stability

The TSR is the most important factor to consider when assessing the moisture damage to asphalt mixes, but occasionally it needs to be more credible because it is only a ratio of two numbers. Also, it does not ensure that the mixture will function properly over the long term. In this study, the authors would instead present the mixtures' retained marshall stability (RMS). **Figure 4.19** shows RMS values for different mixtures of HL and FA. FA fillers contained mixture had lower RMS values than HL-contained mixtures, which explained the dominant performance of HL in resistance to the water. A similar performance could be seen when the TSR was considered. In the case of HL mixtures, 10% HL and 70 % LS had the highest RMS value and water effect resistance. However, adding Ba and DM with HL had a lower RMS value than the mixture prepared with only HL. Whereas, for FA mixtures, a combination of 30% FA and 50 % LS had a higher value of RMS than a mixture prepared with only FA

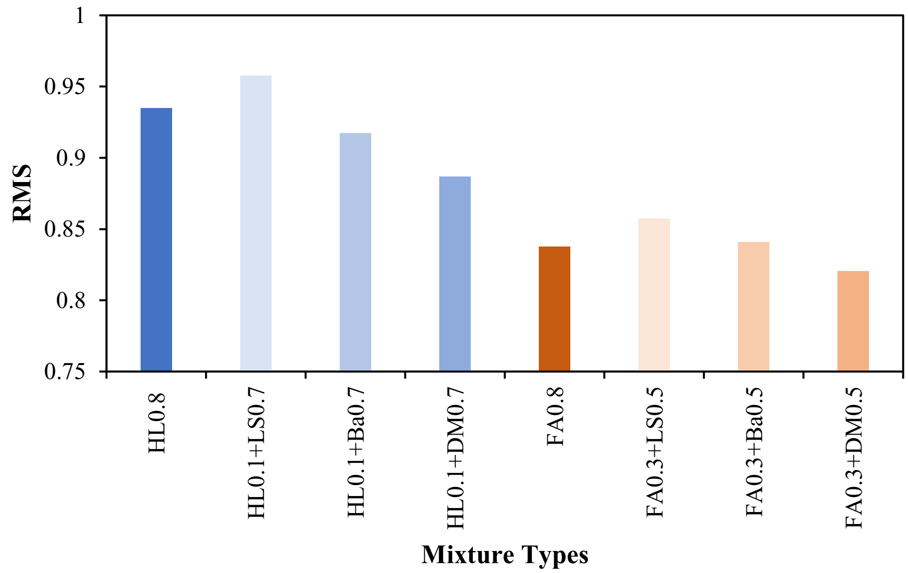

Figure 4.19 RMS values of mixtures at OBC

4.5.8 ANOVA Analysis

From the experimental plan, **Table 4.4** can be derived for ANOVA analysis in Design Expert Software. 8 mixtures were considered for the statistical analysis.

Table 4.4 Parameters for multilevel factorial design

Factor	Name	Unit	Type	Number of Levels	Levels
A [Categoric]	Active Filler	N/A	Nominal	2	HL, FA
B [Categoric]	Inert Filler	N/A	Nominal	4	LS, Ba, DM, N/A

Response	Name	Unit	Type
Stability	Marshall Stability	kN	Continuous
ITS (dry)	IndirectTensile Strength	kPa	Continuous
TSR	Tensile Strength Ratio	Dimensionless	Continuous

4.5.8.1 Analyzed result

The analysis of variance (ANOVA) was conducted for Stability, ITS (dry) and TSR, and the results are presented in **Table 4.5**. For all the models F-value was significant with a p-value of less than 0.05, where the p-value measured the statistical significance of each regression coefficient.

Table 4.5 ANOVA for both models

Source	For response Stability			For response ITS (dry)			For response TSR		
	Sum of Squares	F-value	p-value	Sum of Squares	F-value	p-value	Sum of Squares	F-value	p-value
Model	6.66	43.36	0.0055	16807	53.19	0.0041	0.0114	25.22	0.0121
A-Active Filler	0.4186	10.90	0.0457	1800	22.78	0.0175	0.0091	81	0.0029
B-Inert Filler	6.24	54.19	0.0041	15007	63.32	0.0033	0.0022	6.63	0.0473
Residual	0.1152			237			0.0003		
R^2	0.9830			0.9861			0.9711		
Adjusted R^2	0.9603			0.9676			0.9326		
Predicted R^2	0.8791			0.9011			0.8947		
Adeq. Precision	18.47			21.56			13.42		

Active filler (A) and inert filler (B) had a statistically significant effect on stability, ITS and TSR as the P-values was less than 0.0500. Only the statistically significant terms were included in the model. Significant factors imply a significant change in response to the variation of levels. Factor A, active fillers had the highest value of F for the response TSR, which indicates the maximum effect on TSR. The TSR value would be significantly impacted by the variation of active filler (HL or FA). From the TSR analysis, for HL mixtures, the TSR value ranged between 0.87-0.93, whereas, for FA, the range was .82-85. Factor B, inert fillers, had the maximum effect on ITS. However, inert fillers had a significant effect on stability also. With the inclusion and use of different types of inert fillers, the stability and ITS of the mixtures varied significantly.

For all the responses, the predicted R^2 was in reasonable agreement with the adjusted R^2 and the difference was also less than 0.2 which implies that the data is not over fitting. The lower difference between R^2 and adjusted-R^2 implied that all significant terms were involved in the model (Nayak et al., 2019). Adeq. precision measures the signal to noise ratio. A ratio greater than 4 is desirable (Nayak et al., 2019). For, all the responses the adeq. precision is more than 4 which indicates an adequate signal. These models can be used to navigate the design space. All the assumptions were checked for all the responses and found that all the assumptions are ok. No transformation was needed for any of the responses.

The developed model was optimized to get the parameters to maximize the stability, ITS, and TSR. **Figures 4.20 and 4.21** compare the constraints for the optimization process and showed the best two mixtures to optimize the responses. All the factors significantly influenced the stability, ITS, and TSR. All the factors studied were categorical, and optimized results were found after seven solutions. **Figure 4.20** shows the constraints which will maximize the responses. Combining active filler HL and inert filler LS maximizes stability, ITS, and TSR. The maximum stability value was 13.5787 kN, the maximum ITS was 1349 kPa, and the maximum TSR was .93, with high desirability of 0.935. On the other hand, from **Figure 4.21**, using active filler (HL) without any inert filler had a stability of 12.9288, which was 4.8% less, ITS of 1301.5, which was 3.5% less, TSR of 0.01375, which was 2.3% less than the mixture prepared with the combination of active filler HL and inert filler LS. The desirability of the mix prepared with only HL was 0.753, which is 20.8% less desirable than the mixture prepared with HL and LS.

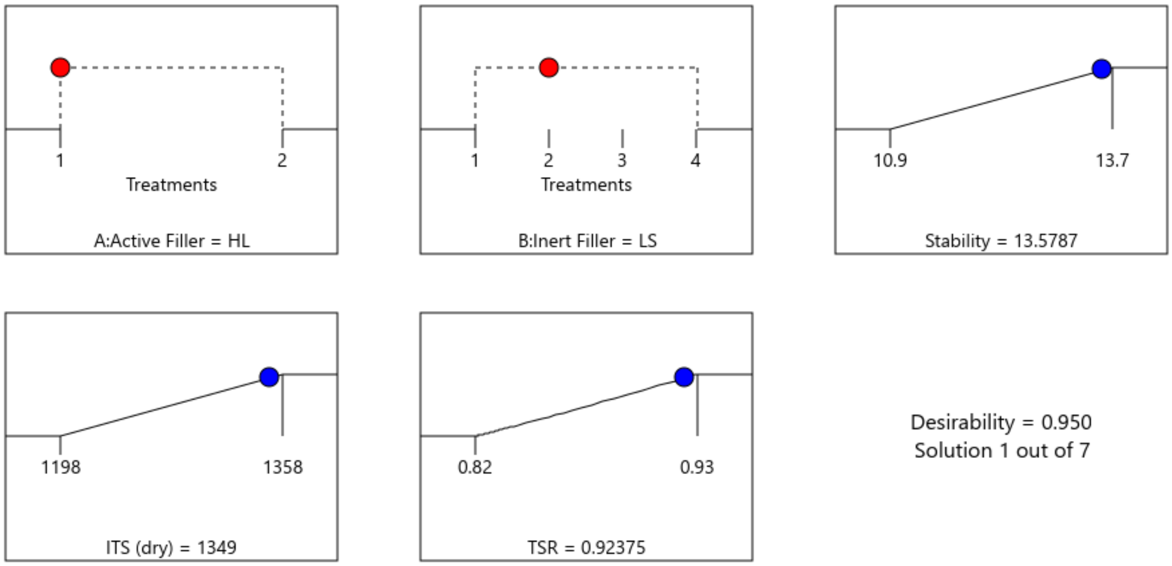

Figure 4.20 Optimization of Stability, ITS and TSR (HL+LS)

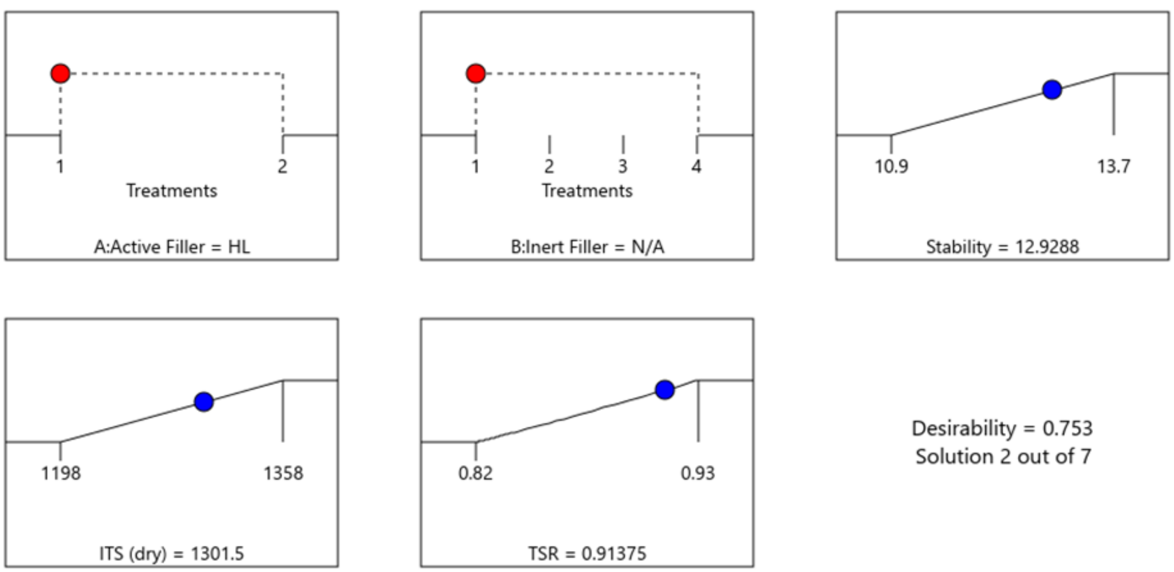

Figure 4.21 Optimization of Stability, ITS and TSR (only HL)

4.6 Conclusions

The following conclusions can be drawn based on the experimental results collected from the mastic and mixture level analysis of different parameters.

- A combination of active filler HL and inert filler LS improved the mastics' rutting resistance compared to those prepared with only active or inert filler by decreasing the J_{nr} value.10% HL+70% LS had a lower J_{nr} value than other mastics. Whereas, for the mastics prepared with the combination of FA and other inert fillers, only 30% Fa+50% LS showed better performance than that prepared with only inert filler Ba.
- The modified method of stress sensitivity analysis showed that all the mastic passed the stress sensitivity criteria, which implied these binders could perform well in high temperatures or heavy loads. However, some mastics failed the previous method of stress sensitivity. All the mastics prepared with the combination of HL+LS and HL+Ba had less stress sensitivity than the aged binder, according to the modified method of stress sensitivity.
- From the polymer modification curve, all the mastics could be graded according to MSCR grading as the J_{nr} value is less than 4.5 kPa^{-1} for all the mastics. In the case of elastomeric performance, the mastics prepared with the combination of HL and other inert fillers (LS, Ba, DM) outperformed the mastics prepared with the combination of FA and other inert fillers. 10% HL+70% LS had better recovery performance compared to the other mastics.
- OBC in a mixture is influenced by the type of active and inert filler that were employed. All the mixtures prepared with FA demonstrated a reduction in OBC compared to the mixtures prepared with HL. Replacing FA with other inert fillers required higher OBC than the mix with only active filler FA.

- Mixtures containing HL had better ITS and TSR value compared to FA mixtures. The combination of HL and LS had the highest ITS and TSR value, implying better cracking and moisture-induced damage resistance. However, this mixture containing FA had a higher MQ value than this mixture containing HL. To better understand the moisture induces damage resistance, the mixture's RMS value was evaluated, and they showed the same results as TSR.
- From the ANOVA analysis, variation of active and inert filler significantly affected the stability, ITS, and TSR value. For TSR, the variation of active fillers had the highest effect, whereas, for stability and ITS, the variation of inert filler had the highest impact.
- From the optimization analysis and performance, the mastics could be ranked (top 4) as HL+LS > HL > HL+Ba > FA+LS, the combination of 10%HL and 70%LS had 4.8% more stability, 3.5% more ITS, and 2.3% more TSR compared to the mixture prepared with only HL.

4.7 Limitations and Recommendations

The above-mentioned results were obtained after completing a limited number of laboratory tests. The performance of the mastics depended on the mixing procedure. The binder, modifiers, and anti-stripping agents were collected from only one source, where they can perform differently depending on the source. Long-term aging, the low-temperature performance of the mixtures, and the chemical interaction of active and inert filler in mastic scales and mixture levels can also be evaluated. Superpave mix design can be considered for future study. Additional lab studies are needed to understand other rheological properties and field tests are needed to obtain more practical results of these alternative fillers.

4.8 Application of the Research

This study used SBS to modify the binder, and the anti-stripping agent Zycotherm was added to the binder. Two active fillers (HL and FA) and three inert fillers (LS, Ba, DM) were combined separately in three different proportions and mixed with the modified binder to fabricate the mastics. RTFO conditioning was employed to evaluate these short-term aged asphalt mastics' creep recovery and mechanical performance in mastic and mixture levels. The combination of active filler HL (10%) and inert filler LS(70%) performed better than other mastics in the mastic scales and mixture level. This mastic passed Extremely Heavy traffic loading criteria based on AASHTO M 332, modified stress sensitivity criteria, and polymer modification curve. While in the mixture level, this mastic showed good cracking and moisture damage-resistant performance. So, this mastic (10%HL+70%LS) is suggested to use in the asphalt mixture instead of using only active, inert, or other combinations of fillers to prepare the mastics to minimize the moisture susceptibility and to strengthen the adhesion of binder with aggregate.

4.9 Acknowledgement

The authors gratefully acknowledge the financial support provided by the City of St. John's and are thankful to Mark White and Gary Caul for their support. Appreciation is also extended to the Center for Research and Education in Advanced Transportation Engineering Systems (CREATES), Rowan University, USA, for their invaluable helps in lab testing. The authors are also grateful to McAsphalt, Yellowline Asphalt Products Limited, Atlantic Minerals, American Gilsonite Company, and Zydex Industries for providing asphalt, modifiers, and anti-stripping agents for the accomplishment of this study.

4.10 Author Contributions

The authors confirm contribution to the paper as follows: study conception and experimental design: Shahrul Ibney Feroz, Dr. Kamal Hossain; data collection: Shahrul Ibney Feroz, Debzani Mitra, Dr. Sk Faisal Kabir; analysis and interpretation of results: Shahrul Ibney Feroz, Dr. Kamal Hossain; draft manuscript preparation: Shahrul Ibney Feroz. Dr. Yusuf Mehta reviewed and improved the manuscript All authors reviewed the results and approved the final version of the manuscript.

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Chapter 5: Conclusions and Application of Research

5.1 Overview

Many researchers and highway agencies have recognized the advantages of utilizing modifiers, anti-stripping agents, and fillers in the binder, mastic, and mixture level to mitigate pavement distresses. The conventional method, like Superpave parameters, cannot fully characterize the modified binders or mastics. Multiple Stress Creep Recovery (MSCR) test can characterize the modified binders and mastics better than the conventional methods.

This research aimed to develop an asphalt mixture that would be suitable for the roads of St John's and could withstand moisture damage, rutting, and cracking distresses. The study had three phases: binder, mastic, and mixture level. In the binder level (study-1), two different modifiers (SBS and Gilsonite) and four anti-stripping agents (Pave Bond Lite, AD-Here, Zycotherm, and Kling beta) were used to evaluate and compare the creep recovery performance. Research on the optimum filler-binder combination and mastic testing had shown more promise than conventional binder testing. The mastic and mixture level analysis were completed using two studies (study-2 and study-3) with different sets of samples. In study-2 (described in chapter 3), two different fillers (HL and LS) with varying ratios of F/B were mixed with modifiers (SBS and Gilsonite) and anti-stripping agents (AD-Here, and Zycotherm) to evaluate the rheological performance in the mastic level and mechanical performance in the mixture level. In study-3 (described in chapter 4), the effect of the combination of different active (HL. FA) and inert (LS, Ba, DM) fillers in modified mastic and mixture levels were evaluated. Statistical analysis was performed in every phase to know the relative impact of each factor and optimize the findings. Dose rate and types of modifiers, anti-stripping agents, types of fillers, and F/B ratios were selected based on the background study and discussed with the industry specialist. This chapter summarizes the key finding from the experimental investigation. This chapter also compares all the results mentioned in chapters 2, 3, and 4.

5.2 Major Findings from the Binder Level MSCR Test

- All the SBS-modified binders containing different dosages of liquid anti-stripping agents showed good rutting performance and passed all the traffic loading criteria based on AASHTO M 332 specification except 1% Pave bond lite. But for Gilsonite-modified binders, 67% binders passed all the traffic loading criteria. SBS is a thermoplastic elastomer polymer, SBS behaves like elastomeric rubbers, improving the binders' elastic behaviour. Consequently, SBS helps to prevent the persistent deformation of the asphalt binders. In case of anti-stripping agents, Zycotherm and AD-Here had lower J_{nr} value than Pave bond lite and Kling beta. The 0.1% Zycotherm modified with SBS had the lowest value of J_{nr} and passed all the traffic loading conditions with the least deformation.
- According to the modified method of stress sensitivity, all the binders had a stress sensitivity less than the aged binder. In contrast, according to the previous method of stress sensitivity, almost all the binders had stress sensitivity more than the aged binder. All the dosages of Gilsonite and SBS modified binders containing different dosages of liquid anti-stripping agents passed the modified method of stress sensitivity. Although SBS modified 0.5 % Ad-Here, 0.075 and 0.1 % Zycotherm modified with SBS binders failed to meet the previous method of stress sensitivity criteria.
- All the SBS and Gilsonite-modified binders containing different dosages of liquid anti-stripping agents could be graded according to MSCR grading as the J_{nr} value was less than 4.5 kPa^{-1} for all the binders. In case of elastomeric performance, all the SBS modified

binders clustered above the polymer modification standard curve and passed the criteria except 1 % Pave Bond Lite. But all the Gilsonite-modified binders clustered below the standard line and failed to pass the criteria. SBS-modified binders outperformed the Gilsonite modified binders in case of recovery performance

- From the quadrant plot analysis, almost all the SBS-modified binders containing different dosages of liquid anti-stripping agents passed the % Recovery and Phase Angle criteria and fell in the "None at Risk" quadrant, except 1% Pave Bond Lite. All these binders showed good % Recovery performance, whereas none of the Gilsonite-modified binders passed the % Recovery and Phase Angle criteria. Most of the Gilsonite-modified binders fell in the "Both at Risk" quadrant.
- From the statistical analysis, modifiers, anti-stripping agents, and dose rate substantially impacted J_{nr} and %R. The creep recovery performance of these binders varies greatly with the variation of modifiers and anti-stripping agents. Modifiers had the most effects for J_{nr} and %R. From the optimization analysis, combination of the SBS modifier with 0.1% Zycotherm minimizes the J_{nr} value rate and maximizes the %R. The creep recovery performance of the binders (Top 2) could be ranked as 0.1% Zycotherm + SBS > 0.5% AD-Here + SBS.

From the above analysis, it can be concluded from the binder level analysis that, 0.1% Zycotherm with SBS and 0.5% AD-Here with SBS showed better resistance compared to other combinations. On the other hand, although Gilsonite modified binders failed to pass some criteria but 0.1% Zycotherm with Gilsonite and 0.5% AD-Here with Gilsonite had a lower value of J_{nr}.

5.3 Major Findings from the Mastic and Mixture Level Analysis of Different Modifiers, Anti-stripping Agents and Fillers

- In comparison to the neat aged binder and the other binders taken into consideration in the binder level study, the non-recoverable creep compliance value, J_{nr}, of the asphalt mastic decreased dramatically with the addition of HL and LS as filler material. SBS-modified mastics had a J_{nr} value range of 0.013-0.07 kPa^{-1}, whereas Gilsonite-modified mastics had a J_{nr} value range of 0.04-0.21 kPa^{-1}. For both the cases of SBS and Gilsonite-modified mastics, HL0.5 containing Zycotherm had the lowest value of J_{nr}, indicating better resistance against permanent deformation and rutting. All the modified mastics containing Zycotherm showed good rut resistance compared to those with AD-Here. Overall, SBS-modified mastics containing Zycotherm, and HL filler had a lower J_{nr} value than the other mastics.
- The modified method of stress sensitivity analysis showed that all the mastic modified with SBS or Gilsonite passed the stress sensitivity criteria, which implied these binders could perform well in high temperatures or heavy loads. However, some mastics failed the previous method of stress sensitivity. All the mastics had less stress sensitivity than the aged binder, according to the modified method of stress sensitivity.
- From the polymer modification curve, all the SBS and Gilsonite modified mastic containing different amounts of HL, and LS could be graded according to MSCR grading as the J_{nr} value was less than 4.5 kPa^{-1} for all the mastics. In the case of elastomeric performance, the SBS modified mastics outperformed the Gilsonite modified mastics. 10 out of 12 SBS mastics passed the criteria, whereas all the Gilsonite mastics clustered below the polymer modification standard curve.

- From the statistical analysis, modifiers, anti-stripping agents, and filler significantly affected J_{nr} and %R. For J_{nr}, modifiers had the highest effect, whereas, for %R, the anti-stripping agent had the highest effect. Both J_{nr} and %R could be used to describe the creep recovery performance of asphalt mastics. 4% SBS modified HL0.5 mastic contains 0.1% Zycotherm minimized the J_{nr} value and maximized the %R.
- OBC is ranging between 6-6.5% for all the mixtures. OBC in a mixture is influenced by the filler, modifier, and anti-stripping agent types that were employed. The addition of SBS and Zycotherm demonstrated a 4% reduction in OBC compared to the control mix, suggesting a considerable reduction in the cost of producing HMA. For both HL and LS, mixtures modified with Gilsonite had more OBC requirement than the control mixtures and SBS modified mixtures due to the affinity of Gilsonite to the asphalt binder and high binder absorption capacity.
- The stability values for all combinations can be observed to meet the Asphalt Institute guideline for the heavy traffic of not less than 8.01 kN. The mixes containing HL and LS as filler had the highest stability values of 13.2 kN and 12.4 kN respectively. Due to decreased OBC requirement of LS, it has higher MQ compare to the HL.
- ITS increased significantly for all the other mixtures compared to the control mix. A mixture with HL filler, SBS, and Zycotherm had the highest ITS value of 1328 kPa, 31% more than the mixture without modifiers and anti-stripping agents implied better cracking resistance. This mixture had the highest TSR value, implying moisture-induced damage resistance. However, this mixture had a lower MQ value than Gilsonite-modified mastics. Zycotherm was showing good performance compared to AD-Here as an anti-stripping

agent. To better understand the moisture induces damage resistance, the mixture's RMS value was evaluated, and they showed the same pattern as TSR.

Finally, it can be concluded that both in mastic and mixture level HL filler modified with SBS and Zycotherm showed better rutting, cracking, and moisture damage-resistant performance. However, most SBS mastics and mixtures passed all the requirements and performed well.

5.4 Major Findings from the Mastic and Mixture Level Analysis of Combination of Different Active and Inert Fillers

- Combination of HL+LS, HL+Ba and FA+LS performed better than other combinations. J_{nr} values were ranging from 0.04-0.14 kPa^{-1} for FA asphalt mastic whereas the J_{nr} values for HL mastics were ranging from 0.012-0.13 kPa^{-1}. A combination of active filler HL and inert filler LS improved the mastics' rutting resistance compared to those prepared with only active or inert filler by decreasing the J_{nr} value.10% HL+70% LS had a lower J_{nr} value than other mastics. Whereas, for the mastics prepared with the combination of FA and other inert fillers, only 30%Fa+50%LS showed better performance than that prepared with only inert filler Ba. DM performed poorly both with HL and FA compared to other mastics.
- All the mastics passed the stress sensitivity criteria according to the modified method of stress sensitivity analysis, which implied these mastics could perform well in high temperatures or heavy loads. However, some mastics failed the previous method of stress sensitivity. All the mastics prepared with the combination of HL+LS and HL+Ba had less stress sensitivity than the aged binder, according to the modified method of stress sensitivity.
- Since the J_{nr} value for all the mastics was less than 4.5 kPa^{-1}, they may all be graded using the MSCR grading system. All the HL+LS, HL+Ba, FA+LS and FA+Ba showed an

excellent recovery. However, in case of elastomeric performance, the mastics prepared with the combination of HL and other inert fillers (LS, Ba, DM) outperformed the mastics prepared with the combination of FA and other inert fillers. 10% HL+70% LS had better recovery performance compared to the other mastics.

- OBC requirement was high for the Stone Mix Asphalt (SMA) mixture due to the gap graded structure, and it ranged between 5.9 to 6.5%. OBC in a mixture is influenced by the type of active and inert filler that were employed. Due to high absorb capacity and porous structure mixtures prepared with HL demonstrated a higher OBC requirement compared to the mixtures prepared with FA. Replacing FA with other inert fillers required higher OBC than the mix with only active filler FA.
- All the mixtures passed the asphalt institute minimum stability criteria. Asphalt mixture containing HL had a higher stability range (13.7-11 kN) than that containing FA (12.5-10.9 kN). The improved adhesion between HL and binder was to blame for the increase in stability. Combination of 10% HL and 70 % LS had the highest stability which is 6.2% more stable than mixtures prepared with only HL
- Mixtures containing HL had better ITS and TSR value compared to FA mixtures. HL particles dispersed well in the HMA mixture due to its large SSA and porous structure. The combination of HL and LS had the highest ITS and TSR value, implying better cracking and moisture-induced damage resistance. However, this mixture containing FA had a higher MQ value than this mixture containing HL. To better understand the moisture induces damage resistance, the mixture's RMS value was evaluated, and they showed the same results as TSR.

- From the ANOVA analysis, variation of active and inert filler significantly affected the stability, ITS, and TSR value. For TSR, the variation of active fillers had the highest effect, whereas, for stability and ITS, the variation of inert filler had the highest impact.
- From the optimization analysis and performance, the mastics could be ranked (top 4) as HL+LS > HL > HL+Ba > FA+LS, the combination of 10%HL and 70%LS had 4.8% more stability, 3.5% more ITS, and 2.3% more TSR compared to the mixture prepared with only HL.

Finally, it can be reported that the combination of 10%HL and 70%LS in mastic and mixture levels demonstrated improved rutting, cracking, and moisture damage-resistant performance. However, the majority of mastics and mixtures met all the criteria and delivered satisfactory results.

5.5 Summary of the Findings

This research aimed to develop an asphalt mixture that can withstand pavement distresses. Three different studies were conducted to meet the goal of this research. Three different levels were considered while planning and preparing the samples. These levels were binder level, mastic level, and mixture level.

Only binder-level analysis was conducted in study-1 (described in Chapter 2). From binder level analysis, 0.1% Zycotherm modified with 4% SBS had better rutting and recovery performance than other binders considered for the study.

In study-2 (described in Chapter 3), mastic and mixture levels were considered, and samples were prepared using with either active or inert filler. From study-2, it was found that HL0.5 modified with 4% SBS and 0.1% Zycotherm had better rutting, moisture damage, and cracking resistance performance.

Like study-2, mastic and mixture-level analysis were considered in study-3 (described in Chapter 4). Combination of active and inert filler was used to prepare the samples. The same methodology, method of analysis, and experiments as study-2 were conducted for study-3. The investigation found that the combination of active filler 10% HL and inert filler 70% LS performed better than the other mastics.

Finally, if study-2 and study-3 were compared, then it could be seen that combination of 10% HL and 70% LS (considered in study-3) had 8.3% lower Jnr value, 3.1 % higher MSCR % Recovery, 3.7% higher stability, 2.2% higher ITS compared to HL0.5 (considered in study-2). So, a combination of active and inert filler had better rutting, moisture damage and cracking resistance performance.

5.6 Application of the Research

In the binder level investigation, SBS and Gilsonite modified binder were combined individually with four different anti-stripping agents in varying doses. The performance of these short-term aged binders' creep recovery performance was assessed using the MSCR test followed by RTFO test. SBS modified binders outperformed Gilsonite modified binders in terms of rutting resistance and recovery performance. Almost all the SBS binders with various liquid anti-stripping agent doses met the "None at Risk" quadrant's criterion, Very High traffic loading criteria, modified stress sensitivity, polymer modification curve. The highest % Recovery and lowest value of J_{nr} are found in 0.1% Zycotherm modified with 4% SBS. To enhance the rutting resistance, elastic recovery and minimize the sensitivity to moisture, this dose of SBS-modified Zycotherm is advised for use with a binder.

In the study-2, different proportions of two different fillers (HL and LS) were mixed with SBS or Gilsonite modified binder containing anti-stripping agent Zycotherm or AD-Here to fabricate the asphalt mastic. RTFO conditioning was employed to simulate asphalt production time aging. MSCR test was employed to evaluate the creep recovery performance of these short-term aged asphalt mastics. SBS-modified HL mastics outperformed the other SBS-modified and Gilsonite-modified mastics. All the SBS modified HL mastics containing Zycotherm passed Extremely Heavy traffic loading criteria based on AASHTO M 332, modified stress sensitivity criteria, and polymer modification curve. SBS modified HL0.5 containg 0.1% Zycotherm showed better performance and suggested to use in the asphalt mixture to reduce the moisture susceptibility and to strengthen the adhesion of binder with aggregate. The mixture level analysis showed this mastics' good cracking and moisture damage-resistant performance.

Only SBS and the anti-stripping compound Zycotherm were employed to modify the binder in the final mastic-level research (study-3). Two active fillers (HL and FA) and three inert fillers (LS, Ba, DM) were combined separately in three different proportions and mixed with the modified binder to fabricate the mastics. RTFO conditioning was employed to simulate the short-term aged asphalt mastics. MSCR employed to creep recovery performance in mastic levels. The mixture of active filler HL (10%) and inert filler LS (70%) outperformed other mastics in both the mastic scales and mixture level. This mastic passed Extremely Heavy traffic loading criteria based on AASHTO M 332, modified stress sensitivity criteria, and polymer modification curve. While in the mixture level, this mastic showed good cracking and moisture damage-resistant performance. So, this mastic(10%HL+70%LS) is suggested to use in the asphalt mixture instead of using only active, inert, or other combinations of fillers to prepare the mastics to minimize the moisture susceptibility and to strengthen the adhesion of binder with aggregate.

5.7 Limitations and Future Study

There were certain restrictions throughout this research even though there were several experimental experiments conducted to meet the goals of this thesis. The following suggestions are provided for further research based on the restrictions.

- The above-mentioned results were obtained after completing limited number of laboratory tests due to lack of a comprehensive asphalt binder lab. These additives were added using laboratory techniques which were done in control environment. Additional lab studies are needed to understand other rheological properties of these alternative additives, modifiers, and fillers
- In this study, an SBS-modified binder was collected from the industry, whereas a magnetic stirrer was used to prepare the Gilsonite-modified binder. The exact mixing procedure should be used to compare the performance of SBS and Gilsonite.
- The performance of the mastics depends on the mixing procedure. A mechanical or electrical mixer would produce a more homogenous mixture than a magnetic stirrer. Some chunks in the mastics affected the performance of the mastics.
- Due to access to equipment restrictions, this research could only simulate the binder's short-term aging (RTFO conditioning) and high temperature performance. Therefore, employing PAV methodology to assess the binder's long-term performance may be preferable. Also, the low temperature cracking performance can also be evaluated.
- Chemical and morphological characterization of the mastics can also be considered for future analysis to explain the interaction between binder and aggregate. The chemical interaction of active and inert filler in mastic scales and mixture levels can also be evaluated.

- In the mixture level investigation, only Marshall mix design was used due to the limitation of other mix design equipment. Superpave mix design can be considered for future study.
- The field application of the recommended asphalt mixture may be highly beneficial in gaining access to the actual situation of the pavement performances, while this investigation was only conducted in a lab setting.

Appendix A: Laboratory Tests

Figure A.1: Mixing of fillers with modified binder

Figure A.2: Pre-heating before mixing

Figure A.3: Filler (Basalt) collected from Enjoy Stone

Figure A.4: Planetary Ball Mill equipment

Figure A.5: Samples prepared for MSCR testing

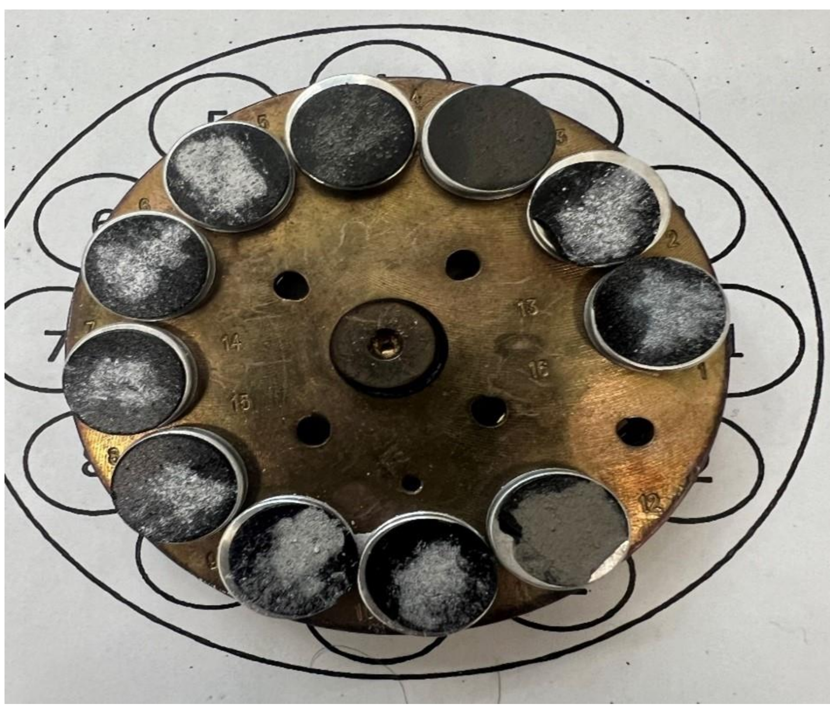

Figure A.6: Fillers ready for SEM and oxide composition

Figure A.7: SEM facility at MUN

Figure A.8: Pre-heating of aggregates and binder for Marshall test

Figure A.9: Weight measurement of aggregate

Figure A.10: Addition of binder to aggregate

Figure A.11: Weight measurement of Marshall sample in water

Figure A.12: Marshall samples following submersion

Figure A.13: Marshall samples after compaction

Figure A.14: Marshall stability and flow test facility at MUN

Figure A.15: Marshall compactor

Figure A.16: Mixer facility at MUN

Figure A.17: Water bath

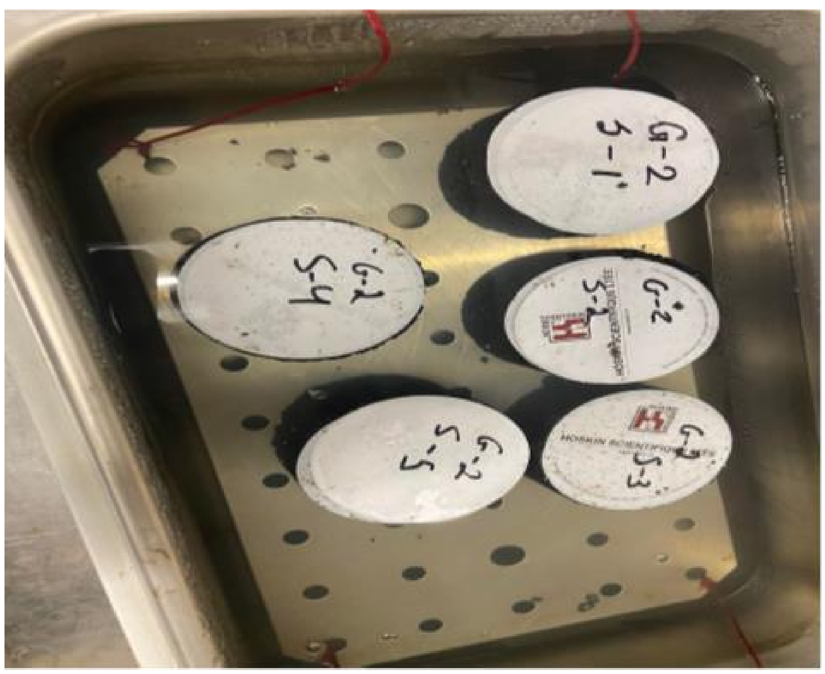

Figure A.18: Marshall samples in submerged condition

Figure A.19: A Malvern Panalytical Kinexus DSR-III Rheometer

Appendix B: Laboratory Test Data

Table B.1: Jnr, Jnr-diff., Jnr-slope and %R of SBS Modified Binders

Anit-Striping Agent	Percentages (%)	Jnr (3.2) (1/kPa)	Jnr, diff. (%)	Jnr, slope (%)	MSCR % R at 3.2 kPa
	0.5	0.2428	33.86	2.65	63.3
Pave Bond	0.75	0.3763	34.71	4.21	55.2
	1	0.9057	27.76	8.11	23.5
	0.5	0.2189	45.68	3.88	80.2
AD-Here	0.75	0.2386	46.10	3.54	68.8
	1	0.3784	26.42	4.19	66.8
	0.05	0.3424	36.77	4.06	58.8
ZycoTherm	0.075	0.2287	36.02	2.60	83.3
	0.1	0.207	45.06	3.01	82.9
	0.5	0.2703	34.59	3.02	63.8
Kling Beta	0.75	0.4293	32.80	4.54	48.6
	1	0.3095	41.84	4.18	55

Table B.2: Jnr, Jnr-diff., Jnr-slope and %R of Gilsonite Modified Binders

Anit-Striping Agent	Percentages (%)	Jnr (3.2) (1/kPa)	Jnr,diff. (%)	Jnr, slope (%)	MSCR % R at 3.2 kPa
	0.5	0.6162	12.26%	2.44	12
Pave Bond	0.75	0.492	9.96%	1.58	14.3
	1	0.5819	10.83%	2.03	12.9
	0.5	0.2761	36.22%	3.22%	26.4
AD-Here	0.75	0.351	40.17%	4.54%	22.7
	1	0.4182	28.34%	4.08%	17.1
ZycoTherm	0.05	0.4664	29.01%	5.30%	12.5

	0.075	0.386	28.26%	3.52%	7.6
	0.1	0.2542	26.55%	2.18%	11.3
	0.5	0.6256	11.06%	2.23%	11.5
Kling Beta	0.75	0.6351	11.68%	2.39%	11
	1	0.3517	26.07%	2.96%	21

Table B.3: Jnr, Jnr-diff., Jnr-slope and %R of Asphalt Mastics

Anti-stripping Agent	Filler	F/B Ratio	Modifier	Jnr (3.2) (1/kPa)	Jnr, diff. (%)	Jnr, slope (%)	MSCR % R at 3.2 kPa
Zycotherm 0.1%	HL	0.3	SBS 4%	0.0221	21.97	10.66129032	84.044
Zycotherm 0.1%	HL	0.4	SBS 4%	0.01975	30.96	12.73225806	93.75
Zycotherm 0.1%	HL	0.5	SBS 4%	0.0132217	46.65	13.35483871	94
Zycotherm 0.1%	HL	0.3	Gilsonite 10%	0.075	71.76	28.10	36.8
Zycotherm 0.1%	HL	0.4	Gilsonite 10%	0.045715	72.63447	34.54	42.55
Zycotherm 0.1%	HL	0.5	Gilsonite 10%	0.04524	92.71662	35.08	47.9
Zycotherm 0.1%	LS	0.8	SBS 4%	0.041555	91.74	30.229	70.85
Zycotherm 0.1%	LS	0.9	SBS 4%	0.033645	93.39	31.013	74.4
Zycotherm 0.1%	LS	1	SBS 4%	0.02529	95.34	32.777	80.65
Zycotherm 0.1%	LS	0.8	Gilsonite 10%	0.1087115	79.67756	37.495	40.75

Zycotherm 0.1%	LS	0.9	Gilsonite 10%	0.094625	98.53175	42.998	43.55
Zycotherm 0.1%	LS	1	Gilsonite 10%	0.081045	98.75501	44.581	45.75
AD-Here 0.5%	HL	0.3	SBS 4%	0.034715	58.95	18.660	75.35
AD-Here 0.5%	HL	0.4	SBS 4%	0.048195	75.17	24.168	72.85
AD-Here 0.5%	HL	0.5	SBS 4%	0.06274	80.73	27.633	43.85
AD-Here 0.5%	HL	0.3	Gilsonite 10%	0.20835	55.63955	33.88	12.8
AD-Here 0.5%	HL	0.4	Gilsonite 10%	0.18105	70.46948	34.54	11.845
AD-Here 0.5%	HL	0.5	Gilsonite 10%	0.17715	76.31386	35.19	12.1
AD-Here 0.5%	LS	0.8	SBS 4%	0.02365	41.22198732	18.314	80
AD-Here 0.5%	LS	0.9	SBS 4%	0.06993	43.51494351	20.981	27.2
AD-Here 0.5%	LS	1	SBS 4%	0.035125	81.22562278	30.920	73.95
AD-Here 0.5%	LS	0.8	Gilsonite 10%	0.2	62.75	30.822	24.25
AD-Here 0.5%	LS	0.9	Gilsonite 10%	0.1294	77.31066	40.722	34.1
AD-Here 0.5%	LS	1	Gilsonite 10%	0.11945	97.08246	45.42887	24.25

Table B.4: Jnr, Jnr-diff., Jnr-slope and %R of Asphalt Mastics Contain Active and Inert Filler

Anti-stripping Agent	Active Filler	Inert Filler	Modifier	Jnr (3.2) (1/kPa)	Jnr, diff. (%)	Jnr, slope (%)	MSCR % R at 3.2 kPa
Zycotherm 0.1%	HL0.8	N/A	SBS 4%	0.0164	51.40	7.20	89.25
Zycotherm 0.1%	HL0.1	LS0.7	SBS 4%	0.012	31.00	1.13	97
Zycotherm 0.1%	HL0.2	LS0.6	SBS 4%	0.014	39.00	3.22	92.4
Zycotherm 0.1%	HL0.3	LS0.5	SBS 4%	0.016	46.00	5.56	90
Zycotherm 0.1%	N/A	LS0.8	SBS 4%	0.041555	91.73	30.22	70.85
Zycotherm 0.1%	HL0.1	Ba0.7	SBS 4%	0.013	35.50	5.58	95.25
Zycotherm 0.1%	HL0.2	Ba0.6	SBS 4%	0.03325	59.50	8.03	86.1
Zycotherm 0.1%	HL0.3	Ba0.5	SBS 4%	0.035225	60.50	8.65	75.85
Zycotherm 0.1%	N/A	Ba0.8	SBS 4%	0.0402829	66.55	10.21	78.4
Zycotherm 0.1%	HL0.1	DM0.7	SBS 4%	0.10645	97.06	32	36
Zycotherm 0.1%	HL0.2	DM0.6	SBS 4%	0.1188	100.25	34	41.4
Zycotherm 0.1%	HL0.3	DM0.5	SBS 4%	0.122935	114.98	37	42.78
Zycotherm 0.1%	N/A	DM0.8	SBS 4%	0.136545	115.27	39	38.67

Zycotherm 0.1%	FA0.8	N/A	SBS 4%	0.116601	84.93	48.10	41.9
Zycotherm 0.1%	FA0.1	LS0.7	SBS 4%	0.052788	70.78	25.19	74.55
Zycotherm 0.1%	FA0.2	LS0.6	SBS 4%	0.05143	68.10	15.57	77.75
Zycotherm 0.1%	FA0.3	LS0.5	SBS 4%	0.0392775	67.00	11.98	81.32
Zycotherm 0.1%	FA0.1	Ba0.7	SBS 4%	0.11395	95.25	44.54	62.8
Zycotherm 0.1%	FA0.2	Ba0.6	SBS 4%	0.073671	86.50	35.08	66.55
Zycotherm 0.1%	FA0.3	Ba0.5	SBS 4%	0.067285	75.56	23.88	78.9
Zycotherm 0.1%	FA0.1	DM0.7	SBS 4%	0.123126333	100.21	51.01	39
Zycotherm 0.1%	FA0.2	DM0.6	SBS 4%	0.123126333	87.00	49.00	44
Zycotherm 0.1%	FA0.3	DM0.5	SBS 4%	0.123126333	85.67	45	48

Table B.5: OBC, Maximum Stability, Maximum Flow, MQ, RMS, ITS and TSR of Asphalt Mixtures

Mixture Types	Filler	OBC	Maximum Stability (KN)	Maximum Flow (mm)	MQ	RMS	ITS dry (kPa)	ITS wet (kPa)	TSR
Control	HL	6.40	8.24	2.9	2.841	0.707	938	807	0.860
SBS+ Zycotherm	HL	6.17	13.2	3.3	4	0.97	1328	1237	0.931
SBS+AD-Here	HL	6.30	12.84	3.5	3.668	0.948	1285	1190	0.926
Gilsonite+ Zycotherm	HL	6.43	11.9	2.5	4.76	0.747	1227	1067	0.869
Gilsonite+ AD-Here	HL	6.47	11.15	2.3	4.847	0.730	1172	1012	0.863
Control	LS	6.33	8.24	2.8	2.875	0.653	867	680	0.784
SBS+ Zycotherm	LS	6.10	13.2	2.9	4.275	0.837	1144	988	0.863
SBS+ AD-Here	LS	6.27	12.84	2.9	3.931	0.933	1034	870	0.841
Gilsonite+ Zycotherm	LS	6.43	11.9	2.2	4.954	0.706	982	805	0.819
Gilsonite+	LS	6.50	11.15	2	5.05	0.699	912	745	0.816

AD-Here

Table B.6: OBC, Maximum Stability, Maximum Flow, MQ, RMS, ITS and TSR of Asphalt Mixtures Contain Active and Inert Filler

Mixture Types	OBC	Maximum Stability (KN)	Maximum Flow (mm)	MQ	RMS	ITS dry (kPa)	ITS wet (kPa)	TSR
HL0.8	6.37	12.9	3.1	4.161	0.934	1298	1190	0.917
HL0.1+LS0.7	6.17	13.7	3.2	4.281	0.957	1358	1255	0.924
HL0.1+Ba0.7	6.30	12.84	3.2	4.012	0.917	1285	1160	0.902
HL0.1+DM0.7	6.43	10.99	2.9	3.789	0.886	1227	1067	0.869
FA0.8	5.90	12.5	2.8	4.464	0.837	1275	1080	0.847
FA0.3+LS0.5	6.10	13	2.98	4.362	0.857	1310	1115	0.851
FA0.3+Ba0.5	6.13	12.2	2.8	4.357	0.840	1265	1070	0.845
FA0.3+DM0.5	6.20	10.9	2.6	4.192	0.820	1198	987	0.823