

Design and Performance Evaluation of Road Base Courses Comprised of Asphalt Emulsion
and Asphaltenes Derived from Alberta Oil sands

by

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Abstract

Stabilization of granular base course materials has proven to be an effective method for enhancing the performance of pavement structure. Among the various methods used in this field, asphalt emulsion stabilization stands out as a widely employed technique that offers several benefits, including the production of high-quality base courses with reduced energy consumption, carbon footprint, and natural material usage. This method leads to increased strength, stability, bearing capacity, and overall mechanical properties compared to granular base layer. However, it comes with certain drawbacks, such as decreased moisture and rutting resistance, early-life performance issues, and longer curing times. Researchers have proposed solutions to address these deficiencies, although not comprehensive enough to eliminate further disadvantages. The addition of cementitious materials as active fillers in asphalt emulsion stabilization yields significant benefits. It leads to higher early-strength gain, increased stiffness, improved rutting resistance, enhanced moisture resistance, and shorter curing and breaking times compared to unmodified layers. These advantages contribute to enhanced structural integrity, load-bearing capacity, and overall performance of the stabilized layer, making it a valuable and effective technique in road construction. A review of existing literature reveals several disadvantages in base layer stabilization, including the low performance properties of base layers stabilized with asphalt emulsion, negative effect of the rigidity of alternative cementitious materials used for stabilization, the lack of proper information regarding the impact of temperature on pavement base layers in current guidelines. In addition, using widely available residue materials like asphaltenes and reclaimed asphalt pavement (RAP) as additives in road construction can improve these layers and reduce the carbon footprint. Integrating these materials into the design process leads to more sustainable

and environmentally friendly practices. In this study, to enhance the performance of the base layer, two different mixtures consisting of natural aggregates or RAP are selected to be stabilized with asphalt emulsion, with the addition of asphaltenes as an additive. Additionally, Portland cement was added to same gradation of aggregates selected and the performance properties were compared to asphaltenes modified mixes. The asphaltenes used in this study are derived from Alberta oil-sands bitumen without any application in industry. The design process of the mixtures is determined based on available guidelines, utilizing tests such as Indirect Tensile Strength (ITS), Tensile Strength Ratio (TSR), and Marshall stability. The performance properties of the mixtures are investigated at high-temperatures with Hamburg wheel tracking, intermediate-temperatures using ITS and TSR, and low-temperatures using creep compliance and strength, and indirect tensile test at low temperatures. Binder testing, and field emission scanning electron microscopy (FESEM) are conducted to analyze the properties of the binders. Additionally, dynamic modulus tests are performed on the mixes to predict the viscoelastic properties of the samples. Furthermore, an analysis of variance (ANOVA) is employed to evaluate the significance of asphaltenes modification. The results of this research demonstrate a significant improvement in the high-temperature properties of both aggregate and RAP layers with addition of asphaltenes and Portland cement. Intermediate-temperature properties also show improvement, and in some cases, the mixes meet the minimum requirements suggested by guidelines after modification. However, no significant changes are observed in the low-temperature properties, indicating that the addition of asphaltenes do not adversely affect the performance in low-temperature conditions, as indicated by the results from ANOVA. However, cement increased the brittleness of the mixes at low-temperatures. The dynamic shear rheometer (DSR) test results also show that addition

of asphaltenes results in stiffening the base binder with FESEM indicating that asphaltenes interact. However, cement does not interact with binder. The dynamic modulus test results demonstrate that both the stabilized base layer and recycled materials exhibit viscoelastic behaviour, with varying moduli at different frequencies and temperatures. This finding indicates that these layers, are viscoelastic materials rather than granular materials in pavement design. The dynamic modulus results of cement modified mixes exhibit elastic behaviour which highlights the unique impact of asphaltenes on the material's viscoelastic properties, providing valuable insights for pavement design and performance. Comparing the positive effects of both cement and asphaltenes at high- and intermediate-temperatures with adverse behaviour of mixes with cement with respect to asphaltenes modified mixes, ultimately, suggested the potential replacement of Portland cement with asphaltenes in asphalt emulsion-stabilized mixes to achieve improved performance in base layers.

Preface

This thesis is an original work conducted by Farshad Kamran under the supervision of Dr. Leila Hashemian. This thesis is organized in a paper-based format.

A version of Chapter 3 titled "Laboratory Evaluation of Stabilized Base Course Using Asphalt Emulsion and Asphaltenes Derived from Alberta Oil Sands" has been published in the Journal of Construction and Building Materials, Volume 283, 122735 in May 2021. The study was conducted by Farshad Kamran, Manjunath Basavarajappa, Dr. Nura Bala, and Dr. Leila Hashemian, with contributions as follows: L. Hashemian conceived and designed the study; F. Kamran and M. Basavarajappa performed data collection; F. Kamran, M. Basavarajappa, N. Bala, and L. Hashemian analyzed and interpreted the results; F. Kamran prepared the initial manuscript draft.

A version of Chapter 4, titled "Performance Evaluation of Stabilized Base Course Using Asphalt Emulsion and Asphaltenes Derived from Alberta Oil Sands," was published in the Transportation Research Record: Journal of the Transportation Research Board, Volume 2675, Issue 10, in May 2021. The study's contributions were as follows: study conception and design by L. Hashemian; data collection by F. Kamran and M. Basavarajappa; analysis and interpretation of results by F. Kamran, M. Basavarajappa, N. Bala, and L. Hashemian; and draft manuscript preparation by F. Kamran.

A version of Chapter 5, submitted for publication as Farshad Kamran, Nusrat Jhora, Dr. Taher Baghaee Moghaddam, and Dr. Leila Hashemian, titled "Assessment of Performance Characteristics of Recycled Base Course Incorporating Reclaimed Asphalt Pavement and Asphaltenes" in the Canadian Journal of Civil Engineering. F. Kamran and N. Jhora were involved in data collection, analysis, and interpretation of results. T.B. Moghaddam and L. Hashemian were involved in the analysis and interpretation of results. The study's conception and design were by L. Hashemian, and the first draft was prepared by F. Kamran.

A version of Chapter 6, submitted for publication as Farshad Kamran and Dr. Leila Hashemian. "Evaluation of Asphaltenes a Potential Alternative for Cement in Stabilized Base Courses Using Asphalt Emulsion" was the title of this research paper in Cleaner Materials Journal of

Elsevier. L. Hashemian supervised, analyzed and interpreted the results and F. Kamran prepared the first draft with analysis and interpretation of results, and data collection.

Appendix contains a research paper submitted to ASTM Journal of Testing and Evaluation by Farshad Kamran, Amirhossein Ghasemirad, Dr. Taher Baghaee Moghaddam, Dr. Alireza Bayat, and Dr. Leila Hashemian titled as "Performance Evaluation of High Modulus Asphalt Concrete (HMAC) prepared using Asphaltenes-Modified Binders". The research was supervised by L. Hashemian and A. Bayat and data collected by F. Kamran and A. Ghasemirad. All the authors contributed in data analysis and interpretation of results.

To my parents, lovely sisters, and life partner

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List of Abbreviations

AASHTO	American Association of State Highway and Transportation Officials
ANOVA	Analysis of Variance
ARRA	Asphalt Recycling and Reclaiming Association
ASTM	American Society for Testing Materials
CI	Colloidal Index
CIR	Cold In-Place Recycling
CSIC	Composite Specimen Interface Cracking
CSS	Cationic Slow Setting
CT-Index	Crack Tolerance Index
DSR	Dynamic Shear Rheometer
FDR	Full Depth Reclamation
FE	Fracture Energy
FESEM	Field Emission Scanning Electron Microscopy
FHWA	Federal Highway Administration
F/T	Freeze/Thaw
G_{ca}	Specific Gravity of Coarse Aggregates
G_f	Failure Energy
G_{fa}	Specific Gravity of Fine Aggregates
GHG	Greenhouse Gas
G_{sb-agg}	Bulk Specific Gravity of Aggregates
GU	General-Use
HMA	Hot Mix Asphalt
HWT	Hamburg Wheel Tracking
IDEAL-CT	Indirect Tensile Asphalt Cracking Test
IDT	Indirect Tensile Test
ITS	Indirect Tensile Strength
LTPPBind	Long-Term Pavement Performance Bind
LVDT	Linear Variable Differential Transformer
MEPDG	Mechanistic-Empirical Pavement Design Guide

MCR	Micro Carbon Residue
MDD	Maximum Dry Density
MQ	Marshall Quotient
OEC	Optimum Emulsion Content
OFC	Optimum Fluid Content
OMC	Optimum Moisture Content
RAP	Reclaimed Asphalt Pavement
RRI	Rutting Resistance Index
RTFO	Rolling Thin Film Oven
Sabita	Southern African Bitumen Association
SARA	Saturates, Aromatics, Resins and Asphaltenes
SBR	Styrene-Butadiene Rubber
SBS	Styrene Butadiene Styrene
SHRP	Strategic Highway Research Program
SIP	Striping Inflection Point
TG2	Technical Guidelines
TSR	Tensile Strength Ratio
TTS	Time–Temperature Superposition
UTM	Universal Testing Machine
WLF	Williams-Landel-Ferry

Chapter 1: Introduction

1.1. Introduction and Problem Statement

The growing industry and expansion of developments have necessitated the establishment of larger and more extensive networks. These networks rely heavily on strong soil as the foundation for roads and transportation methods. However, this has not always been the case, as road bases often require strengthening and improvement to reduce the thickness of pavement layers, resulting in cost savings and minimizing adverse environmental impacts (Barve & Sugandhi, 2022; Christopher et al., 2006; Firoozi et al., 2017; Sheikh et al., 2022; Tremblay et al., 2002; Wirtgen Cold Recycling Manual, 2012). Soil stabilization emerged as a solution to address weak base layers around 5000 years ago in ancient Egypt and Mesopotamia. The Greeks and Romans further advanced this technique by introducing lime as a stabilizing agent in roads (Firoozi et al., 2017; McDowell, 1959). Stabilization of granular base layers using asphaltic or cementitious materials has been shown to increase the tensile strength of unbound layers (I. Thanaya, 2007; White & Weir, 2021). However, the use of calcium-based stabilizers, which were widely employed to overcome initial challenges, posed questions due to issues such as early heaving and premature failures in pavements containing sulfates (Firoozi et al., 2017; Kaminskas & Barauskas, 2014; Saussaye et al., 2015). The rigidity of layers stabilized with cementitious materials, as well as problems such as cracking, shrinkage, slow construction processes, and high greenhouse gas (GHG) emissions associated with cement production, further motivated researchers to explore more viable alternatives (Barcelo et al., 2014; Benhelal et al., 2013; Shah et al., 2022).

To address these shortcomings, asphaltic materials, such as asphalt emulsion, were introduced as alternatives to granular base layers. These materials offer enhanced flexibility, cohesion, shear strength, stiffness, and durability compared to traditional granular bases (Branch, 2005; Kim & Lee, 2012). Additionally, the use of asphalt emulsion brings advantages such as lower temperature application, reduced energy consumption, lower emissions, and decreased hazards compared to conventional hot mix asphalt (HMA) and cut back asphalt, making it more environmentally friendly (Salomon, 2006). However, it is important to note that this method also has its drawbacks. High temperature rutting susceptibility, limited suitability for areas

with heavy traffic, longer curing times, and lower early performance properties are among the disadvantages associated with the use of asphalt emulsion (Du, 2016; Kavussi & Modarres, 2010; Khweir et al., 2004).

Furthermore, the construction industry has witnessed a significant surge in activities since the early 2000s, leading to a global demand for natural aggregates and other construction materials in pavement construction. Unfortunately, the extensive use of these materials also contributes to increased energy consumption and air pollution during their extraction and transportation processes (OECD, 2015). In light of these growing concerns, researchers have explored various methods to enhance the strength of base layers while also promoting the use of recycled construction and demolition materials, such as reclaimed asphalt pavement (RAP) (Chhabra et al., 2021; Mohammadinia et al., 2019). To address these environmental challenges, different agencies and governmental sectors are actively striving to reduce the reliance on natural resources by utilizing low-quality materials and by-products (Lindsey, 2011; Yaowarat et al., 2021). RAP is considered a valuable resource due to its high-quality aggregates and binder content, although it requires some modifications to meet the necessary reconstruction requirements, such as reducing asphalt content, addressing binder brittleness due to aging, and incorporating crushed aggregates with fewer coarse materials (Faramarzi et al., 2018; Kassim et al., 2005). In 2017, Europe and the United States alone generated over 100 million tons of RAP as waste material from rehabilitation projects (Federal Highway Administration, 2008). Utilizing RAP in road construction offers benefits such as improved high-temperature properties, enhanced moisture resistance, better fatigue performance, and a reduced carbon footprint by approximately 50%. It also leads to a 30% reduction in construction costs and promotes the conservation of natural resources (Bizarro et al., 2021; Dughaiishi et al., 2022; Qiao et al., 2019; Yan et al., 2014). However, it is worth noting that RAP usage may lead to higher cracking issues, reduced strength, and a tendency to creep under constant stress (Bleakley & Cosentino, 2013; Willis et al., 2012). The use of asphalt emulsion to stabilize RAP materials further advances these benefits by increasing tensile strength and stiffness. This is achieved through the interaction between the asphalt emulsion residues and RAP during the curing process, facilitating molecular-level exchanges of aromatic molecules (Filho et al., 2020; Tia & Wood, 1983).

In addition to RAP, there are several other by-products that hold potential for utilization in the road industry. One such is crude oil, which is a widely produced material consisting of three phases: gas, liquid, and solid. While the gas and liquid phases have been extensively studied using various chemical analyses, the solid phase, known as asphaltenes, remains relatively unexplored and lacks significant applications in current practices (Mullins et al., 2007; J. G. Speight, 2004). Asphaltenes are the heaviest components and a polar fraction of bitumen. They are characterized by being soluble in toluene, insoluble in n-heptane, and chemically complex, which makes their understanding and treatment challenging (Gharbi et al., 2017; Mullins et al., 2008). Due to their unique properties, asphaltenes has not been fully comprehended, and their potential in the road industry has yet to be fully realized. The polar component of the asphalt binder has the potential to enhance the stiffness and mechanical properties of asphalt mixtures to specific extents (Basavarajappa, 2021). The colloidal index (CI) serves as a valuable indicator for evaluating the stability of the asphaltenes phase within the maltene matrix of the binder. It plays a crucial role in assessing the impact of asphaltenes and is calculated by dividing the weight of asphaltenes and saturates by the weight of resins and aromatics. When the CI exceeds 0.9, it indicates the presence of an unstable asphaltenes phase. Values falling between 0.7 and 0.9 introduce uncertainty regarding stability. Therefore, the CI serves as a useful metric in determining the stability of the asphaltenes component within the binder's maltene matrix (Lesueur, 2009; J. Wang et al., 2019).

Despite the various methods introduced to enhance the performance of granular base layers and address the challenges associated with creating a solid foundation, several unresolved issues persist. Notably, the design of new layers and the lack of established guidelines to consider their behaviour, particularly the impact of temperature, have posed significant challenges (ARRA, 2001; Sabita, 2020; Wirtgen Cold Recycling Manual, 2012). Furthermore, the European Green Deal, negotiated in 2020, has prompted countries to commit to reducing their GHG emissions. Europe, for instance, has pledged to achieve at least a 50% reduction in emissions by 2030 and climate neutrality by 2050, while the United States has committed to an 80% reduction by 2050 (European Commission, 2019). The transportation sector plays a substantial role in these emissions, accounting for approximately one-fifth of the total. Therefore, the adoption of preventive measures such as utilizing waste materials and avoiding

cementitious products becomes an important step in aligning with these environmental goals (Greene et al., 2010).

In summary, a comprehensive analysis of the literature in this field highlights three main gaps that require attention and resolution:

1. The current design approach for asphalt emulsion base layer stabilization and cold recycling materials relies on volumetric and indirect tensile strength test results. However, there is a need to reevaluate this approach and consider performance properties of these mixes to ensure their optimal performance in real-world conditions.
2. Existing pavement design guidelines do not adequately account for the influence of temperature on the behaviour and performance of base layers, necessitating the development of updated guidelines that incorporate this critical factor.
3. Cementitious materials are widely used for stabilization, but they come with disadvantages such as rigidity, high cost, and negative environmental impacts due to their production process. Therefore, there is a need to explore alternative residue materials that can replace cementitious materials while offering better properties. Investigating the potential of residue materials as cement replacements would lead to more cost-effective and sustainable solutions in road construction.

Addressing these gaps through further research, innovation, and the development of new techniques and guidelines will contribute to the advancement of base layer stabilization, environmentally friendly practices, improved pavement design, and the utilization of residue materials in road construction.

1.2. Research Objective

The primary objective of this research was to address the above gaps and improve the design and performance of base layers by focusing on the utilization of asphaltenes and reclaimed asphalt pavement (RAP). To achieve this overarching goal, the following specific objectives were established:

Objective 1. Assessing the impact of incorporating asphaltenes on the performance of base layers stabilized with asphalt emulsion: This objective aimed to evaluate the effects of adding

asphaltenes on the mechanical properties, stability, and durability of base layers stabilized with asphalt emulsion. The research sought to determine the optimal dosage and characteristics of asphaltenes to enhance the performance of these base layers.

Objective 2. Enhancing base layer performance through the utilization of asphaltenes and RAP as residue materials: This objective focused on exploring innovative methods and techniques to effectively incorporate asphaltenes and RAP into base layers. The research aimed to investigate their influence on improving the mechanical properties, load-bearing capacity, and overall performance of the base layers.

Objective 3. Evaluating the impact of temperature on base layers stabilized with asphalt emulsion: This objective aimed to study the influence of temperature variations on the behaviour and performance of base layers stabilized with asphalt emulsion. The research sought to analyze how temperature affects the strength, stiffness, and long-term performance of these base layers, considering their response under different climate conditions.

Objective 4. Investigating the feasibility of replacing cement with asphaltenes in base layer stabilization and road construction: This objective aimed to assess the potential of substituting cement with asphaltenes as a stabilizing agent in base layers. The research aimed to evaluate the performance, environmental impact, and cost-effectiveness of using asphaltenes as an alternative to cement in base layer stabilization, contributing to more sustainable road construction practices.

By addressing these objectives, the research aimed to advance the understanding and utilization of asphaltenes and RAP in base layer design and performance, paving the way for improved road infrastructure with enhanced sustainability and performance characteristics.

1.3. Research Hypothesis

Based on the research objectives outlined and elaborated, the following hypotheses were formulated to investigate during this research:

Hypothesis 1: Asphaltenes can serve as a beneficial additive to enhance the performance of the asphalt emulsion stabilized base layer.

Hypothesis 2: Utilizing asphaltenes in cold recycling process will provide improvement in performance properties of these layers.

Hypothesis 3: Asphaltenes have the potential to be a viable substitute for cement as a residue material with fewer disadvantages.

Hypothesis 4: Modified and unmodified asphalt emulsion stabilized and cold recycled layers are susceptible to temperature and frequency effects, hence the viscoelastic properties should be considered for pavement design.

1.4. Research Methodology

In this study, the project was divided into two phases, and Figure 1-1 presents the flow chart of this research.

Phase 1 involved selecting a range of aggregate gradation based on standard envelopes available. A proctor test was conducted to determine the dry density of several mixtures, and the optimum fluid content and optimum moisture content were chosen prior to the design process. These values helped finalize the optimum emulsion content to be used for performance tests. During the optimization process, indirect tensile strength (ITS) and Marshall stability and flow were used to assess the effect of emulsion content in the mix. Tensile strength ratio (TSR) was employed to evaluate the moisture susceptibility of the mixtures, considering the limitations specified by relevant standards under saturated and freeze/thaw conditions. Since there were no specific recommendations for using asphaltenes a similar percentage for cement was followed and to prepare samples for performance tests, asphaltenes were added at concentrations of 1%, 2% and 3% which was then finalized to 1% and 2% only based on recorded design results. Several samples were then prepared using Marshall, gyratory, and slab compactors. Low-temperature tests were conducted by measuring ITS at 0 °C and -10 °C for both unmodified and modified samples. TSR, ITS, and IDEAL-CT tests were performed to assess intermediate temperature properties, while the Hamburg wheel tracking (HWT) test was employed for high-temperature evaluation. Additionally, dynamic modulus tests were conducted on both modified and unmodified samples to assess the impact of temperature on the mixtures. The research extended beyond investigating the impact of asphaltenes alone. After adding asphaltenes, cement was separately incorporated into the same aggregate gradation, using dosages of 1% and 2%, and the modified mixtures were tested for

similar performance properties. This approach allowed for a direct comparison between the two additives. To assess the performance of the modified asphalt emulsion, rheological tests were conducted on the binder residue using a DSR. This test helped evaluate how the addition of asphaltenes and cement influenced the rheological properties of the asphalt binder. Furthermore, the researchers performed FESEM tests on the modified and unmodified binders. FESEM is a powerful imaging technique that provides high-resolution images of the binder's microstructure. This analysis allowed for a closer examination of the morphological changes and interactions between the additives and the binder, shedding light on the mechanisms that contributed to the observed performance improvements. Finally, an analysis of variance was employed to evaluate the effectiveness of asphaltenes modification on granular base layers.

Phase 2 commenced by utilizing unprocessed reclaimed asphalt pavement (RAP). The physical and rheological properties of this material were determined through grain distribution analysis and the use of a DSR to assess the extracted binders. Optimum fluid and moisture content were then determined using the proctor test, following a similar approach as in phase 1. The design process took into account the available standards for recycled pavements, with ITS measurements under dry, saturated, and freeze/thaw conditions serving as limitations to determine the optimal emulsion content. Based on the findings from phase 1, samples for performance testing were prepared by incorporating 0.5% and 1% asphaltenes content. Following sample preparation using various compactors, including Marshall, gyratory, and slab compactors, the behaviour of the samples at low temperatures was evaluated through ITS measurements at 0 °C, -10 °C, and -20 °C, both for unmodified and modified samples. Intermediate temperature properties were assessed using TSR, ITS, and IDEAL-CT tests, while the Hamburg wheel tracking (HWT) test was employed to evaluate high-temperature performance. Additionally, dynamic modulus tests were conducted on both modified and unmodified samples to examine the impact of temperature on the mixtures. Finally, an analysis

of variance was conducted to assess the effectiveness of asphaltene modification on granular base layers, similar to the approach taken in phase 1.

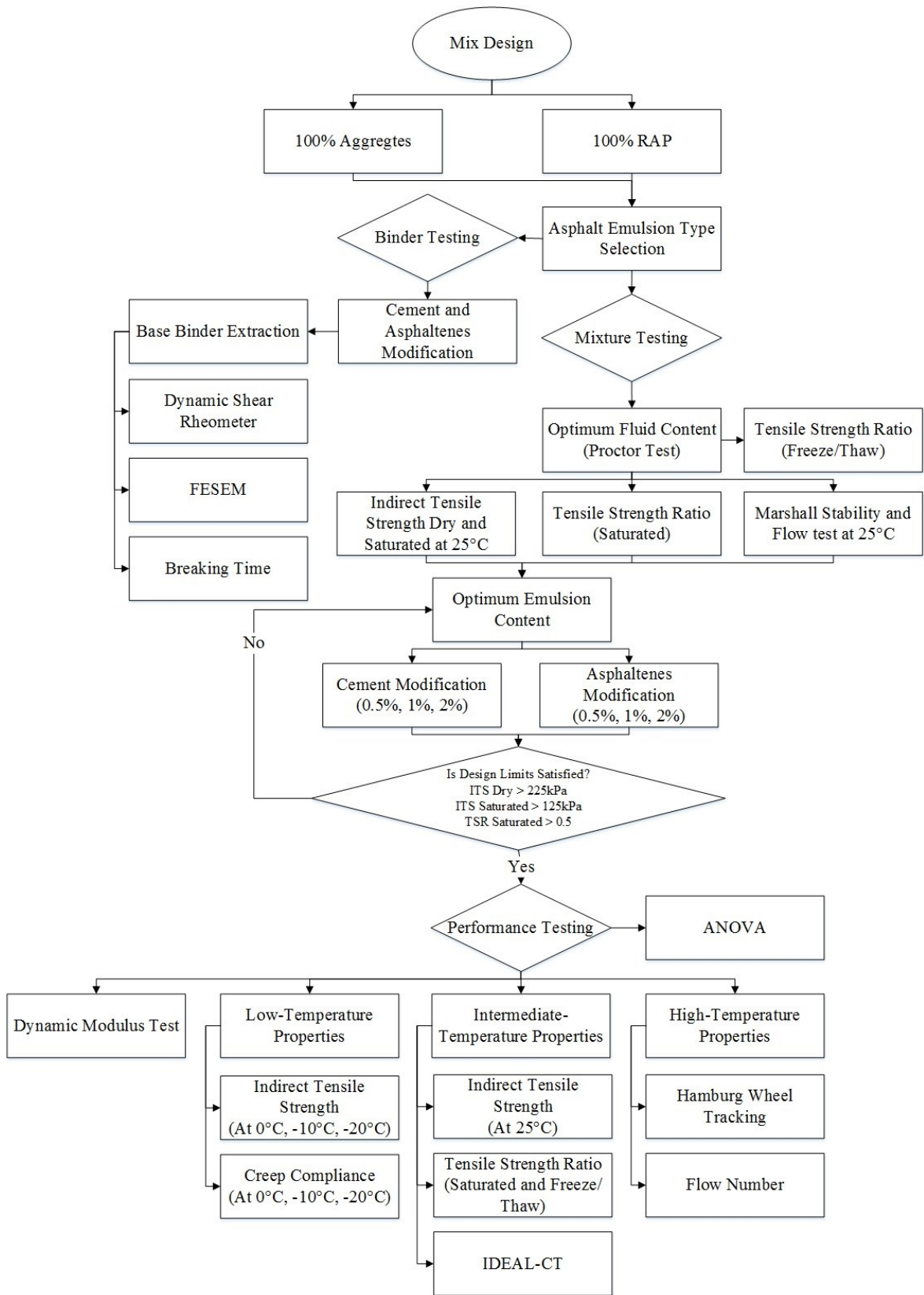


Figure 1-1 Research methodology flow chart

1.5. Thesis Organization

This thesis is structured in a paper-based format, aligned with the research objectives. The chapters are organized as follows:

Chapter 3: This chapter focuses on evaluating the impact of adding asphaltenes to the granular base course stabilized with asphalt emulsion on the performance of these mixes. It assesses the moisture susceptibility, intermediate-temperature behaviour, and high-temperature performance of the stabilized base layer. The selection of a specific aggregate gradation based on existing standards and envelopes is discussed. Various tests, including Marshall stability and flow number, tensile strength ratio (TSR), indirect tensile strength (ITS), and Hamburg wheel tracking (HWT), are conducted to evaluate the properties of the base layer. This chapter addressed the first and fourth objective partly.

Chapter 4: In this chapter, the effect of asphaltenes in the granular base layer is further explored on the performance of the layer. Different percentages and methods of incorporating asphaltenes are investigated, focusing on their impact on low-temperature, intermediate-temperature, and high-temperature properties. Tests such as ITS at low temperatures, HWT at high temperatures, and reporting of chemical properties of the materials are conducted. The optimal percentage of asphaltenes and asphalt emulsion consumption is determined. This chapter addressed the first and fourth objective.

Chapter 5: This chapter delves into the properties of recycling the reclaimed asphalt pavement (RAP) materials using asphalt emulsion and the addition of asphaltenes. The low-temperature, intermediate-temperature, and high-temperature properties of the RAP materials are evaluated through tests including HWT, IDEAL-CT, ITS, TSR, creep compliance and strength tests, dynamic modulus. This chapter sheds light on the behaviour and performance of RAP materials when stabilized with asphalt emulsion and asphaltenes. This chapter addressed the first, third and fourth objective.

Chapter 6: Here, the possibility of replacing cement with asphaltenes in granular base courses stabilized with asphalt emulsion is discussed. Rheological and performance properties are evaluated using techniques such as dynamic shear rheometer (DSR), field emission scanning electron microscopy (FESEM), ITS, TSR, IDEAL-CT, HWT, indirect tensile test (IDT),

dynamic modulus, and flow number. The chapter also examines the breaking time of the asphalt emulsion with the addition of each of these additives. This chapter addressed the first, second, third and fourth objective.

Chapter 7: The final chapter provides a summary of the conclusions drawn from the research presented in the thesis. It discusses the contributions of the research, identifies its limitations, and suggests directions for future studies.

By organizing the thesis in this paper-based format, the research objectives are effectively addressed, and the findings contribute to the understanding and advancement of asphaltenes and RAP in improving the design and performance of granular base layers in road construction.

Chapter 2: Literature Review

2.1. Pavement Structure

In road construction, the structure consists of several layers stacked on top of each other. These layers are commonly referred to as the asphalt layer at the top, the base layer in the middle, and the subgrade at the bottom (Figure 2-1). The main purpose of the base layer is to evenly distribute the load from the vehicles traveling on the asphalt layer down to the subgrade. Each of these general layers may further consist of multiple sublayers to enhance the overall strength and performance of the road (S.-C. Huang & Di Benedetto, 2015).

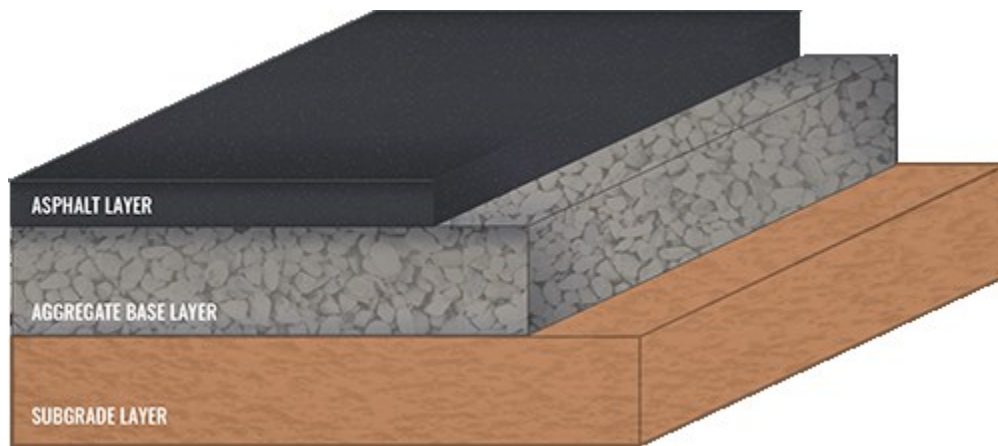


Figure 2-1 Road structure in pavement engineering (Bituminous Roadways, 2023)

Base layers in road construction can be categorized into three types: unbound, bound, and non-continuously bound layers. Unbound layers are composed mostly of granular aggregates, such as crushed stones and gravel. Their primary function is to transfer the load from the upper layers to the subgrade by distributing it through individual particles (S.-C. Huang & Di Benedetto, 2015; Wirtgen Cold Recycling Manual, 2012). Bound layers, on the other hand, utilize cement or asphalt with granular materials to achieve full strength and act as wide beams. As a result, when loaded from the top, these layers experience maximum horizontal stresses at the top and bottom, along with tensile stresses at the bottom of the layer. These interactions can lead to fatigue failure, with vertically propagated cracks from the bottom to the top of the bound layer. The third type of base layer involves granular materials stabilized with asphalt emulsion or foamed bitumen. In these layers, permanent deformation is a primary concern and distress that may occur over time (Wirtgen Cold Recycling Manual, 2012).

2.1.1. Viscoelastic Materials Behaviour

Asphalt binder has both viscous and elastic behaviour. Low temperatures dominates the elastic properties and these properties are responsible for irreversible deformations of asphalt binder. Elastic materials shows strain under the stress and comes back to the original state after removing the load. However, viscous behaviour is more dominated at high temperatures. This will result in a flow of the material under the stress with none recoverable behaviour. Viscoelastic materials such as asphalt binder exhibit both viscous and elastic characteristics undergoing a deformation (Y. Cheng et al., 2019; Mackiewicz & Szydło, 2019; Perilakalathil & Narayan, 2022).

The oscillatory stress and the strain measurement of the materials will be used to analyze the viscoelasticity by a dynamic mechanical analysis. Stress applied to the elastic materials draws an immediate response with a zero degree phase angle. However, viscous materials has a strain lags by 90 degree phase angle with viscoelastic materials having a phase in between the two mentioned (Mills, 2007; Miyake, 2010). In order to present the relations between the oscillating stress and strain, a complex dynamic modulus (G) is used with a G' as storage modulus, and G'' of loss modulus (Bi et al., 2021; Garcia & Thompson, 2007).

Asphalt binder exhibits linear viscoelastic behaviour with high influence of temperature. A large amplitude of strain will results in non-linear mechanical behaviour of the asphalt binder. The glass transition (T_g) is the most important factor in determination of the viscoelastic properties of the amorphous materials (Kriz et al., 2008; Williams et al., 1955). The glass transition is a reversible change in an amorphous domain from a viscous to a relatively brittle glassy state and the other way around and it happens when the characteristic time of molecular motions responsible for structural rearrangements becomes longer than the timescale of the stress. Decreasing the temperature will result in a rapid increase of the timescale for structural relaxation (Moynihan, 1994). An enormous increase of viscosity is the result of transition to glassy state with a sudden change in the mechanical, optical and thermodynamic properties of the materials which results in a brittle and rigid behaviour of materials. In general, lower strains with lower number of cycles lead to linear viscoelastic behaviour and higher strain under lower number of cycles can lead to non-linearity of the materials. This means that permanent deformation happens under an increased number of cycles and high strain levels, whereas the

fatigue failure is caused of higher number of cycles regardless of increased amplitude of the strains (Y. Cheng et al., 2019; Mackiewicz & Szydło, 2019; Perilakalathil & Narayan, 2022).

2.2. Pavement Distresses

Asphalt distress is an important consideration in pavement design. Failure criterion of the pavement should be developed in the mechanistic empirical design method to take care of the specific distress in the pavement (Y. H. Huang, 2004). A distress identification manual should be used to identify the proper distresses with assigning the severity and size for a proper design and effective strategy for maintenance and rehabilitation. Fatigue cracking, block cracking, joint reflection cracking, heave, transverse cracking, water bleeding, rutting, swell, and construction, material, or maintenance related distresses are the main problems related to the pavement (Miller & Bellinger, 2014).

2.2.1. Fatigue Cracking

The fatigue cracking occurs in an area under repeated traffic loading or wheel paths. This cracks could be a series of interconnected cracks in an early stage developments also called alligator cracking. Fatigue cracking is strongly tied to the properties of the asphalt binder in the mix (Norouzi & Richard Kim, 2017; Tschegg et al., 2011). Fatigue cracking shows the asphalt binder and asphalt interfaces behaviours with distinguishing between the long-lasting and short-dated constructions using different performance tests such as three-point and four-point-bending tests (Tschegg et al., 2011).

2.2.2. Block Cracking

The block cracking is a thermal damage occurring in the asphalt pavements. Block cracking is highly affected by traffic volume as an important factor which is a compressive stress from wheel loads healing these cracks. The old low traffic pavements are primarily place for development of block cracking (E. R. Brown et al., 2009; H. Wang et al., 2020). Block cracks divide the asphalt surface to rectangular pieces and it is mainly caused by shrinkage and daily temperature cycling with cyclic stress and strain. This distress is not load associated but can increase the severity by load (Y. H. Huang, 2004).

2.2.3. Thermal Cracking

The thermal cracking or transverse cracking or also called longitudinal cracks are parallel to the pavement centerline and extended across the centerline. Transverse cracking can be caused by a shrinkage of the surface layer because of low temperatures or reflective cracks from the layers underneath and they are usually not load related. The low temperature cracks in the pavement structure creates a conduit for migration of water and fines particles in and out of the pavement. During the winter, entering the water into these cracks and freezing leads to thawing in the pavement and depression at the crack resulting in poor quality and lower pavement service life (Jung & Vinson, 1994).

2.2.4. Permanent Deformation

The wheel paths are the main place of rutting happening in the pavement. A linear, surface depression caused by deformation or consolidation of any pavement layer or subgrade layers. Insufficient pavement thickness, lack of compaction, or weak asphalt mixtures are the reason for the rutting distress in the pavement layers (Y. H. Huang, 2004).

2.3. Performance Tests

2.3.1. Superpave Mix Design

The strategic highway research program (SHRP) developed the Superpave mix design from 1987 to 1993. This research aimed to develop a performance based asphalt binder specification and a mix design system. Performance grade (PG) used to identify the binders is the results of SHRP project (Cominsky, 1994; McCarthy et al., 2016). Based on this research, the attempt to develop a performance based mix design was not very successful, however series of performance tests for asphalt mixtures was developed to predict the mixture response such as strain and stress and performance such as rutting, fatigue cracking, and thermal cracking (Cominsky, 1994). The Superpave PG is based on the idea that asphalt binder should perform based on the conditions which it is under. Existing methods based on penetration and ductility of binders were not very accurate since these tests are empirical and not related to the pavement performance, only one test temperature used for these tests and climate conditions were not considered, and only short-term aging was considered for the testing (Roberts et al., 1991).

The Superpave asphalt mix design uses different steps such as material selection, aggregate structure, asphalt binder content, and moisture sensitivity (Asphalt Institute, 1996). The

Council of Deputy Ministers Responsible for Transportation and Highway Safety introduced the Superpave asphalt design for Canadian roads in 1987.

2.3.2. Mixture Performance Tests

Following the performance graded mix design, asphalt mixes should go under a series of performance mix tests to quantify the mechanistic properties of the mixes prepared. Von Quintus (2009) defined pavement performance as changes in the pavement surface condition over time. Based on this definition, superior performance from a pavement is when it has low surface distresses with a smooth riding surface over the design period. The performance tests evaluate the properties of the mixtures to determine the response of these mixtures for different distresses in the pavement. The key featured performance tests for pavement is introduced in the following and summarized in Table 2-1.

2.3.2.1. Asphalt Pavement Analyzer

Asphalt pavement analyzer is used to determine the rutting susceptibility of the asphalt mixtures. This device is a second generation that was developed in mid-1980s formally named Georgia Loaded Wheel Tester. Although this device can be used for different tests such as fatigue cracking for beams but it is originally designed for measuring the rutting using a loaded aluminum wheel going back and forth on the samples. This test can be used under dry or soaked conditions with slab or cylindrical samples (McCarthy et al., 2016; Skok et al., 2002). This test can simulate field traffic and temperature, simple to perform, 3 to 6 samples can be tested together, the most widely used in USA with widely available standards and guidelines. However, it is relatively expensive (Skok et al., 2002).

2.3.2.2. Hamburg Wheel Tracking

The wheel tracking test is conducted using a wheel-tracking device to run a simulative test by running a rolling small loaded wheel in the specimen repeatedly. Hamburg wheel tracking test is used to evaluate the rutting, fatigue, moisture susceptibility, and stripping predictions. Some of these devices are relatively new and some are updated version of French rutting tester. The Hamburg wheel tracking device was developed in Germany to evaluate the rutting and stripping potential of the slab and cylindrical specimens. HWT test is largely dependent on the performance grade of the binders and use of recycled materials. Higher performance grade will result in lower rutting depth and higher use of recycled materials will result in lower depth

but increased fatigue and low temperature cracking problems (McCarthy et al., 2016). The advantage of using HWT which is widely used in Germany is capable of evaluating moisture susceptibility and can run two samples at the same time but is not widely accepted in USA (Haritonovs et al., 2016; McCarthy et al., 2016).

2.3.2.3. *Superpave Shear Tester*

The Superpave shear tester, developed as part of SHRP research, is capable of conducting four different tests: shear at constant height, frequency sweep at constant height, simple shear at constant height, and repeated shear at constant stress ratio. These tests are described in AASHTO T320 (McCarthy et al., 2016; J. N. Wang et al., 2001).

- Shear at Constant Height Test: This test repeatedly loads the sample in shear and measures cumulative permanent deformation.
- Frequency Sweep at Constant Height: This test measures the shear dynamic modulus, which assesses the stress-strain relationship using continuous sinusoidal shear loading at a constant shear strain.
- Simple Shear at Constant Height: This test measures the maximum shear strain that occurs during the test, along with permanent shear strain at the end of the test.
- Repeated Shear at Constant Stress Ratio: In this test, a specimen is repeatedly loaded in shear, with the ratio of shear load to axial load maintained at a constant value. Cumulative permanent deformation is measured during this test.

This test is sensitive to sample compaction method with samples prepared with gyratory compactor tend to have higher resistance to permanent deformation than extracted cores from field or roller compactor. Timely procedure for this test is a disadvantage that takes up to a week for sample preparation and two days for testing.

2.3.2.4. *Flow Number*

An asphalt mixture performance tester is a machine that is capable of performing several test related to asphalt performance. The flow number test is one the test to be conducted using this device that is repeated constant compressive load on the cylindrical samples under specific temperature conditions with measuring the resulting permanent axial strains for each load

pulse. This test can measure fundamental properties related to rutting and cracking susceptibility of the asphalt mixtures (McCarthy et al., 2016; Withee, 2013).

2.3.2.5. *Texas Overlay*

The Texas overlay test, developed in the 1970s, was originally designed to assess the asphalt mixture's resistance to reflective cracking. Over time, it has been adapted to evaluate resistance to bottom-up fatigue cracking and thermal reflective cracking as well. This test is known for its speed and repeatability, often resulting in the failure of poorly performing samples within minutes. One advantage of this test is its ability to provide results that correlate with both flexible and composite pavements. It is also efficient for samples prepared through gyratory compaction and field cores. However, the sample preparation process can be challenging due to the need for cutting and gluing. The test is conducted at a temperature of 25°C to characterize both crack initiation and propagation properties.

2.3.2.6. *Flexural (Bending) Beam Fatigue*

The determination of the fatigue life of the compacted slab asphalt mixture at intermediate temperatures involves conducting a bending beam fatigue test. Fatigue life is influenced by various factors, including traffic variation, speed, climate conditions, aging, and resting periods between loadings. However, this test simulates the repeated traffic loading experienced during the pavement's fatigue life for the specimen. It is widely accepted in the United States. One disadvantage of this test is the time it can take, which may extend to several days or even months, depending on the chosen strain rate for testing. Additionally, it is more expensive to prepare the samples compared to cylindrical specimens, and the test duration is not ideal for quality assurance control of mix designs for projects with tight timelines (McCarthy et al., 2016).

2.3.2.7. *Disc-Shaped Compact Tension*

The disc-shaped compact tension test is developed to determine the fracture resistance of asphalt mixtures at low temperatures (10°C higher than low PG). Fracture energy is used to evaluate the service life of the mixture under cracking distresses such as thermal, reflective, block cracking. This is easy to conduct with an average specimen preparation of 10 to 15 minutes, which results in about 30 minutes time for testing of each sample including preparation time and relatively cheaper device to obtain.

2.3.2.8. *Indirect Tension Test (IDT)*

Indirect tensile strength test is developed around the 1960s to evaluate the relative quality of the asphalt mixtures for performance and estimating the potential for rutting and cracking. This test was also used to determine the field pavement moisture distresses (Christensen & Bonaquist, 2004). The strength test was done using a constant strain rate until the sample fails by splitting along the diametric axis and horizontal tensile stress is calculated at the center of the test specimen (Blasl et al., 2016; Louw & Jones, 2015; Witczak et al., 2002). In addition, indirect tensile creep and strength test is developed during the SHRP to characterize the resistance of asphalt pavement to low temperature cracking. AASHTO T322 (2020) was developed and reviewed later on with NCHRP 530 report (Christensen & Bonaquist, 2004) for effectivity of this test and prediction of low-temperature properties. A single constant load is used to apply on the sample for 1,000 seconds and horizontal and vertical deformations are recorded.

2.3.2.9. *Thermal Stress Restrained Specimen Test*

The thermal shrinkage during the cooling of the pavement is the reason for low-temperature cracking which results from tensile stresses. The thermal stress restrained specimen test is the function of restraining the deformation in samples while decreasing the temperature with a specific cooling rate. This test measures the tensile load at the failure temperature where the stress on the sample reaches the maximum point before it fractures. The test procedure is based on the hypothesis that the surface layer of the pavement is longitudinally restrained due to the length of the road considered to be infinite (Baumanis et al., 2021; Carter & Paradis, 2010).

2.3.2.10. *Dynamic Modulus Test*

The dynamic modulus was used as a characterization property to assess the asphalt pavement performance, based on its role in mechanistic-empirical pavement design (Jeong et al., 2014; McCarthy et al., 2016). Dynamic modulus values are determined across a spectrum of loading frequencies and temperatures, which can be transformed into a master curve for use in pavement design and performance analysis (McCarthy et al., 2016). This test requires a highly expensive device, high manpower, and high testing proficiency along with longer time for specimen preparation and testing time which makes it a disadvantage of the procedure.

Table 2-1 Performance tests

Test	Purpose of use	Standard
Asphalt Pavement Analyzer	Determining rutting susceptibility	AASHTO TP63
Hamburg Wheel Tracking	Determining rutting and stripping potential	AASHTO T324
Superpave Shear Tester	Characterize a HMA mixture's resistance to permanent deformation	AASHTO T320
Flow Number	Measures fundamental properties related to rutting and cracking susceptibility	AASHTO TP79
Texas Overlay	Determines the bottom-up fatigue cracking and the thermal reflective cracking resistance	Tex-248-F
Flexural (Bending) Beam Fatigue	Characterize the fatigue life of HMA at intermediate pavement operating temperatures	AASHTO T321 ASTM D7460
Disc-Shaped Compact Tension	Determines the fracture resistance of asphalt–aggregate mixtures	ASTM D7313
Indirect Tension Test (IDT)	Measures the low temperature compliance and the fracture strength at low temperatures	AASHTO T322
Thermal Stress Restrained Specimen Test	Determines the low temperature cracking susceptibility of asphalt concrete	AASHTO TP10
Dynamic Modulus Test	Characterize stiffness for the asphalt mixtures at different temperatures and various load frequencies	AASHTO T342

2.4. Asphalt Emulsion

2.4.1. Asphalt Emulsion Fundamentals

Asphalt emulsion is a colloidal dispersion of small droplets of asphalt binder in water, stabilized with the help of an emulsifier. In general, 5-55 μm asphalt dispersed in water composes the asphalt emulsion and water constitutes the continuous phase of the emulsion which results in significant reduction of the original viscosity of asphalt (Gingras et al., 2007; Ronald & Luis, 2016). The coalescence of the droplets is avoided by amphiphile molecule which is called surfactant and it is formulated especially to decrease the interfacial tension

between asphalt and water and emulsify the mix (Ronald & Luis, 2016). The asphalt portion of the emulsion is generally vary based on the application but exist within 50% to 80% (weight percentage). The type and amount of emulsifier used determine the specific asphalt emulsion type and its breaking characteristics. The performance of the emulsion is greatly influenced by the particle size and distribution of the asphalt droplets within the emulsion. However, it's important to note that this emulsion is inherently unstable. Over time, the asphalt droplets within the emulsion will tend to separate from the water, leading to the breakdown of the binder layer (Asphalt Institute, 2008; A James, 2006; Moura et al., 2023). This phenomenon is commonly referred to as "breaking" of the asphalt emulsion. The breaking time of an asphalt emulsion is the duration required for the coalescence of the droplets when the charges on the asphalt binder, introduced by the emulsifiers, begin to diffuse (Asphalt Institute, 2008). As these droplets come into contact with each other, they have enough energy to flocculate, meaning they form clusters, and given enough time, they eventually coalesce, causing the emulsion to break. Once the emulsion has broken, the asphalt binder is released from the water, and the mixture loses its stability. This process is crucial for various applications in the asphalt industry, as it allows the emulsion to effectively coat and bind with aggregates during the construction of roads and other pavement structures. Understanding the breaking characteristics of asphalt emulsions is essential in ensuring proper application and performance of these materials in road construction and maintenance projects. Different types of emulsifiers and control mechanisms can be employed to adjust the breaking time and optimize the performance of the emulsion for specific applications (Asphalt Institute, 2008; A James, 2006).

The properties of the asphalt emulsion (viscosity and stability) is dependent on different factors such as continuous phase properties like salinity and pH, the water/oil ratio, molecular structure or surfactant, size of the droplets, preparation process of the emulsion. Also, application of the asphalt emulsion should be considered for selection of the appropriate properties of the emulsion used such as using high viscosity material for slope stabilization or lower viscosity for irrigation (Ronald & Luis, 2016).

The physicochemical parameters such as surfactant chemical nature, surfactant prevailing affinity, the oil and water phase nature, and temperature are the main parameters that determine the final properties of the asphalt emulsion. Surfactants are the main parts to decrease the

interfacial tension between water and asphalt with dispersing the asphalt during emulsification process. The ionic surfactants form an electrical layers around the asphalt droplets and produce repulsion between the droplets which results in a highly stable emulsion system. A negative hydrophilic-lipophilic deviation is produced to prepare a stable asphalt in water system from several list of cationic, anionic, non-ionic, and even amphoteric surfactants (Ronald & Luis, 2016). Another method for preparing emulsions takes advantage of the precipitation process. Asphalt components can be separated into two main groups: asphaltenes and maltenes. Asphaltenes are soluble in aromatic hydrocarbons but insoluble in low molecular weight paraffins. Additionally, asphaltenes constitute the discontinuous phase of asphalt, with maltenes (comprising resins, aromatics, and saturated compounds) serving as the continuous phase. Resins, found in between, act as intermediates and play a role in homogenizing the asphaltenes into the saturated and aromatic compounds (Salou et al., 1998). Asphaltenes and resins are more acid and basic groups with polar properties and exhibit more interfacial properties. Asphalt in water can result polar fractions to be attracted to migrate to the liquid-liquid interface and act as natural surfactants but asphaltenes and saturated compounds tend to destabilize the emulsion with resins and aromatic helping with emulsification (Ahmed & Al-Sabagh, 1999). In conclusion, asphalt emulsion can be produced by pH change and activating the natural surfactants in asphalt studied by different researchers (Corbett, 1969; Sleem-ur-Rahman & Dwivedi, 1997). This will result in an important factor of resins/asphaltenes ratio in the asphalt with asphalt containing higher asphaltenes and paraffins being hard to emulsify (Holleran & Holleran, 2015; Ronald & Luis, 2016; Salou et al., 1998).

2.4.2. Asphalt Emulsion Classification

In general asphalt emulsions are classified in three major categories of anionic, cationic and nonionic in practice with first two being widely used in road construction based on the materials types used. This classification is based on the surfactant type and electrical charges around the asphalt particles (Asphalt Institute, 2008).

Another classification for emulsion is based on their speed of breaking and setting. These classification are called rapid setting (RS), medium setting (MS), slow setting (SS), and quick setting (QS). The tendency of the emulsion to coalesce is related to the speed of emulsion becoming unstable and break. In addition, different parameters was added to this identification

for more clarifications. The letter “C” was used at the beginning of the speed classification to show the charge of the emulsion as cationic which results in emulsion identification with no “C” considered as anionic type. Furthermore, numbers was added to the identification showing the relative viscosity of emulsion as MS-2 being more viscous than MS-1. The letter “h” is identifier for hard base binder used to produce the emulsion and “s” for softer base binder. Additionally for emulsion with more gel quality prepared by adding certain chemical for production of thicker asphalt film on aggregates to prevent drain off, “HF” or high-float asphalt emulsion is introduced. Several other letters such as “P” as in polymer modification, “L” as in latex polymer modification is introduced (Asphalt Institute, 2008).

2.4.3. Asphalt Emulsion Modification

Asphalt is a byproduct of the crude oil refining process and has been a staple in road construction for many years due to its chemical composition, which makes it highly soluble in all derived fuels. However, this solubility has been a primary cause of various premature distresses, especially in areas prone to accidental fuel spills. These distresses include surface softening, raveling, and deeper damages. To address this issue, coal tar pitches were introduced as an alternative, but their widespread use was hindered by environmental concerns associated with tar. Subsequently, additional solutions were proposed, such as polymer modification, aimed at enhancing the fuel resistance of asphalt binders. However, not all polymer modifications proved equally effective, and some carried disadvantages. Further information about the polymer modification was also provided through different studies and patented (Maldonado et al., 1977; Thornton, 2006). Later, this modification approach transitioned to asphalt emulsions, offering improved workability and broader application possibilities. As an example polymer modified asphalt emulsion by styrene-butadiene rubber (SBR) can only improve the low-temperature crack resistance of the mixture but cannot have better performance at high temperatures (An et al., 2006). Similarly, modification with water-based epoxy resin is only beneficial at high temperature performance (C. Y. Cheng, 2002). Asphalt emulsion with advantages such as cold condition application, higher adhesion and improved mixture uniformity can save about 10% to 20% asphalt binder, extended construction season, and lower GHG emissions but it still has problems such as higher aging potential and temperature sensitivity unlike hot asphalt binder (Abd El-Rahman et al., 2017; Feng, 1998). Therefore, asphalt emulsion modification for improving the flexibility and plasticity in low-

temperatures, improved strength and thermal stability at high-temperatures, enhanced antiaging properties, and higher bonding strength was researched and introduced using different types of polymer modified binder and emulsified (Abd El-Rahman et al., 2017).

The modification of the asphalt emulsion is a common method to improve the physical properties, performance and durability of the emulsion. Different modifiers such as natural rubber and latex, synthetic rubber and latex, block copolymers, reclaimed rubber, plastics and polymer blends can be used to modify the base binder and consequently the asphalt emulsion produced to improve the properties of the asphalt emulsion (Johnston & King, 2008; Kamal et al., 2023). Polymer modifiers are mostly categorized in elastomers and plastomers based on their strain performance and low temperatures. Elastomeric polymers has low elastic modulus that allows the matrix to expand with no failure up to 10 times of its original dimensions (Caltrans, 2003). Elastomers include natural and synthetic rubbers, styrene-butadiene-styrene (SBS), and reclaimed crumb rubber modifiers from scrap tires. However, plastomers has higher strength with a rapid rate along with brittle and resistance to deformation after setting (Johnston & King, 2008). Polyethylene, ethylene-propylene-diene- monomer and ethyl-vinyl-acetate are commonly used plastomers. Comparing the properties of the modified asphalt emulsion and modified asphalt binders shows poor adhesion to aggregates and requirement for using anti-stripping agents for SBS-modified hot mixes in compare to modified asphalt emulsion (Serfass et al., 1992). Moreover, the use of anti-stripping agents are not effective under more adverse climatic conditions for hot mix binders. In addition, other studies show longer application seasons for modified asphalt emulsion. However, modified asphalt emulsion requires longer setting time (Airey et al., 2002; Serfass et al., 1992). Upon the success of polymer modification for hot asphalt binders, asphalt emulsion modification was took interest with producing the asphalt emulsion using the modified binders increasing the properties with lower energy consumption, low carbon emission, and greater workability (Kamal et al., 2023).

A study by Valencia et al. (2007) showed 31% increase in compressive strength of cold mix asphalt using polyvinyl acetate modified asphalt emulsion. Zhang and He (2011) reported increase softening point and mechanical property of asphalt emulsion modified with water-epoxy resin emulsion and styrene butadiene rubber latex. Furthermore, Qinqin et al. (2011) showed a better performance for styrene butadiene rubber latex modified asphalt emulsion

compared to unmodified asphalt emulsion. Kamal et al. (2023) reported the 5% latex modified asphalt emulsion to be the highest quality of the latex polymer modification showing longer adhesion to base and aggregates. In addition, a study conducted by Abd El-Rahman et al. (2017) modified the asphalt emulsion with different types of polymers such as epoxy resin, latex SBS, poly vinyl acetate, and their combinations. Evaluating the physical properties, chemical resistance, chemical durability after thermal cycling, and rheological properties showed higher storage stability, increased softening point, improved kerosene and acid resistance, with improved curing and thermal durability of the asphalt emulsions.

2.5. Asphaltenes

Asphaltenes are a by-product of the deasphalting process of oil sands bitumen, and they are considered residue materials with no significant use in the road industry. The presence of asphaltenes in asphalt can be challenging due to their complex nature and limited compatibility with the asphalt matrix. On the other hand, asphalt is a viscoelastic material composed of polar and non-polar components. The polar components are responsible for the elastic behaviour of the asphalt, providing it with the ability to deform and recover when subjected to load and temperature changes. These polar components contribute to the elasticity and flexibility of the asphalt, allowing it to withstand traffic loads and temperature fluctuations without permanent deformation. In contrast, the non-polar components of asphalt are responsible for its viscous response, determining its flow and resistance to deformation over time. The balance between the elastic and viscous behaviour of asphalt is crucial for its performance as a road construction material (Ramirez-Corredores, 2017; Sultana & Bhasin, 2014). Asphaltenes is the heaviest and most polar component in the crude oil as they consist of various polyromantic structures with aliphatic side chains that increases the crude oil's viscosity and density and responsible for the brown or black color (Hussein, 2023; James G. Speight, 2006; Vargas & Tavakkoli, 2018). Asphaltenes also contain heteroatoms such as sulfur, nitrogen, and oxygen, as well as metals like nickel, vanadium, and iron. These components contribute to their chemistry, rendering it significantly more complex and distinct from that of waxes (Becker, 1997; Bellarby, 2009; Kelland, 2009). Asphaltenes is defined by their solubility characteristics, being the fraction of oil that remains insoluble in light aliphatic hydrocarbons like pentane and heptane, yet soluble in aromatic solvents such as toluene (Hussein, 2023). In a broader context, asphaltenes are considered insoluble in non-polar solvents with a surface tension lower than 25 dyne cm^{-1} at

25°C. Nevertheless, they exhibit solubility in liquids with a surface tension above 25 dyne cm^{-1} at 25°C including substances like pyridine, carbon disulfide, carbon tetrachloride, and benzene (Hussein, 2023; J. G. Speight, 2004). Asphaltenes is materials that can create issues at various stages of the oil production system, leading to challenges like formation damage, flow restrictions, and equipment failures. These problems can result in production losses and additional expenses associated with mitigation and maintenance efforts. It will coagulate and block the flow of oil since called cholesterol of crude oil similar to cholesterol in blood veins (Hussein, 2023).

Asphaltenes precipitation was approached with a regular solution theory first applied by Hirschberg et al. (1984) as a single component. Later on another approached was used with a modified model of Scott and Magat (Scott, 1945; Scott & Magat, 1945) by Kawanaka et al. (1991) for asphaltenes precipitation using a molar mass distribution. Recently, asphaltenes was treated as a mixture of components of different density and molar mass (Yarranton & Masliyah, 1996). In addition, another researcher extended Yarranton and Masliyah's model and Hildebrand and Scott (Hildebrand et al., 1950) to asphaltenes precipitation from Western Canadian heavy oils and bitumen (Alboudwarej et al., 2003).

The asphaltene is believed to improve the elasticity and stiffness of the binder behaviour (Mazalan et al., 2023). Asphalt binder on the other hand, is defined as a composite of two principle components with n-heptane solvent extraction as asphaltene insoluble fraction and maltene soluble fraction (Merino-Garcia et al., 2009). The maltene are separated to three phases of saturates, aromatics, and resins (Keshmirizadeh et al., 2013; Lesueur, 2009). Increasing the asphaltene content in asphalt binder will results in increased hardness and viscosity of binder (Hunter et al., 2015). In addition affecting the ageing sensitivity of binder (Neumann & Braun, 1981). Different studied investigated the impact of asphaltene on the linear viscoelastic behaviour of binder using dynamic shear rheometer for a link between the complex modulus at different temperatures (Hofko et al., 2015; Weigel & Stephan, 2016) which reported the stiffness of asphalt binders affected by the composition and molecular weight (Mazalan et al., 2023). The incorporation of asphaltene in asphalt material for base course stabilization offers both cost-effectiveness and a stiffening effect, potentially enhancing the properties and cost performance of the stabilized mixture. By utilizing residue asphaltene

in this manner, it not only provides a viable solution for their disposal but also contributes to sustainable and environmentally conscious practices in road construction.

2.6. Reclaimed Asphalt Pavement

Reclaimed Asphalt Pavement (RAP) refers to the 100% asphalt material recovered from existing pavements through processes like milling or full-depth removal. It contains well-graded aggregates coated with asphalt binder, making it a valuable resource for sustainable pavement practices (FHA, 2016; Wirtgen Cold Recycling Manual, 2012). The RAP material can be utilized in various pavement applications, contributing to resource conservation, and reducing the need for new materials. Some common uses of RAP include: 1- Reconstruction: RAP can be incorporated into new pavement constructions, helping to reduce the demand for virgin aggregates and binder. 2- Resurfacing: It is often used as a component in the overlay or resurfacing of existing pavements, providing cost-effective and environmentally-friendly solutions. 3- Incorporation with Granular Materials: RAP can be mixed with granular materials to create recycled base or subbase layers, improving the overall strength and stability of the pavement structure. 4- Stabilized Materials: RAP can be combined with stabilizing agents, such as cement or lime, to create a strong and durable base layer for pavements. 5- Hot or Cold In-Place Recycling: In these methods, RAP is processed on-site and blended with new materials, binder, and rejuvenating agents to improve the properties of the asphalt binder. This leads to a sustainable and cost-effective pavement rehabilitation technique. By using RAP in these applications, engineers and pavement professionals can embrace sustainable practices, reduce waste, and conserve natural resources, while still maintaining or improving the performance of the pavement structures (Bleakley & Cosentino, 2013; FHA, 2016).

Vassaux et al. (2023) developed a novel infrared methodology to assess the degree of blending with cold recycled asphalt with bituminous or bio-based binders. RAP with 100% was used to prepare the mixes with 4.51% aged binder and about 3% additional binder. As the conclusion of this study, binder homogeneity was efficient at 3 months for cold mixes with 100% asphalt due to the curing process and higher concentration in non-oxidized virgin binder in the mix. Bio-based binder, remobilization was increased in 3 months but no completed.

2.7. Cold Recycling and Asphalt Emulsion Stabilization

Constructed roads are continually subjected to various types of distress, necessitating regular rehabilitation to maintain their functionality and safety. Cold and hot recycling are two major methods used to address these issues over time, reducing the need for new road construction and the consumption of new materials (Filho et al., 2020; Hilbrich & Scullion, 2008). Cold recycling involves reusing asphalt materials obtained from deteriorated layers. These materials are treated with binding agents and laid down on the road without the need for heating. This process rejuvenates the aged binder and provides a more sustainable approach to pavement maintenance. The choice of cold recycling method depends on the specific types of pavement failure encountered (Dunning & Turner, 1965; Wirtgen Cold Recycling Manual, 2012). For distresses limited to the top layer of the pavement that do not infiltrate the base layer, surface recycling with 100% RAP is an effective solution. However, if the pavement deterioration extends into the lower layers, a combination of RAP and granular material is recommended (Filho et al., 2020). By employing cold recycling techniques tailored to the extent and nature of the distress, road authorities can effectively manage pavement issues, extend the lifespan of the road infrastructure, and promote environmentally friendly practices by reducing waste and utilizing recycled materials.

2.7.1. Cold Recycling

Cold recycling is incorporated with a wide range of stabilizing agents such as modified waxes, natural and synthetic polymers, cementitious materials, and asphalt materials depending on the material characteristics, price, availability, durability and policy of the project jurisdiction. Comparing the unbound granular materials, HMA, asphalt emulsion stabilized, and cement stabilized materials suggests that these methods have structurally different responses (Wirtgen Cold Recycling Manual, 2012). Unbound materials are more prone to shear and permanent deformation due to their stress-dependent behaviour. However, adding a stabilizing material bind the particle together and has different load bearing behaviour. HMA as a fully bound mix acts more like a slab providing a cone like loading distribution. On the other hand, stabilized materials such as asphalt emulsion, provides a non-continuously bound material which results in lower fatigue cracking compared to the cement stabilized material. Stabilization with cement additionally provides rigidity and tends to have shrinkage problems producing block cracking in layers with a bottom-up failure (Sabita, 2020; Wirtgen Cold Recycling Manual, 2012).

Several researchers such as Kim & Lee (2012) has studied the use of cold recycling in road rehabilitation. In this study, a cold in-place recycling (CIR) was prepared with a specific mix design method. Samples were tested for dynamic modulus, flow number, flow time, and raveling test. The results of these tests helped to determine the optimum emulsion content for CIR, and two different asphalt emulsion compared for short term and long-term performance along with raveling loss of two binder material.

Studying the incorporation of RAP with asphalt emulsion stabilized base is studied by Andrews et al. (2023). They used different dosage of RAP with granular aggregates (0%, 25%, 50%, 75%, and 100%) to evaluate the dry density, dry ITS, we ITS, TSR, and rutting resistance. RAP material was also characterized using fragmentation test and cohesion test before blending. Conclusions from this research suggested that physical properties of RAP can affect the blending, cohesion and fragmentation tests. Asphalt emulsion consumption decreasing with higher RAP content and performance slightly increasing up to 75% RAP.

Fatigue cracking is a critical consideration when using 100% Recycled Asphalt Pavement (RAP) in cold recycling applications. Tavassoti et al. (2022) conducted a study to evaluate the fatigue performance of cold recycled mixes through uniaxial tension-compression testing. The research matrix included three cold recycled asphalt mixes: one with a regular cationic slow-setting asphalt emulsion, another with the same emulsion but incorporating 1% Portland cement for stabilization, and the third mix utilizing a polymer-modified asphalt emulsion. The goal was to assess the effect of these additives on fatigue performance. The results demonstrated that the addition of 1% cement significantly improved the fatigue performance compared to the mix with regular cationic asphalt emulsion alone. Furthermore, the inclusion of polymer modification in the emulsion also enhanced the fatigue performance when compared to the regular emulsion mixes. To further assess the impact of polymer modification, the researchers conducted multiple stress creep recovery tests on the residual emulsion binder. The results indicated a potential increase in rutting resistance for the polymer-modified mixes. These findings are of significant importance for the cold recycling industry, as they provide valuable insights into improving the fatigue resistance of recycled asphalt mixes.

2.7.2. Asphalt Emulsion Stabilization

Stabilization of the soil with bitumen can be done using two major additives namely asphalt emulsion and foam bitumen. Both additives agents are made from relatively soft grade binder and the main features of these mixes are significant increase in cohesion compared to granular material with no significant reduction in friction angle, flexural strength due to viscoelastic properties of asphalt, and improved moisture resistance and durability compared to granular materials (Kowalski & Starry, 2007; Sabita, 2020; Wirtgen Cold Recycling Manual, 2012). On the other hand, asphalt stabilization has disadvantages such as high cost due to price of active filler and asphalt, lack of design expertise due to specific behaviour of these material that is not well understood, specific construction expertise, moisture content of untreated material, and asphalt emulsion stability and formulation (Sabita, 2020). Additionally, longer curing times is a considerable disadvantage of asphalt emulsion stabilization that might take up to 3 years for the layer to reach final states of the strength (Du, 2018; Marais & Tait, 1989; Quick & Guthrie, 2011). This will result in low early-stage performance results of asphalt emulsion stabilized mixes. In order to address these issues, active fillers such as lime and cement are recommended to be added on asphalt emulsion stabilization (Du, 2018; Fang et al., 2016). Figure 2-2 presents the performance comparison between different agents used to stabilize the soil considering the resistance to permanent deformation and moisture resistance.

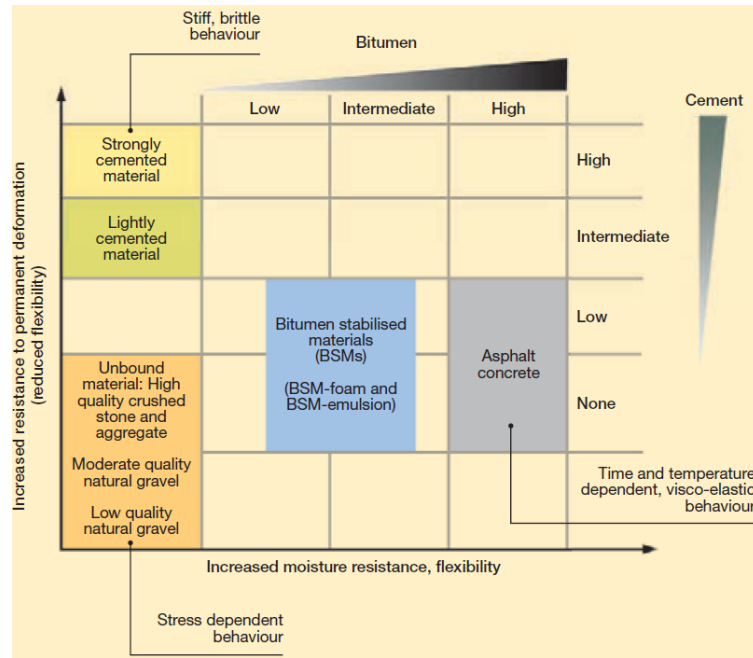


Figure 2-2 Pavement material behaviour and stabilizing agent selection (Wirtgen Cold Recycling Manual, 2012)

Finberg et al. (2007) reported the result of a case study in Las Vegas, Nevada for a granular base stabilization with emulsion. One of the major avenues in Las Vegas was distressed in need of reconstruction. However, decision makers had to reconsider the construction of a new road with a method more timely and low cost. Stabilization of the base layer with a top asphalt layer was selected in this method. The rehabilitation process started by milling the road for about 150mm, lowering the utilities, regarding the aggregates, and adding water for OMC before asphalt emulsion and compaction. As the result of replacing asphalt emulsion stabilization with conventional methods, city saved about \$322,661 (30% cost saving) and 80 days of construction days from 120 days. There was a huge amount of reduction in waste materials (3000 trucks) and less impact on the businesses around the project.

Baghini et al (2015), studied the effect of asphalt emulsion and cement as additive to stabilize the base course materials. Long term properties of these mixes were studied using unconfined compressive strength, flexural strength, wetting and drying, soaked and unsoaked California bearing ratio, dynamic creep, and wheel tracking tests. As the results of this study, cement and asphalt emulsion were presented an effective additives for increasing the strength and permanent deformation of the mixes.

Barbod et al (2014) investigated the laboratory performance of an anionic slow setting asphalt emulsion treated base layer with a dense graded gravel for cold region application. Different curing methods were used to analyze the early strength gain of these mixes, along with dynamic resilient modulus and permanent deformation were performed. As a result, high resilient modulus were observed and deformation was lowered after treating the material with asphalt emulsion. Low temperature at the same time slowed the breaking process of the emulsion causing a lower resistance to repeated loading test.

During the years due to the disadvantages of the asphalt emulsion alone in the stabilization, modification of this binder grows an increasing attraction. Meng et al. (2023) researched different methods used to modify the asphalt emulsion and its performance effects. Commonly used modifiers such as styrene block polymer, polyurethane, styrene butadiene rubber, and waterborne epoxy resin was investigated for effect of modifiers, preparation process, auxiliary additives, and other factors on the performance of modified asphalt emulsions. Result of this research showed that modifiers significantly affect the performance of mixes. Emulsifiers play a great role in determining the breaking speed, adhesion, and stability.

Yan et al. (2017) investigated the early-age strength and long term performance of the asphalt emulsion cold recycled mixes with different cement contents as an additive. Haveem cohesion and raveling tests were used for determining the early-age strength, tensile strength ratio (TSR) for moisture susceptibility, Hamburg wheel tracking test for high temperature properties, and three point bending test was used to determine the low-temperature properties of these mixes. Results from this study showed that addition of cement positively affects the early-age strength and long-term performance of the mixes and a strong linear correlation was proposed between these two factors of the mixes.

Several agencies prepared mix design procedures for asphalt emulsion stabilization and three major ones are Asphalt Institute (2008), Asphalt Recycling and Reclaiming Association (2001), and Southern African Bitumen Association (Sabita) (2020).

2.7.2.1. Asphalt Institute Method

Asphalt institute (MS-19) has introduced the mix design method starting with selection of aggregates type and gradation, asphalt emulsion type suitable for the aggregates. Two design

methods for dense graded and open graded aggregates type is introduced with the requirements for each type of gradation explained.

For dense graded aggregates, SS and MS types of emulsion is recommended with MS used for aggregates having no excessive materials passing a No. 200 mesh sieve. The selection of the asphalt emulsion content is based on the application of the mixture in base or surface mixture according to two different equations and two initial test is required to move forward. The coating and adhesion testing for the mixtures prepared with procedures explained the standard is necessary for this methods (refer to Asphalt institute (2008) for more information). The design step was followed with preparation of minimum three specimens with at least three different emulsion contents one below and one above the determined emulsion content. Each mix is prepared approximately with 1200grams of dry aggregates before adding optimum water content or compacted specimen for 63.5 mm. Samples then compacted using a Marshall hammer with 50 blows on each face and cured at 60°C oven for 24 hours before calculating the volumetrics and Marshall stability test at 25°C in order to determine the optimum emulsion content. The limit for the Marshall stability of the prepared samples determined to be 2224 N or greater for be satisfactory for most of the pavements with low to moderate traffic volumes.

The open graded aggregates mix designs followed similar material preparation procedures using specific table dedicated for the aggregated gradation and specific mixing procedure was followed to determine the optimum emulsion content. In order to determine the optimum emulsion content, the mixture with asphalt residue runoff of 10 grams is selected after adding the aggregates and emulsion at 60°C. The mixture prepared should be transferred to a No. 8 mesh sieve for 30 minutes to drain. The retained mass over the sieve is used to evaluate the coating properties with passing materials dried to a constant mass for calculating the asphalt residue runoff.

2.7.2.2. *Asphalt Recycling and Reclaiming Association Method*

ARRA (2001) followed general steps to prepared the bituminous stabilized mixes for both asphalt emulsion and foam asphalt. First, determining the suitability of the reclaimed materials for the process based on the specification outlined in the manual is required. Secondly, optimum moisture content and optimum fluid content of the materials using proctor test is

needed. Third, optimum binder content is calculated and finally mechanical properties of the mixtures is evaluated.

The gradation and plasticity index are assessed to determine the appropriate asphalt emulsion. In this method, it is recommended that materials passing through a No. 200 mesh sieve fall within the range of 5% to 15% to prevent the formation of agglomerated fines. A higher plasticity index can lead to the presence of cohesive fine particles, which should be avoided.

The optimum fluid content of the materials is determined using a standard modified proctor or similar methods (moisture-density test) with the highest dry density mix having the OFC in the mix. The compatibility of the materials should be determined with a simple coating and adhesion test and assessing the coating degree of the mixtures.

Determining the optimum binder content is based on adding different percentage of the emulsion and water to the oven dried aggregates and compacted using a Marshall or Haveem or other methods. The curing method for these samples consist of 24 hours at room temperature and additional 72 hours at a 40°C oven before using Marshall stability, Haveem stability, or indirect tensile strength at 25°C for strength. For evaluating the moisture sensitivity of the samples a 24 hours of soaking in a 25°C water is recommended before testing the samples with similar strength test and comparing them to dry strength. The highest soaked strength value samples are determined to be the design binder content to be used for further performance tests.

2.7.2.3. *Southern African Bitumen Association (Sabita) Method*

Sabita (2020) provides the most recent mix design for asphalt emulsion stabilization. This method considered different measures such as distress mechanisms and mix types for the process. Permanent deformation and moisture susceptibility are considered as two fundamental distress in this method with binder type selection based on traffic, climate, supporting layers, quality of available aggregates, and economics. In addition, different materials to be stabilized are evaluated from selection from virgin aggregates, recycled granular layers, and reclaimed asphalt with each one to be characterized before using with different tests such as soaked CBR, plasticity index, and gradation. This guidelines determined a minimum mixing temperature to be 10°C for the aggregates without compromising the emulsion dispersion in the mix. The emulsion selected was based on the aggregates types, breaking rate, asphalt emulsion grade,

and category. The OFC should be determined using the equation provided in the guidelines using the maximum dry density method.

Steps following for the preparation of the mixes was started with adding the water at OFC and emulsion to the aggregates and mixing for specific time for a homogenous material. A compaction method is used to prepare the samples with simulating the field compaction in the laboratory with curing the samples an oven for 72 hours at 40°C. More curing is required for the specimens that are not reached to a constant weight.

Determination of the optimum emulsion content is based on indirect tensile strength and triaxial tests. The samples prepared for each emulsion contents are recommend to be minimum of six and tested under dry and soaked conditions. Three samples are to be soaked in a 25°C water for 24 hours prior to test for tensile strength and three more to be test in dry condition s at 25°C. Optimum emulsion content is selected based on the specimens with minimum dry strength of 225kPa and soaked strength of 125kPa whichever has higher values. These minimum limits are changed for different asphalt emulsion types. Furthermore, a triaxial test is used to determine the shear parameters, cohesion, and internal friction angle of the samples for critical performance properties to be evaluated for a reliable mix design in both dry and soaked conditions.

Cement Stabilization and Cement as Active Filler in Asphalt Emulsion Stabilization

Cement stabilization has garnered significant interest among researchers in the past decades. The hydration of particles creates interlocking crystalline bonds between the stabilized materials, resulting in increased compressive and shear strength (Amhadi & Assaf, 2019). Various types of cements are available, offering a broad range of stabilization options for different soil types. However, the use of cement for stabilization raises concerns due to its negative impact on the environment. Cement production in plants is associated with high carbon emissions, contributing to environmental challenges like global warming. Additionally, cost considerations also question the widespread use of this material. While cement stabilization offers notable engineering benefits, it is essential to address its environmental implications and explore sustainable alternatives (Barcelo et al., 2014; Bualuang et al., 2021; Firoozi et al., 2017; Lu et al., 2023; Shah et al., 2022). Research and innovation in low-carbon

cements and environmentally friendly stabilization methods are crucial to strike a balance between engineering advantages and eco-conscious practices in soil stabilization.

The alternative use of cement as an additive to asphalt emulsion stabilized base layers has shown promising benefits. Incorporating active fillers like cement and lime can enhance various properties of the asphalt emulsion layers, including retained strength, stiffness, rutting performance, moisture resistance, and asphalt-aggregate adhesion in the mixes. The addition of active fillers, such as cement, can result in higher strength gain at the early stages of construction. It also leads to decreased breaking and curing time, which contributes to the improved performance and durability of the stabilized layers. Terrell et al. (1971) conducted a study on adding cement as an additive to asphalt emulsion stabilized layers. The research demonstrated that incorporating cement led to notable improvements in curing time, resilient modulus, and overall performance. The optimum amount of cement used in the study was found to be about 1% to 2% of the mixture.

Romeo et al. (2018) studied the effect of active fillers on the cracking resistance of asphalt emulsion stabilized recycled aggregates. Nine types of mixes were used in this study with different blends of cement, lime, and common filler on three experimental road sections. Indirect tensile strength test was conducted on cored samples from these sections in addition to a digital image correlation coded in-house. The results of this study showed an increase in crack resistance for mixtures with active fillers and damages were localized in these mixtures. The optimum values for active fillers were determined to be 1% cement and 2% lime.

Xu et al. (2011) conducted a research on asphalt emulsion stabilized cold recycling mixtures with addition of Portland cement as an active filler. The ITS, bending beam test at low-temperature, dynamic stability rutting were used to evaluate the performance of mixtures with different dosages of cement. The result showed an increase in ITS with higher contents of cement, higher moisture resistance, higher rutting resistance, and higher maximum bending strength. On the other hand, maximum bending strain was decreased. The increase of cement was beneficial for initial strength gain and rutting resistance, but low temperature properties were negatively affected leading to brittleness at low temperatures and optimum cement content was suggested to be around 1.5%.

Barcelo et al. (2014) conducted a study on the effect of cement production on carbon emissions. The research revealed that the manufacturing process of cement generates a significant amount of carbon dioxide, contributing to carbon footprints and environmental concerns. In line with the International Energy Agency's proposal for reducing carbon emissions, addressing this issue necessitates the implementation of substantial reduction plans. To mitigate the environmental impact of cement production, researchers and industry experts have explored various strategies. One approach involves replacing cement with alternative materials that have lower carbon footprints and are more environmentally friendly. Additionally, advancements in production technology, such as utilizing alternative fuels, biomass, and clinker substitution, can significantly reduce carbon emissions associated with cement manufacturing. Addressing the environmental challenges posed by cement production is essential to achieving a more eco-conscious and environmentally responsible approach to infrastructure development and construction projects.

In the study conducted by Firoozi et al. (2017), the researchers focused on addressing the challenges associated with sulfate-rich soils stabilized using cement or lime. These soils often experience significant heaving, leading to pavement failure. The study found that cement or lime treatment can help alleviate certain issues related to soil distress, such as desiccation, swelling, and shrinkage. However, soils that undergo wetting and drying cycles can experience a loss of cohesion, which poses additional challenges. To address this specific problem, the researchers recommended the use of fiber treatment.

The resistance to shrinkage cracking in cement-stabilized aggregates is a critical consideration, and Zhang et al. (2023) conducted a study to investigate this aspect by predicting crack spacing. Field data was collected from several expressways in China, and the maximum crack spacing was utilized to assess the shrinkage crack resistance. The findings of this study revealed that cement stabilization led to a significant reduction in shrinkage cracking, resulting in a 30% decrease in crack occurrence. Furthermore, the study highlighted that proper vibration compaction further improved this shrinkage crack resistance compared to static pressure compaction.

2.8. Pavement Design for Asphalt Emulsion Stabilization

The reliable performance of the asphalt layer hinges on a proper mix design and formulation. It is crucial to conduct a thorough analysis of the ingredients in asphalt emulsion stabilized materials. A complete mix design requires optimization in terms of volumetric properties, workability, compactability, and the consideration of engineering properties and durability (Sabita, 2020). Various guidelines have been developed to provide comprehensive design methods for asphalt emulsion stabilized materials. These include the Asphalt Institute (2008), Asphalt Recycling and Reclaiming Association (ARRA, 2001), and South African Guidelines (Sabita, 2020). While these guidelines offer valuable insights, none of them are entirely complete and did not fully address a behavioural design approach. At some point, the Mechanistic-Empirical Pavement Design Guidelines (MEPDG) were used to design asphalt emulsion stabilized layers. However, due to the lack of information on the viscoelastic behaviour of these layers, they were not considered as typical granular layers or conventional asphalt layers. To enhance the effectiveness of mix design for asphalt emulsion stabilized materials, further research and comprehensive behavioural design approaches are needed. Understanding the viscoelastic properties of these layers can lead to more accurate and robust design guidelines, ultimately improving the performance and longevity of asphalt emulsion stabilized pavements.

Sabita (2020) as the most recent guideline, considered the use of ITS and triaxial tests incorporation in design process of asphalt stabilized materials. A minimum requirement for dry and wet ITS with cohesion, friction angle and retained cohesion values from triaxial test were considered for different types of asphalt emulsions used. Two methods as pavement number and mechanistic-empirical methods were introduced for both asphalt emulsion and foam asphalt stabilized layers. Data from in-service pavement were used to develop pavement number method which gives a good fit to available field data, and it is robust and cannot easily be manipulated to produce in appropriate designs. Mechanistic-empirical design method uses resilient modulus, Poisson's ratio, and layer thickness assigning a long term resilient moduli for asphalt emulsion stabilized layer along with other type of materials to design a pavement layer.

The study conducted by Smith & Braham (2015) focused on the structural design of an asphalt emulsion full depth reclamation (FDR) using AASHTOWare Pavement ME design. As there was limited information available on asphalt emulsion stabilized materials, the authors investigated two different FDR scenarios, considering them as both asphalt mixture and unbound granular material. To develop the design models, the researchers utilized traffic and climate data from two highways located in Arkansas. The results of this study indicated that the asphalt emulsion stabilized layers showed stronger correlations with temperature and modulus, rather than stress state and modulus. This finding suggests that these layers behave more like asphalt concrete than unbound granular materials. By studying the structural design of asphalt emulsion FDR and analyzing the correlations between different parameters, the research provides valuable insights into the behaviour and performance of asphalt emulsion stabilized materials in full-depth reclamation. This information can be significant for improving the design and performance of road pavements, especially when utilizing asphalt emulsion in the stabilization process.

2.9. Summary

The literature review on asphalt emulsion stabilized base layers and cold recycled layers reveals several areas that have not been thoroughly explored and require attention. The significance of the base layer in the pavement structure, which is responsible for load distribution and structural stability, underscores the importance of finding cost-effective, environmentally friendly, and high-performance solutions for its design. The idea of using asphalt emulsion stabilization to improve granular base layer performance was proposed years ago, but it came with drawbacks such as moisture susceptibility, low rutting resistance, early-life low strength, and long-term durability issues. Subsequently, cement stabilization was introduced as a solution to address these problems, leading to increased layer strength, improved durability, reduced moisture-related issues, and better rutting resistance compared to asphalt emulsion stabilization. However, cement stabilization brought its own set of challenges, including the use of large amounts of cement, longer project times for curing, increased costs, environmental concerns, and potential cracking problems. The need to improve cement stabilization with fewer disadvantages was evident, and researchers explored the idea of incorporating cementitious materials as additives in asphalt emulsion stabilized mixes. While this approach was less costly and posed fewer environmental threats compared

to traditional cement stabilization, certain issues persisted, similar to those observed in granular base layers and other stabilization methods. Problems such as cracking, longer curing times, lower breaking times, and brittleness of mixtures at low temperatures were still observed when using cementitious materials in asphalt emulsion stabilized granular base layers and cold recycled materials. Furthermore, performance issues were not adequately addressed, and the design process for these mixes was not studied comprehensively. There is a pressing need for alternative additives to replace cement, and addressing a diverse array of performance issues is crucial.

Over the years, various researchers have attempted to resolve the intricate mix design process of asphalt emulsion stabilized and cold recycled mixes. However, despite these efforts, comprehensive design guidelines have not been established. These mixes do not behave solely as granular base layers or conventional asphalt pavements, necessitating the consideration of their viscoelastic behaviour in the design approach. The existing gap in the design process remains a critical issue that needs to be addressed. Overall, there is a clear need for further research and development in this area to overcome the limitations associated with both asphalt emulsion and cement stabilization, leading to the creation of more efficient and durable base layers in pavement construction with a possible replacement for cementitious materials.

Chapter 3: Laboratory Evaluation of Stabilized Base Course Using Asphalt Emulsion and Asphaltenes Derived from Alberta Oil Sands

3.1. Introduction

Providing load-supporting capacity is the primary function of the base course in flexible pavements. This function improves the pavement by adding stiffness and resistance to fatigue, along with providing a thicker layer to distribute the load throughout the pavement (Christopher et al., 2006). A well designed and constructed base layer with sufficient thickness prevents subgrade from excessive deformation.

Repeated loading on the pavement causes densification and deformation in granular base courses (Lekarp & Dawson, 1998; Wirtgen Cold Recycling Manual, 2012; Yideti et al., 2014), and low resistance to tensile stresses in unbounded material results in tensile failure (Adu-Osei, 2001; S. F. Brown, 1996). The quality of granular material has a direct impact on the resistance of the unbound layer to loading. The unavailability or prohibitively high cost of high-quality materials are potential factors that underscore the need for innovative solutions such as stabilization to enhance the unbound layer properties. Stabilization of base materials in pavements is a common process applied to increase the strength, stability, bearing capacity, and mechanical properties of the layer (Little & Nair, 2009; Patel, 2019; Wegman et al., 2017). According to the Texas Department of Transportation (Branch, 2005), shear strength, stiffness, durability, and resistance to moisture are pavement properties that could be enhanced using stabilization of the base course in asphalt pavement structure.

The application of stabilizing agents must be carefully studied to ensure the proper type and amount of material are selected. Cementitious materials such as hydrated lime, cement, fly ash and various forms of asphalt, including cutback, foamed, and asphalt emulsion, can be used effectively for stabilization depending on the characteristics of the soils to be treated. Asphalt emulsion and cement are the most commonly used material for base course stabilization (Wirtgen Cold Recycling Manual, 2012).

A stabilized layer using asphalt emulsion has higher flexibility compared with a cement stabilized layer (Wirtgen Cold Recycling Manual, 2012). Also, asphalt emulsion-treated courses are more cost-effective compared to hot mix asphalt (Barbod & Shalaby, 2014). Indeed, as asphalt products have broader acceptance in the road construction industry, standard testing methods and specifications available for them, and higher strength gain rate of these materials, an asphalt emulsion is emerging as a preferable option to cement (Barbod & Shalaby, 2014). According to the Asphalt Institute (2008) and Feng et al. (2016), the comparably low energy consumption and pollution associated with asphalt emulsion have also contributed to its increased adoption within the pavement industry.

Asphalt emulsion requires a long curing time to achieve binding properties similar to those of asphalt binders (Du, 2018). Previous studies have suggested that asphalt emulsion sometimes may take up to 3 years to reach a final state of strength (Doyle et al., 2013; Marais & Tait, 1989; Quick & Guthrie, 2011). This long curing requirement in asphalt emulsion-stabilized materials results in lower mechanical strength as well as inadequate performance properties at the early stages of its life (Du, 2016; Khweir et al., 2004). It is thus necessary to investigate novel methods to improve the mechanical strength properties of asphalt emulsion-stabilized mixtures.

Cementitious material could be used to enhance these properties and increase the curing rate of asphalt emulsion-stabilized materials (Terrell & Wang, 1971). Meanwhile, active fillers such as cement or hydrated lime could be added to asphalt emulsion mixtures to enhance the properties of asphalt emulsion-stabilized mixtures (Du, 2018). These active fillers affect the breaking and curing of asphalt emulsions and enhance the retained strength, stiffness, rutting performance, and moisture resistance of asphalt emulsion-stabilized materials; they also improve the adhesion of asphalt to the aggregates and its dispersion in the mixture (Fang et al., 2016).

Generally, the addition of cement into an asphalt emulsion mixes provides improvements in the strength of the mixes at early stages; it also provides more resistance to moisture damages, low-temperature cracking as well as good stability at high temperatures (Oruc et al., 2007; Yan et al., 2017). A study conducted by Terrell et al. (1971) shows that for anionic asphalt emulsion stabilized mixes subjected to cold weather conditions and moisture, the addition of small

quantities of cement significantly improved the curing rate of the mix. It was found that the ultimate resilient modulus of the asphalt emulsion mixture was improved by approximately 200% with the addition of 1% cement. The optimum cement content was found to be about 1% to 2% to improve the performance properties of the mix (Niazi & Jalili, 2009). Romeo et al. (2018) showed that a moderate cement content of 1% could provide a good resistance to cracking in terms of increased tensile limits. A study by Schmidt et al. (1973) indicates that 1.3% cement addition to asphalt emulsion mixture increases the stiffness modulus by five times within a one-day curing period. The study further found an 18% to 70% increase in the ratio of the soaked tensile strength to the dry one. This shows that the addition of 1.3% cement improved resistance to moisture damage of the asphalt emulsion mixture.

Similarly, another study by Jian et al. (2011) showed that the addition of cement to asphalt emulsion mixtures increased the rutting resistance of the stabilized emulsion mixtures. However, the addition of cement content beyond 1.5% might result in an adverse effect with less or no improvement in the rutting resistance of the mixes. This effect is attributed to the high cement content, which makes the stabilized mix very stiff and hard to compacted adequately (Wirtgen Cold Recycling Manual, 2012).

Recently, with the introduction and application of modified asphalt emulsions, the mechanical strength of stabilized asphalt emulsion mixtures has significantly improved and allowed this mixture to be applied in some highway upper layers maintenance (Du, 2014; Zapién-Castillo et al., 2016). The application of polymer modified asphalt emulsion in pavement construction has become very popular, especially because it is used to minimize distresses such as rutting, which enhances pavement service life (Forbes et al., 2001; Labi et al., 2007). A previous study found that pavement constructed using styrene-butadiene rubber modified asphalt emulsion shows a significant increase in rutting resistance (Takamura & Heckmann, 2001). Shafii et al. (2013) showed that the physical properties of stabilized mixes with modified asphalt emulsion using 7% latex were significantly improved. Chen et al. (Chen et al., 2012) performed a study on composite specimen interface cracking (CSIC) test on polymer modified asphalt emulsion mixes under repeated tensile loading. In this study, the rate of cracking damage development was monitored. The findings revealed that the polymer modification of asphalt emulsions significantly increases cracking resistance.

Despite the significant improvement recorded with polymer modification of asphalt emulsions, there are some specific drawbacks with their applications, such as high cost, reduced penetration resistance and higher molecular weight, which makes it difficult to disperse in the emulsion (Yildirim, 2007). Due to these drawbacks, researchers continue to work on finding materials that can replace polymers for emulsion modification. Considering cost-effectiveness as well as environmental protection, now researchers have employed the use of waste materials as substitutes of polymers for asphalt emulsion modification (Ellis et al., 2011).

Asphaltenes extracted through deasphalting of oil sands bitumen are a residue material with no significant use in the road industry. Asphalt is a viscoelastic material made of polar and non-polar components, where the polar components are responsible for the elastic behaviour of the asphalt, while the non-polar components are responsible for viscous response (Ramirez-Corredores, 2017; Sultana & Bhasin, 2014). The main components of asphalt are saturates, aromatics, resins and asphaltenes (often referred to collectively as SARA). Asphaltenes is the most polar fraction of asphalt binder, with a high molecular weight (Behnood & Modiri Gharehveran, 2019; Ramirez-Corredores, 2017). The polar fraction of asphalt binders is composed of asphaltenes and resins, while saturates and aromatics form the non-polar part (Y. Xu et al., 2019). An increase in the polar contents of an asphalt, it should be noted, improves the stiffness and, thereby, the performance of the asphalt (Sultana & Bhasin, 2014; Y. Xu et al., 2019). The cost-effectiveness as well as stiffening effect of asphaltenes in asphalt material if used in base course stabilization could thus enhance the properties and cost performance of the stabilized mixture while at the same time providing a use for residue asphaltenes.

The main objective of our study was to evaluate the impact of adding asphaltenes residue derived from Alberta oil sands for stabilization of a well-graded granular base course material using a cationic slow setting (CSS-1H) asphalt emulsion. After preparing a mix design for a control mix, the same mix design was used for the asphaltenes-modified mixes. The performance properties of the modified base course were evaluated for moisture damage by conducting indirect tensile strength (ITS) tests on dry and conditioned samples and calculating the tensile strength ratio (TSR) and rutting resistance using a Hamburg wheel-tracking test.

3.2. Materials and Experimental Procedures

This experimental study was carried out in three phases. The first phase involved choosing the aggregate gradation, optimum emulsion content (OEC), and optimum moisture content (OMC) for compaction. In the second phase, ITS and Marshall stability and flow tests were performed to evaluate the strength and stability of the mixtures prepared. In the third phase of the study, TSR tests for both saturated and freeze/thaw (F/T) samples, as well as Hamburg wheel tracking tests, were conducted in order to evaluate the performance properties of mixtures. Figure 3-1 shows the test matrix defined for this research providing the steps from the mix design till the performance tests.

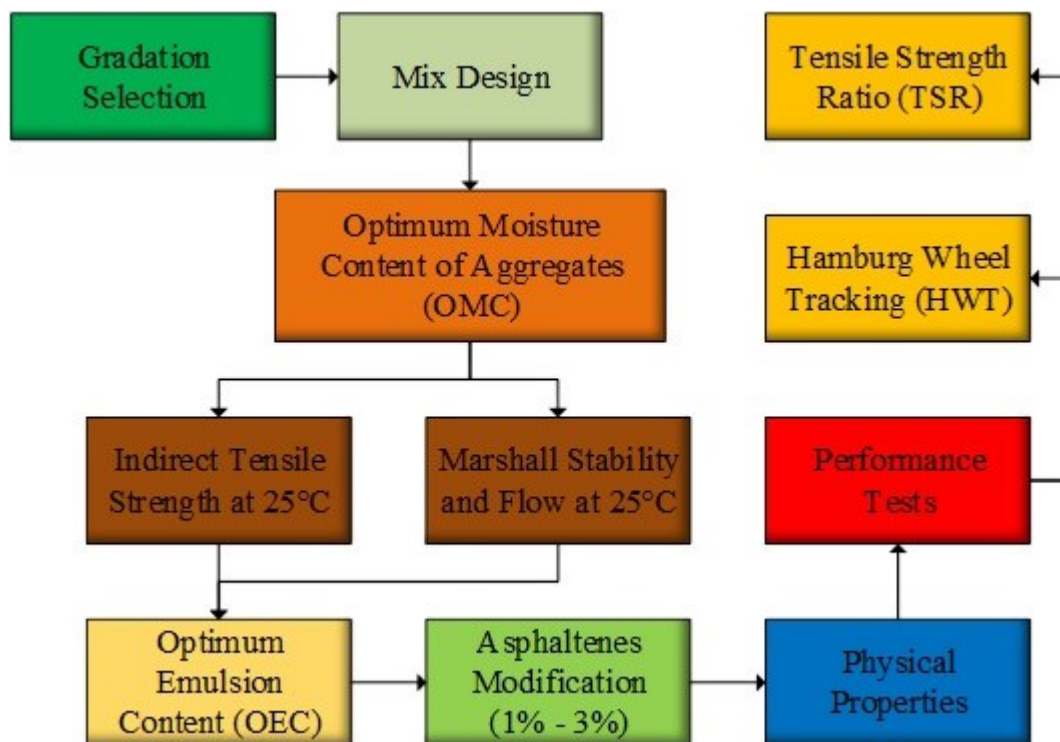


Figure 3-1 Test matrix flow chart

3.2.1. Materials

3.2.1.1. Aggregates

The aggregates used for the mixture preparation were obtained from a single source (provided by Lafarge Canada). Aggregate gradation specifications were selected based on the parameters set out in Wirtgen Cold Recycling Manual (2012), in City of Edmonton regulations (2015), and in Alberta Transportation documentation as per Soliman et al. (2014). A well-graded

aggregate gradation that satisfied the parameters was selected. Table 3-1 presents the selected aggregate gradation skeleton consisting of 57.27% coarse aggregates, 36.73% fine aggregates, and 6% filler, while the physical properties of the aggregates based on different test are presented in Table 3-2 and the bulk specific gravity of the aggregates (G_{sb-agg}) was found to be 2.601. The amount of filler used in stabilization of the granular base layers and the Los Angeles test result for aggregates were both found to satisfy the specifications of Wirtgen Cold Recycling Manual (2012) and the City of Edmonton (2015).

A proctor test was carried out to obtain the OMC of the aggregates in accordance with ASTM D698 (2021).

Table 3-1 Selected aggregate gradation

Sieve size (mm)	% Passing	% Retained	Coarse - Fine	
20.000	100	0.00		
12.500	75	24.83		
10.000	61	13.94	57.27%	Coarse
8.000	55	6.23		
6.300	48	7.00		
5.000	42	5.27		
2.500	32	10.65		
1.250	25	6.47		
0.630	18	6.68	36.73%	Fine
0.315	13	5.93		
0.160	9	4.00		
0.080	6	3.00		
Filler (Pan)	0	6.00	6%	Filler

Table 3-2 Physical properties of the aggregates

Property (unit)	Standard	Result	Limitation
Amount of material finer than 75- μ m (No. 200) sieve in aggregate (%)	ASTM C117 (2017)	6	2-9
Fine aggregates	Specific gravity (G_{fa})	2.604	-
	Absorption of water (%)	0.624	
Coarse aggregates	Specific gravity (G_{ca})	2.598	-
	Absorption of water (%)	0.870	
Abrasion of coarse aggregates (%)	ASTM C131 (2020)	23	Max 40

Proctor test	OMC (%)	ASTM D698	6.3	-
	Dry Density (kN/m ³)	(2021) (Modified D1557 (2000))	15.4 max.	-

3.2.1.2. Asphalt Emulsion

Typically, the asphalt emulsion used in base stabilization is cationic slow setting (CSS). This type of emulsion has workability time to have a well dispersed mixture with higher stability. The emulsion used in our study was prepared with a hard binder as CSS-1H consisting of 61% asphalt and 39% water. The properties of the 80/100 penetration asphalt used to produce the asphalt emulsion are shown in Table 3-3, while properties of the asphalt emulsion are outlined in Table 3-4.

Table 3-3 Properties of asphalt binder used to produce emulsion (Husky Energy, 2018)

Property (unit)	Standard	Specification		Typical Analyses
		Min.	Max.	
Specific gravity (density) at 15°C (kg/L)	ASTM D70 (2021)	-	-	1.0341
Penetration at 25°C (100 g, 5 s) (dmm)	ASTM D5 (2020)	80	100	90
Flash point (COC) (°C)	ASTM D92 (2018)	230	-	276
Ductility at 25°C (5 cm/min) (cm)	ASTM D113 (2018)	100	-	150+
Solubility in trichloroethylene (%)	ASTM D2042 (2022)	99.5	-	99.9
Absolute viscosity at 60 °C (Pa·s)	ASTM D2171 (2018)	150	-	183
Viscosity at 135 °C (Pa·s)	ASTM D4402 (2006)	-	3.00	0.42
RTFO mass loss (%)	ASTM D1754 (2020)	-	1.00	0.37

Table 3-4 Asphalt emulsion properties (Husky Asphalt, 2019)

Property (unit)	Standard ASTM/AASHTO	Specification		Typical Analyses
		Min.	Max.	
Tests on Emulsion				
Specific gravity (Density) at 15.6 °C (kg/L)	D6937 (2016)/T59 (2022)	-	-	1.020
Residue by distillation (% by mass)	D6997 (2012)/T59 (2012)	57	-	61
Viscosity at 25 °C (S.F.S)	D7496 (2020)-D88 (2019)/T59 (2022)	20	100	22
Oversized particles (sieve) (% by mass)	D6933 (2022)/T59 (2022)	-	0.300	0.008

Settlement (24 hr) (% by mass)	D6930 (2019)/T59 (2022)	-	1.0	0.5
Particle charge test	D7402 (2017)	Positive		Positive
Tests on Asphalt Residue				
Penetration at 25 °C (100 g, 5 s) (dmm)	D5 (2020)/T49 (2019)	40	125	95
Ductility at 25 °C (5 cm/min) (cm)	D113 (2018)/T51 (2018)	40	-	>40
Solubility in Trichloroethylene (% by mass)	D2042 (2022)/T44 (2018)	97.5	-	>97.5

3.2.1.3. *Asphaltenes*

In our study, two asphaltenes from different sources were used for sample preparations. For determining the asphaltenes content of each, SARA tests were conducted for both samples in order to determine the relative content of saturates, asphaltenes, resins, and aromatics (Table 3-5). The ratio of asphaltenes in Source 2 was found to be 1.05% more than in Source 1.

Asphaltenes from Source 1

Asphaltenes samples from Source 1 were obtained in chunk form (as shown in Figure 3-2a) through a deasphaltation process using n-pentane condensate. These chunks were crushed into powder form and sieved through a No. 100 mesh sieve. The asphaltenes content in Source 1 was determined to be 79.62% according to the SARA test, while saturates, aromatics, and resins accounted for the rest of the sample.

Asphaltenes from Source 2

Although the Source 2 asphaltenes were obtained already in powder form as shown in Figure 3-2b, for consistency they were also passed through a No. 100 mesh sieve. Asphaltenes from this source were vacuum-dried, and it was sourced from Cold Lake bitumen. A SARA test showed that asphaltenes accounted for 83.63% of the content in Source 2. Value Creation Inc. provided us with the properties of the Source 2 asphaltenes (Table 3-6).

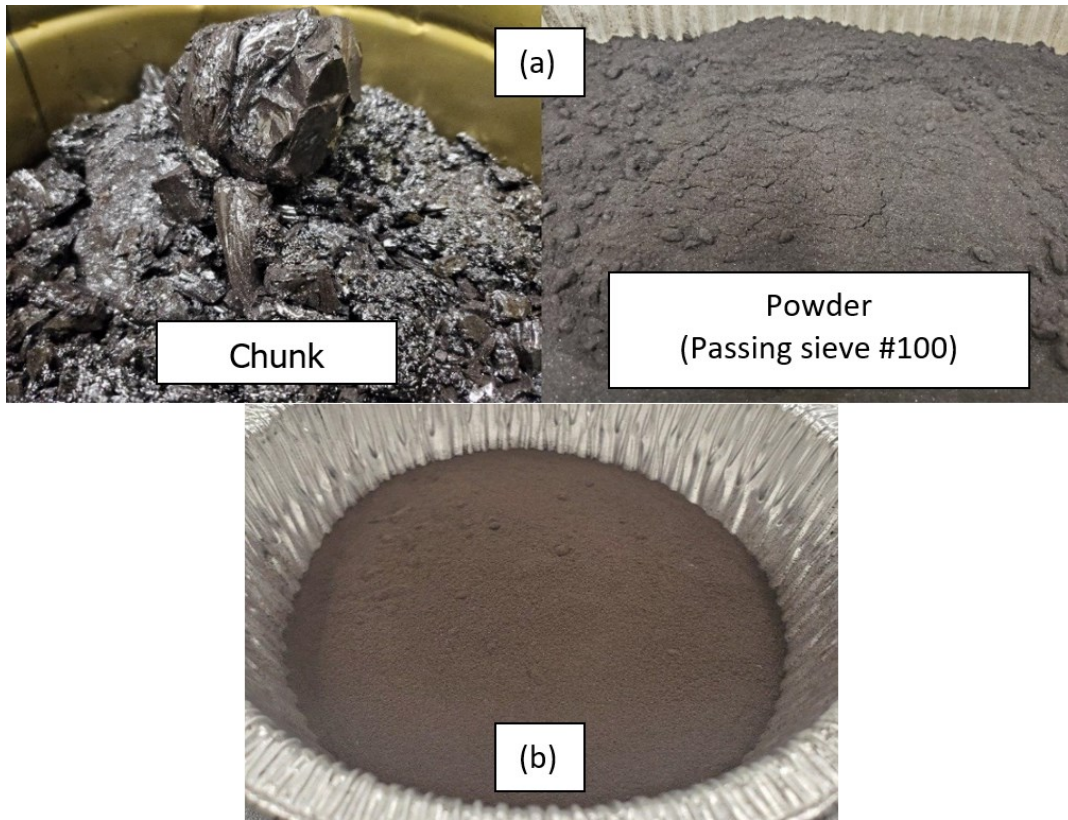


Figure 3-2 (a) Asphaltenes provided for mixing Source 1 and (b) Source 2

Table 3-5 SARA test result for asphaltenes

Description	Saturates (%)	Aromatic (%)	Resin (%)	Asphaltenes (%)
Source 1	6.85	9.68	3.84	79.62
Source 2	2.35	7.3	6.62	83.63

Table 3-6 Properties of Source 2 asphaltenes (Value Creation Inc., 2006)

Property (unit)	Standard ASTM	Typical Analyses
Original of Sample	-	Cold Lake bitumen
Bulk Density (kg/m ³)	-	403.6
True Density (kg/m ³)	-	1,135
Gross Calorific Value (MJ/kg)	ASTM D5865 (2019)	38.6
Net Calorific Value (MJ/kg)	-	36.4
Micro Carbon Residue (MCR)	ASTM D4530 (2008)	37.2

3.3. Testing Program

3.3.1. Mix Design and Mechanical Properties of Unmodified Samples

3.3.1.1. Design Matrix for Optimum Emulsion Content

The design method used for the base stabilization in this study was based on the method described by the Asphalt Institute (2008), considering we were working with well-graded granular aggregates. The amount of asphalt emulsion to be used in this method was calculated in reference to the relationship characterized in Equation (3-1 for well-graded aggregate gradation subject to ASTM D6997 (2012)). The test showed the value of A, which is the amount of the material retained after distillation, to be 61%.

$$\text{Base mixture: Asphalt Emulsion \%} = \frac{(0.06B + 0.01C)100}{A} \quad (3-1)$$

where: A = Percentage of residue of asphalt emulsion remaining after distillation (as determined using ASTM D6997 (2012)), B = Percentage of dry aggregate passing through a No. 4 sieve, C = Percentage of dry aggregate retained on a No. 4 sieve.

The approximate asphalt emulsion content was determined using this method as 5.14% per total aggregates, and four different asphalt emulsion contents with 1% intervals were prepared for the purpose of determining the OEC with respect to the Marshall stability and flow results.

In our study, Marshall stability and flow and ITS tests were utilized for OEC determination as per the design matrix shown in Table 3-7. The mixing process was initiated with the addition of water to the oven-dried aggregates to reach the OMC, after which it was mixed until the water was uniformly distributed throughout the aggregates. The aggregates were allowed to cool down to ambient temperature before adding the water in order to prevent the loss of moisture. Asphalt emulsion was introduced to the wet aggregates according to the contents determined in Table 3-7 and compacted using a Marshall hammer with 50 blows applied to each side of the sample.

Table 3-7 Emulsion and additional water contents for design matrix

Emulsion content (% per total mix)	Additional water (% per total aggregates)
3.04	5.1
3.98	4.7
4.89	4.3

5.78	3.9
6.66	3.5

3.3.1.2. *Marshall Stability and Flow Test for OEC*

The Marshall stability test was conducted in accordance to ASTM D6927-15 (2015) for the different contents of asphalt emulsion determined in the design matrix for OEC (Table 3-7). The samples compacted using a Marshall hammer were cured inside the compaction molds for 48 hr in an oven at a temperature of 60 °C. For extracting them from the molds after conditioning, the samples were cooled down for at least 2 hr. Three replicates for each content level were prepared. The Marshall stability test, it should be noted, was conducted following conditioning in a 25 °C air bath for 3 hr (Asphalt Institute, 2008).

3.3.1.3. *ITS Test for OEC*

The ITS test was conducted in accordance with AASHTO T283 (2021). The samples for the ITS test were prepared using the same asphalt emulsion concentrations specified in the design matrix for OEC. Concentrations were chosen that would ensure consistency with the results of the Marshall stability tests. Three samples were prepared for each asphalt emulsion concentration. The samples were prepared using a Marshall hammer and the same compaction procedure as for the Marshall stability tests. These samples were cured following the same process and conditioned for 3 hr in an air chamber at 25 °C, in accordance with AASHTO T283 (2021). After conditioning, a load was applied to the samples at a rate of 50 mm/min. The maximum load applied to the sample before failure was recorded in order to determine the ITS according to the following equation:

$$S_t = \frac{2000P}{\pi tD} \quad (3-2)$$

where: S_t = Indirect tensile strength (kPa), P = Maximum applied load (N), t = Average height of specimen (mm), D = Diameter of specimen (mm).

3.3.1.4. *Tensile Strength Ratio*

Moisture susceptibility is the ability of mixtures to resist the stripping action of water that causes separation of the asphalt from the mineral aggregates. Soaking a mixture in water and

subjecting it to a F/T process prior to conducting the ITS test allows the moisture susceptibility to be evaluated (i.e., TSR test) (Chehelgo et al., 2018).

Samples subject to freeze/thaw cycle

The TSRs of the control and modified samples were tested as per AASHTO T283 (2021). Three replicates were prepared for both the dry and F/T-conditioned samples of the selected mixtures. The ITS of the samples was determined using a Universal Testing Machine (UTM) at 25 °C and subject to the parameters of the standard mentioned above. The TSR values were calculated using Equation (3-3).

$$\text{TSR} = \frac{S_2}{S_1} \quad (3-3)$$

where: S_1 = Average tensile strength of the dry subset (kPa), S_2 = Average tensile strength of the conditioned subset (kPa)

Water saturated samples

Another set of samples with three replicates prepared and saturated for 24 hr in 25 °C water was tested for ITS. The TSRs of the saturated samples were also calculated using the dry subset.

3.3.1.5. Hamburg wheel tracking test

AASHTO T324-19 (2019) was followed in performing the wheel-tracking test for slab samples with dimensions of 400 mm (length), 300 mm (width), and 80 mm (height) considering the nominal maximum aggregate size (Figure 3-3). The selected mixtures were prepared and compacted using a slab compactor and a similar curing process to the one followed for the Marshall samples. The test temperature was set at 40 °C, considering the binder grade and base layer. The samples were preconditioned in water at this temperature for 45 minutes prior to testing. A 705±4.5N, 47 mm-wide steel wheel with a frequency of 52±2 passes per minute and a maximum speed of 0.305 m/s at midpoint was used for the test, with the termination point set at 20,000 passes or a 12 mm rutting depth, whichever was achieved first. The striping inflection point (SIP) and rutting resistance index (RRI) were determined for each sample

based on the rutting depth and number of passes in order to evaluate the rutting potential and susceptibility to moisture damage (Aschenbrener, 1995).



Figure 3-3 Slab sample compacted by roller compactor

3.4. Results and Discussion

3.4.1. Optimum Moisture Content

A proctor test was carried out, in accordance with the parameters set out in ASTM D698 (2021), to determine the OMC, (which, in turn, is necessary for the purpose of calculating the amount of additional water that needs to be added to the aggregates for mixing with the asphalt emulsion). Figure 3-4 presents the OMC plot based on the proctor test, where the results indicate the dry density of the compacted aggregates at different water contents (e.g., material with 6.3% water content has a maximum dry density of 15.4 kN/m^3).

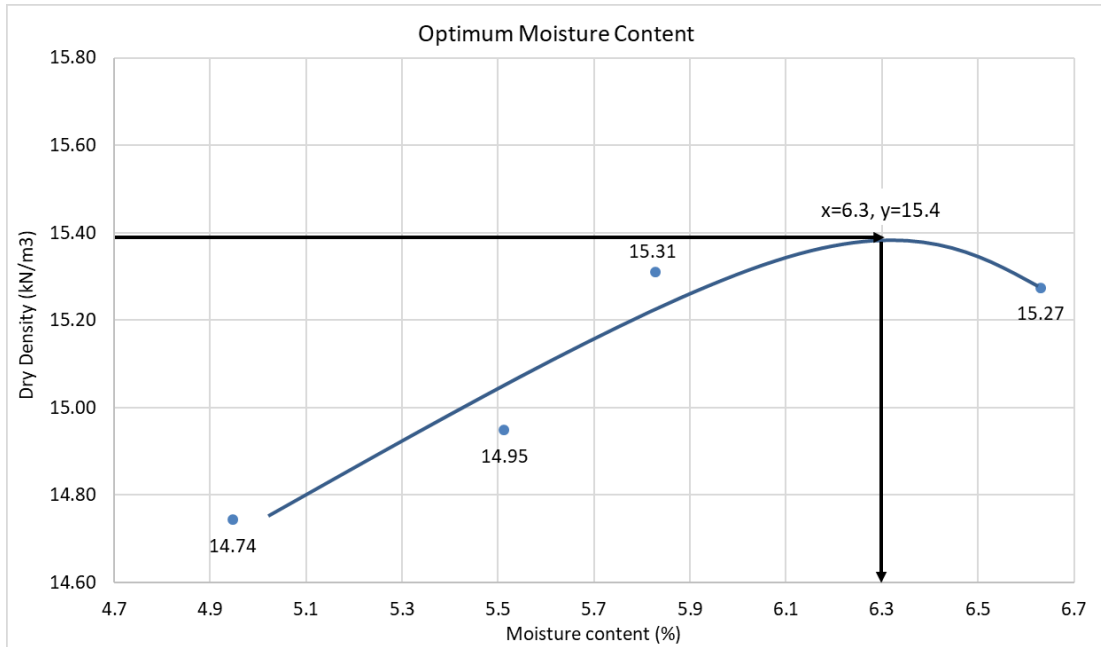


Figure 3-4 Proctor test results

3.4.2. OEC Determination with Marshall Stability Test

The results of the Marshall stability, flow and density for determination of OEC are presented in Figure 3-5a, Figure 3-5b, and Figure 3-5c, respectively. The samples prepared with 3.7% asphalt emulsion (by weight of total mixture) was found to be the OEC according to this test. The minimum acceptable Marshall stability for the stabilized base courses, meanwhile, was found to be 2.2 kN for low-volume roads. However, past studies have recommended adjusting the minimum value based on the given mix type and application (Asphalt Institute, 2008). The Marshall stability and flow values obtained were found to be comparable to those associated with hot mix asphalt. According to the results, the maximum Marshall stability and density values were achieved at an asphalt emulsion content of 3.85% per total weight of aggregates, equal to 3.7% per total mixture. Hence, in reference to the Marshall stability test, this amount was considered the OEC (Table 3). However, in order to confirm this result, ITS test was applied to the new samples that had been prepared for determining the OEC, as recommended in the literature (Du, 2014; Wirtgen Cold Recycling Manual, 2012). The results of both tests were considered in ultimately determining the OEC. ITS, it should be noted, is an indirect measure of the tensile strength and reflects the flexibility and flexural characteristics of the stabilized mix.

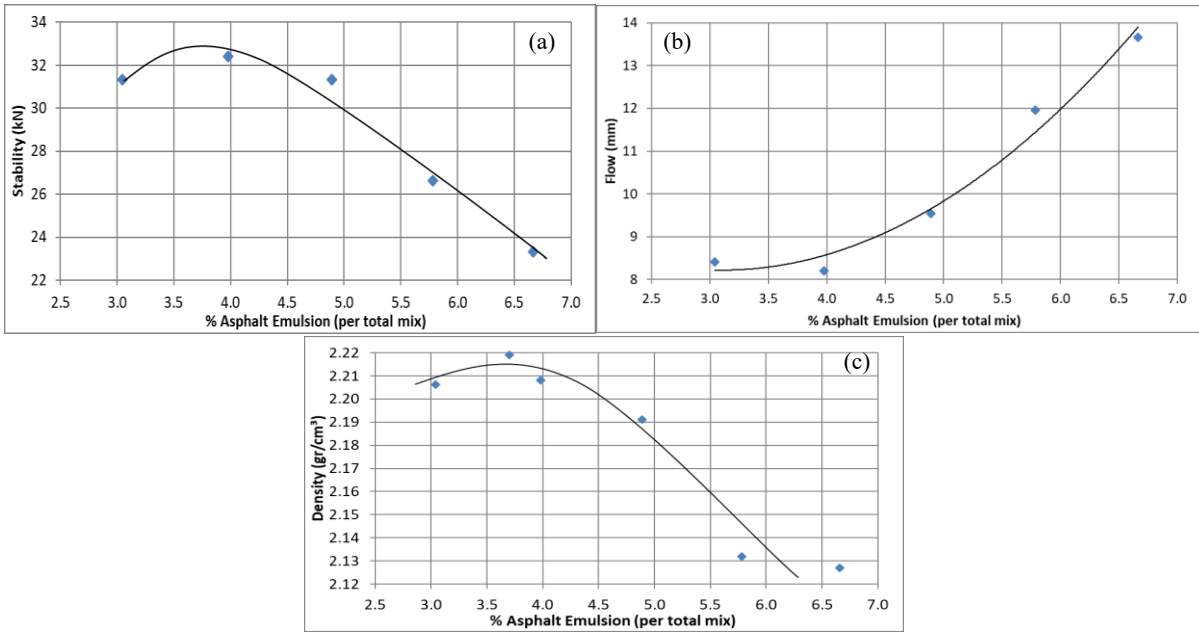


Figure 3-5 (a) Stability, (b) Flow and (c) Density values with respect to asphalt emulsion content

3.4.3. OEC Determination Using ITS Test

Figure 3-6 presents the results of the OEC determination using the ITS test. As the results demonstrate, the maximum ITS was achieved after adding 3.85% asphalt emulsion by weight of aggregates, a finding similar to that observed in the Marshall stability tests. According to this result, the OEC was found to be 3.7% by weight of total mixture. A value of 225 kPa was determined as the lower limit for the ITS value of asphalt emulsion-stabilized material per Wirtgen Cold Recycling Manual (2012), and all the levels of emulsion content under study were found to satisfy this minimum value.

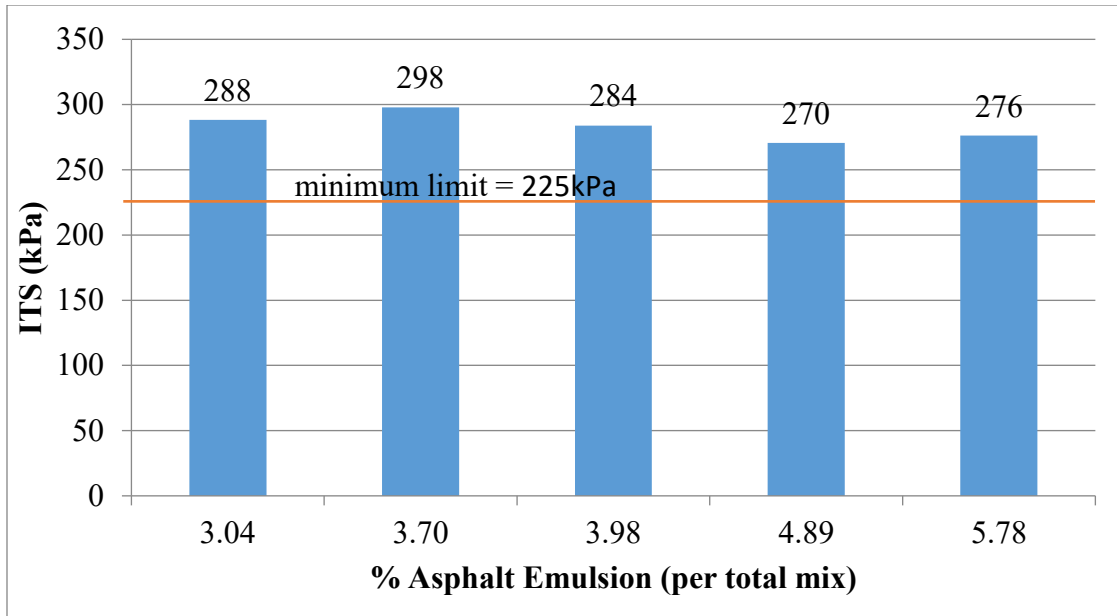


Figure 3-6 ITS results for mix design samples

3.4.4. Mix Design and Mechanical Properties of Asphaltenes-modified Samples

3.4.4.1. Preparation of Asphaltenes-modified Samples

For adding asphaltenes into the mixture, the OEC that had been determined was selected as the constant content for the entire mixtures, given its high stability and strength.

This step was carried out in several stages and with different levels of emulsion content. The control samples were prepared following the same process as for the optimum asphalt emulsion (without any asphaltenes) in order to compare them with the modified samples and thereby gain understanding of the impact of asphaltenes modification.

Asphaltenes were added to the asphalt emulsion prior to mixing with the aggregates. According to the experimental observations during the preparations of the mixture, this mixing process was selected due to the relative ease of mixing asphaltenes in asphalt emulsion compared to in aggregates. Finally, the samples were prepared by adding 1%, 2%, and 3% asphaltenes content (as a percentage of the total mixture weight) into the asphalt emulsion and then mixing the modified asphalt emulsion with the aggregates. Four different mixes were prepared. The samples with 1% asphaltenes content were found to be the easiest modified samples to prepare, as the relatively low quantity of asphaltenes made mixing with the asphalt emulsion easier. Given that increasing the asphaltenes content would cause the asphalt emulsion to fast break

and make mixing more difficult, water was added (25% and 50% by total asphalt emulsion weight, as per Table 3-8) to the asphalt emulsion in order to make it less viscous and easier to mix. Samples S1-1-0, S1-2-25, S1-2-50, and S1-3-50 were prepared using Source 1 asphaltenes, while S2-1-0 was used to confirm the results using an alternate asphaltenes source (Source 2). Samples IDs are defined as SX-Y-Z where SX is the asphaltenes source used (Source 1 and Source 2). Y indicated the percentages of the asphaltenes used in the sample (1%, 2%, and 3%) and finally Z is the percentages of the water added to the emulsion per total emulsion (0%, 25%, and 50%)

Table 3-8 Design matrix for asphaltenes modification

Sample ID	Asphaltenes (% per total mix)	Extra water (% per total emulsion)
S1-1-0	1	0
S1-2-25	2	25
S1-2-50	2	50
S1-3-50	3	50
S2-1-0	1	0

3.4.4.2. *Indirect Tensile Strength*

ITS testing was conducted for the mixes presented in Table 3-8, with the results shown in Figure 3-7. Looking at the results it is apparent that the samples modified with asphaltenes show higher ITS than do the control samples. Sample S1-2-25 was found to have the best response to tensile strength testing, and increasing the asphaltenes content from 2% to 3% (i.e., comparing S1-3-50 to S1-2-50) resulted in a slight increase in ITS. Comparing S1-2-25 with S1-2-50, meanwhile, indicates that increasing the water has an adverse effect on the tensile strength of the samples, even though extra water is necessary in some cases to ease the mixing process. According to these results, and balancing ITS with consideration of the mixing process, three samples were selected for continuation: the control, S1-1-0, and S1-2-25. The aim of these selections was to minimize the extra water needed while maximizing the asphaltenes content with respect to gain the higher tensile strength resistance of the mixture. Figure 3-8 shows modified samples after ITS test in dry condition.

Table 3-9 compares the air void content and ITS results for the selected samples, where a slight increase in air void content is seen when adding asphaltenes to the samples. Figure 3-7, meanwhile, presents the maximum tensile strength for each sample prepared. As can be seen, all results were found to satisfy the lower limit for asphalt emulsion-stabilized layers as determined in reference to Wirtgen Cold Recycling Manual (2012).

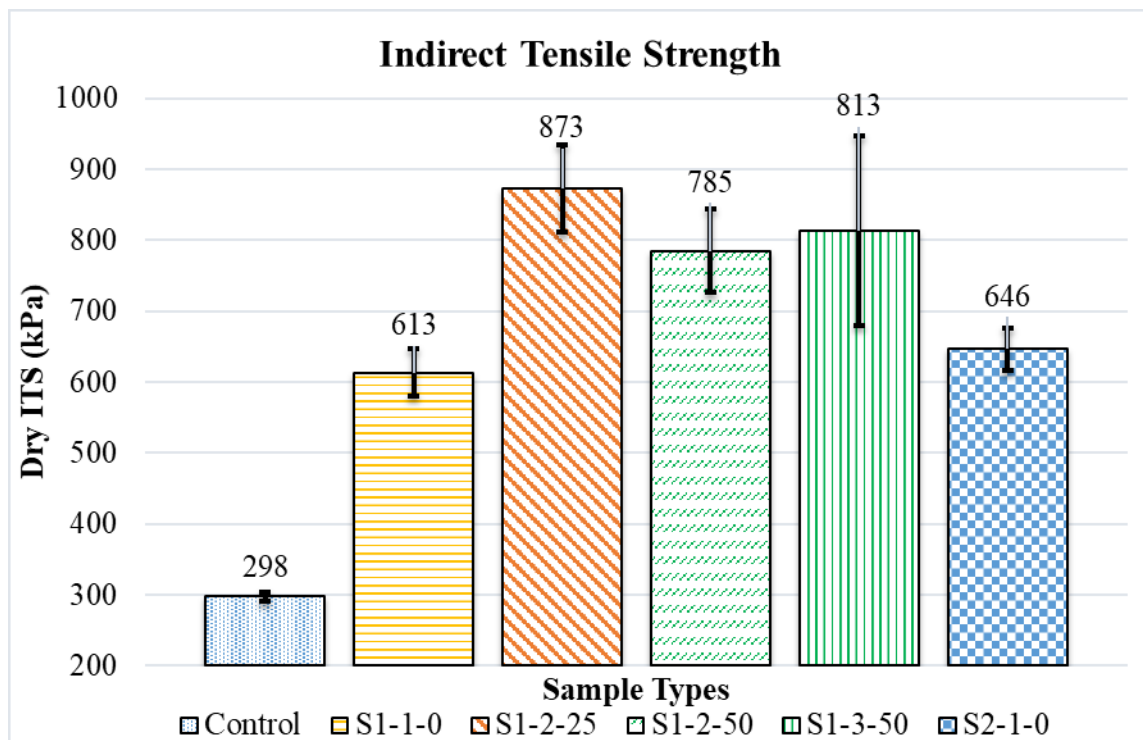


Figure 3-7 Resistance to tensile strength for samples of testing matrix



Figure 3-8 Compacted samples by Marshall hammer after performing ITS test

Table 3-9 ITS and air void content for selected samples

Sample ID	ITS (kPa)	Air (% per total mix)	voids
Control	298	10.934	
S1-1-0	613	12.187	
S1-2-25	873	12.994	
S2-1-0	646	13.908	

3.4.4.3. *Marshall Stability and Flow Test*

Marshall stability and flow tests were conducted to compare the three selected samples in terms of stability. For this test, Source 2 asphaltenes was also used in order to compare the effect of the two different asphaltenes types. Table 3-10 gives the average values for stability, density, flow, and air void content of the mixtures. It can be seen that asphaltenes increase the stability of the mixtures (comparing the control samples with 2% and 1% asphaltenes samples), while the asphaltenes-modified samples were found to have lower density than the control. On the other hand, asphaltenes source was not found to have a significant effect on the Marshall stability. For the samples with the same asphaltenes content (1%), Marshall stability is lower when the air void content is higher.

Table 3-10 Marshall test results and air void content

Sample ID	Stability (kN)	Density (gr/cm³)	Flow (mm)	Air voids (% per total mix)
Control	11.536	2.197	5.545	11.113
S1-1-0	17.059	2.150	5.263	11.395
S1-2-25	22.718	2.143	7.482	12.574
S2-1-0	16.730	2.094	4.575	13.798

3.4.4.4. *Performance tests*

To determine the moisture susceptibility and rutting performance of the samples, TSR and Hamburg wheel tracking tests were applied to the control, S1-1-0, and S1-2-25 samples.

Moisture susceptibility

The TSR test results obtained for the samples are shown in Figure 3-9. In addition to the TSR test with conventional F/T cycles, samples were tested with only 24 hr of saturation in 25 °C water. The ITS of the samples was measured, with the TSR of the saturated and conditioned samples in comparison to the control samples shown in Figure 3-9. Looking at the results, the saturated control sample was found to have almost the same tensile strength as dry control sample, whereas the F/T cycle was found to decrease the tensile strength resistance of control sample to about 50%. For the samples with 1% asphaltene content, the moisture susceptibility was observed to increase, while the TSR values for the samples modified with 1% asphaltene under saturated and F/T cycle conditions were found to be 90% and 36%, respectively. These effects became more pronounced with higher asphaltene content, with the 2% asphaltene samples exhibiting TSRs of 75% and 30% TSR for saturated and F/T cycle conditions, respectively. These results indicate that modification of the asphalt emulsion-stabilized aggregates with asphaltene increases the moisture susceptibility of the mixture.

While the dry subsets of the modified samples were found to satisfy the minimum ITS of 310 kPa specified in AASHTO MP 31-17 (2017), the control samples failed to satisfy this design limitation. Meanwhile, the samples subject to F/T cycle failed to satisfy the TSR value of 0.7 specified in AASHTO T283 (2021), while saturated samples met the specifications of AASHTO MP 31-17 (2017) for TSR value.

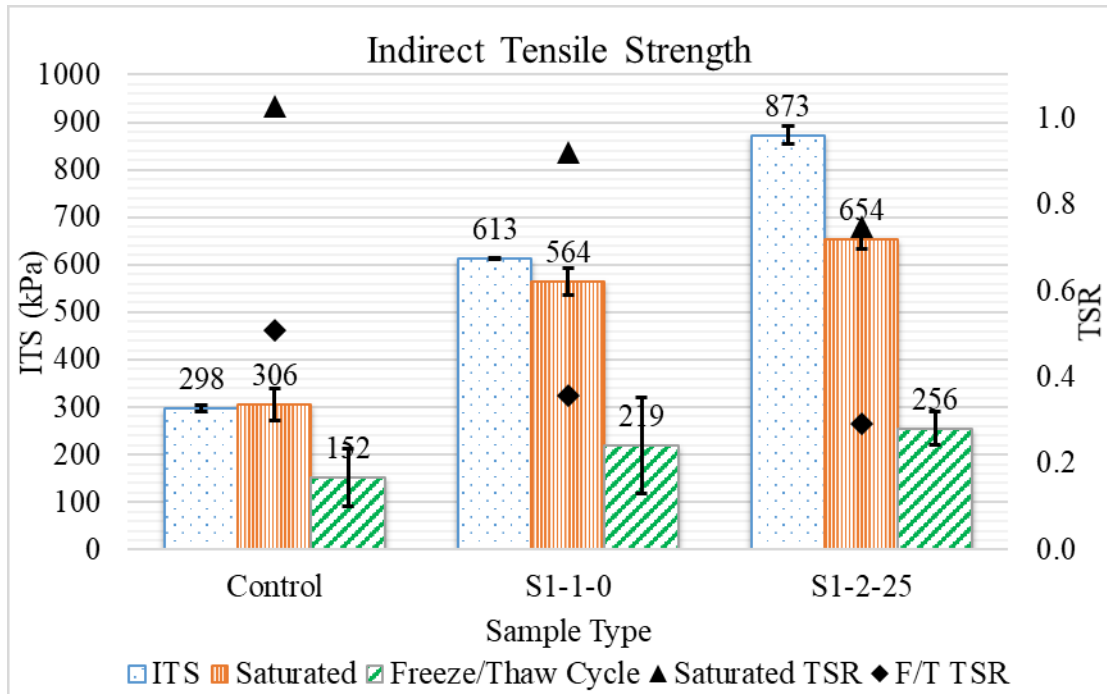


Figure 3-9 Moisture susceptibility of modified samples

A wheel-tracking test was performed to determine the moisture resistance and rutting performance of the different samples. The moisture susceptibility results in Figure 3-10 show that the inflection point for the control samples was found to be at approximately 3,800 passes, while adding asphaltenes moved this point to 8,200 and 7,400 passes for 1% and 2% asphaltenes, respectively. However, considering that these values represent a relatively low number of load cycles, the mixtures could be susceptible to moisture damage at this point. Also, moisture sensitivity increases for asphaltenes modified samples compared to the control samples.

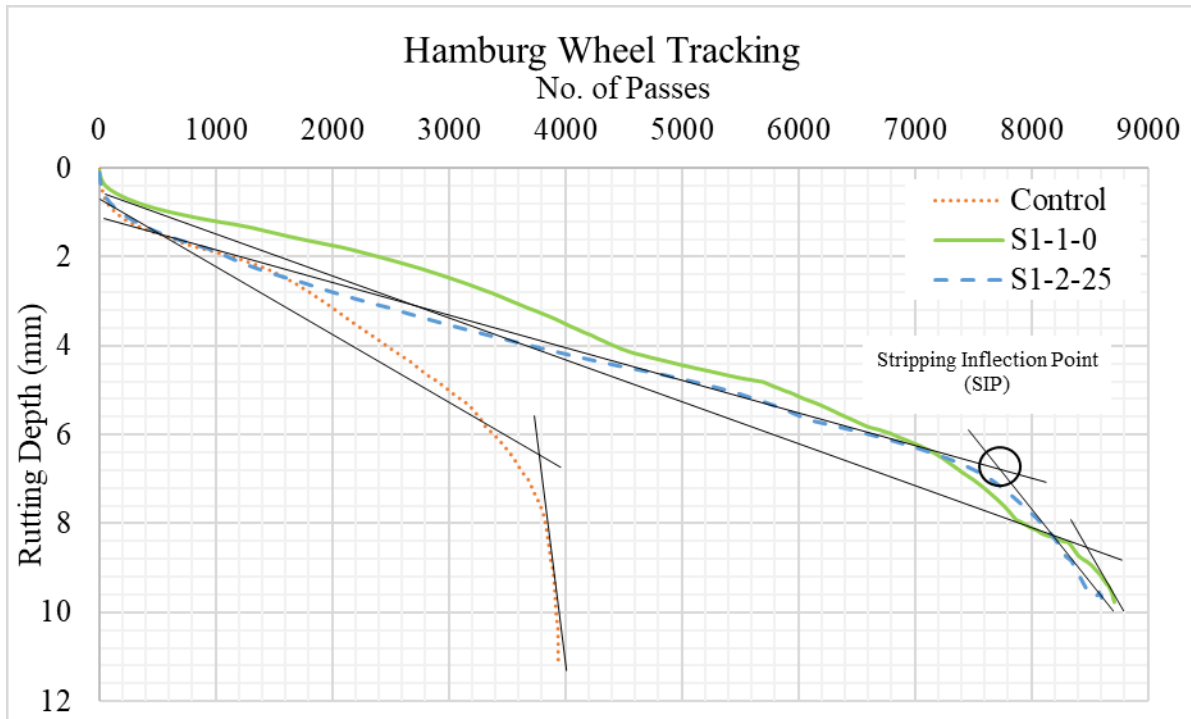


Figure 3-10 Hamburg wheel tracking results

Rutting performance

The Marshall stability and flow results were used to determine the Marshall quotient (MQ) as an indicator of rutting performance, while the Hamburg wheel tracking test was used to provide further insight into the performance features of the mixtures under higher temperatures. The wheel tracking test results for the mixes are presented in Figure 3-10. Looking at these results, modification with asphaltenes was found to have a positive impact on the rutting resistance of the material stabilized by asphalt emulsion. Figure 3-11 presents the MQ and RRI calculated using the wheel tracking results, where the RRI calculated based on the number of passes and the depth of rutting helps to form a clearer picture of the effect of asphaltenes modification. The results of the two tests show similar trends, with an increase in RRI for the modified mixtures compared to the control (with a slight decrease for 2% asphaltenes-modified mixtures as compared to 1%). Considering the lower compactability and slight increase in air void content of the 2% asphaltenes mixture during the sample preparation process compared to the 1%, the difference in resistance of the 2% and 1% samples can be justified. In addition, the 56% increase in MQ for the 1% asphaltenes samples and the 46% increase for the 2% asphaltenes samples relative to the control samples are indicative of a higher stiffness for the

samples modified with asphaltenes. The MQ and RRI results exhibit similar trends, supporting the idea of higher resistance of the asphaltenes-modified samples to permanent deformation compared to the control samples.

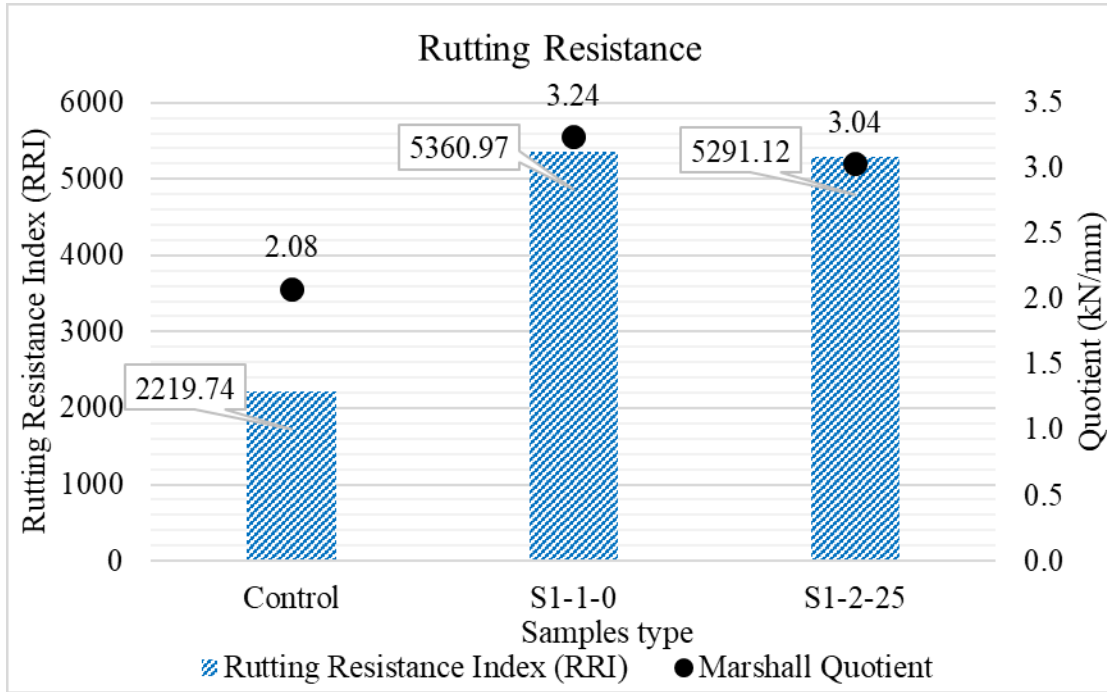


Figure 3-11 Rutting resistance properties

3.4.5. Discussion

Table 11 provides a comparison between the modified sample and control samples. According to the presented results, the addition of asphaltenes to the stabilized mixes has increased the tensile strength of the mixes in all dry, saturated and freeze/ thaw conditions. The maximum improvement is observed for dry mixes, followed by saturated and freeze/thaw conditioned samples. It is also concluded that higher asphaltenes content is more effective in improving tensile strength. However, the results show that the addition of asphaltenes could have a negative impact on TSR value, especially for the samples with freeze/thaw conditioning. The moisture sensitivity of the sample containing 2% asphaltenes is higher compared to those of 1% asphaltenes.

From Table 3-11, it is concluded that Marshall stability of asphaltenes modified samples is higher than the control mix, and increasing the asphaltenes content is increasing the stability. Both Marshall Quotient and RRI are significantly improved after the addition of 1%

asphaltenes. However, by increasing asphaltenes content from 1% to 2%, there was no improvement in these parameters and, consequently, in rutting resistance of the mixes. In conclusion, the addition of 1% asphaltenes seems to be more effective in enhancing the stabilized mixes performance properties compare to 2% asphaltenes.

Table 3-11 Comparison of performance parameters of modified samples to the control samples

Sample ID	Control	S1-1-0	S1-2-25
ITS Dry (%)	100.0	205.7	293.0
ITS Saturated (%)	100.0	184.3	213.7
ITS F/T (%)	100.0	144.1	168.4
Saturated TSR (%)	100.0	89.5	73.0
F/T TSR (%)	100.0	70.1	57.5
RRI (%)	100.0	241.5	238.4
Marshall Stability (%)	100.0	147.9	196.9
Marshall Quotient (%)	100.0	155.8	146.2

3.5. Conclusions

Based on the results and analysis described above, the following conclusions have been drawn:

- Using the mix design procedure recommended in MS-19, the OMC and OEC for this study were determined to be 6.3% per aggregate and 3.7% per total mixture, respectively. Furthermore, asphaltenes were found to increase the tensile strength and stability of the mixtures. Both levels of asphaltenes content (1% and 2%) were found to have higher tensile strength and stability than the control samples. In fact, the tensile strength and stability, respectively, were found to increase by 300% and 200% for 2% asphaltenes, and by 200% and 150% for the 1% asphaltenes. However, it was found that 2% asphaltenes in asphalt emulsion requires extra water to ease the mixing process.

- An increase in air void content was observed for the mixtures modified with asphaltenes. As a result, there was a slight decrease in the density of the asphaltenes-modified mixtures as compared to the control samples (decreases in density of 2%, 2.5%, and 5% were observed for S1-1-0, S1-2-25, and S2-1-0, respectively). The two asphaltenes sources were found to have similar effects on the stability of the mixtures, an observation, which confirms the repeatability and reliability of these effects.
- The TSR results for both saturated and F/T samples indicate that asphaltenes-modified mixes are slightly more sensitive to moisture damage compared to unmodified mixes. In addition, the SIP results from the wheel-tracking test showed same effects in moisture resistance of the asphaltenes-modified mixes in comparison to the control samples.
- Comparing the wheel tracking test results and MQ values within the mixtures prepared, a considerable increase in rutting resistance of the samples modified with 1% asphaltenes compared to the control mix was observed. However, the 2% asphaltenes did not improve the rutting resistance significantly compared to the samples modified with 1% asphaltenes. This could be related to the difficulties of compaction and higher air void content of the samples modified using 2% asphaltenes.

Chapter 4: Performance Evaluation of Stabilized Base

Course using Asphalt Emulsion and Asphaltenes

Derived from Alberta Oil Sands

4.1. Introduction

One strategy to enhance load distribution within the pavement to increase its bearing capacity and fatigue resistance is to strengthen the base course by increasing its thickness (Christopher et al., 2006). However, increasing the thickness has some drawbacks, such as increased cost, time, and environmental impact due to the additional materials used. Using higher quality material in the base course will result in higher resistance but will also increase the cost of construction of the layer. Base stabilization, on the other hand, is a promising solution that can overcome these problems (Little & Nair, 2009; Patel, 2019; Wegman et al., 2017). A well-designed base layer with sufficient thickness can reduce distress on the pavement layers (Wirtgen Cold Recycling Manual, 2012), while the tensile strength of the base layer can be improved through stabilization using an asphaltic or cementitious material (Christopher et al., 2006; Wirtgen Cold Recycling Manual, 2012).

Unbounded base courses generally consist of crushed stones and gravels that transfer the load to lower layers and resist it within the particles (Wirtgen Cold Recycling Manual, 2012; Yideti et al., 2014). However, these layers have low resistance to the tensile load (Adu-Osei, 2001; S. F. Brown, 1996). In this regard, studies have shown that stabilization of aggregate layers will result in higher shear strength, stiffness, durability, and resistance to moisture in asphalt pavements (Branch, 2005). Asphalt emulsion could be used for stabilization of these layers due to its advantages such as low-temperature application, less energy usage, lower emissions, and the fact that it is less hazardous than hot mix asphalts and more environmentally friendly than cutback asphalts (Salomon, 2006). However, disadvantages such as low rutting resistance, higher cracking, longer curing time for asphalt emulsion (to increase the bond between particles), and comparably poor early performance properties have limited its application (Du, 2016, 2018; Khweir et al., 2004). Indeed, some studies have indicated that it may take up to

three years for an asphalt emulsion-stabilized layer to reach maximum strength (Doyle et al., 2013; Marais & Tait, 1989; Quick & Guthrie, 2011).

The selection of the proper additive and the adequate amount to be added for stabilization has an important role in the performance properties of the stabilized layers (Betti et al., 2016; Patel, 2019; Wegman et al., 2017). Active fillers provide higher mechanical properties, stiffness modulus, permanent deformation resistance, moisture resistance, and fatigue strength when used for asphalt emulsion stabilizations (S. F. Brown & Needham, 2000; Giuliani, 2001; Hodgkinson & Visser, 2004). Another significant impact of active fillers on asphalt emulsions is that they reduce the breaking and curing time for asphalt emulsions (Terrell & Wang, 1971). Cement and lime as active fillers result in relatively slow strength gain and longer curing time. Environmental hazards of some active fillers due to the nature of their production is another drawback (Fang et al., 2016; Gutiérrez et al., 2012; Modarres & Ayar, 2016; Paoli et al., 2014). In addition, using cement and lime in stabilization for low-temperature conditions will not be as effective as conventional asphalt layers, as the low temperatures have adverse impacts on the strength gain of the stabilized layers (Soliman et al., 2014). However, the high-temperature properties of the mixtures with active fillers such as cement, hydrated lime, and slag could be enhanced with specific contents of additives (Du, 2015; Gutiérrez et al., 2012; Modarres & Ayar, 2016; Paoli et al., 2014; Soliman et al., 2014). The rigidity of the mixture after stabilization, adverse effects on the environment, shrinkage cracking, as well as several other drawbacks in cement stabilization make it unsuitable for application in cold climate regions (Perraton et al., 2011; M. A. Shafii et al., 2011).

Asphaltenes are residue material produced through deasphalting process of bitumen that has no significant application in the road construction industry. In addition, asphaltenes are a component of asphalt binders alongside saturates, resins, and aromatics (collectively referred to as “SARA”) (Sultana & Bhasin, 2014). Asphalt as a viscoelastic material is made of polar and non-polar components, the polar components being responsible for the elastic behaviour while the non-polar components govern the viscous behaviour (Behnood & Modiri Gharehveran, 2019; Ramirez-Corredores, 2017; Sultana & Bhasin, 2014; Y. Xu et al., 2019). Adding asphaltenes to asphalt materials will result in a stiffening of the mixture, thereby enhancing its mechanical properties (Sultana & Bhasin, 2014). The use of asphaltenes as an

additive in asphalt emulsion-stabilized mixtures has not yet been studied, and, considering the effects of this material on the asphalt binder properties, similar enhancements to other additives are expected in stabilized mixtures. The presence of asphaltenes in the asphalt binder also raises the matter of the compatibility between the two materials, which could be a key factor in the flexibility of the final mixture.

4.1.1. Objective and Scope

The main objective of this study was the evaluation of the low and high-temperature properties of a well-graded granular base course stabilized with a slow setting (CSS-1H) asphalt emulsion. The layer was modified with a source of asphaltenes derived from Alberta oil sands bitumen. A mix design for the stabilized base course was determined and compared to the modified mixes using the asphaltenes. Indirect tensile strength (ITS), creep compliance and strength, and Hamburg wheel-tracking tests were conducted in order to evaluate the low and high-temperature mechanical properties of the mixtures.

4.2. Materials and Mix Design

4.2.1. Aggregates

Single source aggregates were used in the mixture preparation for this study. Based on the limitations provided by Wirtgen Cold Recycling Manual (2012), the City of Edmonton (2015), and Alberta Transportation in accordance with Soliman et al. (2014), the aggregate gradation to be used was determined to be well-graded and within the limitations stated. Figure 4-1 illustrates the final gradation and aggregate envelopes for three different specifications. The aggregate gradation skeleton consisted of 57.27% coarse aggregates, 36.73% fine aggregates, and 6% filler. The aggregates were tested to determine their physical properties, with the results presented in Table 4-1. The bulk specific gravity of the aggregates (G_{sb-agg}) was also determined and was found to be 2.601. The filler amount and Los Angeles abrasion value for the aggregates were controlled to be within the parameters specified in the Wirtgen Cold Recycling Manual (2012) and City of Edmonton standards (2015).

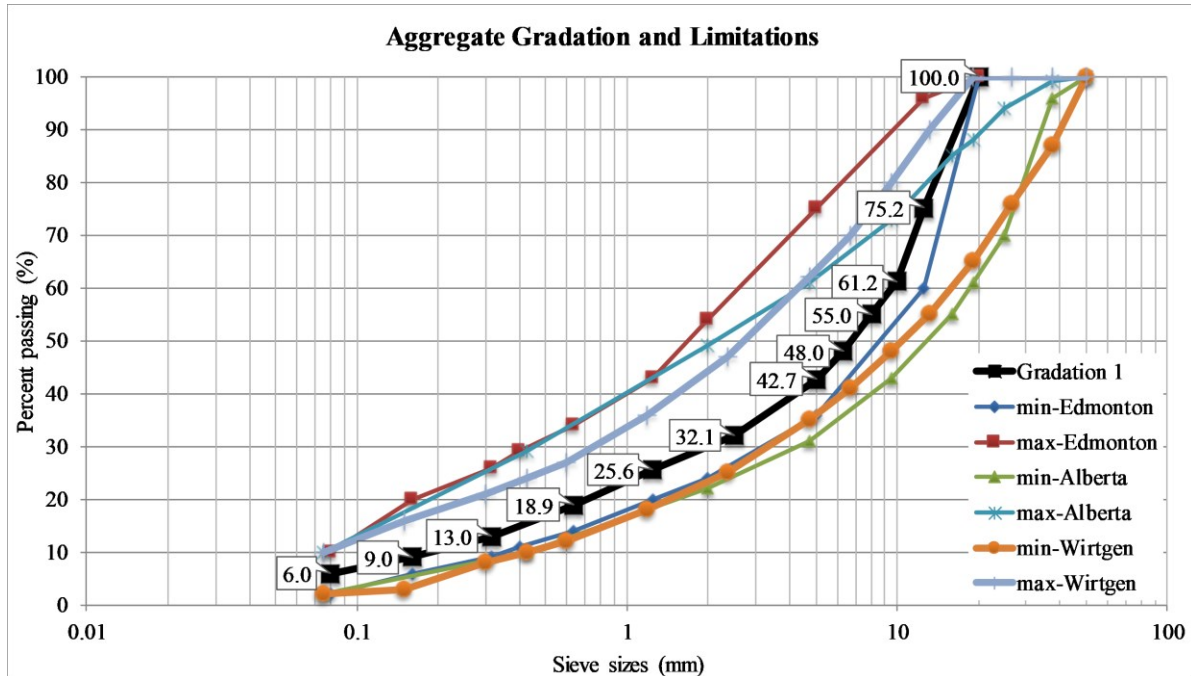


Figure 4-1 Target aggregate gradation and limitations

Prior to mixture testing, the optimum moisture content was determined in accordance with the proctor test with respect to ASTM D698 specification (2021) for the aggregate gradation and the results shown in Table 4-1.

Table 4-1 Aggregate properties

Property (unit)	Standard	Result
Amount of material finer than 75- μ m (No. 200) sieve in aggregate (%)	ASTM C117 (2017)	6
Fine aggregates	Specific gravity (G _{fa})	2.604
	Absorption of water (%)	0.624
Coarse aggregates	Specific gravity (G _{ca})	2.598
	Absorption of water (%)	0.870
Abrasion of coarse aggregates (%)	ASTM C131 (2020)	23
Proctor test	OMC (%)	6.3
	Dry Density (kN/m ³)	(Modified D1557 (2000)) 15.4 max.

4.2.2. Asphalt Emulsion

Cationic slow setting (CSS) is the typical asphalt emulsion used for base stabilization due to the aggregate charge and the minimum time required for the mixing and laying of the base layer (Wirtgen Cold Recycling Manual, 2012). Workability time to allow asphalt emulsion

sufficiently to disperse in the mixture is an essential parameter to be considered in this type of mixture. The asphalt emulsion used in this study contains 61% hard binder and 39% water, and it is classified as CSS-1H. The asphalt binder that was used to prepare the emulsion has a penetration of 80/100 and a viscosity of 0.42 Pa·s at 135 °C. Various tests were conducted on the asphalt emulsion, and the results are presented in Table 4-2.

Table 4-2 Properties of CSS-1H asphalt emulsion (Husky Asphalt, 2019)

Property (unit)	Standard ASTM/AASHTO	Specification		Typical Analyses
		Min.	Max.	
Tests on Emulsion				
Specific gravity (Density) at 15.6 °C (kg/L)	T59 (2022)	-	-	1.020
Residue by distillation (% by mass)	T59 (2022)	57	-	61
Viscosity at 25 °C (S.F.S)	T59 (2022)	20	100	22
Oversized particles (sieve) (% by mass)	T59 (2022)	-	0.300	0.008
Settlement (24 hours) (% by mass)	T59 (2022)	-	1.0	0.5
Particle charge test	D7402 (2017)	Positive		Positive
Tests on Asphalt Residue				
Penetration at 25 °C (100 g, 5 s) (dmm)	T49 (2019)	40	125	95
Ductility at 25 °C (5 cm/min) (cm)	T51 (2018)	40	-	> 40
Solubility in Trichloroethylene (% by mass)	T44 (2018)	97.5	-	> 97.5

4.2.3. Asphaltenes

The asphaltenes used in this study were sourced from Alberta oil sands bitumen, having been processed through a solvent deasphalting process. The asphaltenes were obtained in solid form as shown in Figure 4-2 and, to achieve adequate dispersion, they were crushed into powder and sieved through a No. 100 sieve. Considering that the properties of the asphaltenes from different sources depend on the oil and processing method used to produce it, initial tests had to be conducted to determine its percentage purity as well as its chemical composition.

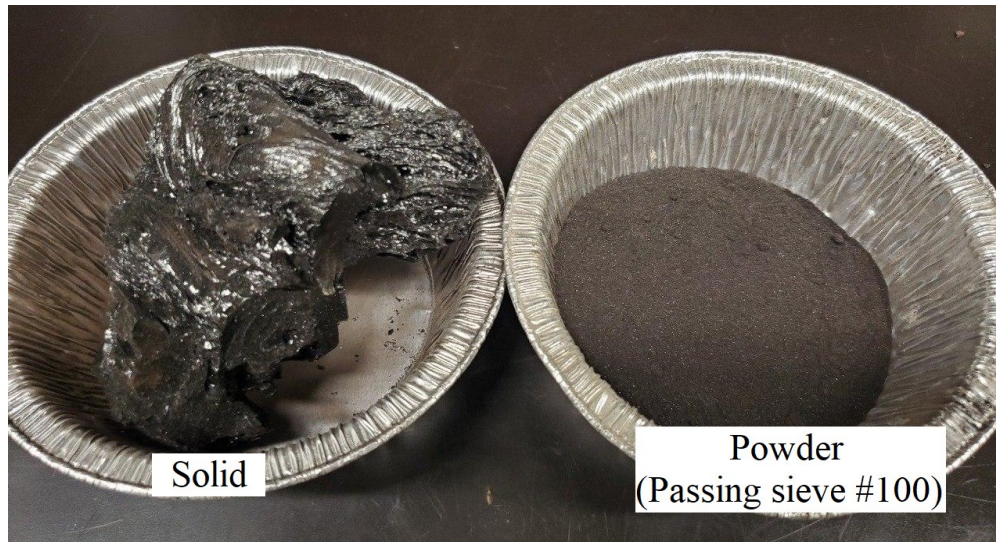


Figure 4-2 Asphaltenes provided for mixing

In addition, the SARA test was conducted for the asphaltenes sample to determine the relative quantities of saturates, asphaltenes, resins, and aromatics. According to the results of the SARA content analysis, it was found to be composed of 79.63% asphaltenes, and 9.68% aromatics, 6.85% saturates, and 3.84% resin.

4.2.4. Composition of Mixtures and Optimum Emulsion Content (OEC)

Asphalt Institute specifications (2008) were used to design the mixtures for the well-graded granular base aggregate stabilization. The approximate asphalt emulsion percentage to be used based on this method was calculated with respect to the relationship described in Equation (4-1) using the result from the ASTM D6997 (2012). The amount of the material retained after distillation was determined to be 61% according to the test results.

$$\text{Base mixture: Asphalt Emulsion \%} = \frac{(0.06B + 0.01C)100}{A} \quad (4-1)$$

where: A = Percentage of residue of asphalt emulsion remaining after distillation (as determined using ASTM D6997 (2012)), B = Percentage of dry aggregate passing through a No. 4 sieve, C = Percentage of dry aggregate retained on a No. 4 sieve.

Using this method, the approximate asphalt emulsion content was determined to be 4.89% per total mixture, and four different contents with 1% intervals were prepared in order to determine

the OEC with respect to Marshall stability and flow as well as ITS test. Considering the result from the proctor test for the optimum moisture content (OMC) of aggregates, additional water was used in conjunction with the oven-dried aggregates after cooling down to room temperature and mixed until the water was uniformly distributed; this was done in order to achieve the optimum moisture content in ambient temperatures to prevent loss of moisture. Asphalt emulsion was introduced to the wet aggregates in the contents determined in the design matrix and compacted using a Marshall hammer with 50 blows on each side of the sample.

4.2.4.1. Marshall Stability and Flow Test for OEC

ASTM D6927-15 (2015) standard was used to perform the Marshall stability test for the samples of different asphalt emulsion contents as determined in the design matrix in order to select the OEC. The samples were cured in the compaction molds for 48 hours in an oven at 60 °C following the compaction with the Marshall hammer. After conditioning, the samples were extracted from the molds and cooled at room temperature for at least two hours. For each of the emulsion contents, three replicates were prepared for the purpose of Marshall stability testing. The stability testing was conducted after conditioning of the samples in a 25 °C air bath for a period of 3 hours (Asphalt Institute, 2008).

According to the results of the stability test, 3.98% asphalt emulsion content per total weight of the mix provided the highest stability for the mixtures; however, the trends for density and stability show that maximum values were attained at about 3.7% asphalt emulsion (Figure 4-3). This proportion of asphalt emulsion per total mix was thus chosen as the OEC for the performance tests. It should be noted that ITS test has been suggested by different researchers as an effective means of confirming the results obtained from Marshall stability testing (Du, 2015; Wirtgen Cold Recycling Manual, 2012).

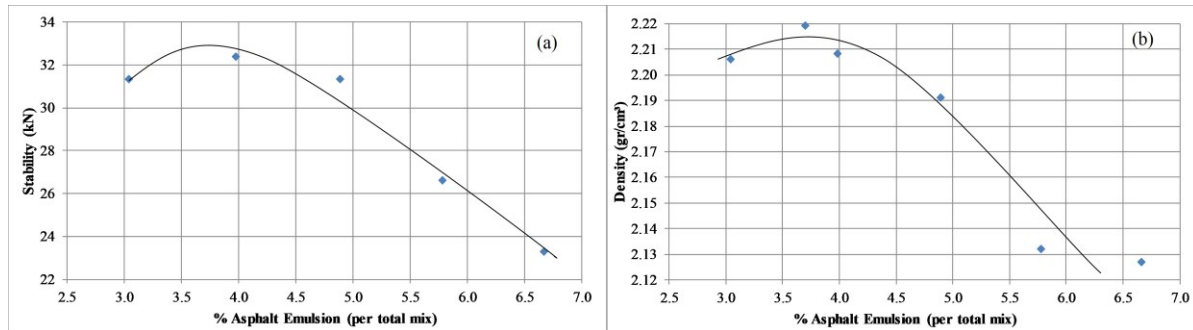


Figure 4-3 (a) Stability and (b) density values for asphalt emulsion design matrix

4.2.4.2. Indirect Tensile Strength

ITS test was conducted in accordance with AASHTO T283 (2018). Similar contents of asphalt emulsion as determined in the design matrix were used to prepared samples for this test. Each sample was prepared with three replicates. Marshall hammer was used for compaction of the samples, which were cured using the same process as explained in the description of the Marshall test above. The samples were subjected to 3 hours of conditioning in an air chamber at 25 °C, in accordance with AASHTO T283 (2018), prior to testing under a load applied at a rate of 50 mm/min. The maximum load applied to the samples before the failure point was recorded in order to determine the ITS. The ITS for each of the samples was calculated using Equation (4-2).

$$S_t = \frac{2000P}{\pi tD} \quad (4-2)$$

where: S_t = Indirect tensile strength (kPa), P = Maximum applied load (N), t = Average height of the specimen (mm), D = Diameter of the specimen (mm)

The same asphalt emulsion concentrations were used to prepare the samples for the ITS test, and the results from the ITS test showed the same trend as those from the Marshall test (Table 4-3). Considering results from both the Marshall and ITS tests, a 3.7% of asphalt emulsion per total mixture weight for OEC was selected. A minimum value of 225 kPa was identified as the lower limit for asphalt emulsion-stabilized material, in accordance with the Wirtgen Cold Recycling Manual (2012). The ITS was calculated as 298 kPa for the optimum emulsion

content (3.7% of asphalt emulsion per total mixture weight) that satisfies the minimum requirement.

Table 4-3 ITS values for mix design

Asphalt emulsion (per total mix)	ITS (kPa)	Air void (%)
3.04	288	13.302
3.70	298	10.934
3.98	284	14.165
4.89	270	15.702
5.78	276	16.641

4.2.4.3. *Asphaltenes Modification Matrix and Sample Preparation*

The OEC of 3.7% by weight of total mixture was used to prepare the asphaltenes-modified mixtures throughout the study. The control samples were prepared in a manner similar to the way the design samples for OEC had been prepared and were compared to the asphaltenes-modified samples. According to the mix design test results, asphaltenes could be added to the dry aggregates before mixing with asphalt emulsion or added to the asphalt emulsion prior to mixing with the aggregates. Due to the relative ease with which asphaltenes can be mixed in the asphalt emulsion compared to mixing with aggregates, this method was selected with respect to the experimental observations. Proportions of 1%, 2%, and 3% of asphaltenes by total mixture weight were added into the asphalt emulsion and mixed with the aggregates. Three replicates prepared for each level of asphaltenes content. Among the proportions, the 1% asphaltenes samples were found to be the easiest to prepare, as the asphaltenes content is relatively little. Increasing the asphaltenes content made preparation more difficult, as the asphaltenes caused the emulsion to fast-break and made mixing more difficult. To ease the mixing process, then, more water was added to the asphalt emulsion for the 2% and 3% asphaltenes samples to make them less viscous and easy to mix. Table 4-4 presents the test matrix for the performance tests.

Table 4-4 Design matrix for asphaltenes modification

Sample ID	Asphaltenes (% per total mix)	Extra water (% per total emulsion)
S1-1-0	1	0
S1-2-25	2	25
S1-2-50	2	50
S1-3-50	3	50

4.3. Testing Program

4.3.1. Indirect Tensile Strength

ITS test was conducted in accordance with the AASHTO T283 standard (2018) using the design matrix presented in Table 4 and the OEC of 3.7% per total mixture. Three replicates of the samples were prepared at each of the asphalt emulsion contents, and Marshall hammer was used for compaction using the same procedure described above. The samples also underwent 3 hours of conditioning in an air chamber at 25 °C prior to testing. A load with a rate of 50 mm/min was applied on the samples after conditioning, and the maximum value of strength before failing was recorded for each sample to determine the ITS of the samples using Equation (4-2).

In order to calculate the Tensile strength ratio of modified and unmodified samples, 3 extra samples were prepared and cured in the same process. Samples were tested in similar condition as dry subsets according to standard after saturation at 25°C for 24hours in water and TSR value calculated using the ITS of dry subsets over saturated subsets. Wirtgen cold recycling manual (2012) used to control the limits that determined to be 50% for TSR values.

4.3.2. Creep Compliance and Indirect Tensile Test

AASHTO T322-07 (2020) was used to perform the creep compliance and strength test of the mixtures using the ITS test setup. The purpose of this test is to capture the low-temperature performance of the mixtures. Marshall samples with 3 replicates for each of the asphaltene contents were prepared. However, in this case the surface of the samples was cut to the height of 38 mm to 50 mm. The test temperatures used in this study were 0 °C and -10 °C considering the base layer and PG grading of the binder used to prepare the asphalt emulsion. The samples were conditioned in the air chamber for 3±1 hours prior to testing. A fixed static load was applied on the specimens for 100±2 seconds, and Linear Variable Differential Transformers (LVDTs) were used to record the deformation of the samples along both the horizontal and vertical axes. After completion of the creep test, an ITS test using a rate of 12.5 mm/minute was applied to the samples until the failure point of the samples had been reached. Figure 4-4 presents the test setup and shows images of a sample prior to and

after testing. The fracture energy (FE) and the ITS of the samples for both temperatures was calculated using the load-deformation values obtained from the test.

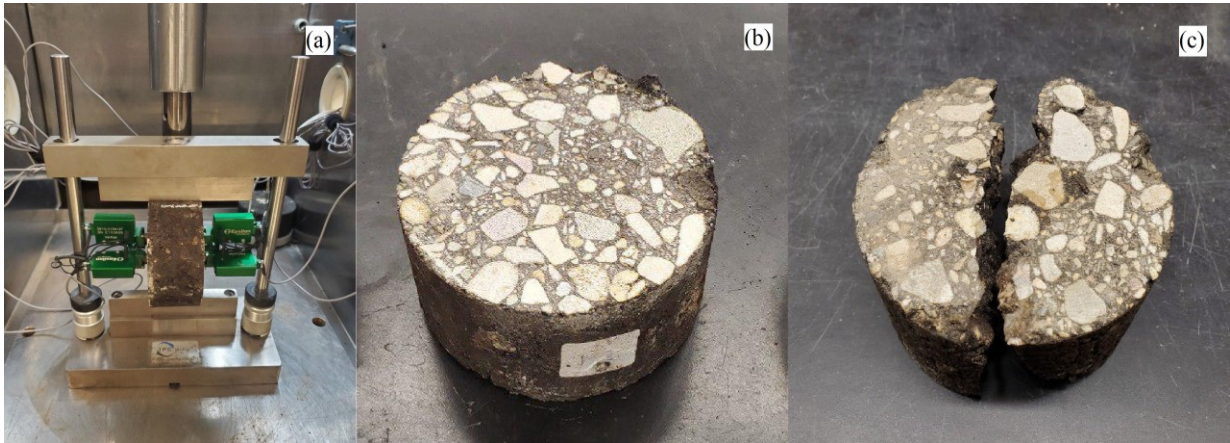


Figure 4-4 (a) IDT test setup, (b) sample prior to testing, and (c) sample after testing

4.3.3. Hamburg Wheel-Tracking Test

A Hamburg wheel-tracking test was performed in accordance with AASHTO T324-19 (2019). Considering the nominal maximum aggregate size, slab samples with dimensions of 400 mm length, 300 mm width, and 80 mm height were used for the test. A roller compactor was used for compaction of the samples, and a same curing process was followed. The test temperature for the wheel-tracking test was determined to be 40 °C considering the binder grade and base layer location. Following the standard noted above, the samples were preconditioned in water for 45 minutes prior to testing. A steel wheel with 47 mm width and 705 ± 4.5 N weight at frequency of 52 ± 2 passes per minutes and a maximum speed of 0.305 m/s at midpoint was used for this test. A maximum of 20,000 passes or 12 mm rutting depth, whichever was achieved first, was considered as the termination point for the test. The rut depth value and number of passes were used to determine the rutting resistance index (RRI) and stripping inflection point (SIP) in order to evaluate the rutting potential and susceptibility of the mixtures to moisture damage.

4.4. Results

4.4.1. Indirect Tensile Strength

The ITS test was conducted on both the modified and unmodified mixture samples, with the results presented in Figure 4-5. The results from the ITS test show that modification with

asphaltenes had a significant effect on the strength of the samples. The S1-2-25 samples with 2% asphaltenes were found to have the best response in terms of tensile strength; however, an increase in asphaltenes content to 3% was found to cause a decrease in tensile strength of the modified mix. To understand the effect of adding extra water to the mix, samples with 2% asphaltenes but different amounts of extra water content (S1-2-25 and S1-2-50) were compared, and the results indicate that this increase in water content had an adverse effect on the tensile strength of the modified samples. However, as a practical consideration with respect to ease of mixing, this extra water would seem to be necessary for higher levels of asphaltenes content.

Figure 4-5 also presents results for tensile strength ratio test for samples tested after conditioning at 25°C inside water bath for 24hrs. Control samples for saturated samples had almost similar results as dry subsets which indicates that TSR is about 1. However, this values decreases after modification of samples to 0.9 and 0.7 for 1% and 2% samples. This indicates that moisture sensitivity of the modified samples increases. Considering the Wirtgen cold recycling manual (2012), this values should not be lower than 0.5 for saturated stabilized mixtures with emulsified asphalt. It can be seen that the results are satisfying the 50% lower limits for base courses in the targeted mixtures.

The ITS test results for the set of samples with better performance were used to select samples for performance testing. In this respect, S1-1-0, S1-2-25, and the control samples were selected for further performance testing to evaluate their relative performance. The criteria underlying this selection with respect to the resistance of the samples to tensile strength were a higher asphaltenes content and minimal added water. The air void contents was calculated to be 11.4, 11.6, and 13.0, for the control, S1-1-0, and S1-2-25, respectively. These results indicate an increase in air void for the asphaltenes-modified samples. The ITS test results show that all samples satisfied the lower limit for asphalt emulsion stabilization (225 kPa) as determined by Wirtgen Cold Recycling Manual (2012).

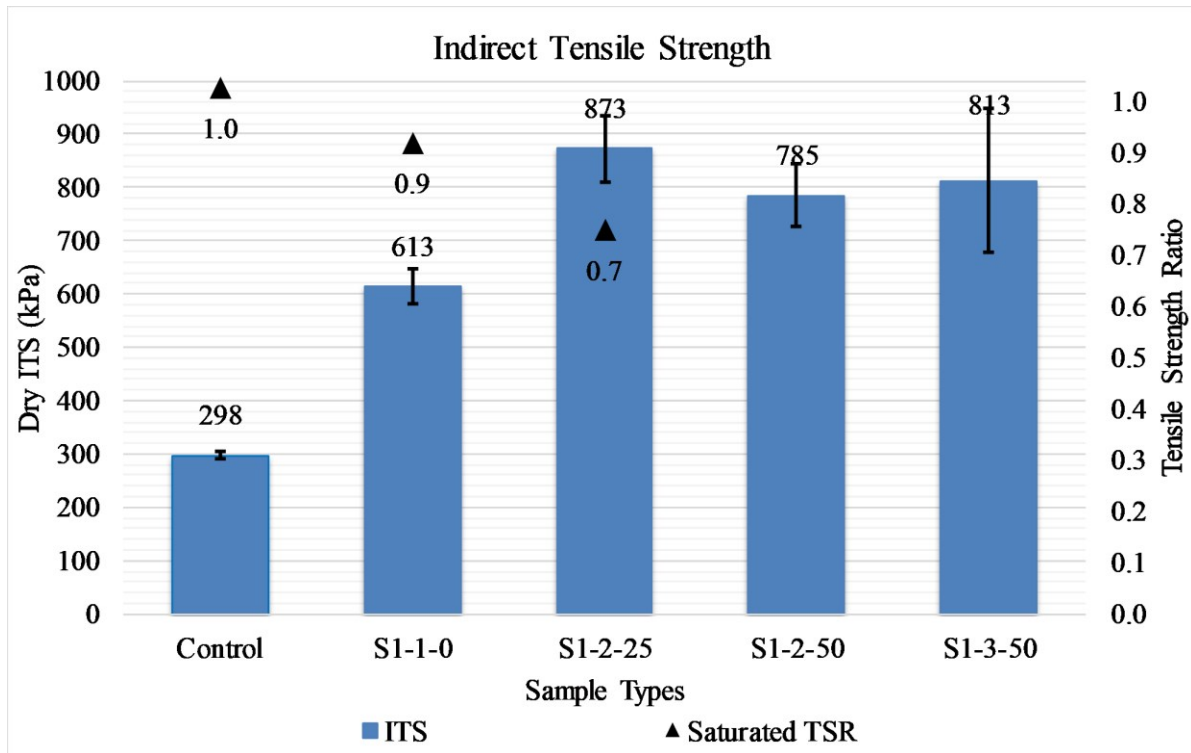


Figure 4-5 Indirect tensile strength and tensile strength ratio for control and asphaltene modified mixes

4.4.2. Creep Compliance and Indirect Tensile Test

The creep compliance test (AASHTO T 322, 2020) was conducted at temperatures of 0 °C and -10 °C for both the modified and unmodified samples. The vertical load versus deformation graphs are presented in Figure 4-6 for 0 °C and Figure 4-7 for -10 °C. Table 4-5 presents the ITS and FE values calculated from the load-deformation graphs for the samples at both temperatures. The results of the ITS at 0 °C show that the maximum failure load increased slightly, while the FE for the modified samples decreased (8% and 7.5% increases in tensile strength and 24% and 17.7% decreases in FE of the 1% and 2% asphaltene samples, respectively, were observed). Comparing these results at 0 °C for the different levels of asphaltene content, it can be seen that there was not a significant difference observed with respect to either tensile strength or FE as a result of an increase in asphaltene content from 1% to 2% asphaltene. However, the slope of the graphs after the peak point for the modified samples are steeper, and this is indicative of more rapid crack propagation. According to these observations and the given mixing procedure, the samples with 1% asphaltene content were found to be more efficient than the samples with 2% asphaltene content. These results are

indicative of stiffness in the asphaltenes-modified samples, as was expected. The test results at $-10\text{ }^{\circ}\text{C}$ showed higher values for ITS and FE as compared to those at $0\text{ }^{\circ}\text{C}$, except for 2% asphaltenes content that had a decrease for both tensile strength and FE. Increasing the asphaltenes content in $-10\text{ }^{\circ}\text{C}$ results in decrease of tensile strength in contrast to $0\text{ }^{\circ}\text{C}$. The reduction in FE was also observed to be more pronounced in the 2% asphaltenes samples than in the 1% asphaltenes samples. The reductions in tensile strength were found to be approximately 7.5% and 23.9% for the 1% and 2% asphaltenes samples, respectively, while the FE decreased by 25.1% and 21.9%. With respect to these results, it is apparent that modification of mixtures with a higher concentration of asphaltenes content makes the sample weaker and more prone to cracking at lower temperatures.

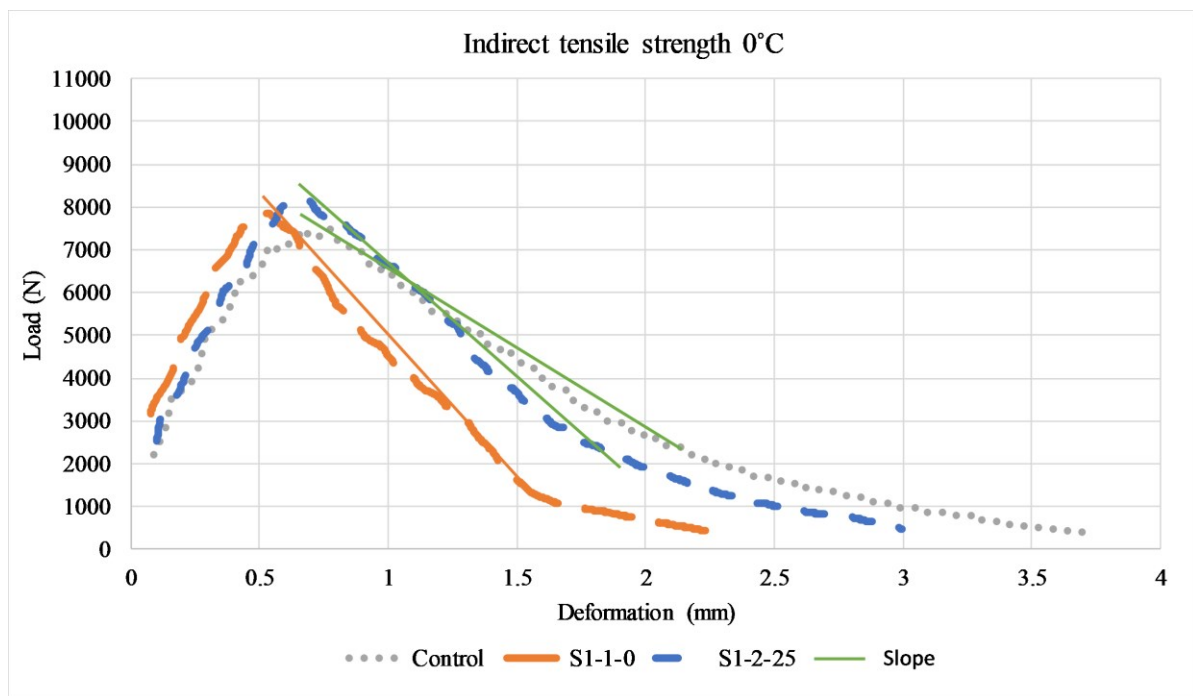


Figure 4-6 Load-deformation results for $0\text{ }^{\circ}\text{C}$

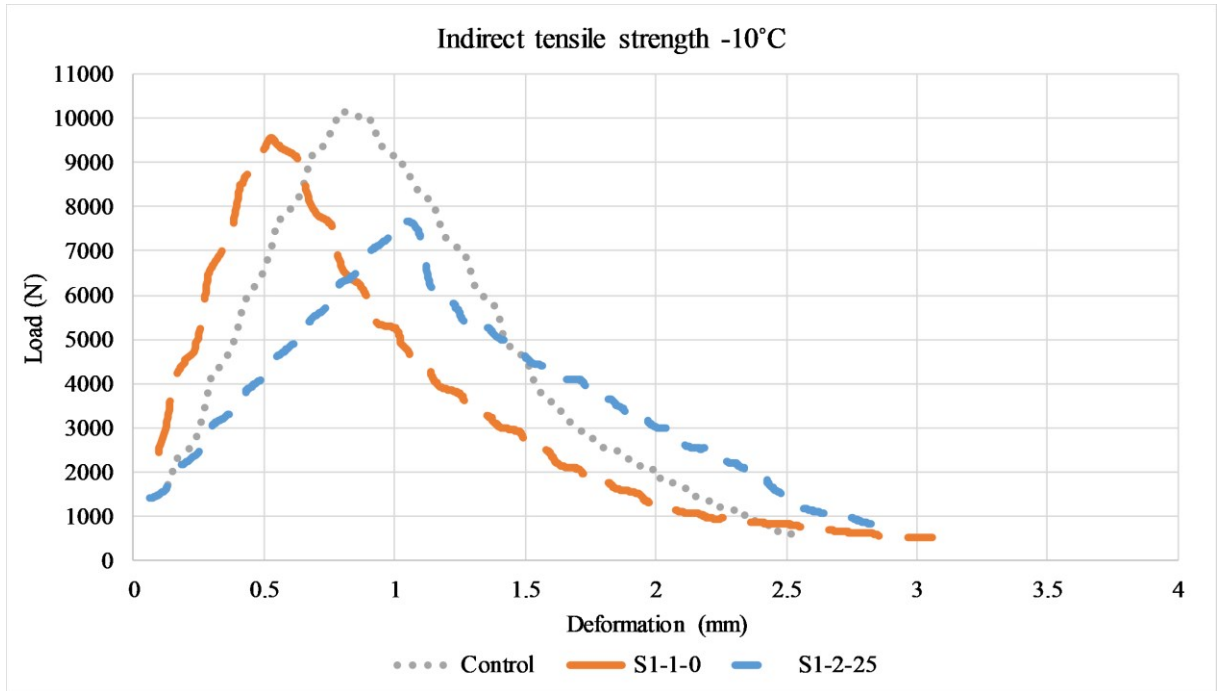


Figure 4-7 Load-deformation results for $-10\text{ }^{\circ}\text{C}$

Table 4-5 Tensile strength and fracture energy for creep test samples

Sample ID	Temperature ($^{\circ}\text{C}$)	Tensile Strength (kPa)	Fracture Energy (J/m^2)
Control		1083.09	2437.33
S1-1-0	0	1170.05	1852.65
S1-2-25	0	1163.99	2006.42
Control		1357.34	2713.02
S1-1-0	-10	1255.27	2032.22
S1-2-25	-10	1033.29	2118.12

4.4.3. Hamburg Wheel-Tracking Test

The Hamburg wheel-tracking test (AASHTO T 324, 2019) was used to determine both the moisture susceptibility and the rutting parameter of the samples. Figure 4-8 presents the prepared slab samples before and after conducting the test, while Table 4-6 presents the test results, which show an increase in the number of passes before failure for the modified samples as compared to the control. This increase for the modified mixes indicates a higher resistance to rutting. In addition, the SIP value for the control samples was found to be approximately 3,800 passes, whereas adding asphaltenes increased this value to 8,200 and 7,400 passes for the 1% asphaltenes and 2% asphaltenes samples, respectively. Furthermore, both the 1% and

2% asphaltenes samples showed a significant increase in rutting resistance performance compared to the control samples. The moisture sensitivity of the samples, calculated by dividing the SIP values by the number of passes, had a decreasing trend and indicates the sensitivity of the modified samples to moisture. This index for control samples is about 0.96 and for modified samples decreased to 0.94 and 0.86 for 1% and 2% asphaltenes respectively. The RRI was also calculated using the number of passes and the depth of rutting for the samples. As mentioned above, these high values for the modified sample indicate a higher rutting resistance for the asphaltenes-modified samples in comparison to the control samples. In fact, an increase in rutting resistance was observed from 2219.74 for control samples to 5360.97 and 5291.12 for 1% and 2% samples respectively. Both the 1% asphaltenes content and 2% asphaltenes content samples showed better rutting resistance compared with the control samples; however, for the 2% asphaltenes, a slight decrease of about 69.85 RRI was observed compared to the 1% asphaltenes sample. This decrease may be attributable to the slight increase in air voids and decrease in compactibility of the 2% asphaltenes samples compared to the 1% asphaltenes samples. As a result of this test, it can be concluded that asphaltenes modification significantly increased the permanent deformation resistance in the samples.

Table 4-6 Rutting resistance and moisture sensitivity results

Sample ID	SIP	Number of passes at 12 mm rutting	RRI	Moisture sensitivity index
Control	3,800	3,940	2,219.74	0.96
S1-1-0	8,200	8,712	5,360.97	0.94
S1-2-25	7,400	8,604	5,291.12	0.86



Figure 4-8 Slab samples compacted by roller compactor (a) prior and (b) after test

4.5. Discussion

Figure 4-9 presents the comparative data for the Creep Compliance, ITS, and Hamburg wheel-tracking tests. The control samples for each test were taken into consideration in comparison to the S1-1-0 and S1-2-25 samples in order to gain understanding of the effect of modification for the base layer with asphaltenes. The strength of the samples at 25 °C was found to be significantly higher about 106% and 193% for the modified samples at both 1% and 2% asphaltenes contents compared to control samples. Comparing the results of the ITS at 0 °C, a slight increase in strength of the mixtures was observed as a result of increasing the asphaltenes content. This improvement was about 8.0% and 7.5% for 1% and 2% asphaltenes. Whereas a decrease in the strength of the samples as a result of asphaltenes modification was observed at -10 °C about 7.5% and 24% for 1% and 2% asphaltenes, respectively. In contrast to the results for the intermediate temperature, which showed an increase of about 63% for 1% asphaltenes and 106% for 2% asphaltenes in FE, the results for both low temperatures show a slight decrease in FE for the modified samples. This decrease for 0 °C was 24% and 18% for 1% and 2% asphaltenes and for -10 °C was about 25% and 22% for 1% and 2% asphaltenes, respectively. These outputs from the creep test indicate an increase in brittleness of the mixes after adding the asphaltenes to the asphalt emulsion-stabilized samples. Moreover, while increasing the asphaltenes content has a considerable increasing effect on the strength of the samples for intermediate temperatures, a similar effect was not observed at the lower temperatures.

The results of the Hamburg wheel tracking test and RRI values show that modification of asphalt emulsion-stabilized mixtures with asphaltenes has a significant impact on the rutting resistance (e.g., with respect to the high-temperature performance of the mixtures, increased 141.5% for 1% asphaltenes and 138.4% for 2% asphaltenes in rutting resistance). The samples with 1% asphaltenes content were also found to be more effective compared to 2% asphaltenes in terms of improving rutting resistance. This could be related to the ease of mixing and compaction of modified mixes using 1% asphaltenes, no need to extra water during the mixing process and consequently, lower mixes air void content.

The stiffness of the modified samples was found to increase at high temperatures according to the wheel tracking results. This stiffening trend continued for the intermediate temperatures as per the results of the ITS test. However, the ITS results for the low temperatures do not show a significant variation in stiffness in the modified samples corresponding to the trends in the ITS results. Considering the overall results for the low, intermediate and high-temperature tests, it can be concluded that the samples with 1% asphaltenes content are more effective at enhancing the performance properties than the other samples. Also, the impact of 1% of asphaltenes on improving mixes intermediate and high temperature properties overweighs its adverse effect on low temperature properties of the mixes.

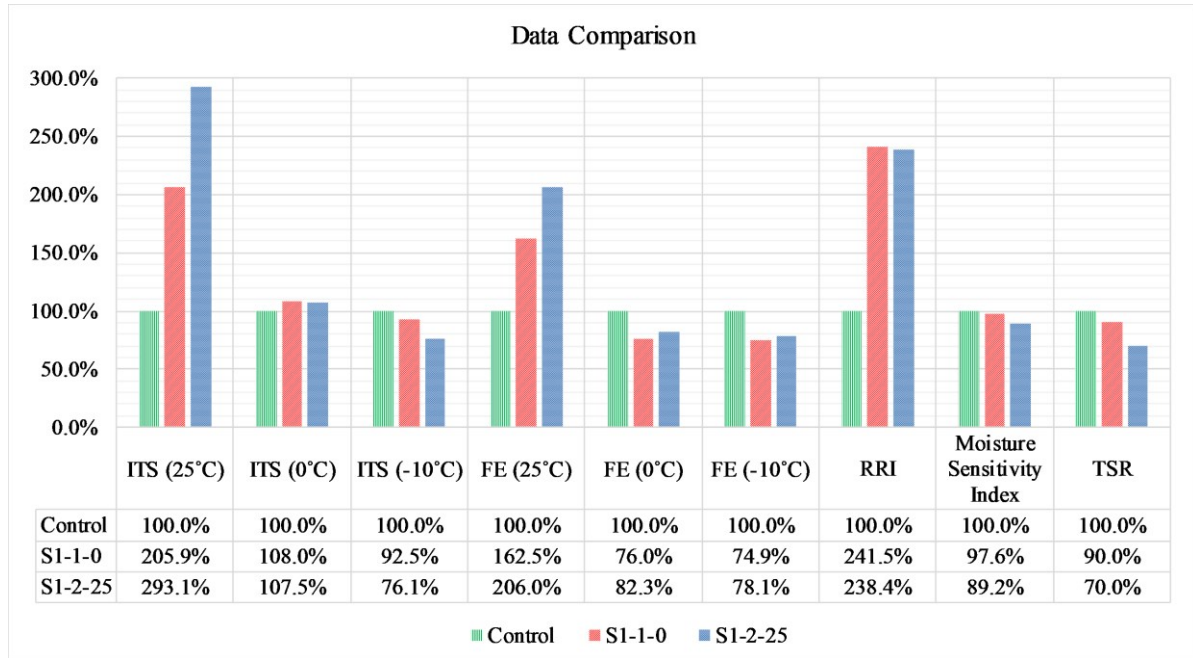


Figure 4-9 Comparison of performance properties parameters of asphaltens modified mixes with the control samples

4.6. Conclusions

Based on the results of the various performance tests and analyses conducted in this study, the following conclusions have been drawn:

- The tensile strength at 25 °C was found to increase by 105.9% and 193.1% for the samples with 1% and 2% asphaltens content, respectively. However, it should be noted that the samples with 2% asphaltens content required extra water to increase the viscosity during mixing with aggregates.
- The tensile strength ratio decreased by 10% and 30% for 1% and 2% asphaltens contents, respectively. It shows that addition of asphaltens will increase the moisture sensitivity of the mixes. However, it was not significant for modified mixes using 1% of asphaltens.
- IDT results showed that modification of the asphalt emulsion-stabilized material with asphaltens resulted in lower fracture energy values and consequently, increase in the samples brittleness at lower temperatures. However, the tensile strength of the modified samples were slightly lower than the control samples at lower temperatures.

- The Hamburg wheel-tracking test results are indicative of a notable improvement in rutting resistance of the modified mixtures compared to the unmodified samples. The RRI index was found to increase by 141.5% and 138.4% for both the 1% and 2% asphaltenes content samples compared to the control samples.
- According to the SIP and moisture sensitivity values, all the samples (both the control samples and the different modified samples) were found to be sensitive to moisture and asphaltenes modification was found to decrease this parameter by about 2% and 10% for the 1% and 2% asphaltenes-modified samples, respectively. The results show the same trend that was observed from the TSR values for 1% and 2% asphaltenes content.
- According to the results of the performance tests at different low, intermediate and high-temperature conditions, it can be concluded that 1% asphaltenes content is more effective than 2% asphaltenes content for asphaltenes-modified asphalt emulsion-stabilized base courses.
- Asphaltenes as a residue material have similar, or in some cases, better impacts on the asphalt emulsion-stabilized courses in comparison to the various commonly used active fillers. This material could be used as an inexpensive and environmentally friendly alternative to improve the properties of mixtures.

Chapter 5: Assessment of Performance Characteristics of Recycled Base Course Incorporating Reclaimed Asphalt Pavement and Asphaltenes

5.1. Introduction

The road construction industry has made great advances in recent years. However, factors such as low-quality materials and inadequate quality control have resulted in serious distresses in pavements that necessitate significant maintenance or reconstruction. Meanwhile, the replacement of deteriorated asphalt pavement generates a considerable amount of waste materials, known as reclaimed asphalt pavement (RAP). Moreover, reconstruction necessitates the use of large volumes of virgin aggregates. The Federal Highway Administration (FHWA), in the early-1990s, estimated that more than 90 million tons of asphalt pavement were being reclaimed annually at that time (Copeland, 2011; Federal Highway Administration, 1993). This number increased to 100 million tons annually by 2008 (Federal Highway Administration, 2008). RAP is a valuable material consisting of aggregates and asphalt binder that can replace expensive virgin aggregates and binders. Looking at the cost breakdown of asphalt mixture production, materials, plant production, trucking, and laydown; materials are the most expensive portion, accounting for 70% of the cost (Copeland, 2011). Replacing virgin aggregates with RAP brings economic and environmental benefits, and is a sustainable option for road construction since it reduces the amount of material directed to landfills and mitigates the greenhouse gas emissions associated with road construction (McDaniel & Anderson, 2001; Mohd Hasan & You, 2015; Newcomb et al., 2016; Y. Wang et al., 2018). Recycling methods such as replacing the RAP with virgin aggregates have been gaining popularity in recent years due to growing concerns about the adverse effects of using natural resources in the pavement construction industry being among the largest consumers of raw materials (Horvath, 2003; Sangiorgi et al., 2017). Recycling of asphalt pavement dates back to as early as 1915, but it was not a common practice until the Arab oil embargo of the 1970s (Kandhal, 1997; West & Willis, 2014). During this crisis, the highway industry developed milling machines to remove the distresses from asphalt layers and recycle the existing asphalt. RAP recycling technology

today consists of hot in-plant recycling, hot in-place recycling, full-depth reclamation, cold in-plant recycling, and cold in-place recycling (Miliutenko et al., 2013).

Cold recycling is a reliable method that, compared to hot recycling, is a more environmentally friendly method that also leverages the benefits of asphalt emulsion as a recycling agent (Miliutenko et al., 2013). The higher cost of hot mix asphalt (both plant production and the cost of trucking it to the site) also underscores the benefit of using cold-recycled RAP as an alternative to hot mix asphalt (Copeland, 2011).

RAP recycling has been used in different layers and mixes at various concentrations ranging from 10% to 100% (Arimilli et al., 2015; Dal Ben & Jenkins, 2014; Flores et al., 2019; Harshita et al., 2022; Taha et al., 2002). In pavement construction in the United States, a RAP content of 20% to 30% is typically recommended for the wearing and binder asphalt layers. However, European countries allow up to 50% RAP for the base layers of the pavement, and the production of these mixtures with RAP is growing (Dinis-Almeida et al., 2016; López et al., 2019). Despite the benefits of using RAP materials in asphalt mixtures, though, several studies have questioned the performance properties of these mixes. Due to the aged binders in these materials, poor low-temperature cracking resistance, poor fatigue resistance, partial blending, higher air voids, and higher moisture susceptibility have been some of the main points of concern (Needham, 1996; Read & Whiteoak, 2003).

Stabilization and recycling of the asphalt layers have been used as a substitute for other pavement construction methods, this having been achieved through measures such as increasing the thickness of layers and using high-quality materials (I. Thanaya, 2007). Using higher thicknesses and better-quality materials have evident drawbacks, though, such as increased cost, construction time, and adverse environmental impacts (Barve & Sugandhi, 2022; Basack et al., 2021; Christopher et al., 2006; Little & Nair, 2009; Patel, 2019; Wegman et al., 2017; Wirtgen Cold Recycling Manual, 2012; Yideti et al., 2014). Meanwhile, alternative methods using asphaltic or cementitious materials can be used to stabilize the pavement layers, increasing the tensile strength of the unbound granular base layers (S. F. Brown, 1996; I. Thanaya, 2007; Uddin et al., 2021; White & Weir, 2021). The use of asphalt emulsion in cold-recycled RAP enhances flexibility and cohesion; however, asphalt mixes prepared with asphalt emulsion can be prone to rutting under high service temperatures and

slow-moving traffic (Kavussi & Modarres, 2010). Higher shear strength, stiffness, durability, compatibility of RAP with aggregates, and resistance to moisture in asphalt pavements are other advantages of the stabilization of asphalt pavements (Branch, 2005; Kim & Lee, 2012).

Using RAP in pavement construction offers several advantages. It is a sustainable method that helps to conserve natural resources, lowers the overall cost of construction (a minimum of a 30% reduction in the cost of the base layer) (Dughaiishi et al., 2022; Qiao et al., 2019), reduces the landfill burden, and reduces carbon footprint (including conservation of natural aggregate resources and a reduction in greenhouse emissions by up to 50%) (Aurangzeb et al., 2014; Bizarro et al., 2021). In turn, one of the main advantages of stabilization techniques, beyond those mentioned above, is that they enable the use of higher RAP content (50% to 100%) in the base and sub-base layers. In this regard, in some jurisdictions, the asphalt industry faces the problem of excessive material storage/disposal due to the limited quantities of RAP that can be reused in new asphalt mixes. For example, an estimated 500,000 tons of surplus RAP is generated every year in Calgary, Canada, and the same amount is currently stockpiled at various facilities across the city. One of the principal limiting factors in the overall use of RAP is the concentration of RAP used in the mix, and this is generally dependent on the gradation, the properties of the extracted aggregates, and the properties of the recovered binders (Pradyumna et al., 2013). Moreover, a high concentration of RAP increases the chance of cracking distress in the pavement, while oxidized binder in the material also decreases the utilization rate of this material in roads (Willis et al., 2012).

Asphaltenes are produced in the deasphalting of bitumen as a residue material with no significant application in current practice. Asphaltenes are also one of the components of asphalt binders, along with saturates, resins, and aromatic (collectively referred to as "SARA"). Asphalt binder as a viscoelastic material contains both polar and non-polar components, and asphaltenes are one of the polar components (Behnood & Modiri Gharehveran, 2019; Ramirez-Corredores, 2017; Sultana & Bhasin, 2014; Y. Xu et al., 2019). The addition of asphaltenes to the binder is expected to increase the stiffness of the mixture (since polar components are mainly responsible for the elastic behaviour of the binder), thereby improving the mechanical properties. These conclusions have been drawn based on the research results available for the

use of asphaltenes in the stabilization of the granular base course with asphalt emulsion (Kamran et al., 2021) and rheological tests done in previous studies (Basavarajappa, 2021).

5.2. Objectives and Scope

The main objective of the research presented in this paper was to combine the use of high-RAP-content materials with a cationic slow-setting (CSS) asphalt emulsion stabilization and improve the properties of these mixtures with asphaltenes, pushing the strength values of the 100% RAP to the mix design limits through the use of an asphaltenes-modified asphalt emulsion stabilizer. In addition to improving or keeping constant the low-, intermediate- and high-temperature properties of mixtures compared to unmodified mixtures. Indirect tensile strength, Hamburg wheel tracking (HWT), creep compliance and strength, and dynamic modulus tests were performed to evaluate the performance factors.

5.3. Materials

5.3.1. RAP

The unprocessed RAP material was collected from a source near Edmonton, Canada. The RAP material was sieve-analyzed and was found to nearly satisfy the limitations provided by the City of Edmonton (2015). Figure 5-1 illustrates the final gradation and envelope for the City of Edmonton specification. The gradation skeleton consisted of 26.0% coarse materials, 67.9% fine materials, and 6.1% filler. Penetration, performance grading, and binder content tests were performed on the recovered RAP binder, with the results presented in Table 5-1. Prior to mixture testing, the optimum fluid content (OFC) and the optimum moisture content (OMC) were determined by the proctor test with respect to ASTM D698 (2021) specification. The RAP gradation and the City of Edmonton envelope (2015) are also provided in Table 5-1.

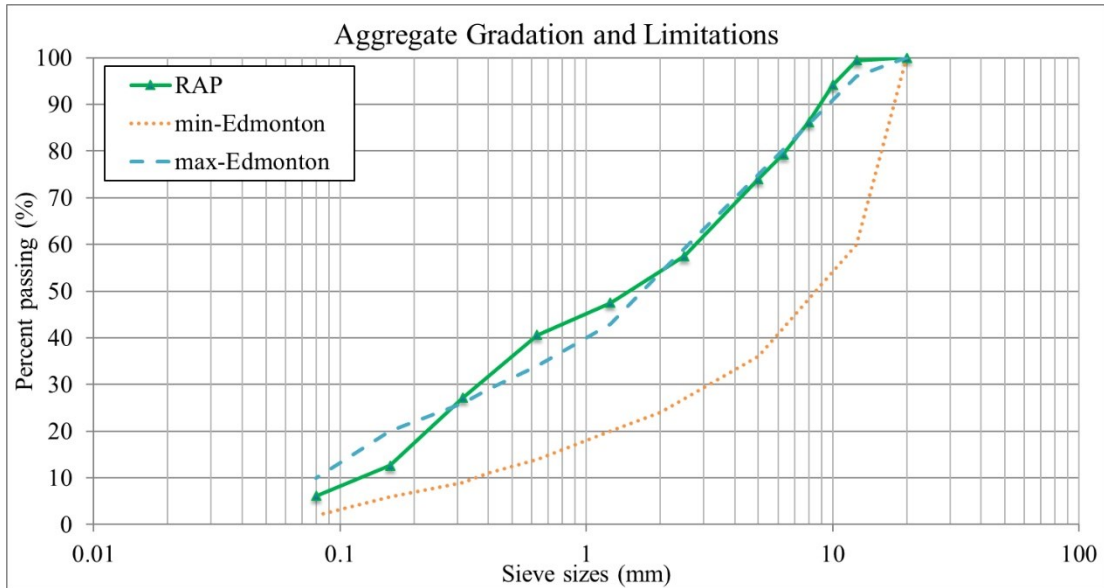


Figure 5-1 RAP gradation and limitations

Table 5-1 RAP binder properties

Binder	Test Name	ASTM	Test Temperature (°C)	Test Result	Unit	Pass/Fail
Original Binder	Original Dynamic Shear Rheometer	D7175 (2015)	-	-	-	-
	Specific Gravity	D70 (2021)	25	1.05	-	-
			15	1.05	-	-
	Penetration A	D5 (2020)	25	55	mm/10	-
	Penetration B		25	53	mm/10	-
Penetration C	25		53	mm/10	-	
RTFO Residue	RTFO Dynamic Shear Rheometer	D7175 (2015)	76	2.690	kPa	PASS
			82	1.380	kPa	FAIL
	Predicted Failure Temperature	-	-	77.81	°C	-
PAV Residue	Aging Temperature	D6521 (2022)	-	100	°C	-
	PAV Dynamic Shear Rheometer	D7175 (2015)	31	1,450	kPa	PASS
28			2,040	kPa	PASS	

Predicted Failure Temperature	-	-	20.18	°C	-
Bending Beam Rheometer (Creep)	D6648 (2016)	-18	142.0	MPa	PASS
Predicted Failure Temperature		-24	322.0	MPa	FAIL
Bending Beam Rheometer (Slope)	D6648 (2016)	-18	0.3	-	PASS
Predicted Failure Temperature		-24	0.2	-	FAIL
True Grade	-	-	77.9-28.5	-	-
Standard Performance Grade (PG)	-	-	76-28	-	-

5.3.2. Asphalt Emulsion

The typical asphalt emulsion used for the stabilization of materials (i.e., CSS) was selected for stabilization in our study due to the aggregate charge and the minimal time required for the mixing and laying of the base layer (Wirtgen Cold Recycling Manual, 2012). The time needed for the asphalt emulsion to mix with materials before breaking and dispersing in the mixture is an essential parameter to be considered in cold mixture design. The asphalt emulsion used in the present study contains 61% hard binder and 39% water and is classified as CSS-1H. The asphalt binder that was used to prepare the emulsion, meanwhile, has a penetration of 80/100 and a viscosity of 0.42 Pa·s at 135 °C. To determine the asphalt emulsion properties, various tests were conducted and the results are presented in Table 5-2.

Table 5-2 Properties of CSS-1H asphalt emulsion (Husky Asphalt, 2019)

Test type	Property (unit)	Standard	Specification		Typical Analyses
			Min.	Max.	
Tests on Emulsion	Specific gravity (Density) at 15.6°C (kg/L)		-	-	1.020
	Residue by distillation (% by mass)	AASHTO T59 (2022)	57	-	61
	Viscosity at 25 °C (S.F.S)		20	100	22
	Oversized particles (% by mass)		-	0.300	0.008

Tests on Asphalt Residue	Settlement (24 h) (% by mass)		-	1.0	0.5
	Particle charge test	ASTM D7402 (2017)		Positive	Positive
	Penetration at 25 °C (dmm)	AASHTO T49 (2019)	40	125	95
	Ductility at 25 °C (cm)	AASHTO T51 (2018)	40	-	> 40
	Solubility in Trichloroethylene (% by mass)	AASHTO T44 (2018)	97.5	-	> 97.5

5.3.3. Asphaltenes

The asphaltenes used in this study were sourced from Alberta oil sands bitumen, having been processed through a solvent deasphalting process. The asphaltenes were obtained in powder form and, to achieve adequate dispersion, they were sieved through a No. 100 sieve. Considering that the properties of the asphaltenes from different sources depend on the oil and processing method used to produce it, tests were conducted to determine its purity (expressed as a percentage) as well as its chemical composition (Table 5-3). In addition, the SARA test was conducted to determine the relative quantities of saturates, asphaltenes, resins, and aromatics. According to the results of the SARA analysis, it was found to be composed of 79.63% asphaltenes, 9.68% aromatics, 6.85% saturates, and 3.84% resin.

Table 5-3 Properties of asphaltenes

Property	ASTM standard	Typical analysis
Origin of Sample	-	Cold Lake bitumen
Bulk Density, kg/m ³	-	403.6
True Density, kg/m ³	-	1135
Gross Calorific Value, MJ/kg	D 5865 (2019)	38.6
Net Calorific Value, MJ/kg	-	36.4
MCR (Micro Carbon Residue), Wt%	D 4530 (2008)	37.2

5.3.4. Composition of Mixtures and Optimum Emulsion Content

The technical guideline for bitumen stabilized materials (“TG2”) published by the Southern African Bitumen Association (Sabita) (2020) and the basic asphalt recycling manual published by the Asphalt Recycling and Reclaiming Association (ARRA) (2001) were used to design the stabilized mixtures containing RAP. The proctor test was conducted on the 100% RAP material

to determine the OFC and based on this, the OMC. The amount of emulsion to be added to the mixtures was then calculated accordingly.

Using this method, four different percentages of asphalt emulsion and water increasing at 0.5% intervals (1%, 1.5%, 2%, and 2.5%) were added to the mixtures in order to determine the optimum emulsion content (OEC). Dry and soaked indirect tensile strength (ITS) tests were used to finalize the OEC value for the prepared mixtures. The RAP was oven-dried at 110 °C to remove any existing moisture, and water was then added to the materials once they had cooled to room temperature. The materials were then mixed until the water was uniformly distributed. Heated Materials were cooled down to achieve the OMC in ambient temperature to prevent loss of moisture. Finally, the asphalt emulsion was introduced to the wet RAP at the selected concentrations, and the resulting mixtures were compacted using a Marshall hammer with 50 blows on each side of the sample.

5.3.5. Indirect Tensile Strength Test

The ITS test was conducted in accordance with AASHTO T283 (2021). Six replicates for each asphalt emulsion concentration (3 replicated for dry and 3 for soaked) were prepared for this test. A Marshall hammer was used for the compaction of the samples, which were then cured for 48 h at 60 °C (ARRA, 2001). Three samples in dry conditions were subjected to 3 h of conditioning in a controlled temperature environmental chamber at 25 °C, prior to testing, while the other three samples were soaked in a 25 °C water bath for 24 h. Both sets of samples were tested after the conditioning under a load applied at a rate of 50 mm/min. The maximum load applied to the samples before the failure point was recorded to determine the ITS. The ITS for each of the samples was calculated using Equation (5-1).

$$S_t = \frac{2000P}{\pi t D} \quad (5-1)$$

where: S_t = Indirect tensile strength (kPa), P = Maximum applied load (N), t = Average height of the specimen (mm), D = Diameter of the specimen (mm).

Examining the ITS results from both the dry triplicates and the soaked triplicates, the mixture with 1.5% asphalt emulsion per total mixture weight was found to have the OEC (Figure 5-2).

Minimum values of 225 kPa and 125 kPa for ITS-dry and ITS-soaked, respectively, were identified as the lower limits for asphalt emulsion-stabilized material, in accordance with TG2 (Sabita, 2020). The OEC was determined based on the higher value of the ITS-dry and ITS-soaked samples with the minimum asphalt emulsion content (Based on the objectives of the present research that want to improve the performance with lowering the cost of construction, there was no need to consider the ITS-dry design limit in determining the OEC). The tensile strength ratio (TSR) was also controlled for the design limits, based on TG2 and the Wirtgen cold recycling manual (Sabita, 2020; Wirtgen Cold Recycling Manual, 2012).

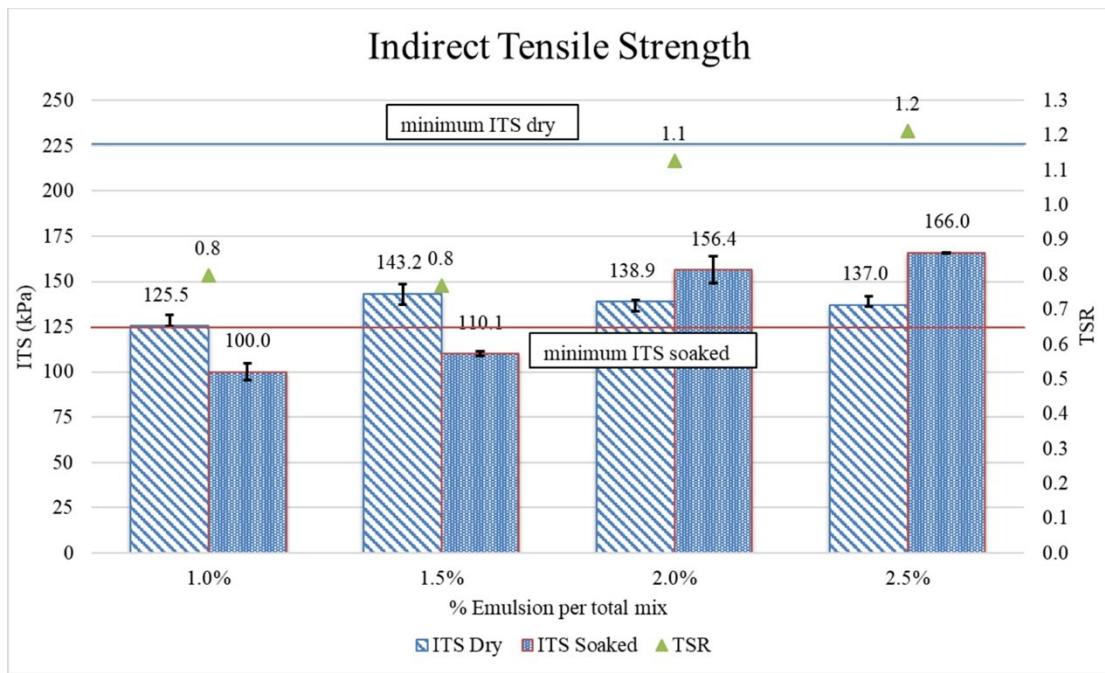


Figure 5-2 ITS results to determine the OEC

5.3.6. Asphaltene Modification and Sample Preparation

The OEC of 1.5% by weight of the total mixture, determined as described above, was used to prepare the asphaltene-modified mixtures used in the study. Control samples were prepared, in a similar manner to the design samples for OEC, for the purpose of comparison to the asphaltene-modified samples. According to the mix design test results, asphaltene was added to the RAP material dried at 110 °C and cooled to room temperature before mixing with asphalt emulsion and water. Concentrations of 0.5% and 1% asphaltene by total mixture weight were also added to the RAP before adding the asphalt emulsion, based on previous research finding 1% to be the optimum content for asphaltene modification (Kamran et al.,

2021). Three replicates were prepared for each concentration of asphaltenes. Samples were labeled as EX-AY, where E refers to the asphalt emulsion, X is the percentage of emulsion added, A is the asphaltenes, and Y is the percentage of asphaltenes added to the mix.

5.4. Testing Program

5.4.1. Tensile Strength Ratio

The ITS test, the purpose of which was to analyze the design limits and moisture susceptibility of the samples at intermediate temperatures, was conducted in accordance with the AASHTO T283 standard (2021) for dry, soaked, and freeze/thaw conditions. Nine replicates of the samples (3 replicates for dry, 3 replicates for soaked, and 3 replicates for freeze/thaw) were prepared at each of the asphalt emulsion concentrations, and a Marshall hammer was used for compaction using the same procedure as described above. The dry samples were subjected to 3 h of conditioning in an air chamber at 25 °C prior to testing. The soaked samples, meanwhile, were conditioned in a water bath at 25 °C for 24 h. The freeze/thaw, finally, were prepared in accordance with AASHTO T283 (2021). A similar procedure for loading as the one followed when using a universal testing machine (UTM) was carried out, and the ITS and strength was calculated using Equation (5-1).

In order to calculate the tensile strength ratio of modified and unmodified samples, the ITS of soaked and freeze/thaw-conditioned subsets were divided by dry subsets separately. The specifications in TG2 were used to determine the limit (i.e., 50% for the TSR values).

5.4.2. Ideal-CT Analysis

The IDEAL-CT analysis was also conducted on the samples tested for ITS using ASTM D8225-19 (2020) standard. The crack tolerance index (CT-Index) was calculated for all the samples based on the fracture energy (FE) theory using Equation (5-2).

$$CT_{Index} = \frac{t}{62} \times \frac{L_{75}}{D} \times \frac{G_f}{|m_{75}|} \times 10^6 \quad (5-2)$$

where: CT-Index = Cracking tolerance index, G_f = Failure energy (Joules/m²), $|m_{75}|$ = Absolute value of the post-peak slope (N/m), L_{75} = Displacement at 75% post-peak load (mm), D = Specimen diameter (mm), t = Specimen thickness (mm);

5.4.3. Hamburg Wheel Tracking Test

AASHTO T324-19 (2019) was followed to conduct the HWT test. The HWT test, it should be noted, was designed to evaluate the high-temperature properties of a mixture simulating the real-life span of the roads. Considering the nominal maximum aggregate size and curing conditions, slab samples with dimensions of 400 mm length, 300 mm width, and 80 mm height were prepared for the test. Similar curing conditions for Marshall samples were used for the samples compacted with a roller compactor. The binder grade and origin of the layer being base dictated the test temperature of 40 °C for the samples (Beecroft & Petho, 2015). Following the standard noted above, the samples were preconditioned in water for 45 minutes prior to testing. A steel wheel with 47 mm width and 705 ± 4.5 N weight at a frequency of 52 ± 2 passes per minute and a maximum speed of 0.305 m/s at the midpoint was used. A maximum of 20,000 passes or 12.5 mm rutting depth, whichever was reached first, was considered as the termination point for the test. The rutting resistance index (RRI) was calculated by multiplying the number of passes to one inch subtracted by rut depth (in inches). The stripping inflection point (SIP) was determined in order to evaluate the rutting potential and susceptibility of the mixtures to moisture damage.

5.4.4. Creep Compliance and Strength Test

The low-temperature properties of the mixture are important factors to be considered in pavement construction, especially in cold climates. In this regard, the creep compliance test is designed to evaluate the behaviour and properties of an asphalt mixture at low temperatures under a constant creep load. A creep compliance and strength test based on AASHTO T322-07 (2020) was performed in this research using the ITS test setup. This test simulates the low-temperature performance of the mixtures by recording the load applied and deformations in the horizontal and vertical directions of the samples. The test temperatures used in this study were 0 °C and -20 °C, based on the performance grading of the binder used to prepare the asphalt emulsion. Marshall samples with six replicates (3 replicates for each test temperature) for each of the asphaltene concentrations were prepared. Samples were surface cut to heights ranging from 38 mm to 50 mm. The samples were conditioned in an air chamber for 3 ± 1 h prior to testing. A constant static load of 1 kN was applied on the specimens for 100 ± 2 s, and extensometers, mounted on each side of the test specimen, were used to record the deformation of the samples along both the horizontal and vertical axes. After completion of the creep test,

the ITS test using a loading rate of 12.5 mm/minute was applied to the samples until the failure point of the samples had been reached. Figure 5-3 presents the test setup and shows images of a sample prior to and after testing. From the data collected, the fracture energy, and creep compliance were calculated. Creep compliances were calculated using Equation (5-3). Equation (5-4) was used to determine the pass or fail criteria for the creep compliance correction factor.

$$D_t = \frac{\Delta X_{tm,t} \times D_{avg} \times b_{avg}}{P_{avg} \times GL} \times C_{cmpl} \quad (5-3)$$

Where: D_t = Creep compliance at time t (1/kPa), GL = Gauge length in meters (25×10^{-3} for 100mm diameter specimens), D_{avg} = Average diameters (mm), b_{avg} = Average thickness (mm), P_{avg} = Average force (kN), $\Delta X_{tm,t}$ = Trimmed mean of the $\Delta X_{i,t}$ arrays, C_{cmpl} = Creep compliance correction factor.

$$\left[0.704 - 0.213 \left(\frac{b_{avg}}{D_{avg}} \right) \right] \leq C_{cmpl} \leq \left[1.566 - 0.195 \left(\frac{b_{avg}}{D_{avg}} \right) \right] \quad (5-4)$$

where: D_{avg} = Average diameters (mm), b_{avg} = Average thickness (mm), C_{cmpl} = Creep compliance correction factor

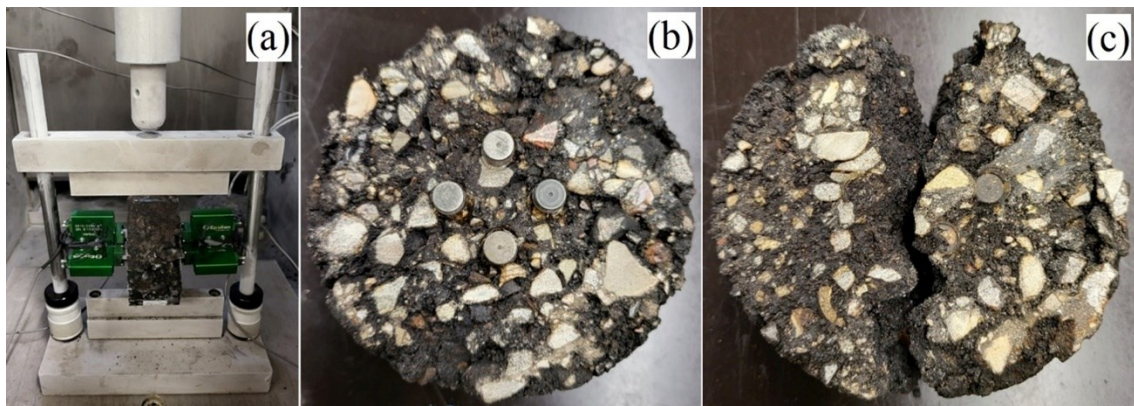


Figure 5-3 (a) Indirect tensile test (IDT) setup, (b) sample prior to testing, and (c) sample after testing

5.4.5. Dynamic Modulus

The Dynamic modulus $|E^*|$ test was performed on the samples in order to determine whether the stress and strain responses in the asphalt mix correlate with the time- and temperature-dependent properties of the mix in terms of field performance. A universal testing machine (UTM) based on the AASHTO T378 (2021) standard. Linear Variable Differential Transformers (LVDTs) attached to the Superpave gyratory compactor prepared samples after cutting and coring them into required dimensions (Cylindrical specimens of 100 mm diameter and 150 mm height). Sinusoidal axial compressive stress at loading frequencies of 0.01 Hz, 0.1 Hz, 0.5 Hz, 1 Hz, 5Hz, and 10 Hz was applied to the specimens at temperatures of 4 °C, 21 °C, and 37 °C following the conditioning times suggested in the literature for each temperature. The applied stresses and corresponding strain responses of the specimens were measured continuously during testing using a data acquisition system, while the dynamic modulus values were calculated by dividing the stress magnitude by the average strain magnitude. Two replicates for each mixture were prepared and tested.

5.5. Results

5.5.1. Tensile Strength Ratio

Figure 5-4 presents the ITS and TSR values corresponding to each of the three conditioning methods. The results from the ITS test show that modification with asphaltenes significantly increases the strength of the mixture, allowing it to satisfy the minimum mix design limit (i.e., 225 kPa for dry subsets and 125 kPa for soaked samples). The E1.5-A1 samples with 1% asphaltenes were found to have the best response in terms of tensile strength; however, an increase in asphaltenes content to 3% was found to cause a decrease in tensile strength of the modified mix. Considering the criterion for minimum ITS dry, only E1.5-A1, among the dry samples, was found to satisfy the limit. However, the ITS-soaked value was satisfied for both the 0.5% and 1% asphaltenes. The TSR values were found to be over 0.5 for all the soaked samples, meaning that they satisfied the limit indicated in the specifications (Sabita, 2020). The TSR values were found to be 0.685 and 0.923 for 0.5% and 1% asphaltenes concentrations, respectively, compared to a TSR value of 0.769 in the case of the control sample. From these TSR test results, we can infer that the mixtures are not susceptible to moisture damage.

The air void content was found to be 20.1%, 19.6%, and 20.5%, for the control, E1.5-A0.5, and E1.5-A1 samples, respectively. This value was high for all the mixtures due to the high content of RAP, and it was found to be similar for both the modified and unmodified samples.

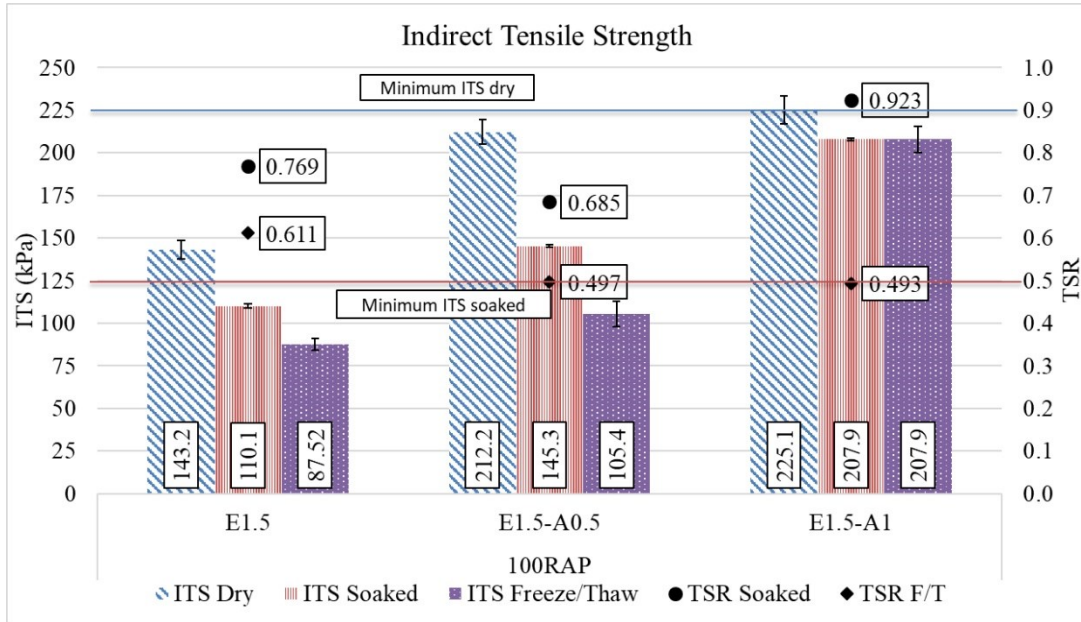


Figure 5-4 ITS and TSR for control and asphaltene-modified mixes

5.5.2. Ideal-CT Analysis

The load-deformation graph was used to calculate the FE, and CT-Index for the samples (Table 5-4). FE values were determined to be 437.2, 1324.9, 1495.6 (Joules/m²) for E1.5, E1.5-A0.5, E1.5-A1, respectively. Accordingly, CT-Index was increased from 46.2 of E1.5 to 134.3 and 131.4 for E1.5-A0.5 and E1.5-A1, respectively. The increase in FE and CT-Index values shows better crack resistance at intermediate temperatures of modified mixtures with asphaltene.

Table 5-4 IDEAL-CT analysis

Sample ID	Fracture Energy (J/m ²)	STDV	COV	CT-Index	STDV	COV
Control	437.2	40.9	9.4	46.2	0.13	0.3
E1.5-A0.5	1,324.9	65.1	4.9	134.3	30.5	22.7
E1.5-A1	1,495.6	11.7	0.8	131.4	3.29	2.5

5.5.3. Hamburg Wheel Tracking Test

The HWT test (AASHTO T 324, 2019) was used to determine both the moisture susceptibility and the rutting resistance of the samples. The moisture sensitivity of the samples, calculated by dividing the SIP value by the number of passes, was determined to evaluate the sensitivity of the unmodified and modified samples to moisture; it was found to be 1 for all the mixtures.

The RRI, meanwhile, was calculated based on the number of passes and the depth of rutting. Figure 5-5 presents the test results, which show an increase in the number of passes before failure for the asphaltenes-modified samples compared to the control sample (indicating higher resistance to rutting). Furthermore, both the 0.5% and 1% asphaltenes samples showed a significant increase in RRI compared to the control samples. The samples with 1% asphaltenes had the best performance, with the test reaching 20,000 wheel passes before the samples had reached the 12.5 mm rut depth limit. As mentioned above, the high RRI values achieved by the modified samples indicate a higher rutting resistance for the asphaltenes-modified samples in comparison to the control sample. In fact, an increase in RRI from 1,925.86 in the case of the control samples to 6,674.48 and 16,267.72 for the 0.5% and 1% asphaltenes-modified samples, respectively, was observed, representing respective increases of 346.6% and 844.7% in rutting resistance compared to the control samples. In addition, no SIP was detected for the E1.5-A1 sample and test termination point was over 10,000 passes, meaning that this sample is not susceptible to moisture damage within the limits of the test (Aschenbrener, 1995). As a result of this test, it can be concluded that asphaltenes modification significantly increased the permanent deformation resistance in the 100% RAP samples.

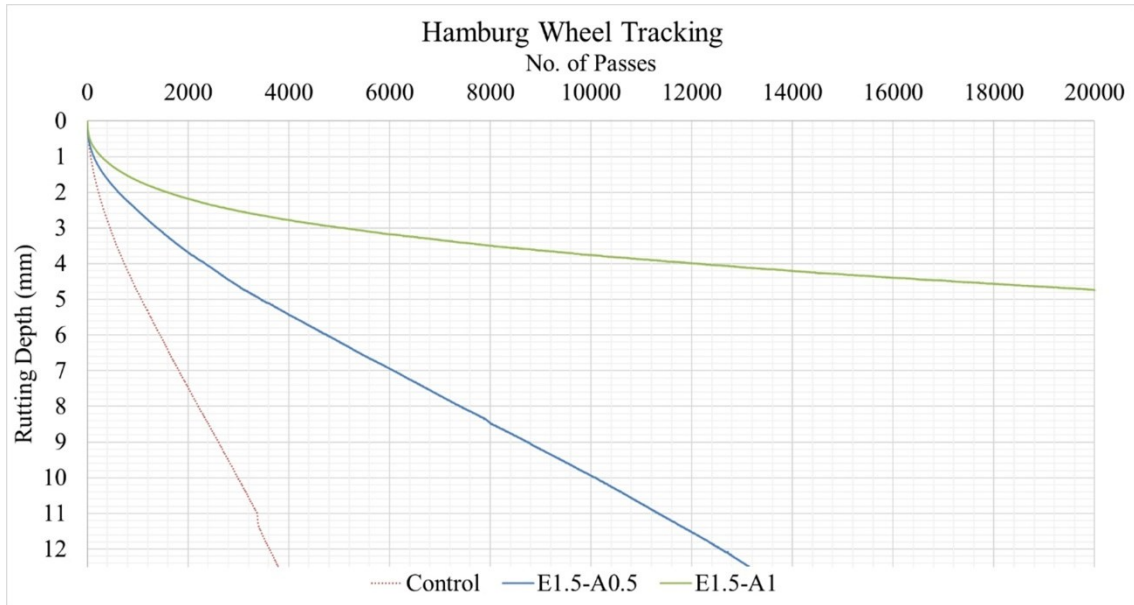


Figure 5-5 Slab samples compacted by roller compactor (a) prior and (b) after test

5.5.4. Creep Compliance and Strength Test

The creep compliance test (AASHTO T 322, 2020) was conducted at temperatures of 0 °C and -20 °C for both the modified and unmodified samples. The vertical load versus deformation graphs at each temperature are presented in Figure 5-6 and Figure 5-7. The tensile strength, FE, and creep compliance values were calculated as listed in Table 5-5. The control samples that underwent the creep test at 0 °C had strength values of about 542.9 kPa for 100% RAP with no asphaltenes. Adding 0.5% and 1% asphaltenes increased this value by about 7.9% and 11.7%, respectively. FE values for control samples decreased for 0.5% asphaltenes and 1% asphaltenes about 19.4% and 58.0%, respectively. Meanwhile, adding asphaltenes to the mixture by up to 1% increased the resistance of the mixture to creep and decreased the compliance to the creep loading (Table 5-5). As can be seen, the slope of the strength test graph (Figure 5-6) after failure for the sample with 1% asphaltenes is steeper compared to the sample containing 0% and 0.5% asphaltenes, and this is indicative of rapid crack propagation in the case of the samples with higher asphaltenes concentration. These creep and strength test results for E1.5-A1 samples are indicative of the superior stiffness achieved by the asphaltenes-modified samples compared to the control samples at this temperature, which was expected.

The strength values at -20 °C increased about 4.8% and 7% by adding 0.5% and 1% asphaltenes, respectively, compared to the control samples (Figure 5-7). FE also decreased at

this temperature, by about 24.7% and 34.6% for 0.5% asphaltenes and 1% asphaltenes modified samples, respectively, compared to the control samples. The creep compliance decreased about 22% by adding 0.5% asphaltenes and 61.6% by adding 1% asphaltenes at this temperature, underscoring the improved resistance of the modified samples to the creep loading compared to the control samples. The slopes of the load-displacement graphs at -20°C were higher for the modified samples, suggesting that crack propagation was increasing by asphaltenes modification at this temperature. In addition, the test results show that, as the temperature decreases, the ITS values increase while the FE values decrease.

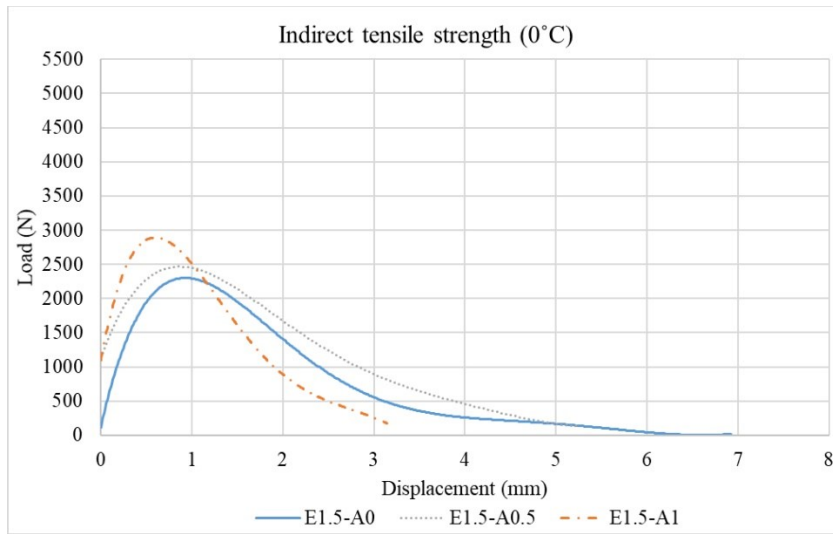


Figure 5-6 Load–displacement results for 0°C

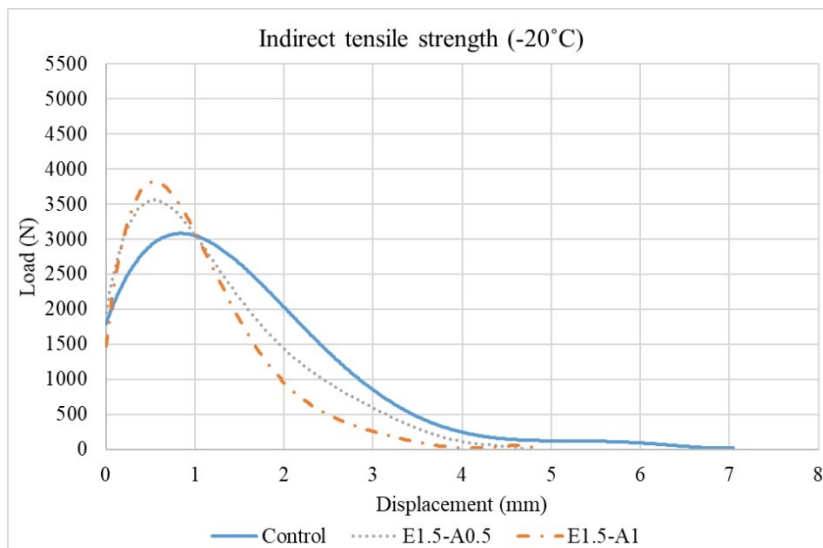


Figure 5-7 Load–displacement results for -20°C

The creep compliance results for the samples at different temperatures over a period of 100 s were recorded as shown in Figure 5-8 and Figure 5-9. As can be seen, for the modified samples, the creep compliance changes were found to be similar at 0 °C compared to each other. The control sample results were much higher with increased loading time with increasing slope. Comparing the slopes of the graphs for all samples at both 0 °C and -20 °C temperatures, only mixture with 1% of asphaltenes had less steep slope at -20 °C. This observation illustrates the stiffening effect of asphaltenes at lower temperatures for high contents of asphaltenes. This means at -20 °C, the 1% asphaltenes showed less creep compliance in comparison to the control and 0.5% asphaltenes samples. The control samples at -20 °C, meanwhile, had a higher creep compliance than the modified samples at this temperature and had a steeper slope.

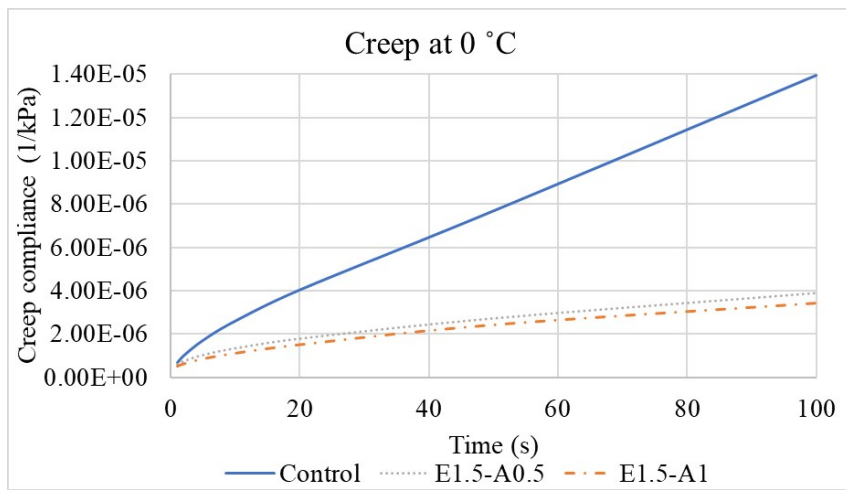


Figure 5-8 Creep compliance–time results for 0 °C

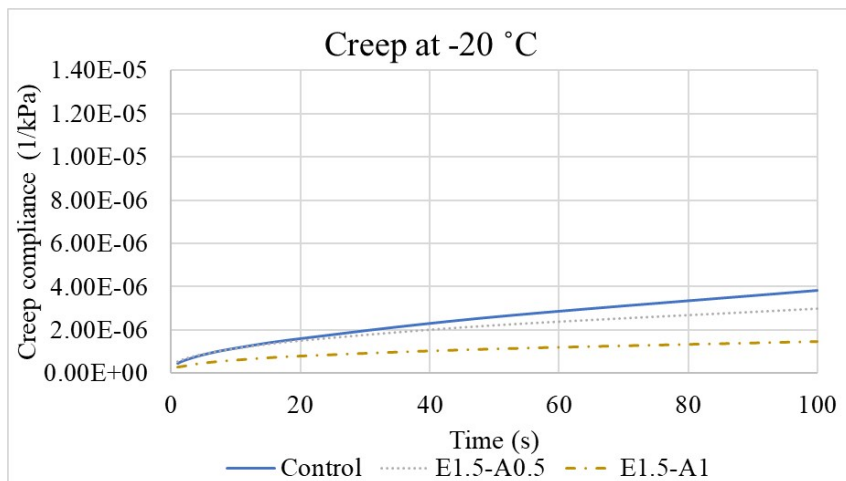


Figure 5-9 Creep compliance–time results for -20 °C

Table 5-5 Tensile strength and fracture energy for creep test samples

Sample ID	Temperature (°C)	Tensile strength (kPa)	Fracture Energy (J/m ²)	Creep compliance at 100 s (1/kPa)
Control	0	542.9	1,299.4	1.39×10^{-5}
E1.5-A0.5		585.9	1,046.9	3.88×10^{-6}
E1.5-A1		606.4	545.4	3.43×10^{-6}
Control	-20	711.1	1,989.3	3.82×10^{-6}
E1.5-A0.5		745.3	1,498.7	2.99×10^{-6}
E1.5-A1		761.2	1,300.3	1.47×10^{-6}

5.5.5. Dynamic Modulus

Dynamic modulus test results are presented in Figure 5-10 and Figure 5-11 as dynamic modulus and phase angle master curves obtained from Mechanistic-empirical Pavement Design Guide (MEPDG) method. These results show high modulus values for E1.5-A0.5 and E1.5-A1 at low frequencies, indicating that the modified mixtures have better performance in terms of high temperature properties compared to E1.5-A0. Samples modified with 0.5% asphaltenes show higher modulus values compared to the samples modified with 1% asphaltenes. As the frequency increases, the dynamic modulus and viscoelasticity of the asphalt mixtures also increase. However, at high frequencies, these values converge for both modified and unmodified samples suggesting no significant changes in the low-temperature properties of the modified mixtures. The almost straight line for E1.5-A1 indicates that the addition of 1% asphaltenes increases the elastic behaviour of the mixtures more than the viscous behaviour. Analysis of the phase angle data (Figure 5-11) shows decrease in the phase angle of the modified mixture at high frequencies and an increase at low frequencies compared to E1.5-A0. A higher phase angle for the modified mixtures at low frequencies indicates a more viscous behaviour, while unmodified mixtures exhibit more elastic behaviour. Increasing the frequency, however, results in a decrease in the phase angle of the modified mixture compared to the unmodified mixture, indicating that the modification slightly reduces the viscous behaviour of the mixtures at low temperatures.

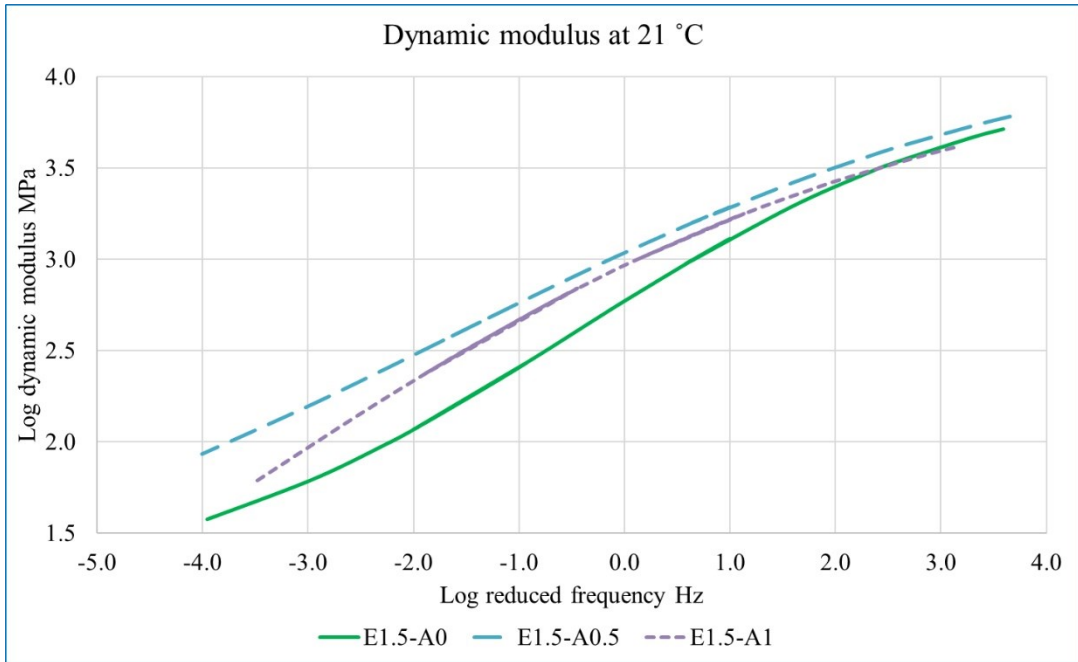


Figure 5-10 Dynamic modulus master curve at 21 °C

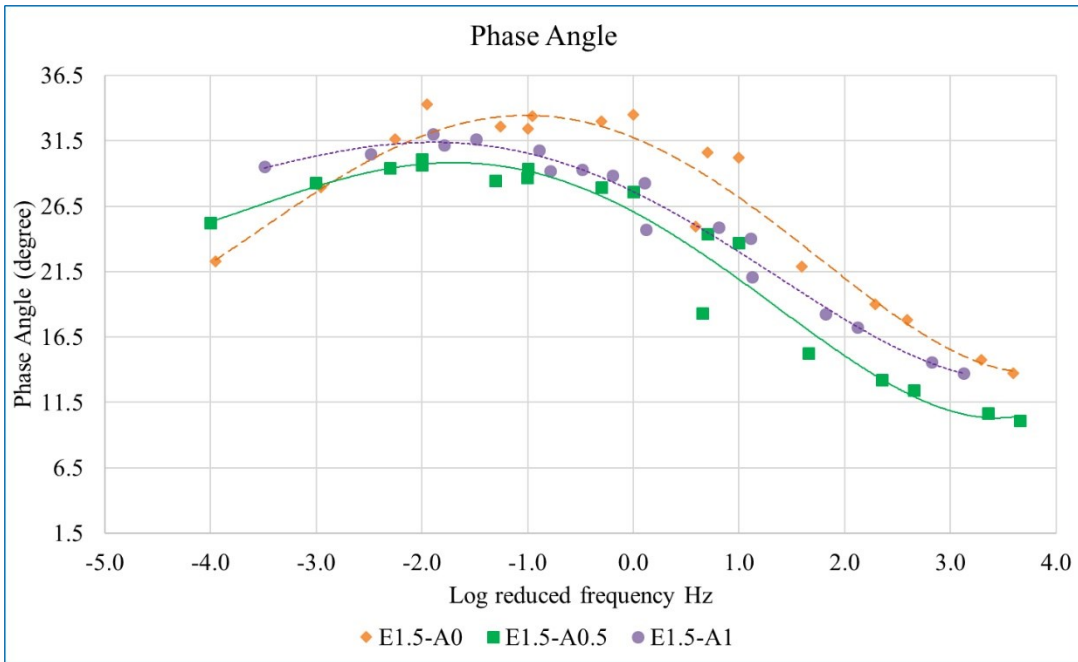


Figure 5-11 Phase angle master curve at 21 °C

5.6. Discussion

Table 5-6 presents the comparative data for the intermediate-, low-, and high-temperature properties of the mixtures based on the applicable test results. In conducting the ITS dry test

at intermediate temperature, the tensile strength values were found to be about 50% higher in the case of the modified samples compared to the control samples, resulting in about 203% and 242% increases in FE for the 0.5% and 1% asphaltenes-modified samples compared to control, respectively. CT-Index values also increased by about 191% and 185% for the 0.5% and 1% asphaltenes-modified samples compared to the control, respectively. These results show that the tensile strength increased due to the addition of asphaltenes, with the modified samples exhibiting increased strength compared to the control mixtures. The higher FE value of the modified samples compared to the control samples indicates their higher capacity to absorb energy and show improved resistance to cracking. The ITS soaked and freeze/thaw values also increased after asphaltenes modification, with 32% and 89% increases in ITS soaked for the 0.5% and 1% asphaltenes concentrations, respectively, and 20% and 27% increases in ITS freeze/thaw for the 0.5% and 1% asphaltenes concentrations, respectively, compared to the control samples. The TSR values were about the same for the control and modified samples, confirming the SIP values from the rutting test, as well as confirming satisfaction with the limits specified in the relevant design methods. Meanwhile, the TSR values for the freeze/thaw cycle decreased by about 20% compared to the control sample, indicating that asphaltenes modification does not have a significant impact on the moisture susceptibility of the mixture in the base layer which is resulting in higher rate of increase for ITS dry compared to ITS freeze/thaw and soaked.

The results with respect to the low-temperature properties of the mixtures, meanwhile, are indicative of the high strength and low compliance of the samples to creep. The test results for the unmodified (control) and asphaltenes-modified mixtures were compared to gain an understanding of the effect of modification in the base layer with asphaltenes. This results are also to be analyzed with analysis of variance (ANOVA) to consider the significance of the changes exhibited at low temperatures. The strength of the samples at 0 °C was found to be almost similar for both modified samples (i.e., asphaltenes concentrations of 0.5% and 1%). The creep compliance had a decrease of about 75% indicating that modification of samples results in less creep compliance compared to control samples. The FE of the mixture modified with asphaltenes was found to be lower compared to those obtained for the unmodified mixtures which we can infer that a higher asphaltenes concentration results in a higher rigidity and stiffness in the mixtures.

The ITS values increased, and FE decreased at $-20\text{ }^{\circ}\text{C}$ by approximately 6% and 25%, respectively. The decrease in FE for 1% (35% lower FE) was more in compare to 0.5% (25% lower FE) asphaltenes modification. Followingly, the creep compliance was lower for the mixture with 1% asphaltenes compared to the mixture containing 0.5% asphaltenes. These creep test results indicate that the modified samples reach a lower creep compliance than the control samples while maintaining approximately the same strength. Further analysis in ANOVA reveals the significance of these changes statistically.

The high-temperature properties were evaluated based on the HWT test results. The HWT test results and RRI values show that asphaltenes modification has a significant impact on the rutting resistance (e.g., rutting resistance increased by 247% in the case of the mixture with 0.5% asphaltenes and by 745% in the case of the mixture with 1% asphaltenes). No SIP was observed in the 20,000 passes and 12.5 mm rutting as the limits of the test for any of the modified or unmodified samples. The higher rutting resistance values achieved by the modified mixtures as noted above emphasize the higher stiffness of the asphaltenes-modified mixtures and confirm the intermediate-temperature test results regarding the moisture susceptibility of the mixture.

Dynamic modulus and phase angle master curves showing higher changes at low frequencies and no significant changes at high frequencies.

Table 5-6 Comparison of performance properties of modified and unmodified mixes

Test	Parameter	E1.5-A0.5	E1.5-A1
		Result improvement	
ITS	ITS Dry (25 °C)	48.2%	57.2%
	FE Dry (25 °C)	203.1%	242.1%
	CT-Index (25 °C)	190.8%	184.6%
	ITS Soaked	32.0%	88.8%
	ITS Freeze/Thaw	20.4%	26.9%
	TSR Soaked	-11.0%	20.1%
	TSR Freeze/Thaw	-18.7%	-19.3%
Creep compliance	ITS (0 °C)	7.9%	11.7%

	FE (0 °C)	-19.4%	-58.0%
	Creep compliance (-0 °C)	-72.1%	-75.3%
	ITS (-20 °C)	4.8%	7.0%
	FE (-20 °C)	-24.7%	-34.6%
	Creep compliance (-20 °C)	-21.7%	-61.6%
HWT	RRI	246.6%	744.7%

Table 5-7 presents the results of the one-way ANOVA conducted on the test results of different mixes. The purpose of this analysis was to examine the effect of modification on each of the test results by comparing the modified and unmodified samples. Single-factor ANOVA was performed on the data from three groups to determine the statistical significance of the differences among their means, considering the variability within each group and between groups. The null hypothesis assumes that there is no significant difference among the means of the groups, suggesting that any observed differences are due to random chance or sample variability. The F-statistic, which divides the variability between groups by the variability within the groups, was used to assess the significance of the differences. Additionally, the p-value indicates the strength of evidence against the null hypothesis and represents the probability of obtaining the observed results if the null hypothesis is true. A smaller p-value suggests stronger evidence against the null hypothesis. For this analysis, a significance level or alpha of 0.05 was selected as the predetermined threshold for determining statistical significance. If the p-value is lower than 0.05, the null hypothesis is rejected.

The modification of the recycled RAP with asphalt emulsion using 0.5% and 1% asphaltenes showed no significant changes in the properties of ITS and FE at -20 °C, as indicated by the acceptance of the null hypothesis. Lowering the temperature did not reveal any significant impact of the modification on the performance of the samples. This conclusion was drawn by comparing the F-values for ITS at low temperature (-20 °C), which was 0.14 to the F-critical value of 5.14. However, when comparing the F-values to the F-critical value for other properties and considering p-values lower than the alpha level of 0.05, there was a significant impact of the modification, leading to the rejection of the null hypothesis. The results demonstrated that asphaltenes modification had a significant effect on ITS and FE at 25 °C,

with F-values of approximately 55.7 and 60.9, respectively, compared to the F-critical value of 5.14 in this analysis. The next highest impact of asphaltenes modification was observed in CT-Index at 25 °C, with F-values of approximately 9.7, compared to the F-critical value.

Table 5-7 ANOVA for modified and unmodified mixes

Property	Asphaltenes content			
	F-Value	P-Value	F-Critical	Null Hypothesis
Dry ITS (25 °C)	55.67736	0.00013	5.14325	Rejected
Dry FE (25 °C)	60.90928	0.00010		Rejected
CT-Index (25 °C)	9.70195	0.01317		Rejected
ITS Soaked	23.35275	0.00147		Rejected
ITS (-20 °C)	0.14413	0.86868		Accepted
FE (-20 °C)	1.88089	0.23220		Accepted

5.7. Conclusions

The objective underlying the research presented in this paper was to design cold asphalt mixtures including asphalt emulsion and 100% RAP and improving their performance by adding asphaltenes. The performance of mixes was evaluated using a testing matrix for low-, intermediate-, and high-temperature properties. Based on the results of the various performance tests and analyses conducted in this study, the following conclusions can be drawn:

- Asphaltenes modification improved the strength of samples significantly at intermediate temperatures and satisfied the design limits for cold mix asphalt.
- The HWT test results, meanwhile, are indicative of a notable improvement in the rutting resistance of the modified mixtures compared to the unmodified samples. The RRI index was affected by asphaltenes modification showing better high-temperature properties.
- According to the TSR, SIP and moisture sensitivity values, all the samples (both the control samples and the different modified samples) were found to be unsusceptible to moisture.

- The tensile strength of the samples was almost the same for the modified and unmodified samples at low temperatures showing no significant changes in the strength of samples.
- The creep compliance results at low temperatures showed that modification of the RAP material with asphaltenes resulted in slightly lower FE and creep compliance values. However, it was not significant.
- ANOVA results showed that asphaltenes modification has not a significant effect on low temperature properties of mixtures, but high and intermediate performance of modified mixtures was affected significantly.
- The dynamic modulus test revealed a significant increase in the modulus of the asphalt mixtures at high temperatures, while a comparatively lower increase was observed at low temperatures. These findings are consistent with the results obtained from performance tests. Furthermore, by analyzing the variations in the modified mixtures modulus and phase angle at different frequencies and temperatures, it was confirmed that the mixes possess viscoelastic properties.
- Utilizing asphaltenes, a residue material derived from oil sands processing, as a modifier in asphalt emulsion demonstrated positive effects on the properties of 100% RAP mixtures.

Construction of a test section to validate the test results and long-term monitoring of the performance of the asphaltenes-modified layer to validate the test results is the future goal of this study.

Chapter 6: Evaluation of Asphaltenes a Potential Alternative for Cement in Stabilized Base Courses Using Asphalt Emulsion

6.1. Introduction

Climate change and unchecked consumption of viable resources are critical and dangerous environmental phenomena that require a worldwide response and solution. Moreover, these environmental issues are accompanied by economic crises, societal demand for continued development, and geopolitical tensions that underscore the pressing need for a more sustainable method of securing our resources (Plati, 2019). Transportation technologies and pavement, as a crucial part of economic sector, total about 21 million kilometers worldwide (Costa et al., 2020), and result in considerable greenhouse gas (GHG) emissions and adverse environmental impacts (Plati, 2019). Sustainability and judicious use of natural resources have been crucial considerations in road construction in recent years. The expansion of road networks in many developed countries in recent decades has led to excessive use of natural resources and resulted in severe damage to the natural environment (Nazarko et al., 2015). In this regard, many governments and industrial sectors worldwide are prioritizing research on decreasing the use of natural resources by moving to low-quality materials and industrial by-products (Lindsey, 2011; Yaowarat et al., 2021). These studies have proposed methods to improve low-quality materials using industrial by-products to reduce the use of natural resources, energy consumption, and GHG emissions, resulting in more green technologies and fewer adverse effects on the environment (Mirzababaei et al., 2013; Puodziukas et al., 2016).

Technologies such as using recycled waste materials as an alternative to virgin aggregates and cold recycling methods have been widely studied and proven effective (Softić et al., 2020; Yaowarat et al., 2021; Yoobanpot et al., 2017). Reclaimed asphalt pavement (RAP), for instance, is a waste material produced widely in the rehabilitation of roads, and over 100 million tons of production of this material were reported in Europe and the United States in 2017 (Costa et al., 2020; Federal Highway Administration, 2008). Incorporating a recycled asphalt layer in pavement construction may results in a shorter construction time (due to the

time savings associated with minimizing the removal, transport, and deposit of the existing pavement), in addition to the environmental benefits noted above (Puodziukas et al., 2016). Asphalt emulsion stabilization is another measure taken to minimize the excessive consumption of raw material by decreasing the layer thickness and replacing low-quality materials (I. Thanaya, 2007). Asphalt emulsion stabilization brings environmental advantages and a more economical solution than conventional asphalt mixtures such as hot mix asphalt (HMA) (A James, 2006; Pérez et al., 2020). However, these methods have several drawbacks, such as weak early-life strength, lower rutting resistance, longer curing time, moisture susceptibility, and high porosity (Du, 2021; Fang et al., 2016; Kavussi & Modarres, 2010; Pérez et al., 2020). In order to overcome such disadvantages in asphalt emulsion stabilization, additives such as cement, fly ash, biomass ash, and hydrated lime have been recommended by several agencies and researchers (Fang et al., 2016; AD James et al., n.d.; Jiang et al., 2022; I. N. A. Thanaya et al., 2009; Wirtgen Cold Recycling Manual, 2012; Yaowarat et al., 2021; Yoobanpot et al., 2017; Zhao et al., 2022). However, cement, among these additives, is not considered a sustainable and environmentally friendly solution due to the high carbon dioxide gas emission rate during its production (Barcelo et al., 2014; Benhelal et al., 2013; Shah et al., 2022). Additional disadvantages of using cement include cracking problems at low temperatures, shrinkage, slower construction process, and high cost (Thom & Dawson, 2019). A more sustainable replacement for cement could result in an annual global GHG reduction of up to 1.3 gigatons (Shah et al., 2022).

Crude oils consist of three different phases: gas, liquid, and solid. The gas and liquid phases are well studied and open to testing with various chemical analyses. However, solid particles, known as asphaltenes, are not well understood, and this residue of the deasphalting process has no significant application in current practice (Mullins et al., 2007; Sheu & Mullins, 1998). This material is the heaviest component and part of the polar fraction of bitumen, surrounded by aliphatic and heteroatomic chains, and may contain metals (Mullins et al., 2008). Asphaltenes are toluene-soluble, n-heptane-insoluble, and not chemically defined, making it difficult to understand and treat. Moreover, its chemical composition may vary depending on the variety of crude oil (Gharbi et al., 2017; Mullins et al., 2008). The asphalt binder used in road construction is a viscoelastic material that contains both polar and non-polar components, whereas asphaltenes is one of the polar components of this material (Behnood & Modiri

Gharehveran, 2019; Ramirez-Corredores, 2017; Sultana & Bhasin, 2014; Y. Xu et al., 2019). According to Ghasemirad (2020), resins and asphaltenes are considered more polar fractions in the molecular structure of binder, whereas saturates and aromatics are non-polar, with aromatics being slightly more polar than saturates. The difference in polarity between asphaltenes and saturates, along with resins and aromatics, has varying effects on the elastic and viscous characteristics of asphalt binder, with the latter exhibiting a lesser impact (Y. Xu et al., 2019). When considering the influence of polar components on asphalt stiffness, it is worth noting that an increase in asphaltenes content is expected to enhance the mechanical properties of mixtures to some extent (Basavarajappa, 2021). However, it is crucial to take into account the colloidal index (CI) of the binder. The colloidal index is a value used to assess the stability of the asphaltenes phase within the maltene matrix of the binder. It is calculated by dividing the weight of asphaltenes and saturates by the weight of resins and aromatics. A CI value greater than 0.9 indicates an unstable asphaltenes fraction, while a value lower than 0.7 signifies stability. CI values between 0.7 and 0.9 indicate uncertain stability of the asphaltenes fraction (Lesueur, 2009; J. Wang et al., 2019). Therefore, in assessing the effects of asphaltenes on binder properties, it is important to consider the colloidal index to determine the stability of the asphaltenes phase within the binder matrix.

Many countries have plans in place to reduce their GHG emissions in the near future. For instance, Europe has committed to reducing its emission by at least 50% by 2030 and to achieving climate neutrality by 2050 as part of the 2020 European Green Deal (European Commission, 2019). Additionally, the United States has committed to decreasing its emissions by up to 80% by 2050. The transportation sector is crucial to these undertakings, since it is the most prominent GHG emitter, accounting for a fifth of global GHG emissions (Greene et al., 2010). Wide adoption of cold recycling, the use of residue materials, and avoiding materials such as cement can be effective in this regard.

The main objective of this study was to evaluate the possible replacement of cement with asphaltenes as an additive in base course stabilization with asphalt emulsion. Asphaltenes were added to the asphalt emulsion, and the rheological properties were assessed using a dynamic shear rheometer. In addition, mixes modified with both asphaltenes, and cement were prepared to determine the mechanical properties using a series of performance tests, then compared to

unmodified mixes. Various mechanical properties of these materials, such as indirect tensile strength (ITS), tensile strength ratio (TSR), crack tolerance, low-temperature cracking, rutting resistance with Hamburg wheel tracking (HWT), and structural behaviours using dynamic modulus test, were investigated.

6.2. Materials

6.2.1. Aggregates

The materials used in this research were aggregates with a specific gradation collected from sources near Edmonton, Canada. Both materials were sieve-analyzed, and mixtures were prepared within the limitations provided by the City of Edmonton (2015) and Alberta Transportation (Soliman et al., 2014) for the base material. The optimum fluid content (OFC) of the materials was determined using the proctor test and, based on this, the optimum moisture content (OMC). The emulsion percentage to be added to both mixtures was calculated, and mixtures were prepared using the process mentioned above.

The gradation of the aggregates is provided in Figure 6-1, with the limitations specified in the standards available noted. A number of different tests were performed on the materials. The aggregates had a specific gravity of 2.601 and optimum moisture content of 6.3%. The maximum dry density (MDD) was determined to be 15.4 kN/m³, with a Los Angeles abrasion of 23% and primarily siliceous material.

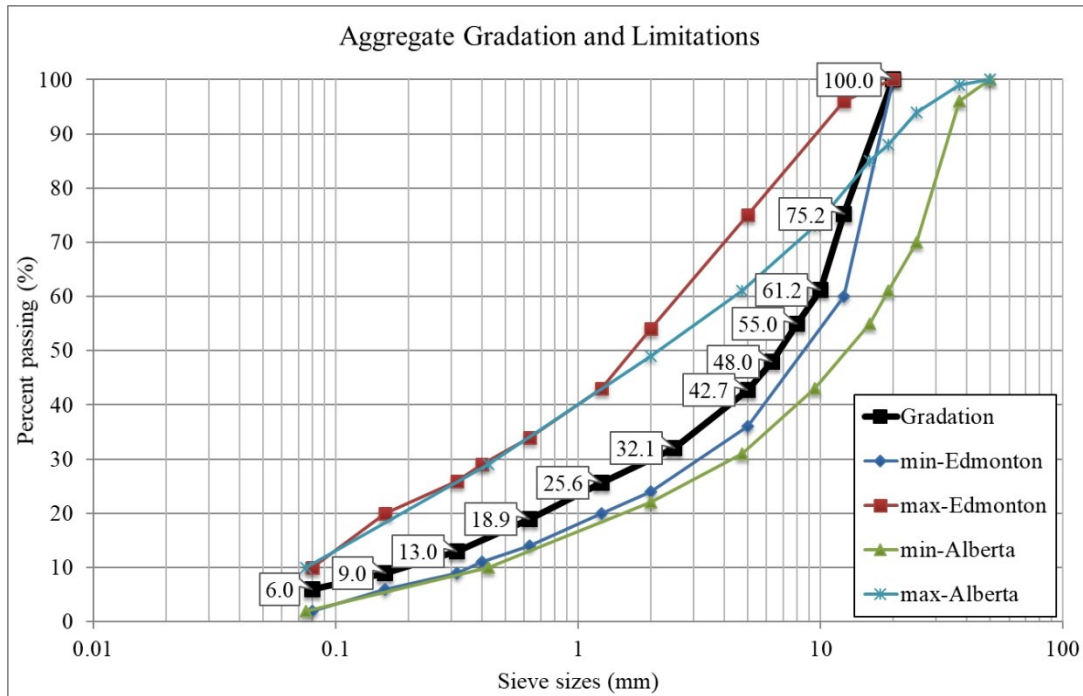


Figure 6-1 Gradation of the aggregates

6.2.2. Asphalt Emulsion

Asphalt emulsion was used as a binding material with aggregates, and, based on material properties such as aggregate charge and minimum time required for the mixing and for laying of the base layer (Wirtgen Cold Recycling Manual, 2012), a cationic slow setting (CSS-1H) asphalt emulsion was selected. Cold mixtures are dependent on the emulsion type due to the time needed for the materials to be mixed before breaking and dispersing. The asphalt emulsion used in this study contained 61% hard binder and 39 % water with a base binder of 80/100 penetration and a viscosity of 0.42 Pa-s at 135 °C. The results of the test conducted on the asphalt emulsion are presented in Table 6-1.

6.2.3. Asphaltenes

Alberta oil sands bitumen was the source of the asphaltenes used in this research, obtained by processing through a solvent deasphalting process. One source of asphaltenes was in solid form, sieved through a No. 100 mesh sieve after more powderization. Asphaltenes used in this study has density value lower than the density of water about 403.6 kg/m³. In addition, relative quantities of saturates, asphaltenes, resins, and aromatics were determined using the SARA

test. This result indicated that asphaltenes used was composed of 79.6% asphaltenes, 9.7% aromatics, 6.9% saturates, and 3.8% resins.

6.2.4. Cement

A commercially available general-use (GU) cement meeting the specifications of CSA A3000 was used as another additive with the mixtures. The properties of the cement are presented in Table 6-1.

Table 6-1 Properties of asphalt emulsion and cement

Material	Property (unit)	Standard	Specification		Typical Analyses
			Minimum	Maximum	
Asphalt Emulsion	Specific gravity (Density) at 15.6°C (kg/L)		-	-	1.020
	Residue by distillation (% by mass)	AASHTO T59 (2022)	57	-	61
	Viscosity at 25 °C (S.F.S)		20	100	22
	Oversized particles (% by mass)		-	0.300	0.008
	Settlement (24 h) (% by mass)		-	1.0	0.5
	Particle charge test	ASTM D7402 (2017)	Positive		Positive
	Initial setting time (min)		45		-
Final setting time (min)		420		-	
Cement	Compressive strength at 3 days (MPa)	ASTM 1157 (2020)	13		-
	Compressive strength at 7 days (MPa)		20		-
	Compressive strength at 28 days (MPa)		28		-

6.3. Methods

6.3.1. Impact of Additives on Breaking Time of Asphalt Emulsion

Prior to conducting various tests, it was essential to analyze the breaking time of the asphalt emulsion, as it plays a crucial role in cold mix stabilization. Asphalt emulsion is designed to break when it comes into contact with external substances such as aggregates (Asphalt Institute, 2008). The breaking time refers to the duration required for the asphalt emulsion to break and set. This time can vary depending on the type of emulsion being used. The concept of dimulsibility is employed to describe the chemical breaking of the emulsion, and ASTM

D244-09 (2017) is the standard method used to measure this factor for rapid setting and medium setting asphalt emulsions. This method involves the use of a calcium chloride solution to separate the water from the oil in the emulsion. However, when it comes to determining the breaking time of the emulsion, there is no specific standardized method available. Additives such as hydrated lime and cement contains calcium chloride that can cause the asphalt emulsion to break and reduce the curing time (S. F. Brown & Needham, 2000). The addition of cement to the asphalt emulsion has dual effects. Firstly, it absorbs the water present, and secondly, it increases the pH of the emulsion, leading to its breaking (Niazi & Jalili, 2009). Researchers like Terrell and Wang [48] have investigated the effect of adding cement to asphalt emulsion by adding different amounts of cement (ranging from 0% to 3%) to evaluate the curing time. Furthermore, other studies have been conducted to assess the impact of cement on asphalt emulsion (Head, 1974; Schmidt et al., 1973). In this study, a comparative approach was adopted to evaluate the effects of different modifiers. Equal quantities of filler (passing 80 μ m), filler/asphaltenes, and filler/cement were added to fixed amounts of asphalt emulsion, and the breaking time was observed. Specifically, when filler and cement were added to the asphalt emulsion, the suspension broke after approximately 19 hours. Similarly, the addition of filler and asphaltenes resulted in a breaking time of approximately 27 hours. In contrast, when only filler was added to the selected asphalt emulsion (CSS-1H) in this project, the suspension remained unbroken for about 44 hours.

6.3.2. Rheological Analysis on the Residue of Asphalt Emulsion

The asphalt emulsion residue was collected using a low-temperature evaporation technique (AASHTO PP72-11 procedure A (2013)) to maintain the asphalt emulsion's rheological properties. The materials were poured into a silicon mat at a spread rate of 1.5 to 2.0 kg/m². The mat was left in an oven for 24 h at 25 °C, then cured at 60 °C for another 24 h. Afterward, the cured material was left at room temperature to cool down and then carefully peeled off the silicon mat and stored in a container for further binder testing.

The frequency sweep test on the unmodified binder and 26.95% asphaltenes-modified binder equal to 1% by weight of the total mixture was used to analyze the rheological performance of the residues at various temperatures and loading frequencies. A dynamic shear rheometer (DSR) with 8 mm diameter plates and a gap of 2 mm was used for this test. The collected

asphalt emulsion residues were heated until fluid enough to pour inside an 8 mm diameter DSR mold. Test temperature loops of 4 °C, 10 °C, 20 °C, 25 °C, 30 °C, and 40 °C, with an angular frequency range of 0.1 to 100 rad/s, were used in this experiment. Three replicates were prepared for each of the binders. The time–temperature superposition (TTS) principle and Williams-Landel-Ferry (WLF) regression analysis were applied using RheoCompass software to plot the master curves for the complex modulus and phase angle at 25 °C over a wide range of frequencies.

6.3.3. Field Emission Scanning Electron Microscopy of Recovered Binders

Asphalt emulsion was modified by adding similar amount of filler, filler/cement and filler/asphaltenes separately and then analyzed with field emission scanning electron microscopy (FESEM) after recovering the binder. A Hitachi S4800 used to run FESEM on the samples after a 10-nanometer gold coating on the samples using a Denton Sputter coater.

6.3.4. Optimum Emulsion Content and Mix Preparation

The procedure proposed by Asphalt Institute (2008) and by Sabita (2020) were used to design the stabilized mixture with aggregates. The first step was to conduct the proctor test on the aggregates to determine the OFC and, based on that, the OMC. This method used four different percentages of asphalt emulsion and water with increasing increments of 1% (i.e., 3%, 4%, 5%, and 6% per total aggregates). Also, further to the test results a trend line suggested an OFC of 3.7% and added to the mix design to confirm. The ITS test was used to finalize the OEC for mixtures. Sample preparation began with oven-drying the materials overnight and allowing them to cool to room temperature before mixing to remove any existing moisture. Sufficient quantities of water and asphalt emulsion to reach the OFC were added to the mixture and mixed until they were uniformly distributed. The prepared mixture was compacted using a Marshall hammer with 50 blows on each sample side. After the compaction stage was complete, prepared samples were left to cure in a 60 °C oven for 48 h (ARRA, 2001) and then de-molded and cooled down before running the volumetric and performance test.

Additionally, for the modified samples, asphaltenes were added before adding the water in 1 % content to the aggregates with OEC. This asphaltenes content was selected based on previous research finding 1 % to be the optimum content for the modification (Kamran et al., 2021). In addition, cement was introduced to the aggregates before adding water, and a similar

process for the rest of the sample preparation to the original method was followed, with a notable difference being the removal of filler equal to the weight of cement added to the mixture. Three replicates for each sample were prepared and labelled as EX-(A or C)Y, where E refers to the asphalt emulsion, X is the percentage of emulsion added, A is the asphaltenes, C is cement, and Y is the percentage of asphaltenes, or cement added to the mix. For example, E3.7-A1 refers to a mix with 3.7% of asphalt emulsion and 1% asphaltenes modification.

6.3.5. Indirect Tensile Strength Test

The ITS test was performed per AASHTO T283 (2021). The samples containing the aggregates were prepared in triplicate and tested in dry conditions according to the instructions provided by the Asphalt Institute (2008) and Sabita (2020). The samples were conditioned in a temperature-controlled environmental chamber at 25 °C for 3 h prior to testing. Load was applied to the samples at a constant rate of 50 mm/min, and the maximum load before the failure point was recorded to determine the ITS. Equation (6-1) was used to calculate the ITS for the samples:

$$S_t = \frac{2000P}{\pi tD} \quad (6-1)$$

where: S_t = Indirect tensile strength (kPa), P = Maximum applied load (N), t = Average height of specimen (mm), D = Diameter of specimen (mm).

6.3.6. Tensile Strength Ratio

The ITS test determines the OEC, and modification of the mixture with asphaltenes was continued on samples with OEC. Six replicates of samples (3 for ITS-dry and 3 for ITS-saturated) were prepared, compacted, and cured in a similar manner to that described in the design section. AASHTO T283 (2021) was followed for the ITS tests on all of the prepared samples. ITS-dry and ITS-saturated were conducted in a similar manner to the design sample preparation. The ITS for all the samples was calculated using Equation 1, and then TSR was determined as the ratio of ITS-saturated to ITS-dry. The limitation of 50% for the TSR values was drawn from Sabita (2020).

6.3.7. Indirect Tensile Asphalt Cracking Test (IDEAL-CT)

The IDEAL-CT as a simple and practical method is among the most popular tests for determining cracking potential of asphalt mixes (Zhou et al., 2016). This test has been conducted using the traditional ITS test setup on the sample by calculating the fracture energy (FE) and, accordingly, the crack tolerance index (CT-Index). A constant 60mm/min load applied to the samples and load-displacement values were recorded until the load reduced to 0.1kN. This parameter was derived from the load-displacement curve of the samples after ITS testing. For analysis purposes it was considered that a higher CT-index value is indicative of a higher fatigue resistance for a given sample. Several researchers has studied the effectiveness of using CT-Index as a fatigue cracking parameter and this parameter has been used for field fatigue cracking results (Alae et al., 2023; Saha Chowdhury et al., 2022). Equation (6-2) was used to calculate this parameter.

$$CT_{Index} = \frac{t}{62} * \frac{l_{75}}{D} * \frac{G_f}{|m_{75}|} * 10^6 \quad (6-2)$$

where: CT_{Index} = Crack tolerance index, G_f = Fracture energy (Joules/m²), $|m_{75}|$ = Absolute value of the post-peak slope (N/m), l_{75} = Displacement at 75% of post-peak slope (mm), D = Specimen diameter (mm), t = Thickness of specimen (mm)

6.3.8. Hamburg Wheel Tracking (HWT) Test

AASHTO T 324-19 (2019) was followed for the HWT test. High-temperature properties of the mixtures were evaluated using this test to simulate the real-life span for a base layer prepared using these mixtures. Slab samples were prepared for this test with dimensions of 400 mm length, 300 mm width, and 80 mm height, taking into consideration the nominal maximum aggregate size and curing conditions of the samples. The samples were compacted using a roller compactor and cured in a similar manner to Marshall samples. Considering the binder grades and base layer as the location of our mixture, the test temperature was set at 40 °C. The standard prescribed 45 min of preconditioning prior to testing, and a steel wheel with 47 mm width and 705 ± 4.5 N weight at a frequency of 52 ± 2 passes per minute and a maximum speed of 0.305 m/s at midpoint, were used to run the test. The termination point for the test was set to 20,000 passes or 12.5 mm rutting depth, whichever was reached first. Two indicators were

calculated using the results from the test. The rutting resistance index (RRI) was calculated by multiplying the number of passes by one inch subtracted by the rut depth (in inches), and the stripping inflection point (SIP) was determined based on the HWT standard in order to evaluate the rutting potential and moisture susceptibility of the mixtures.

6.3.9. Indirect Tensile Strength Test at Low Temperatures

The ITS test was conducted per AASHTO T322-07 (2020) at 0 °C and –10 °C for both unmodified and modified mixtures with asphaltenes and with cement. Three replicates of samples for each testing temperature and additive, along with control samples without any asphaltenes or cement, were prepared for this test. A load at the constant rate of 12.5 mm/min was applied to the sample prepared after 3 h of conditioning at the specified temperature. The ITS and FE values were calculated based on the load-deformation data collected from the test and using Equation 1.

6.3.10. Dynamic Modulus and Flow Number

AASHTO T342-11 [59] and T378-17 (2021) were followed to establish the dynamic modulus and flow number test results for the mixtures prepared. Based on the dynamic modulus $|E^*|$ test, stress and strain responses of the mixtures and correlation of the time–temperature-dependent properties to field performance could be determined, and the master curve was calculated using the AASHTO R 62 (2021). Dynamic modulus results, it should be noted, are the critical inputs for the design of flexible pavements based on the mechanistic-empirical pavement design guide (MEPDG). These results depend on different elements of the layer, such as asphalt binder, aggregate structure, and physiochemical interactions between them. In the mixture, aggregates are the primary particles, as they play a significant role in the dynamic modulus test results (Baghaee Moghaddam, 2019; Yu & Shen, 2012). A universal testing machine (UTM) was used to conduct the test with linear variable differential transformers (LVTDs) attached to the sample to record the deformation under a sinusoidal axial compressive stress. Two replicates of samples were prepared for each mixture using a gyratory compactor set at a height of 170 mm and a diameter of 150 mm. Before the test, all samples were cut and cored to a height of 150 mm and a diameter of 100 mm. The test temperatures were set at 4 °C, 20 °C, and 45 °C and testing frequencies of 0.01 Hz, 0.1 Hz, 1 Hz, and 10 Hz were used for the dynamic modulus.

The flow number test was run at 45 °C, i.e., the high adjusted performance grade temperature estimated by long-term pavement performance bind (LTPPBind) based on the project location (Edmonton, Canada). A contact load of 3 kPa and a deviator load of 69 kPa were selected for the test, with a haversine axial compressive loading pattern with a loading period of 0.1 s and a resting period of 0.9 s. The termination point for the test was set at 20,000 cycles or a maximum of 50,000 microstrains—whichever was reached first.

6.4. Results and discussion

6.4.1. Rheological Analysis on the Residue of Asphalt Emulsion

The frequency sweep test data was used to calculate the master curves for the control and asphaltenes-modified samples. The complex shear modulus and phase shift angle master curves were evaluated using the time–temperature superposition (TTS) principle and Williams-Landel-Ferry (WLF) regression analysis at 25 °C. Figure 6-2 shows the phase shift angle master curve, where the results indicate a decrease in the phase angle of the modified binder compared to the control samples without any asphaltenes. A phase angle of 70° was observed for control samples at 1 Hz angular frequency compared to 45° for modified samples. This decrease suggests that asphaltenes reduces the viscous behaviour of modified binders.

Figure 6-3 presents the complex shear modulus master curve at 25 °C. Significant improvement in the complex shear modulus of the asphaltenes-modified binder compared to the control samples can be seen from these data at different frequencies. However, the lower frequencies show significant improvement compared to the higher frequencies. Based on these results, the complex shear modulus of the control sample is in the range of 105 and 106 at 1 Hz compared to the asphaltenes-modified binder, which is between 106 and 107. These results are similar to the phase angles results and show a stiffening effect of the binder with asphaltenes modification. However, the samples at low frequencies (higher temperatures) were found to be affected more compared to samples at high frequencies (low temperatures).

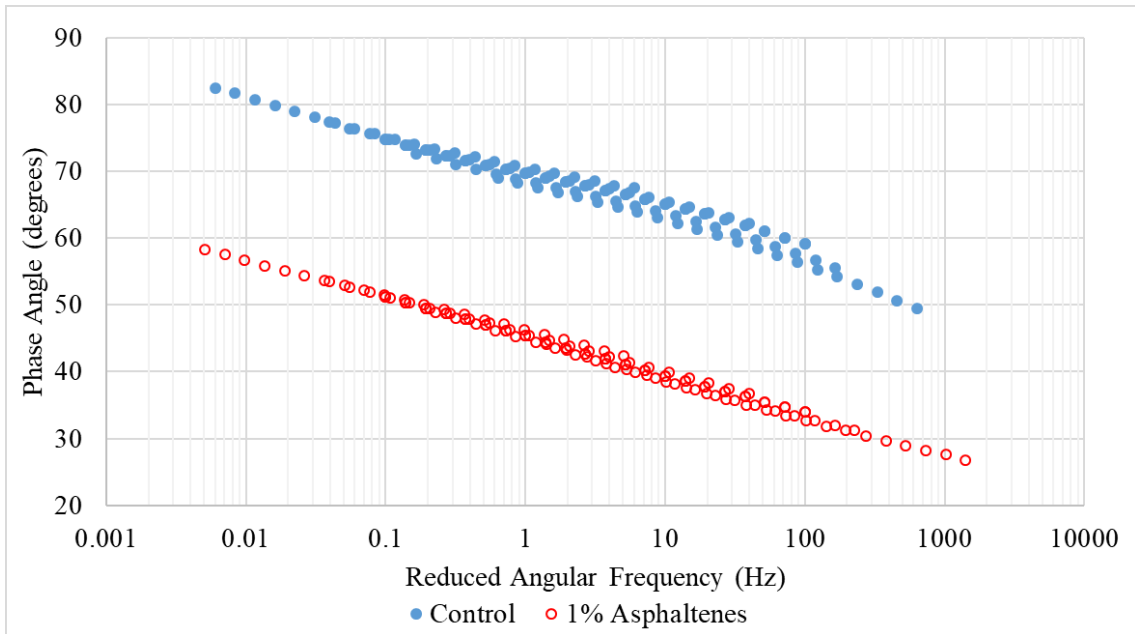


Figure 6-2 Phase angle master curve at 25 °C

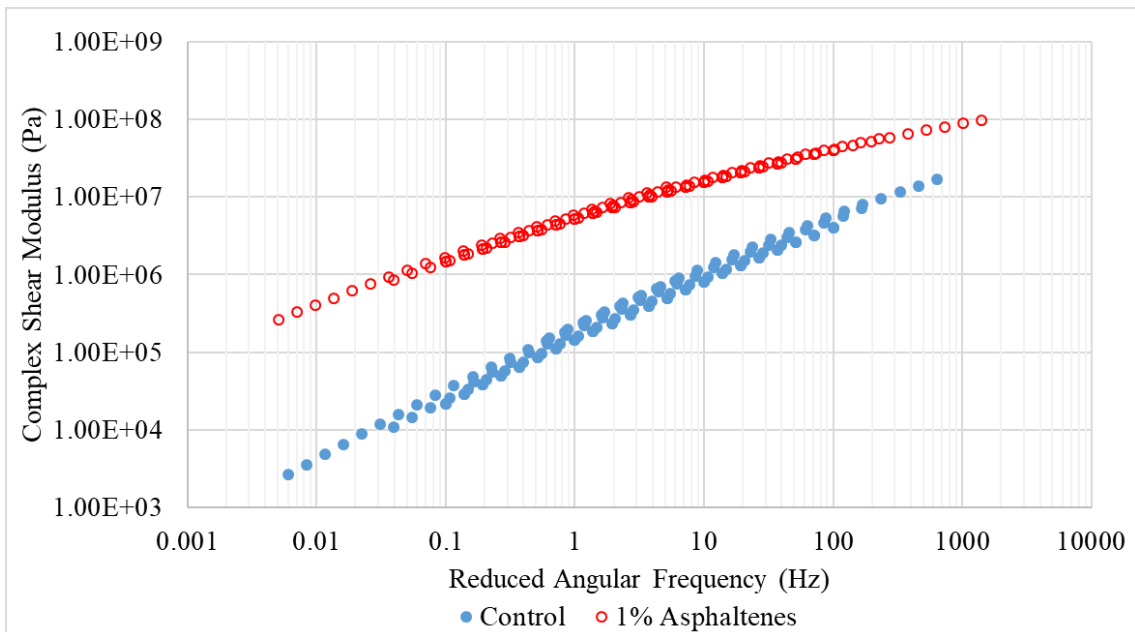


Figure 6-3 Complex shear modulus master curve at 25 °C

6.4.2. FESEM

Figure 6-4 presents the results obtained from FESEM at 5000 magnification for asphalt emulsion residue modified using different substances. Figure 6-4a illustrates the presence of filler particles that have passed through an 80 μm sieve and are mixed with asphalt emulsion after curing. These particles are coated with asphalt binder, appearing as several small pieces

rather than being agglomerated to sizes smaller than the sieve openings they passed through. In Figure 6-4b, asphaltene particles are fully coated with asphalt binder, forming chunks or separate particles smaller than 150 μm (sieve size passed through). Finally, Figure 6-4c demonstrates a fully agglomerated chunk of filler with cement components, which bind the particles together. These findings indicate that the filler itself does not directly interact with the binder and is mostly dispersed within the mixture. However, asphaltene particles are effectively covered by the binder, leading to interactions that alter the binder properties, as observed in the frequency sweep tests. Furthermore, comparing these two results with the cement-modified binder reveals that cement does not directly react with the binder but primarily adheres to the fine particles, facilitating their agglomeration and contributing to the overall stiffness of the mixture beyond the binder's influence. Furthermore, comparing these two results with the cement-modified binder reveals that cement does not directly react with the binder but primarily adheres to the fine particles, facilitating their agglomeration and contributing to the overall stiffness of the mixture beyond the binder's influence. Cement reacts with water and hydrates, forming bonds with the filler material and providing structural integrity to the mixture. This outcome is anticipated due to the distinct properties of asphalt and cement, particularly at normal temperatures. However, asphaltene particles are inherent components of asphalt, and their incorporation alters the asphalt's composition, enhancing the structural strength of the binder rather than the entire mixture. The inclusion of more polar particles and a stiffer binder subsequently contributes to increased strength and improved resistance to rutting compared to unmodified mixtures. Figure 6-4d presents the samples after gold coating.

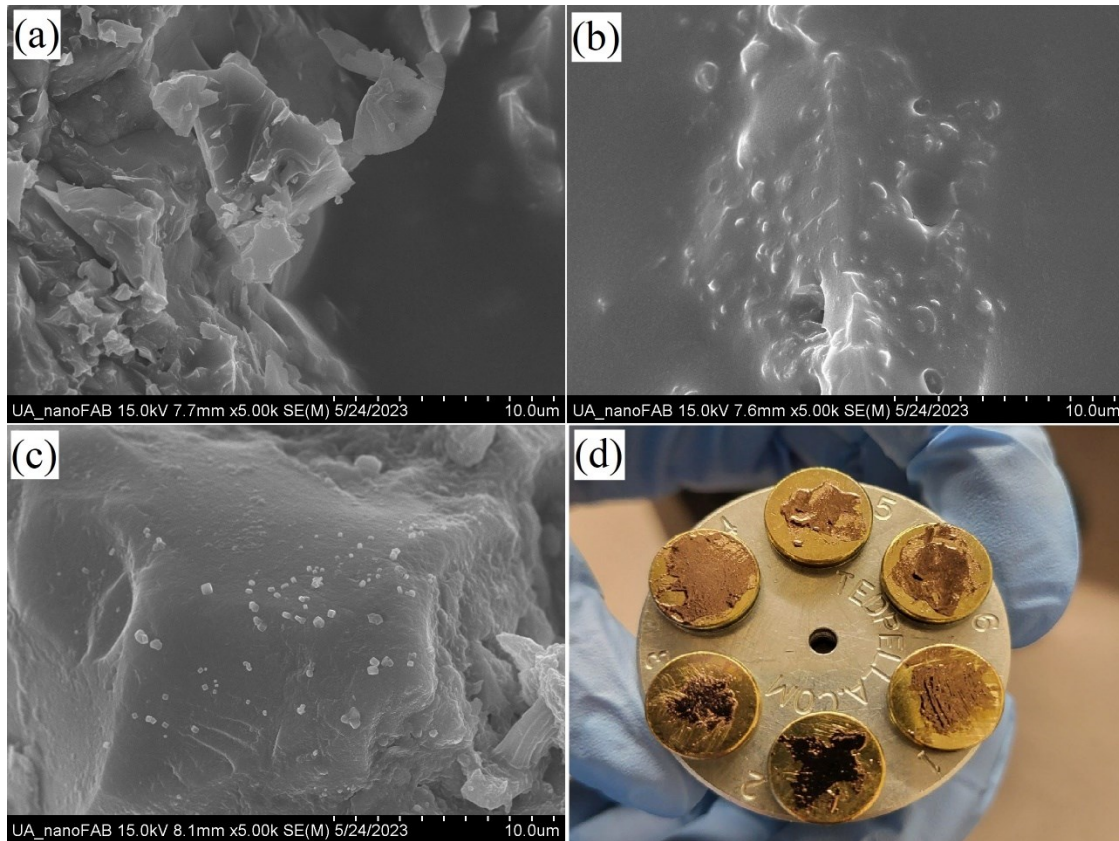


Figure 6-4 FESEM samples (a) filler, (b) asphaltenes, (c) cement, (d) prepared samples, and result at x5.0k magnification

6.4.3. Indirect Tensile Strength Test for Design Mixtures

The ITS and TSR results for the mix design samples prepared with unmodified aggregates were evaluated to determine the OEC for the continuation of the study. The minimum limitations for the ITS-dry and ITS-saturated were set to 225 kPa and 125 kPa, respectively, based on Sabita (2020). Figure 6-5 shows the results for the tested samples, reflecting the OEC for the mixtures. The results indicate that 3.7% of asphalt emulsion was the OEC for these mixtures. All the mixtures prepared with different percentages of the asphalt emulsion satisfied the design limits, showing considerable strength under the load applied. These results were confirmed using different sources of asphaltenes, suggesting that the different source do not correspond to significant changes in performance.

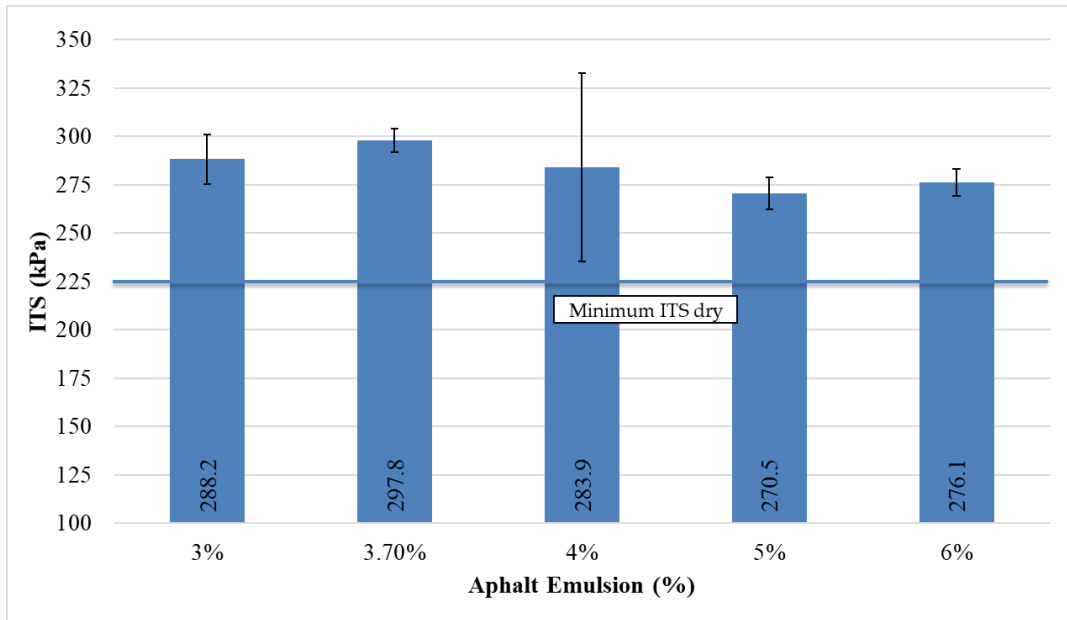


Figure 6-5 Mix design ITS results for mixtures

6.4.4. TSR, ITS, and IDEAL-CT Test for Modified Mixtures

The OEC was adopted to prepare the modified samples, which were then tested for ITS. IDEAL-CT, ITS, and TSR values were reported for both the 1% asphaltenes and 1% cement samples. The average air voids for these samples was 10.9%, 11.2%, and 11.5% for E3.7, E3.7-A1, and E3.7-C1, respectively. Table 6-2 presents the results for these mixtures with the design limits specified. Asphaltenes and cement modification of the aggregates was found to significantly increase the ITS-dry and -saturated, with these values for asphaltenes increased by about 315 kPa and 258 kPa, respectively. However, cement modification only increased these values by about 152 kPa and 154 kPa, respectively, for the parameters mentioned above. Moreover, these results indicate the higher strength for the modified mixtures compared to the unmodified mixtures. The TSR values for mixtures modified with asphaltenes, meanwhile, decreased, whereas cement modification increased this value. However, all the saturated-TSR values were higher than the 50% limit specified. IDEAL-CT test result was determined for the evaluation of FE and CT-Index. The FE values for asphaltenes-modified samples increased by about 660 J/m², while the cement-modified samples decreased by about 112 J/m² relative to the control samples. Despite the differing changes in FE observed, the CT-index decreased for both methods of modification, by about 14.5 for asphaltenes and 15.1 for cement relative to the control samples. These results indicate that cement has a more adverse impact on the fatigue

resistance of stabilized samples compared to asphaltenes. Figure 6-6 presents the load-displacement graphs for all three samples with the absolute value of post-peak slope $|m_{75}|$ and displacement at 75% of peak load. These graphs show the crack propagation of each sample, and it can be seen that the asphaltenes-modified samples exhibit the highest slope, followed by the cement-modified and control samples.

Table 6-2 ITS, TSR, and IDEAL-CT results for asphaltenes modified mixtures

Sample ID	Dry	IDEAL-CT		Saturated			
	ITS (kPa)	STDV ¹	FE (J/m ²)	CT-Index	ITS (kPa)	STDV	TSR
E3.7	297.8	6.1	1,056.4	22.7	305.8	1.4	1.027
E3.7-A1	613.2	33.1	1,716.8	8.2	563.7	28.4	0.919
E3.7-C1	449.7	3.3	944.2	7.6	459.9	13.4	1.020
Minimum	225.0	-	-	-	125.0	-	0.500

¹ STDV: standard deviation.

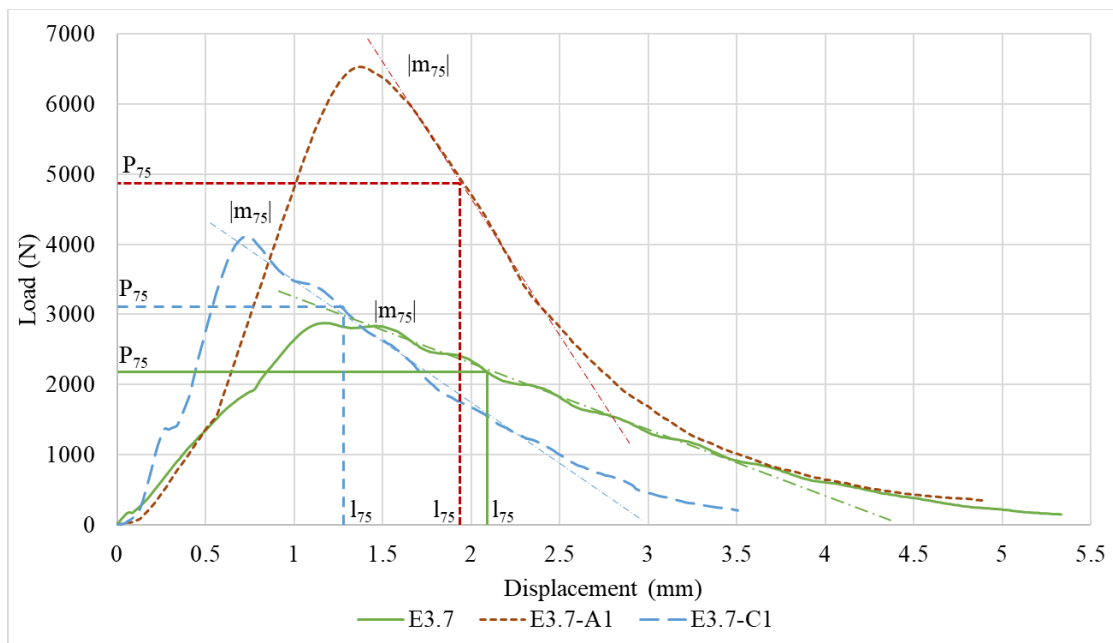


Figure 6-6 Load-displacement graph

6.4.5. Hamburg Wheel Tracking Test

The HWT test samples were prepared in slabs and tested according to AASHTO T324-19 (2019) to determine the rutting resistance and moisture susceptibility of the mixtures. The moisture sensitivity of the samples was calculated by dividing the SIP values by the maximum number of passes. Figure 6-7 and Table 6-3 present the results of the HWT test for the asphaltenes-modified, cement-modified, and unmodified mixtures. About 3,940 passes for

E3.7 at maximum rutting depth, 8,712 for E3.7-A1, and 11,220 passes for E3.7-C1 was observed. Because the test was terminated at different rutting depths for the different samples and more reliable values were needed in order to compare the rutting resistance, RRI was calculated. Improvements of about 142% and 157% were observed for E3.7-A1 and E3.7-C1 in RRI results, respectively. There was an increase in the number of passes before failure for both of the modified mixtures, indicative of a stiffer sample following modification and delayed failure compared to the control samples. The SIP values also increased for the modified samples, from 3,800 passes for E3.7 to 6,800 for asphaltenes-modified mixtures and 6,500 for cement-modified mixtures. Considering that all the SIP values were under 10,000 passes, there is a possibility for moisture susceptibility of all the mixtures (Aschenbrener, 1995). The moisture sensitivity index also supports this decreasing result for TSR of asphaltenes-modified mixtures.

Table 6-3 Wheel tracking test result

Sample ID	SIP	Number of passes at 12.5 mm rutting	RRI	Moisture sensitivity index
E3.7	3,800	3,940	2,219.74	0.96
E3.7-A1	6,800	8,712	5,360.97	0.78
E3.7-C1	6,500	11,220	5,698.35	0.58

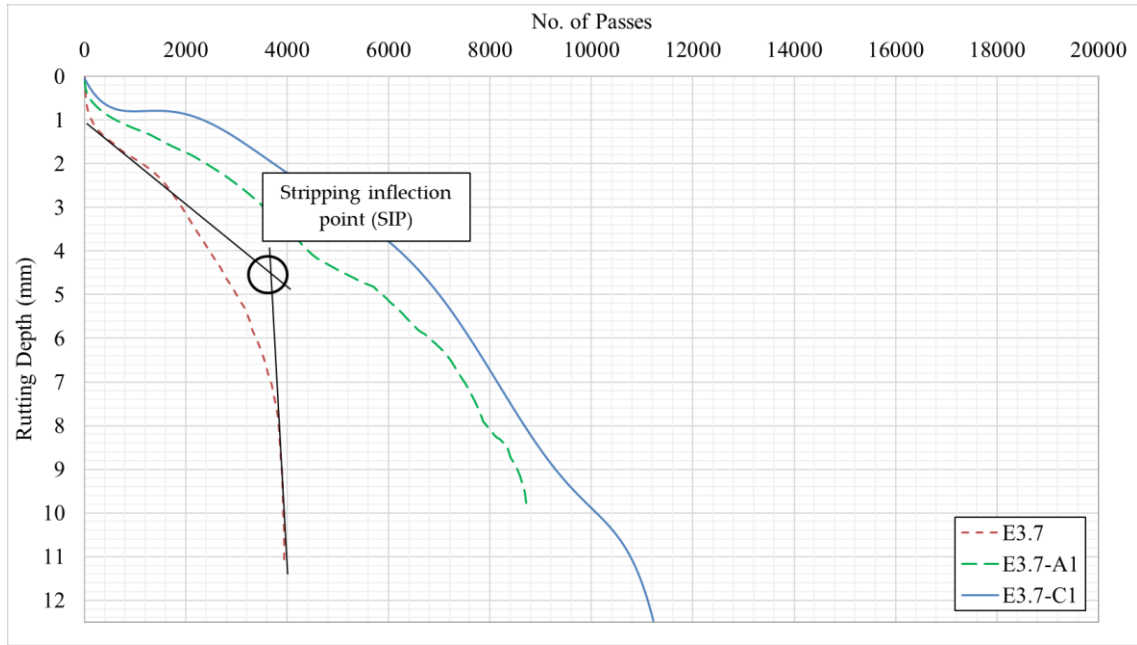


Figure 6-7 Hamburg wheel tracking results

6.4.6. Indirect Tensile Strength Test at Low Temperatures

The low-temperature properties of the modified mixture were evaluated using the ITS test at 0 °C and -10 °C. ITS and FE were calculated for all of the samples using the load-deformation data. These results reveal the low-temperature and crack propagation properties of the mixtures (Figure 6-8). The ITS test results at 0 °C show an increase for asphaltene-modified samples compared to the E3.7 from 1,083.1 kPa to 1,170.1 kPa. A similar value was seen for the cement-modified mixtures compared to E3.7 as 1,080.5 kPa. However, a comparison of the asphaltene and cement shows higher values for asphaltene modification, about 89.6 kPa. A comparison of the FE for the asphaltene and cement to E3.7 shows a decreasing trend from 2,437.3 J/m² to 1,852.7 J/m² and 1,538.3 J/m², respectively. Decreasing the temperature to -10 °C resulted in an increasing trend for both ITS and FE compared to the results at 0 °C, as expected. On the other hand, the ITS at -10 °C decreased for both asphaltene- and cement-modified samples compared to the control sample (E3.7), from 1,357.3 kPa to 1,255.3 kPa and 1,159.8 kPa, respectively. Analyzing the FE for these mixtures at -10 °C, decreases from 2,713.0 J/m² to 2,032.2 J/m² and 1,510.0 J/m² were observed for asphaltene-modified and cement-modified mixtures, respectively, compared to the control samples. It can be concluded from these results that the modified mixtures became stiffer at low temperatures, while crack propagation increased with these changes. The cement-modified mixtures were found to be

more susceptible to low temperature cracking than were the asphaltenes-modified mixtures. These mixtures are strong and at the same time, susceptible to shrinkage cracking and fast crack propagation. As a result, comparing these two additives, asphaltenes modification has a less negative effect low temperature cracking. Observing the variability of data in the samples, it's evident that there is not a substantial difference between the ITS values at 0°C, however the difference is more pronounced at -10°C. This could be related to the higher susceptibility of cement treated material to shrinkage cracking at lower temperatures. When we consider fracture energy and compare the error bars, a different pattern emerges. At both testing temperatures, the fracture energy of cement modified mixes is the lowest which shows these mixes are more prone to crack propagation.

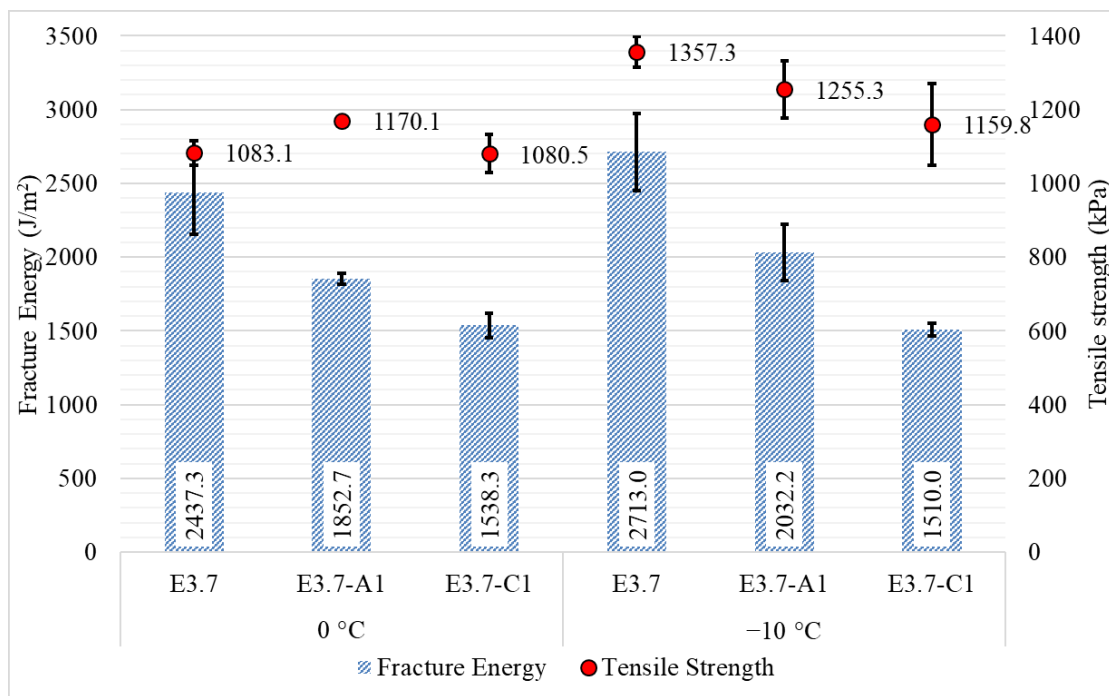


Figure 6-8 Low-temperature test results

6.4.7. Dynamic Modulus and Flow Number

The dynamic modulus test was conducted based on AASHTO T342-11 (2019) and T378-17 (2021), and the master curve presented was obtained using AASHTO R 62 (2021) and the MEPDG method. Figure 6-9 presents the master curves for three different mixtures prepared at a reference temperature of 21 °C. The results for these mixtures show an increase for the modified mixtures compared to the E3.7 as the control sample. E3.7-C1 has higher modulus

values than E3.7-A1, suggesting higher stiffness for the cement-modified mixture. The shape of the dynamic modulus master curve of E3.7-C1, with its almost straight line, suggests that cement modification increases the elastic behaviour of the mixtures more than the viscous behaviour compared to the asphaltenes-modified samples. The modified mixtures were found to be stiffer at low temperatures; meanwhile, a comparison of the cement-modified samples to asphaltenes modification shows a lower stiffness and, therefore better low-temperature properties in the case of asphaltenes modification. Overall, these results are similar to those from the low-temperature indirect tensile strength (IDT) and high-temperature HWT results. The dynamic modulus test results show an improvement from both asphaltenes and cement modification. These values at 10 Hz increased about 17% and 22% for asphaltenes and cement, respectively, compared to the control samples.

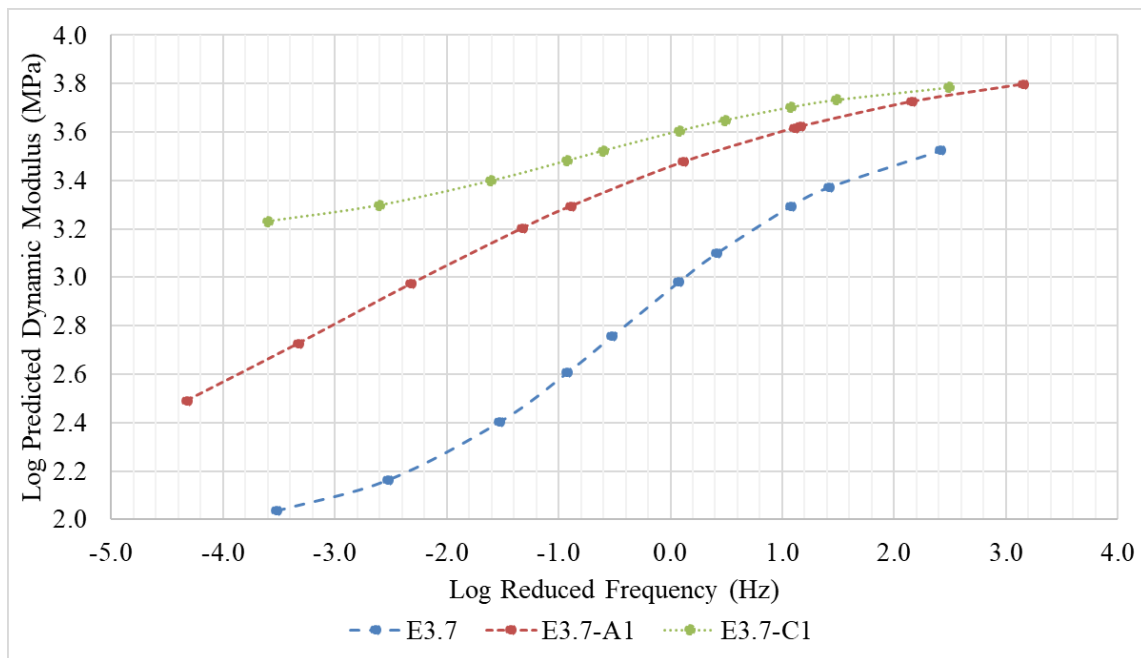


Figure 6-9 Dynamic modulus master curve

Figure 6-10 presents the phase angle master curve, which shows a decrease in the phase angle of the modified samples compared to the unmodified E3.7. At lower frequencies, the asphaltenes-modified mixtures showed the highest phase angle, indicating more viscous behaviour than cement and unmodified mixtures. The cement-modified mixtures had the lowest phase angle at low frequencies (high-temperature properties), showing more elastic behaviour. Increasing the frequency to 10 Hz resulted in an increase in the phase angle of the

mixtures, while E3.7-A1 did not follow this increasing trend all throughout, instead starting to decrease after about 0.01 Hz and ultimately dropping lower than the control samples. Moreover, the decreasing trend in E3.7-A1 did not continue with increasing frequencies. Furthermore, it never dropped below that of E3.7-C1 until higher frequencies or low-temperature properties. It is worth noting that based on this results binder helps with the viscous behaviour of mixture rather than aggregates interlocks at lower frequencies of asphaltenes-modified mixes. The control mixtures and E3.7-C1 show similar phase angle trends with changing frequencies, with lower values observed for cement-modified mixtures than for the control sample throughout. The lower phase angle values at higher frequencies for modified mixtures compared to control mixtures suggest that modification decreases the viscous behaviour of the mixtures at low temperatures. There was a decrease by about 33% and 52% in phase angle for asphaltenes and cement, respectively, compared to the control samples. The dynamic modulus and phase angle changes suggest increasing elastic behaviour for both asphaltenes modification and cement modification, with cement having a greater impact.

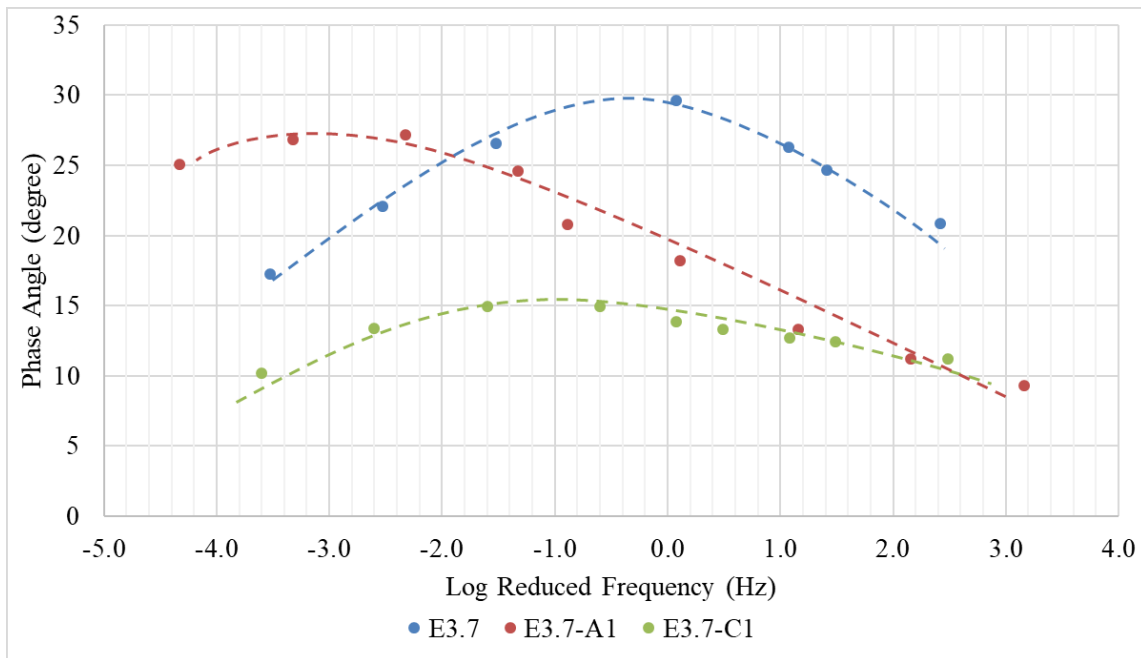


Figure 6-10 Phase angle master curve

Comparing the asphalt binder frequency sweep results and dynamic modulus tests, the low frequency (or high temperature) properties improved for the modified mixtures relative to the control. Both mixes became stiffer with modification, decreasing the viscous behaviour and

increasing the elastic behaviour. These findings demonstrate the superior high-temperature properties of the modified mixtures, could be confirmed by the findings of other tests such as HWT and flow number. Comparing the phase angle results, asphaltene-modified binders show stiffer properties at lower frequencies. This behaviour is not consistent for the mixture phase angle, where we see a higher phase angle for asphaltene-modified mixture, indicating a higher effect of binder than the aggregate skeleton in the mixture after modification. However, for cement-modified mixtures, the phase angle of the mixture is lower than that of the control sample, suggesting a more elastic behaviour and that the aggregate skeleton interlocks are helping with the high-temperature properties. As a result of these tests, we can infer that cement interacts more with aggregates and improves the aggregate structure, whereas asphaltene affects the binder phase of the mixtures.

A flow number test was conducted based on AASHTO T378-17 (2021) for all the mixtures, with the results presented in Figure 6-11. None of the samples moved to the tertiary zone, and the test reached the cycle number limit prior to failure in every case. However, it is worth noting that the control samples accumulated more microstrains than did the modified samples. The E3.7-C1 mixture with 275.5 microstrain deformation had the lowest deformation compared to the 884.3 for E3.7-A1 and 4,760.8 for E3.7 samples, indicating similar results from HWT and dynamic modulus for high-temperature properties of the mixtures modified with cement. Asphaltene and cement both decreased the deformation of the stabilized mixtures at higher temperatures, with cement slightly more effective than asphaltene.

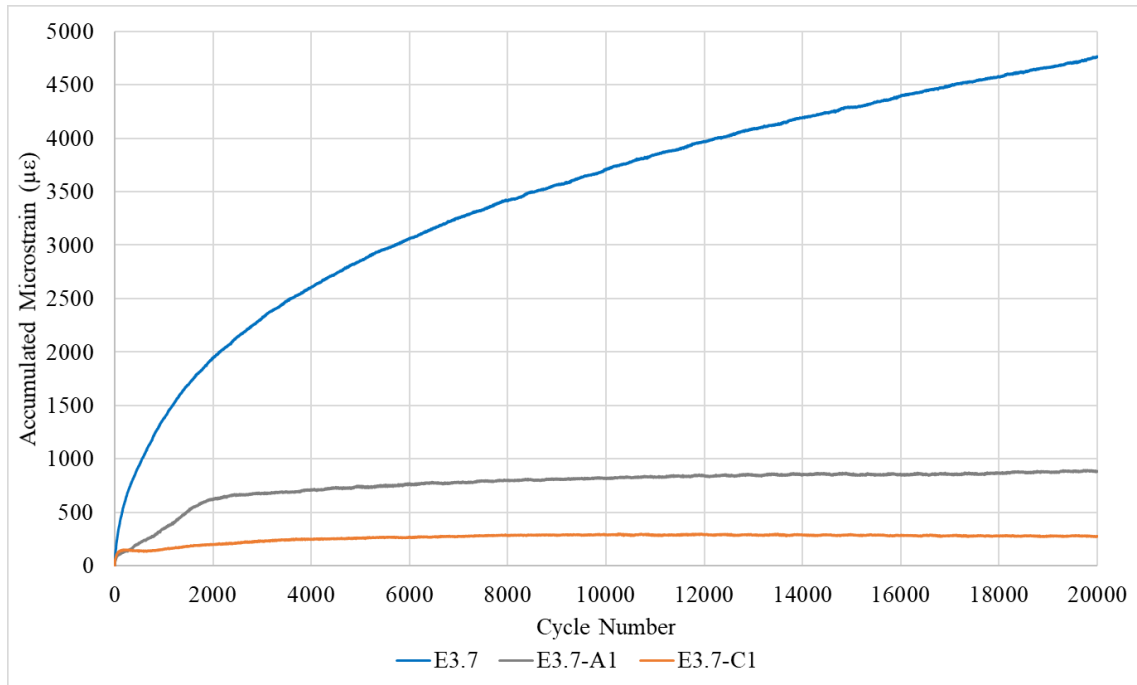


Figure 6-11 Flow number test results

6.5. Conclusions

The test matrix designed for this research topic to evaluate the possibility of replacing asphaltenes with cement led to the following conclusions:

- The measurement of breaking time for asphalt emulsion modified with cement and asphaltenes revealed a faster breaking time for asphaltenes modified asphalt emulsion compared to regular emulsion, but slower when compared to cement and asphalt emulsion. In addition, FESEM and DSR analysis showed asphaltenes modifies asphalt emulsion rheological properties, while cement mostly reacts with fine particles and makes the mix stiffer.
- Based on IDEAL-CT results, the cracking resistance of the asphaltenes modified mixes is higher than that of cement-modified mixes. However, moisture sensitivity of the cement-modified mixes is slightly less than asphaltenes modified mixes.
- According to HWT and flow number test results, cement modified mixes have slightly higher rutting resistance compared to asphaltenes modified mixes. Based on dynamic

modulus test results, this could be related to greater elastic properties of the cement modified mixes compared to asphaltenes modified mixes.

- Based on the results of the low-temperature tests, it is evident that both the modified mixes retain their strength compared to the unmodified mixes. However, it is notable that crack propagation in the modified mixes occurs more rapidly after reaching maximum strength, with the cement-modified samples being more adversely affected than the asphaltenes-modified samples.

It is worth mentioning that the results of this study is limited to one type of granular material and laboratory investigation. It is essential to validate all these results in the real world application.

The future objective of this research is to establish a test road for verifying the accuracy of the test outcomes, as well as to conduct continuous monitoring of the asphaltenes-modified layer's performance for validation purposes.

Chapter 7: Conclusions, Limitations, and Future

Directions

7.1. Research Summary

The background on cold mix asphalt and base layer stabilization highlighted some drawbacks of existing methods and identified gaps in the research that need attention. To address these issues, this study aimed to evaluate the performance of a granular-based layer and RAP stabilized with asphalt emulsion, using asphaltenes as an additive. The objective was to explore the possibility of replacing cement, a commonly used stabilizing additive with negative environmental and economic impacts. Furthermore, the impact of temperature on the mix design of base layers stabilized with asphalt emulsion was investigated.

Several laboratory tests were employed for the evaluation process. The ITS test was used in the mix design process in addition to assess intermediate temperature performance. Additionally, the Hamburg wheel track test was conducted at high temperatures to evaluate permanent deformation. To address low-temperature properties, the prepared mixes underwent creep compliance and strength tests at negative temperatures. The dynamic modulus test was also performed to generate modulus and phase angle master curves, enabling performance prediction for the prepared mixes. The dynamic modulus results were further analyzed to understand the temperature effect on the mixes and its influence on the mix design.

By conducting these comprehensive laboratory tests and analyses, this research sought to enhance the understanding of asphalt emulsion-stabilized base layers and identify alternative, more environmentally friendly additives to replace cement. The findings aim to contribute to the development of improved and sustainable base layer stabilization methods for road construction and maintenance.

Chapter 3: In this chapter, the study investigated the application of asphaltenes, derived from Alberta oil-sands bitumen as residue material, to enhance the mechanical properties of stabilized base courses. The research involved adding 1% to 3% of asphaltenes by the total weight of the mix to a granular aggregate stabilized with asphalt emulsion. The impact of asphaltenes on the tensile strength, moisture susceptibility, and rutting performance of the

modified mixtures was thoroughly examined. The results revealed that the OMC and OEC for the granular base layer were 6.3% per aggregates and 3.7% per total mixture, respectively. Comparing the wheel tracking test results and Modified Quotient (MQ) values within the prepared mixtures, a significant increase in rutting resistance was observed in the samples modified with 1% asphaltenes, as compared to the control mix. However, the addition of 2% asphaltenes did not yield a significant improvement in rutting resistance when compared to the samples modified with 1% asphaltenes. Overall, the asphaltenes-modified base courses exhibited higher tensile strength and rutting resistance than the control mix. Furthermore, their moisture susceptibility was found to be slightly lower than that of the control mix. These findings suggest that incorporating asphaltenes as an additive in stabilized base courses has the potential to enhance the pavement's mechanical properties, contributing to improved performance and durability. Additionally, this research demonstrates the feasibility of utilizing residue materials, such as asphaltenes, in sustainable road construction practices.

Chapter 4: In this chapter, the goal was to enhance the performance of asphalt emulsion-stabilized base courses by incorporating asphaltenes into the mix. Different amounts of asphaltenes were added to the mix at ambient temperatures to create modified mixtures. The research evaluated the Marshall stability and indirect tensile strength of these mixtures using varying contents of asphalt emulsion and asphaltenes. The investigation also delved into the low-temperature performance properties of selected mixtures through the indirect tensile test, while the high-temperature properties were assessed using the wheel tracking test. The study's findings indicate that the addition of asphaltenes improved the intermediate temperature properties and indirect tensile strength, while slightly reducing moisture resistance. However, this modification significantly enhanced the rutting resistance of the mixtures. On the other hand, the indirect tensile strength at low-temperatures revealed that the modified mixes exhibited a slightly higher susceptibility to low-temperature cracking when compared to the unmodified ones. Upon comparing the different dosages used, it was observed that the inclusion of 1% asphaltenes resulted in optimum performance results compared to using 2% of this material. Overall, the addition of asphaltenes proved to be beneficial for improving the asphalt emulsion-stabilized base course's rutting resistance and intermediate temperature properties. However, there was a trade-off with a slight increase in vulnerability to low-temperature cracking. The study's findings highlight the importance of carefully selecting the

appropriate dosage of asphaltenes to achieve optimal performance for specific pavement applications.

Chapter 5: In this chapter, the objective was to enhance the performance of asphalt emulsion recycled base courses prepared with RAP by incorporating asphaltenes into the mixture. Various performance properties of the selected mixtures were thoroughly investigated, including ITS, TSR, IDEAL-CT, HWT, dynamic modulus, and creep compliance and strength tests. The testing results, along with statistical analysis, revealed that the addition of asphaltenes significantly improved the high and intermediate temperature performance of the modified mixes. The enhanced rutting resistance and stability indicated the positive effects of using asphaltenes in the cold recycling process with asphalt emulsion and RAP. Importantly, the moisture sensitivity of the modified RAP mixes with asphaltenes remained unaffected by the additive, which is a favorable outcome in terms of the mix's durability and performance under varying moisture conditions. While the low-temperature creep compliance of the asphaltenes-modified mixtures was slightly lower than that of the unmodified mixtures, the low-temperature properties were not significantly affected. The dynamic modulus test results and master curve prediction demonstrated the temperature's importance in the design process of cold recycled mixes with asphalt emulsion. Overall, the study's outcomes emphasized the importance and positive effects of utilizing asphaltenes and 100% RAP in cold recycling, showcasing their potential to enhance the performance and durability of asphalt emulsion recycled base courses.

Chapter 6: Throughout this chapter, the impact of asphaltenes on improving the rheological properties of asphalt binder was thoroughly investigated. A series of binder characteristic tests using a dynamic shear rheometer, breaking time analysis, and microscopic evaluation were conducted on modified asphalt emulsion with asphaltenes. The performance of asphalt emulsion-stabilized granular materials with asphaltenes was compared with mixtures prepared using cement. These two mixes were then compared to unmodified mixtures, serving as the base of the research. To assess the performance of the modified mixtures, Hamburg wheel tracker and flow number tests were employed to evaluate permanent deformation and moisture susceptibility. The low-temperature properties of the mixes were investigated using indirect tensile strength tests, while dynamic modulus analysis was used to analyze the viscoelastic

behaviour of the mixtures. The conclusions drawn from this chapter are as follows: 1- The breaking time of asphalt emulsion decreased with the addition of modifiers, with cement having a faster effect on the emulsion than asphaltenes. 2- Rheological and chemical tests on binders revealed an increase in the performance of binders modified with asphaltenes compared to unmodified binders and cement. The asphaltenes reacted better with fine particles than the cement binder. 3- The moisture resistance of the mixtures was not significantly affected by the modification, while rutting resistance improved considerably with the addition of 1% of both additives (by total weight of the mix). 4- Some slight adverse effects on low-temperature properties were observed with the asphaltenes modification, showing less adverse impacts than the cement modification. 5- Elastic behaviour was observed in the modified mixes, with cement exhibiting more considerable changes compared to asphaltenes. Overall, the results demonstrate that asphaltenes, as residue material with a high production rate, hold promise as a viable alternative to cement in the stabilized base layer. The positive effects on rutting resistance and improved performance of asphalt emulsion-stabilized mixes make asphaltenes a valuable option for enhancing road construction practices.

7.2. Academic and Industry Contributions

This research contribution of industry and academia in the pavement engineering is summarized as following section.

7.2.1. Academic Contributions

- A new materials is introduced to improve the performance properties of the cold mix and stabilized asphalt mixes with a high impact comparable to widely used additive such as cement.
- A new design approached is introduced to be evaluated for further research topics. This approached should be more performance based that the design criterions already existed for the stabilized or cold mixes.
- This research could be the initial base for further analysis on improve the design methods for cold mix asphalt and consideration of viscoelastic properties for the mixes.

7.2.2. Industry Contributions

- A possible economic and environmental friendly replacement conditional for further analysis for the widely used additive in industry for a higher performance and more acceptable methods for the industry is introduced.

7.3. Research Limitations

- One limitation of this research is the lack of real-time data to evaluate the behaviour of the mix designs under actual traffic loads. This absence of field data makes it challenging to fully verify and validate the obtained results in real-world conditions. Laboratory testing and simulations provide valuable insights into the performance of the materials, but they may not fully capture the complex interactions and variations that occur in the field.
- This research focused on a specific set of materials, including one type of aggregate and gradation, one type of RAP, and two types of asphaltenes. By adopting a more inclusive approach, the research outcomes can be better applied to a wider range of scenarios and contribute to a more robust understanding of asphalt emulsion-stabilized base layers' performance and applicability.

7.4. Future Directions

For future research in this area, the following directions are suggested:

- To generalize the results and draw comprehensive conclusions, it is essential to incorporate a more diverse range of materials in future studies. Including various aggregate types, gradations, and sources of RAP, as well as exploring a broader selection of asphaltenes, will help account for the inherent variability encountered in real-world road construction projects.
- Addressing the addition of asphaltenes to the mix and the compaction process is another critical challenge that future studies should focus on. The utilization of asphaltenes as an additive in asphalt emulsion-stabilized base layers may require adjustments to the cold recycling and base stabilization equipment. Comprehensive investigations in this area will help pave the way for smoother implementation of asphaltenes as additives in road construction practices.

- Design a pavement section with available guidelines such as MEPDG using the layer coefficients derived from the tests conducted on both granular and RAP base layers. This will enable a comprehensive evaluation of the behaviour and performance of these layers, taking into account their specific characteristics and properties.
- Implement a test road that incorporates the asphaltene-modified granular base layer. This test road will serve as a practical application to verify the accuracy and reliability of the test outcomes obtained from laboratory testing. Monitoring the performance of this test road over an extended period will provide valuable insights into the long-term behaviour and durability of the asphaltene-modified granular base layer.
- Similarly, construct a test road that incorporates the asphaltene-modified recycled RAP layer. This test road will serve as a validation platform for the laboratory test outcomes, specifically related to the performance and behaviour of the asphaltene-modified recycled RAP layer. Monitoring and assessing the performance of this test road will provide real-world data on the effectiveness and durability of this modified layer.
- Conduct continuous monitoring of these test roads to validate the laboratory findings. Continuous monitoring allows for the collection of data on the performance, distresses, and overall condition of the asphaltene-modified granular base layer and asphaltene-modified recycled RAP layer under real traffic loads and environmental conditions throughout a long range of time. This data can be used to assess the long-term effectiveness, sustainability, and structural integrity of these modified layers.

By pursuing these future research directions, there will be a greater understanding of the performance and behaviour of asphaltene-modified granular and recycled RAP layers, allowing for more accurate design guidelines and improved decision-making in road construction practices.

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Appendix

Performance Evaluation of High Modulus Asphalt Concrete (HMAC) prepared using Asphaltenes-Modified Binders

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Abstract

High strength, extended fatigue life, and improvement in rutting resistance are the main advantages of using high-modulus asphalt concrete as a base course material in the pavement structure. The primary goal of this paper was to investigate the performance properties of the high modulus base course using different asphaltenes-modified binders. A crude oil binder and two different asphalt binders from Alberta oil sands sources were used to prepare the mixtures. To prepare hard-grade asphalt binders, all binders were modified using asphaltenes, a waste by-product of the deasphalting of Alberta oil sands. The performance grades of the modified and unmodified binders were determined, and a mix design was developed for the high modulus asphalt concrete mixes. To evaluate the performance properties of high modulus asphalt concrete mixes composed of unmodified and asphaltenes-modified binders, Hamburg wheel tracking, dynamic modulus and flow number, and indirect tensile strength at low temperature were conducted. The high-temperature performance test results, including Hamburg wheel tracking and flow number tests, indicated that asphaltenes-modified mixtures show higher resistance to permanent deformation. Meanwhile, the indirect tensile strength test results at low temperature showed higher tensile strength and lower fracture energy for the asphaltenes-modified mixtures compared to the unmodified samples. Moreover, according to

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the dynamic modulus test results, the asphaltene-modified mixtures exhibited higher modulus values (stiffness) than the unmodified samples at different loading frequencies compared to the unmodified samples.

Keywords

High-modulus asphalt concrete, Modified asphalt binder, Asphaltene, Dynamic shear rheometer, Rutting, Indirect tensile strength, Dynamic modulus, and flow number

1.0 Introduction

High-modulus asphalt concrete (HMAC) contains hard grade binder and first used in France in the 1960s to increase the rutting resistance of pavement. This technology was originally developed by the Laboratoire Central des Ponts et Chaussées (LCPC) known as French Road Institute in France and was not applied on reinforcement and rehabilitation projects without excavation restrictions until the 1980s^{1, 2}. HMAC's comparably low permanent deformation and moisture susceptibility and superior fatigue resistance have made it a valuable asphalt mix for the pavement industry, as noted in previous studies²⁻⁶. HMAC has been widely used as a base layer, often topped with a thin asphalt concrete (AC) layer as a wearing course. The reason HMAC is typically topped with a thin wearing course is that HMAC contains a hard grade binder with low air voids, resulting in a low surface texture and less thermal protection². HMAC also has a longer design life compared to the conventional asphalt mixtures, as reported by various researchers^{7, 8}. Moreover, preparing the base layer with HMAC can reduce the thickness of the asphalt layer by 20% to 25%, thereby reducing the need for asphalt binder and natural aggregates accordingly^{9, 10}.

HMAC mixes were produced with binders from different sources, including straight-run hard grade binders and binders modified with natural asphalt or polyolefin¹¹. These modified binders are referred to as high-modulus asphalt binders (HMAB). Due to the method's origin being France with its mild temperate climate, low-temperature properties were not a key consideration in the original design^{1, 12}. Corte¹² reported that a trial section of HMAC featuring a binder with initial characteristics of 5/10 mm penetration and a softening point of 88 °C (as determined using a ring and ball test) exhibited a Young modulus of 21,600 MPa at 15 °C.

Observing the pavement section after one winter with minimum temperatures typically ranging from $-10\text{ }^{\circ}\text{C}$ to $-13\text{ }^{\circ}\text{C}$, the HMAC layer exhibited cracking. Other studies have shown that the likelihood of critical failure at low temperatures increases in high-modulus asphalt mixture compared to asphalt mixtures with conventional binders^{13, 14}. Judycki et al.¹³ compared the performance of pavements constructed by typical HMAC base roads with conventional asphalt concrete mixes following the first trial applications of HMAC mixes in Poland in 2001. They conducted a field investigation in one of Poland's coldest regions, employing a falling weight deflectometer (FWD) test, and several instances of low-temperature cracking were observed. Their study revealed that the load transfer efficiency was lower in HMAC than in conventional asphalt mixtures. Vervaecke & Vanelstraete¹⁴ studied the low-temperature properties of HMAB and mixtures separately, employing a bending beam rheometer (BBR) for the asphalt binders and a restrained cooling test (RCT) for the asphalt mixtures. Looking at correlations in the results for unaged, short-term aged, and recovered binders, it was concluded that the critical temperature of the recovery binders was higher than that of the short-term aged binders. The RCT, meanwhile, indicated that the failure temperature of the HMAC was $8\text{ }^{\circ}\text{C}$ higher than that of normal-density asphalt.

Some studies have proposed using various additives, such as polyolefin and other polymer modifiers, to overcome this problem¹⁵⁻¹⁸. In this regard, polymer modification has some drawbacks, though, such as high cost (mainly when used in the base layer) and poor storability¹⁹⁻²³. Meanwhile, using natural asphalt cement is not always practical since it is a highly viscous material and cannot be used as a direct binder^{9, 24}. Moreover, HMAC must meet the given performance requirements due to the performance-based nature of this design method^{10, 25}. In this regard, dynamic modulus, workability of the mixture, resistance to permanent deformation, and fatigue resistance are the essential properties to be considered at the mix design stage¹⁰.

Alberta oil sands facilities produce a considerable amount of asphaltenes. The bitumen processed in these refineries is composed of approximately 17.5% asphaltenes, and this material has no significant use in the pavement industry²⁶. Asphaltenes is a by-product of deasphalting oil sands and is generated at a high rate. Given its ubiquity and the lack of practical applications in the current practice, this material could pose problems for the oil

recovery process through precipitation, flocculation, and deposition in reservoir pores, wellbores, or pipelines. Dissociation of asphaltenes prior to any such occurrences is thus critical^{27,28}. Asphaltenes is characterized as homogenized solid in crude oil, n-alkane insoluble, highly polar, and heteroatomic. Determining the composition of the source bitumen, particularly the proportions of saturates, aromatics, resins, and asphaltenes these components often being referred to collectively as "SARA" is essential to identify the quantity and separate these components and find a practical and environmentally friendly solution to this material is essential^{27,29}. Bitumen is also often characterized in terms of polar and non-polar components. Asphaltenes, as one of the polar fractions of bitumen, is responsible for its elastic behaviour, while non-polar components such as maltenes govern its viscous behaviour^{19, 28, 30-32}. Based on this, it is expected that the addition of asphaltenes in the asphalt binder will stiffen the binder, thereby enhancing the mechanical properties and decreasing creep^{30, 33, 34}. Improving the binder properties is common practice to enhance the mechanical properties of asphalt mixes at high and low temperatures^{35,36}. Studies have also observed lower temperature susceptibility with the addition of asphaltenes, resulting in better thermal stability^{37, 38}.

2.0 Research Objectives and Scope

Existing HMAC asphalt design methods, except for the Enrobés à Module Elevé (EME), though subject to limitations, provide reasonable estimations for preparing an efficient mix. These methods mainly use the modulus as the design criterion. However, the low- and high-temperature properties such as fatigue and rutting of these mixes are not considered in the design process, and this warrants further investigation, especially in the context of cold region applications. The main objective of the study described in this paper, then, was to evaluate the low- and high-temperature properties of an asphaltenes-modified HMAC base course layer and compare them to those of an unmodified asphalt binder. Asphaltenes sourced from Alberta oil sands bitumen was used to modify a straight-run binder as a control binder and two other binders from separate oil sands bitumen sources to evaluate the possibility of using these binders in asphalt pavement. Therefore, this research aims to incorporate the asphaltenes from oil sands in HMAB and HMAC design procedure. Indirect tensile strength at low temperature, flow number, and Hamburg wheel-tracking tests at high temperatures were conducted in order to evaluate the mechanical properties of the mixtures.

3.0 Materials and Methods

3.1 Aggregates

The minimum dynamic modulus required for the HMAC determined the minimum binder performance grade of the mixture. To assess this value for the binder performance, an aggregate gradation with a nominal maximum size (NMAS) of 19 mm was selected³⁹. The aggregates to prepare the mixtures for this study were obtained from a single source. The samples were prepared based on the gradation provided by Levia-Villacorta et al.³⁹, and the same volumetric parameters were used as the reference properties. The French envelope²⁵ used for the HMAC is provided (AC20-EME), along with the targeted gradation, in Figure 1, while reference volumetric properties are listed as 5.7% for binder grade of a PG 88-16 with design air voids of 1.5%. Voids of mineral aggregates are 15%, and voids filled with asphalt are 90%³⁹. The aggregate gradation skeleton consisted of 39.7% coarse aggregates, 52.4% fine aggregates, and 7.9% filler. The aggregates were tested to determine their physical properties, with the results presented in Table 1. The bulk specific gravity of the aggregates (G_{sb-agg}) was also determined and was found to be 2.547.

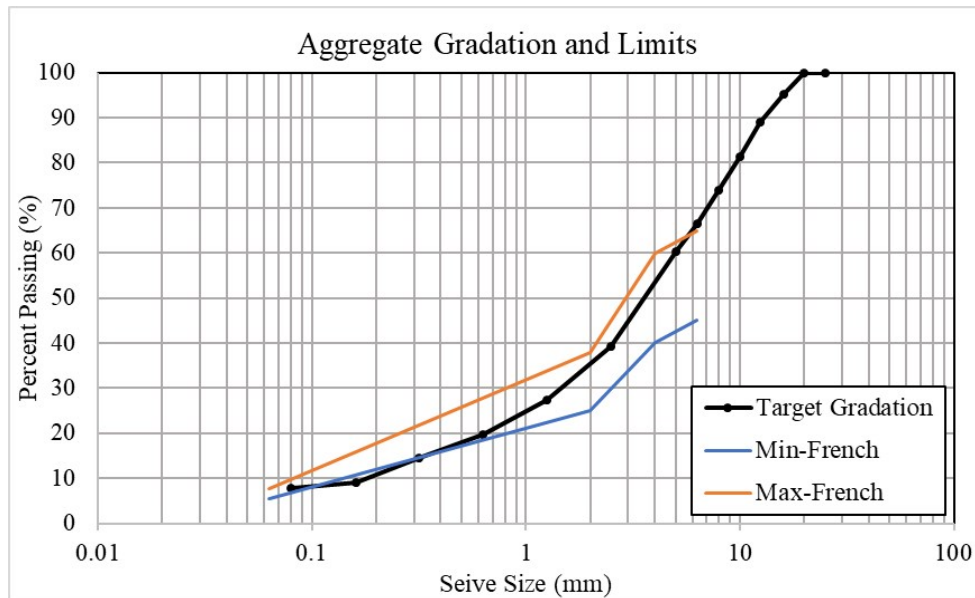


Figure 1: Target aggregate gradation and limitations

Table 1: Aggregate properties

	Property (unit)	Standard	Result
Fine aggregates	Specific gravity (G_{fa})	ASTM C128 ⁵⁵	2.618
	Absorption of water (%)		2.229
Coarse aggregates	Specific gravity (G_{ca})	ASTM C127 ⁵⁶	2.502
	Absorption of water (%)		0.428
	Abrasion of coarse aggregates (%)	ASTM C131 ⁵⁷	23.000

3.2 Asphalt Binder

An asphalt binder from a crude oil source with a performance grading (PG) of 70-22⁴⁰ was used as the base control binder in order to modify with asphaltenes. Two different unmodified asphalt binders from different sources of Alberta oil sands bitumen were also used (referred to as binders A and B) in this study. The bitumen, distilled at 460 °C to obtain these binders, originated from Alberta oil sands. Table 2 presents the properties of the control straight-run binder, which is PG 70-22, a common choice for surface layer applications.

Table 2: PG 70-22 control asphalt binder properties

Property (unit)	ASTM Standard	Specification		Value
		Min.	Max.	
Density @ 15°C, Kg/L	D70 ⁵⁸	---	---	1.0341
Penetration @ 25 °C (100 g, 5 s), dmm	D5 ⁵⁹	80	100	90
Flash Point (COC), °C	D92 ⁶⁰	230	---	276
Ductility @ 25 °C (5 cm/min), cm	D113 ⁶¹	100	---	150+
Solubility in trichloroethylene, %	D2042 ⁶²	99.5	---	99.9
Absolute viscosity @ 60 °C, Pa·s	D2171 ⁶³	150	---	183
Viscosity @ 135 °C, Pa·s	D4402 ⁶⁴	---	3.00	0.42
Mass loss, %	D1754 ⁶⁵	---	1.0	0.37

3.3 Asphaltenes

Solvent deasphalting was used to extract the asphaltenes from the Alberta oil sands bitumen. This material, it should be noted, is a macro-polar structure that forms within the oil matrices. The solvent deasphalting process triggers the precipitation of the asphaltenes using n-alkanes as an anti-solvent⁴¹. Figure 2 shows the asphaltenes obtained in solid form. They were

subsequently crushed into powder and passed through a No. 100 sieve for consistency and to achieve a higher specific surface area. It is also worth noting that the composition and properties of asphaltenes depend on the characteristics of the source oil and the method of processing used to extract the asphaltenes. In the present case, the chemical composition of the material was analyzed, and the SARA proportions of saturates, aromatics, resins, and asphaltenes were found to be 6.85%, 9.68%, 3.84%, and 79.63%, respectively.



Figure 2: Asphaltenes used for modification

3.4 Asphalt Binder Modification and Mix Design

Ghasemirad⁴² used a dynamic shear rheometer (DSR) and bending beam rheometer (BBR) to determine the performance grade of modified and unmodified binders with different concentrations of asphaltenes. In our study, different proportions of asphaltenes were added to the binders accordingly based on Ghasemirad's results and mixed with aggregates. Figure 3 shows the viscosity graphs as well as the mixing and compaction temperatures of the modified and unmodified binders. AASHTO T315⁴³ specification was followed to characterize the viscoelastic behaviour of all the modified and unmodified binders at high and intermediate temperatures, while a BBR was used to ascertain the low-temperature properties in accordance with AASHTO T313⁴⁴. Asphaltenes were mixed in the binder with a high shear mixer, and in order to avoid aging during the mixing process, the temperature was maintained at $140\pm 5^{\circ}\text{C}$. The samples with modified binders were mixed at a speed of 2,000 rpm for 60 minutes to ensure a uniform mixture based on the experience to properly disperse the asphaltenes in the

binder. The performance grading of the modified and unmodified binders is presented in Table 3.

Table 3: Performance grading results for modified and unmodified binders

Binder	Asphaltenes Content (%)	True High Temperature PG ¹ (°C)		Int. PG (°C)	True Low Temperature PG (°C)	PG (°C)	Selection for Mixture Samples
		Unaged	RTFO-Aged				
PG 70-22 (M1)	0	70.5	71.1	19.2	-27.1	70-22	Yes (control)
PG 70-22 (M2)	12	82.9	84.6	29.0	-21.8	82-16	Yes
Oil sands A	0	70.1	69.0	17.7	-26.6	64-22	No
Oil sands A (M3)	12	82.2	86.1	30.4	-21.2	82-16	Yes
Oil sands B	0	83.8	82.6	33.5	-18.3	82-16	No
Oil sands B (M4)	6	88.2	88.5	37.1	-16.6	88-16	Yes

¹PG: Performance Grade

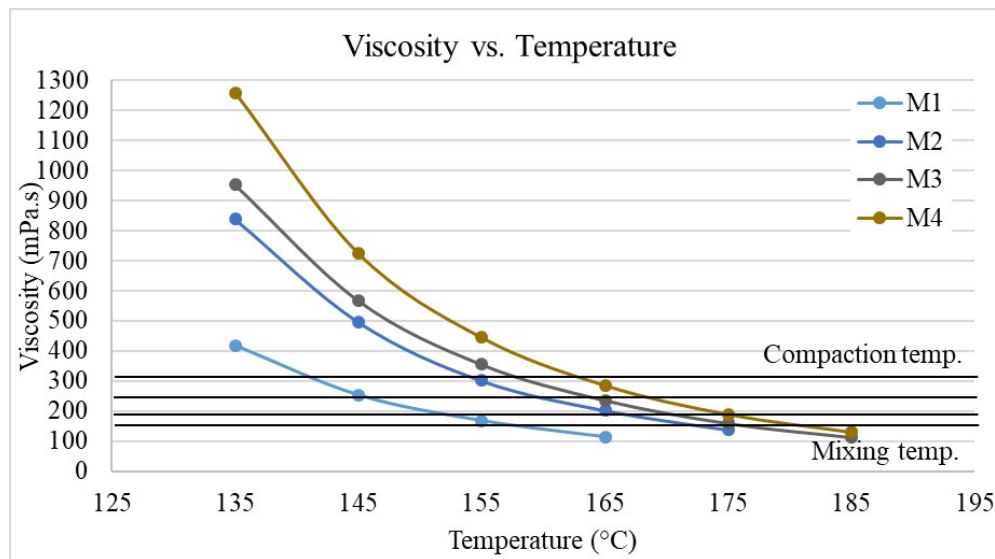


Figure 3: Viscosity-temperature ratio for binders

Applying the minimum dynamic modulus requirement for the HMAC asphalt based on the LPC bituminous mixtures design guide²⁵ yielded a dynamic modulus value of 14 GPa at a loading frequency of 10 Hz and a temperature of 15 °C, a result consistent with the values

reported in other studies on HMAC^{13, 39, 45, 46}. The targeted dynamic modulus of 13.79 GPa was calculated based on the binder with a PG grade of 82-16 in this study and, accordingly, PG 70-22 (M1) was used as the base binder, along with PG 70-22 + 12% asphaltenes (M2), Oil sand A + 12% asphaltenes (M3), and Oil sands B + 6% asphaltenes (M4), which all binders except M1 had PG grading higher than the calculated dynamic modulus⁴⁷.

4.0 Testing Program

4.1 Dynamic Modulus Test

The M1, M2, M3, and M4 binders used to mix with the selected gradation and dynamic modulus $|E^*|$ tests were applied to the samples in order to determine whether the stress and strain responses in the asphalt mix correlate with the time- and temperature-dependent properties of the mix in terms of field performance. Different loading frequencies and temperatures were applied using a universal testing machine (UTM) based on the AASHTO T378⁴⁸ standard. Figure 4 shows the UTM setup for the dynamic modulus test with Linear Variable Differential Transformers (LVDTs) attached and ready to test, as well as the samples prepared, cut, and cored for testing. The samples were prepared in a Superpave gyratory compactor after being cored and cut into standard cylindrical specimens of 100 mm diameter and 150 mm height. Sinusoidal axial compressive stress at loading frequencies of 0.01 Hz, 0.1 Hz, 1 Hz, and 10 Hz was applied to the specimens at temperatures of 4 °C, 10 °C, 20 °C, and 45 °C following the conditioning times suggested in the standard (AASHTO T342⁴⁹) for each temperature. These temperatures and frequencies were selected based on the AASHTO R84-17⁵⁰ with an extra temperature of 10°C. The applied stresses and corresponding strain responses of the specimens were measured continuously during testing using a data acquisition system, while the dynamic modulus values were calculated by dividing the stress magnitude by the average strain magnitude. Two replicates for each mixture were prepared and tested.



Figure 4: Cored and cut sample (A) dynamic modulus test setup (B)

4.2 Indirect Tensile Strength Test at Low Temperatures

The low-temperature properties of the mixtures prepared with the unmodified and modified samples were tested per AASHTO T322-07⁵¹ using an indirect tensile strength (ITS) test setup and a UTM. Marshall samples with three replicates for each of the mixtures (i.e., the control and the different asphaltenes-modified mixtures) were prepared. The samples were surface cut to heights in the range of 38 mm to 50 mm (Figure 5B). Test temperatures of 0 °C and -10 °C were selected because the specimens were designed for use in the base layer and consideration of the PG grading of the binders. An air chamber was used to condition the samples for 3±1 hrs prior to testing. According to the relevant standard, a fixed static load was applied on the specimens for 100±2 s, and LVDTs were used to record the deformation of the samples along both the horizontal and vertical axes (Figure 5A). ITS testing was carried out following completion of the creep test using a load with a rate of 12.5 mm/minute until the failure point of the samples had been reached. Figure 5C shows the samples after testing. Load-deformation values were used to calculate the fracture energy (FE) and the ITS of the samples at both temperatures. FE was calculated based on the ASTM D8225-19⁵² as an area under the load-deformation graph divided by sample cross section.

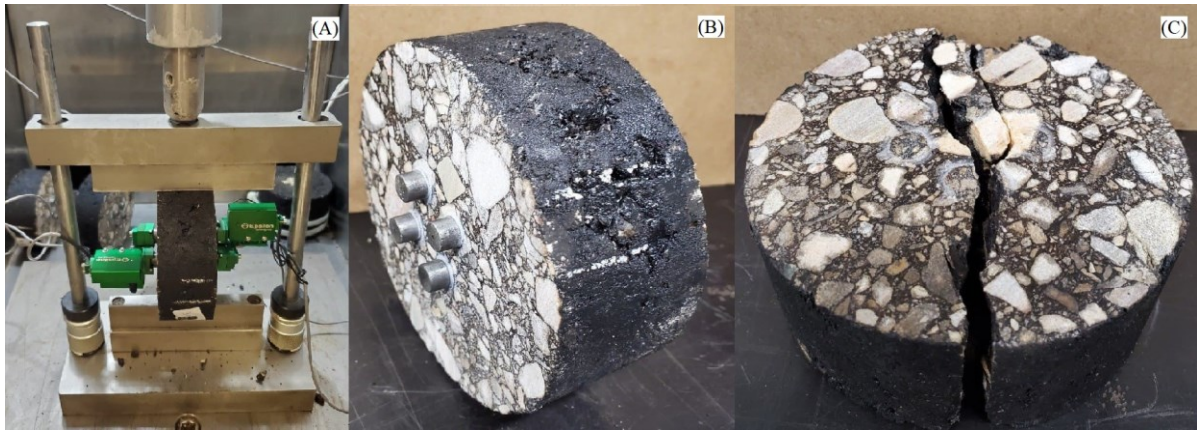


Figure 5: (A) IDT test setup, (B) sample prior to testing, and (C) sample after testing

4.3 Flow Number Test

The flow number test was conducted at a temperature of 60 °C using the test setup specified in AASHTO T378⁴⁸. Temperatures for the flow number test were selected based on climate data for the assumed project location (Edmonton, Canada) and in consultation with the long-term pavement performance (LTPP) program. The contact stress and deviator stress for the testing were 3 kPa and 69 kPa, respectively. The loading pattern for the test was a haversine axial compressive pattern with a loading period of 0.1 s and a resting time of 0.9. Finally, the test termination criterion was set as 20,000 cycles or a microstrain of 50,000, whichever was reached first.

4.4 Hamburg Wheel-Tracking Test

The high-temperature performance of the samples was assessed using a Hamburg wheel-tracking test per AASHTO T324-19⁵³. One set of Superpave gyratory compacted samples with a diameter of 150 mm and a height of 60 mm were used for this test. The samples were saw cut along the edge and placed in high-density polyethylene molds (Figure 6A). Considering the binder grade and comparing the results to conventional asphalt concrete, the test temperature was set to 55 °C⁵⁴. The samples were conditioned in water for 45 minutes prior to testing. For the test, a steel wheel with a width of 47 mm and weighing 705±4.5 N was applied at a frequency of 52±2 passes per minute and a maximum speed of 0.305 m/s at the midpoint. Testing continued until reaching a rutting depth of 12 mm or until 20,000 passes, whichever was reached first. Rutting resistance index (RRI) and stripping inflection point (SIP) were obtained using the rutting depth values and the number of passes to evaluate the rutting

potential and susceptibility of the mixtures to moisture damage. Figure 6B shows the samples after testing. Using the maximum number of passes for each sample, permanent deformation under repeated wheel tracking load cycles was calculated in terms of RRI, which was the number of passes multiplied by subtracting the rutting depth from the 1-inch value.

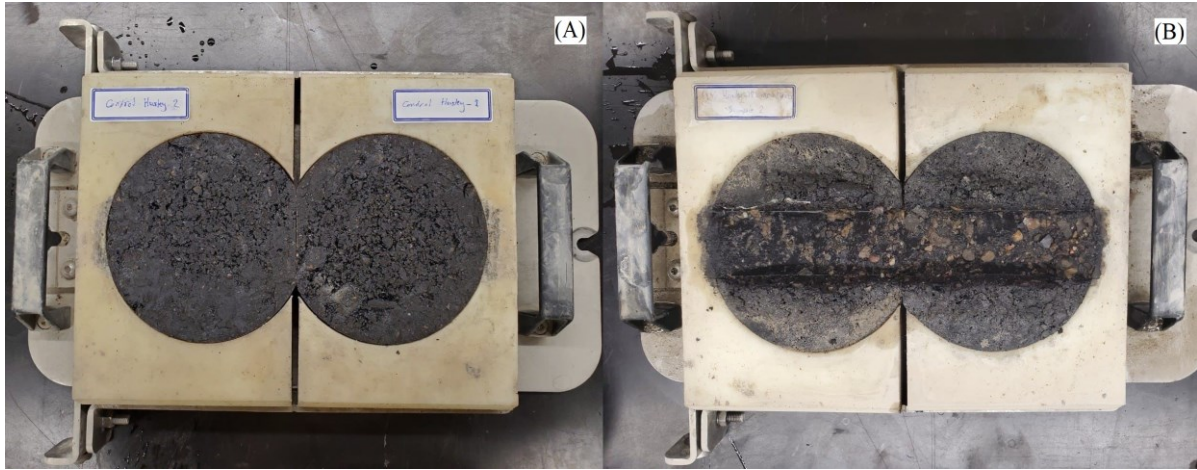


Figure 6: Hamburg wheel tracking samples before (A) and after (B) testing

5.0 Results

5.1 Dynamic Modulus Test Results

The dynamic modulus test results show high modulus values for all modified samples under high loading frequencies, meaning that, as the frequency increases, the dynamic modulus, and therefore the viscoelasticity and stiffness of the asphalt mixtures, also increases (Figure 7). Meanwhile, increasing the temperature significantly decreased the dynamic modulus values, as reflected in the results. M1, as the control sample, was found to have a modulus of 8,505.5 MPa, which is lower than the threshold specified by Delorme et al.²⁵ at a temperature of 15 °C and a loading rate of 10 Hz (14,000 MPa). Meanwhile, the modified samples achieved the minimum requirement for a high-modulus asphalt application of 14,000 MPa at 15 °C temperature and 10 Hz loading frequency as set out in the European standard. Mixture M3 showed a similar result to M2, with only negligible improvement. However, M4 showed a significant improvement of 119% in dynamic modulus at a temperature of 15 °C and a frequency of 10 Hz. Analysis of the phase angle data shows that the samples became more viscous and less elastic when increasing the temperature and decreasing the frequency. Figure

8 presents the phase angle results for the modified and unmodified samples at 10 Hz frequency, which shows that binder modification also decreased the phase angle. These values decreased for all the modified materials at each temperature, suggesting that asphaltenes modification improved the elastic behaviour of the asphalt binder under loading. M1 as the control sample had the highest phase angle at all temperatures, while M4 had the lowest phase angle among the specimens under investigation.

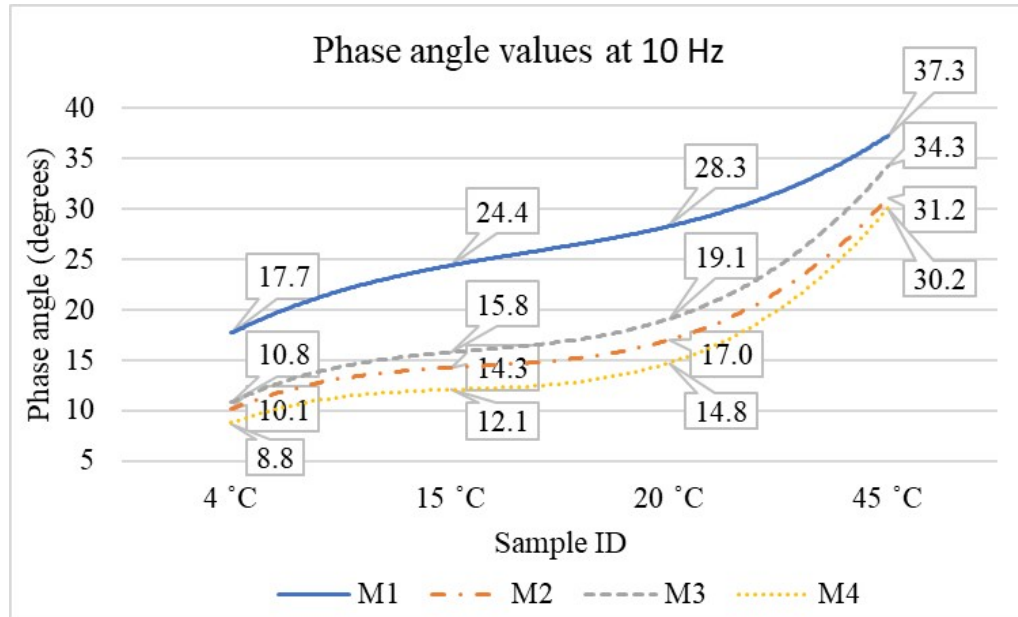


Figure 7: Dynamic modulus master curve for selected mixture at a reference temperature of 15 °C

Figure 7 shows the dynamic modulus master curves for the mixtures with different binders. It can be seen that higher dynamic modulus values were observed for the higher frequencies. At the same time, the modified binders were found to exhibit higher modulus (stiffness) values along with the different frequency ranges compared to the control (i.e., M1). As with the other tests, M4 showed the highest values among the examined specimens.

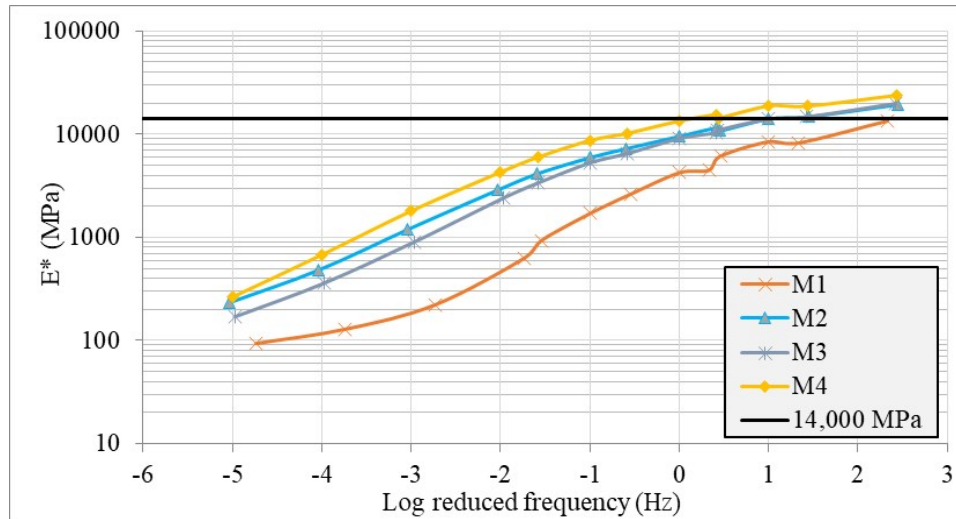


Figure 8: Phase angle values in 10 Hz frequency

5.2 Indirect Tensile Strength Test Results at Low Temperatures

The indirect tensile test (IDT) was conducted based on AASHTO T322⁵¹ for the modified and unmodified samples. Figure 9 and Figure 10 present the load-deformation results for the temperatures of 0 °C and -10 °C, respectively. The ITS and FE values for the tested sample were calculated using the extracted data, with the results for both temperatures presented in Table 4. As can be seen, the ITS results at 0 °C show higher values for all of the modified samples in comparison to M1 (i.e., the control sample). The FE values of the modified sample were lower than the control sample, by margins of 19.8%, 21.5%, and 51.9% for M2, M3, and M4, respectively. Increasing the tensile strength and decreasing the FE values at 0°C show that the modified mixes are stronger but are more prone to cracking, which could be due to the higher stiffness of the samples. Rapid crack propagation was expected for the modified samples, considering the steep slope of the modified samples after reaching the peak point compared to the control sample. M4 was found to have the highest stiffness among the mixtures, with the steepest slope and highest crack propagation.

Similarly, Table 4 shows that the modified samples have slightly higher ITS values and slightly lower FE values at -10 °C as compared to the control sample. These values are slightly higher for ITS and lower for FE by comparing -10 °C to 0 °C. The results also show that asphaltene modification of the binders increased the strength and crack propagation of the mixtures. The increases in tensile strength were found to be 29.9%, 25.1%, and 26.8% for M2, M3, and M4,

respectively. FE decreased by 32.4%, 37.2%, and 38.7% for M2, M3, and M4, respectively, because of asphaltenes modification.

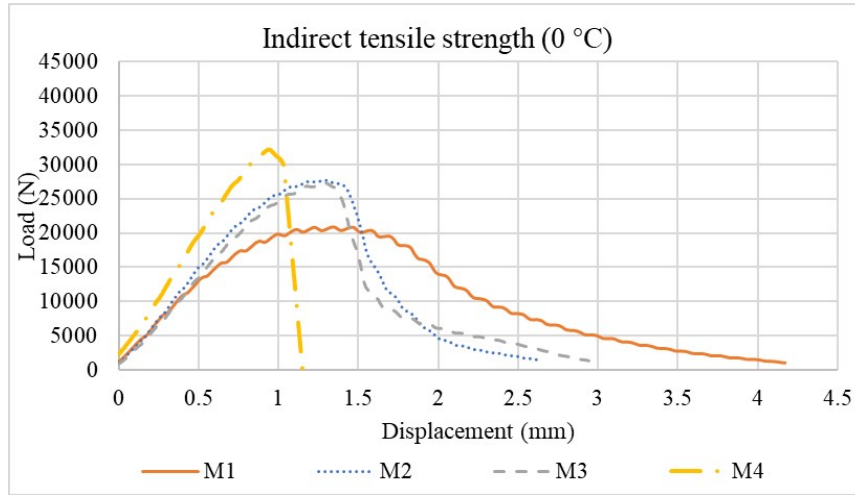


Figure 9: Load-deformation results at 0 °C

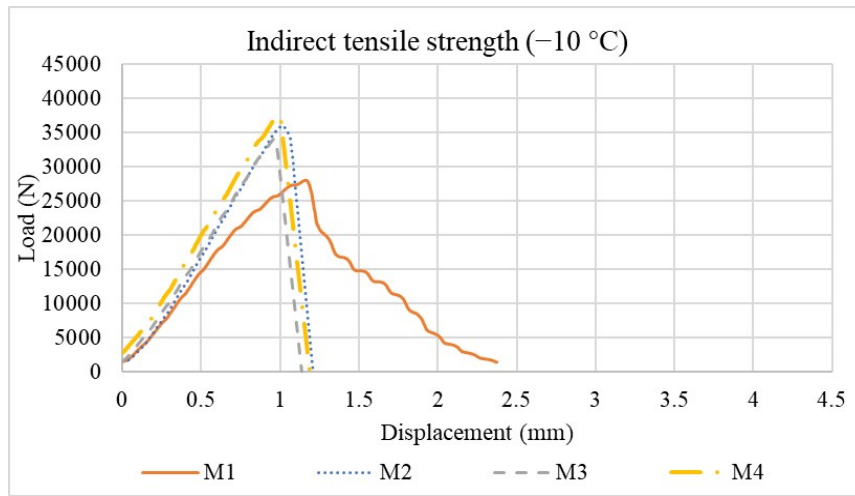


Figure 10: Load-deformation results at -10 °C

Table 4: Performance grading results for modified and unmodified binders

Sample ID	Temperature (°C)	Tensile Strength (kPa)	Fracture Energy (J/m ²)	Air voids (%)	VMA (%)	VFA (%)
M1	0	2,921.34	11,054.33	1.90	11.27	83.21
M2		3,655.74	8,860.68	2.08	11.43	81.86
M3		3,666.63	8,673.33	2.25	11.59	80.67
M4		4,369.38	5,320.94	2.62	11.93	78.01
M1	-10	3,853.02	8,052.17	1.76	11.15	84.22
M2		5,003.67	5,445.90	2.00	11.36	82.41

M3	4,820.76	5,055.25	2.02	11.38	82.23
M4	4,884.43	4,933.47	1.84	11.22	83.59

VMA: Voids in mineral aggregates; VFA: Voids filled with asphalt.

5.3 Flow Number Test Results

Figure 11 presents the results for the flow number test carried out at 60 °C per AASHTO T378⁴⁸. All the tests were terminated at the maximum number of cycles because the maximum strain level of 50,000 microstrains was not reached after 20,000 cycles. Considering the accumulated microstrain value for the samples, M4 had the lowest deformation, and therefore the highest rutting resistance, at 1,704.49 $\mu\epsilon$. M1, as the control mixture, had the highest microstrain, 346.6% higher than M4, while M2 was found to be 50.4% higher and M3 81.2% higher in comparison to M4. Meanwhile, the flow point was calculated using the Francken model, and the fitted flow point was not reached for the samples at 20,000 cycles. This outcome is because none of the samples reached the tertiary stage of the deformation. The accumulated microstrain values for M1, M2, and M3 were found to be 7,612.04 $\mu\epsilon$, 2,562.88 $\mu\epsilon$, and 3,088.96 $\mu\epsilon$, respectively.

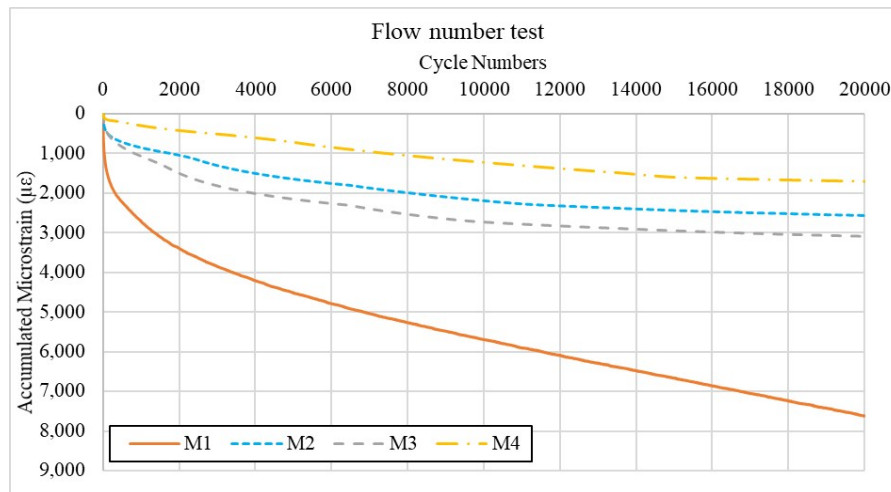


Figure 11: Flow number test results

5.4 Hamburg Wheel-Tracking Test Results

The samples' rutting performance and moisture susceptibility were tested using a Hamburg wheel-tracking test⁵³. Table 5 presents the test results, which show that all the samples, except for M1, did not reach the maximum allowable rutting depth of 12 mm after 20,000 wheel

passes. According to the results, the rut depth would remain less than 4 mm for the mixes with asphaltenes modified binder. Accordingly, the SIP was calculated to be 15,376 for the control sample and 19,999 for all the modified mixtures (Figure 12). The RRIs were higher for the modified samples than for M1, which was 7,803. The RRIs of the modified samples, calculated using the number of passes and the depth of rutting, were 18,566.9 (M2), 17,236.2 (M3), and 19,622 (M4). The moisture sensitivity index for the samples, calculated by dividing the SIP values by the number of passes, increased for the modified mixtures to 1.00, meaning that all the modified samples were found to be resistant to moisture damage. The corresponding index for the control was approximately 0.70. Based on the results of this test and the flow number test, it can be concluded that asphaltenes modification significantly increased the permanent deformation resistance in the samples.

Table 5: Performance grading results for modified and unmodified binders

Sample ID	SIP	Number of passes	Rutting depth (mm)	RRI	Moisture sensitivity index
M1	10,692	15,376	12.00	7,803.0	0.70
M2	19,999	20,000	1.82	18,566.9	1.00
M3	19,999	20,000	3.51	17,236.2	1.00
M4	19,999	20,000	0.48	19,622.0	1.00

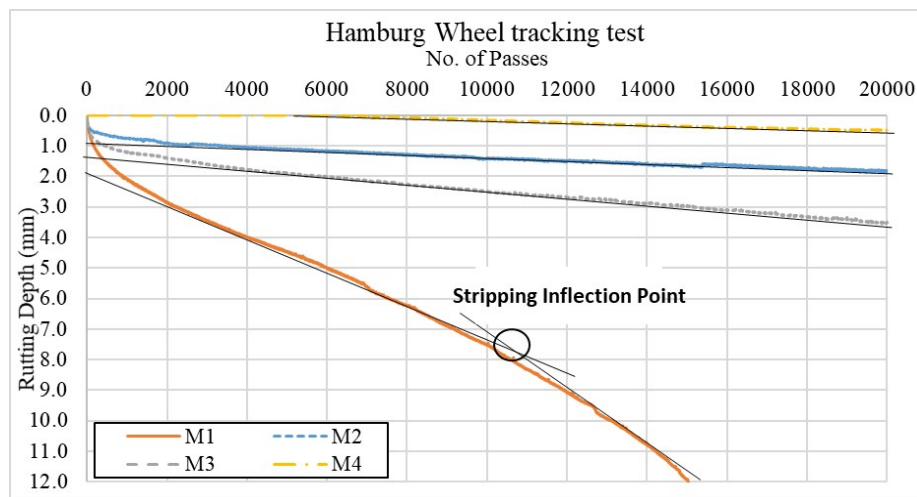


Figure 12: Hamburg wheel-tracking test results

6.0 Discussion

A summary of the results from the performance tests is presented in Figure 13. The tensile strength of the samples at both 0 °C and -10 °C was found to increase due to asphaltene modification. This value at 0 °C increased by 25.1%, 25.5%, and 49.6% for M2, M3, and M4, respectively, and at -10 °C increased by 29.9%, 25.1%, and 26.8%, respectively. Comparing the results for M1 and M2, which have a similar base binder, indicates that asphaltene modification increases the tensile strength. In contrast to the ITS results, the FE values decreased for the modified mixtures compared to M1, indicating that the modified samples exhibit increased crack propagation and brittleness. FE decreased about 19.8%, 21.5%, and 51.9% at 0 °C and 32.4%, 37.2%, and 38.7% at -10 °C for M2, M3, and M4, respectively. As a result, it could be observed that although asphaltene modification increases the tensile strength of the mixtures, this comes at the cost of decreased resistance to the crack propagation. This result indicates the effect of increased asphaltene resulting in higher stiffness of the samples compared to the unmodified binders, which subsequently will help with the resistance of the sample to higher loading. However, the cracking propagation is faster once it fails.

The Hamburg wheel tracking test assisted with calculating the RRI values, with the modified samples showing significant improvements in RRI compared to the base binder (M1)—a 137.9% improvement for M2, 120.9% for M3, and 151.5% for M4. Similar to the low-temperature properties, higher asphaltene content helps with the stiffness of the binder and consequently mixtures. Asphaltene modification also significantly improved high-temperature properties, as confirmed by the flow number test. All the mixtures reached 20,000 cycles in the flow number test, and the accumulated microstrain of each sample under the compressive deformation showed rutting resistance. This value was the highest for M1, as expected, and it decreased for M2, M3, and M4 by 66.3%, 59.4%, and 77.6%, respectively, meaning that the modified mixtures were found to be more resistant to rutting in comparison to the control. A comparison of the moisture sensitivity index shows that modification of the binders with asphaltene decreased the susceptibility of the mixture to moisture damage by approximately 42.9% for all mixtures.

Meanwhile, the dynamic modulus test results show the corresponding changes in mixture properties at different temperatures. Figure 13 presents the dynamic modulus values of the mixtures within the parameters of the LPC bituminous mixtures design guide²⁵ at a frequency

of 10 Hz and a temperature of 15 °C. As explained above, all the samples satisfied this threshold except M1, which reached only 8.5 GPa due to the low value of high PG for this asphalt binder. Comparing M2, M3, and M4 to the control mixture (M1) shows respective increases of 64.8%, 64.7%, and 119.2%.

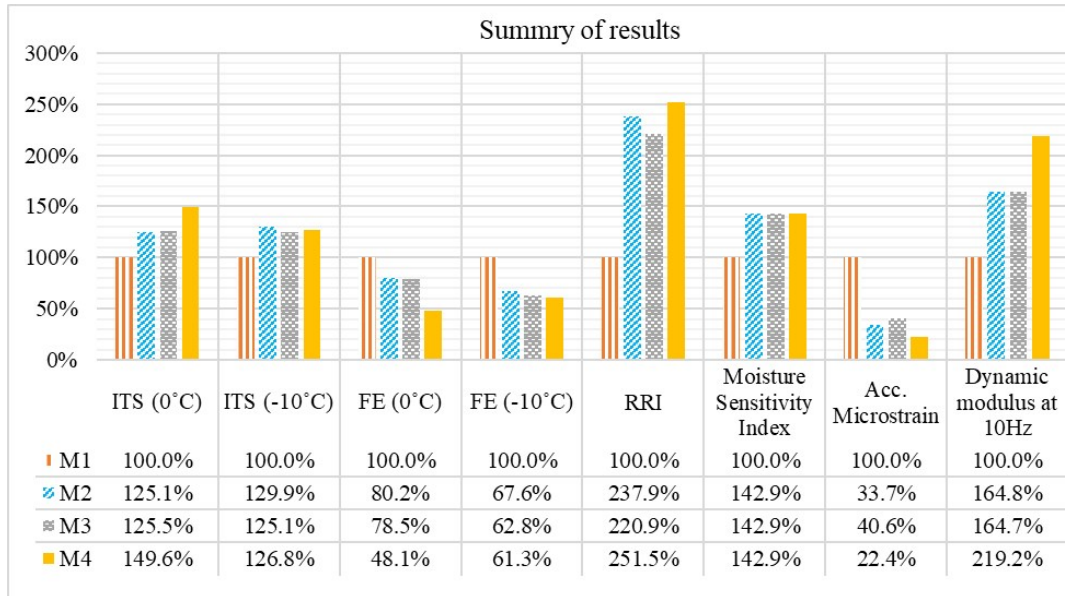


Figure 13: Summary of the performance test results

7.0 Conclusions

This study investigates the use of asphaltenes to develop high-modulus asphalt concrete (HMAC) for cold climatic regions. To achieve this objective, mechanical testing was used to evaluate the performance of the developed HMAC mixtures at both high and low temperatures. According to the mixture performance test results, the following conclusions can be drawn:

- According to the performance results, it could be concluded that both straight-run binder and oil sands binders modified with asphaltenes had enhanced high-temperature performance properties compared to the unmodified straight-run binder.
- The asphaltenes-modified samples could satisfy the stiffness criterion of HMAC mixtures which is 14 GPa at 15 °C and a frequency of 10 Hz. However, the unmodified sample failed to satisfy this requirement.

- The asphaltenes modification makes the samples more elastic based on the phase angle results.
- Based on the IDT test results, modification of the binders with asphaltenes results in a more brittle fracture, making the mixture more prone to low-temperature cracking than the unmodified binders.
- The moisture sensitivity analysis indicated that the modification of samples with asphaltenes would improve the moisture resistance of the mixtures, confirming the SIP results.
- The Hamburg wheel tracking test result indicated improved rutting resistance of the mixtures at higher temperatures due to the asphaltenes modification.
- The flow number test results confirmed the improvement in the rutting performance because of asphalt modification.

Future research will investigate the effect of using fibers to enhance the cracking properties of designed mixes.

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