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Thesis Report

Title: Effect of Lignin Content on the Performance Grade and Stability of Asphalt Binders used in Thunder Bay

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Abstract

The design roads relies solely on bitumen as a binding agent because it provides superior adhesive properties and waterproofing capabilities. The main source of bitumen production from crude oil oil refining results in expensive production costs and supply chain problems and reduced refining capabilities. The worldwide transition toward sustainable infrastructure solutions and environmentally responsible construction materials has become more urgent because of these challenges. The paper industry produces lignin as a renewable biopolymer which shows promise as a bitumen extender because of its structural compatibility and sustainability advantages. The research evaluates the addition of kraft lignin at 10%, 20%, and 30% levels in asphalt mixtures to determine their performance against Ontario's Ministry of Transportation (MTO) Performance Grade (PG) standards for cold climate applications in Thunder Bay. The primary objective of this study is to determine whether lignin-modified asphalt (LMA) meets structural and performance standards while providing a sustainable alternative to traditional binders. The primary objective is to determine whether lignin-modified asphalt (LMA) can meet structural and performance standards while providing a sustainable alternative to traditional binders. The primary objective is to determine whether lignin-modified asphalt (LMA) meets structural and performance requirements while serving as a sustainable alternative to conventional binders. A hybrid methodology was adopted, involving both practical and theoretical approaches. The experimental component consisted of mix design tests using PG 52-34 bitumen and kraft lignin, including Marshall Stability and Superpave Gyratory Compactor methods. These tests examined workability, compaction, and strength parameters. For the theoretical analysis, secondary data from FPInnovations was used to evaluate rheological properties, oxidative aging resistance, and PG classification shifts. Results indicated that a 20% lignin substitution level provided the most balanced performance, achieving optimal Marshall stability, acceptable air voids, and good compaction. Theoretical data supported improvements in high-temperature PG ratings (e.g., PG 58-28) and enhanced resistance to rutting and oxidative aging at moderate levels of substitution. This research will provide valuable insights into the mechanical viability and environmental potential of LMA. The findings support the use of Lignin as a partial bitumen substitute and offer a foundation for future research and performance-based implementation strategies in cold-region pavement applications.

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List of Abbreviations

A.A.S.H.T.O.	American Association of State Highway and Transportation Officials
AC.	Asphalt Cement i.e. Bitumen
H.M.A.	Hot Mix Asphalt
PG.	Performance Grade
PVN	Penetration Viscosity Number
VTS	Viscosity Temperature Susceptibility
HMA	Hot mix asphalt
WMA	Warm Mix Asphalt
CMA	Cold Mix Asphalt
SHRP	Strategic Highway Research Program
VTS	Viscosity Temperature Susceptibility
PMB	Polymer-modified Binder
SHRP	Strategic Highway Research Program
NCHRP	National Cooperative Highway Research Program
PCC	Portland Cement Concrete
PG	Performance Grade

PVN	Penetration Viscosity Number
RHD	Roads and Highway Division
RPM	Revolutions Per Minute
SHRP	Strategic Highway Research Program
VTS	Viscosity Temperature Susceptibility

Chapter 1: Introduction

The durability and performance of pavement materials are critical to the long-term functionality of transportation infrastructure. Pavements play a crucial role in industrial development and global economic growth. The essential role of pavements as communication and mobility corridors enables the efficient movement of goods and people between different points. The basic knowledge of pavement construction techniques and sustainable design development remains crucial for contemporary development. The basic structure of asphalt pavements consists of bitumen and aggregates, with bitumen serving as the primary adhesive component. The production of bitumen relies on refined crude oil which is a non-renewable fossil fuel that generates economic and environmental problems because of decreasing global resources This research investigates the possibility of using lignin as a renewable biopolymer byproduct from paper and bio-refining operations to replace part of bitumen. Understanding the properties of lignin and how it influences asphalt performance may help promote more secure, eco-friendly, and sustainable pavement systems.

1.1 Background

Pavements are integral to modern infrastructure, enabling transportation, commerce, and economic growth [1]. These engineered surfaces typically consist of asphalt, a composite material formed by blending aggregates with bitumen, a petroleum-derived binder known for its adhesive and waterproofing properties [2]. Flexible pavements have multiple layers that distribute loads to the subgrade, reducing deformation and extending service life [3]. The pavement industry faces increasing sustainability challenges regarding petroleum-based materials, despite the performance benefits of bitumen.

The worldwide decrease in crude oil supply poses a threat to bitumen production, as it leads to higher expenses, inconsistent product quality, and additional environmental issues associated with the use of fossil fuels. The current challenges underscore the need for developing sustainable and renewable alternatives to preserve or enhance pavement performance [4].

Lignin, a natural polymer and byproduct of the pulp and paper industry, has emerged as a potential partial substitute for bitumen [5]. Its complex aromatic structure bears chemical similarities to bitumen and has shown promise in improving binder stiffness, oxidative stability, and resistance to deformation [6]. Moreover, lignin is a renewable resource, locally available in Canada, and aligns with climate change mitigation goals by reducing dependence on fossil fuels [7]. This research examines the application of lignin-modified asphalt binders in cold climate regions, specifically by the performance grading standards mandated in Ontario [8]. By assessing lignin at 10%, 20%, and 30% substitution levels, this study focuses on creating sustainable pavement design and has the potential to contribute construction materials, which will help in a eco-friendly future. [9]

1.2 Problem Statement:

Asphalt pavements are a cornerstone of modern transportation infrastructure, traditionally constructed using bitumen as a binder due to its excellent adhesive and waterproofing properties [1][2]. However, bitumen is derived from crude oil, a non-renewable and increasingly scarce resource. The global decline in crude oil supply, combined with geopolitical and environmental pressures, has led to fluctuating costs, reduced refining capacity, and uncertainty in long-term bitumen availability [4][8]. These challenges are particularly critical in cold regions, such as Ontario, where asphalt must meet stringent Performance Grade (PG) standards to withstand extreme temperature variations [9].

The search for sustainable and renewable bitumen alternatives has gained prominence due to these issues. The pulp and paper industry byproduct lignin shows promise as a sustainable alternative to bitumen because it shares a bitumen-like structure and demonstrates potential to improve asphalt binder properties. [5][6]. While some international studies have explored the use of lignin in asphalt, there is limited empirical data on its mechanical performance and suitability within Canada's cold-climate pavement design standards [7].

This research is exploring possible solutions to that gap by evaluating the performance of asphalt mixtures modified with kraft lignin at substitution levels of 10%, 20%, and 30%. It aims to determine whether lignin-modified binders meet Ontario's PG requirements while offering a more sustainable, cost-effective solution for future road construction [7][9]. The study also provides foundational insights into how lignin affects blending behaviour, compaction, and mix design integrity, contributing to long-term material innovation in the pavement engineering field [3][6].

1.3 Objectives

The primary objectives of this research are to verify the conformance of the lignin-modified asphalt with the target specifications of Ontario's asphalt binder and to determine if this asphalt can be used with the Performance Grading System. To address the overarching goal of evaluating the potential of lignin as a partial substitute for bitumen in asphalt mixtures, this research sets out to achieve the following specific objectives:

- To perform a comprehensive literature review to establish a foundational understanding of pavement engineering principles, bitumen characteristics, and the potential benefits of lignin as a bio-based modifier.
- To examine how lignin can influence and potentially improve current pavement design methodologies by analyzing its structural, environmental, and economic implications. This will be achieved by reviewing existing case studies, such as the life cycle assessments (LCAs) of lignin-modified asphalt, evaluating the chemical and mechanical compatibility of lignin with asphalt, and analyzing published data on its cost competitiveness and environmental footprint.
- To assess the feasibility of replacing conventional bitumen with lignin-modified alternatives by evaluating their compatibility with cold-climate pavement requirements. This was achieved by conducting mix design testing (Marshall and Superpave Gyratory) using PG 52-34 asphalt modified with 10%, 20%, and 30% kraft lignin, and comparing the results with MTO's Superpave PG specification for cold regions, such as Thunder Bay.
- To analyze the effects of lignin content on critical performance indicators such as stiffness, compaction, temperature sensitivity, and structural integrity of the asphalt binder.
- To synthesize the findings from both theoretical research and practical testing regarding the engineering viability of lignin-modified asphalt and propose recommendations for future research and implementation.

1.4 Structure of the Report:

The report is organized into five distinct sections. Chapter 1 marks the outset, introducing the subject matter. Chapter 2 provides an in-depth exploration of existing literature. Chapter 3 focuses on methodology, which outlines the research process, the techniques employed to analyze the data, and the conclusions drawn from the research. Chapter 4 will consolidate the data from the previous chapter and focus on analyzing the results of the reading. This chapter will also conclude the analysis of the research, binding everything together. Chapter five includes a summary, conclusions and recommendations of this thesis.

Chapter 2: Literature Review

Bitumen is derived from the distillation of crude oil during the refining process. Its formulation aligns with a spectrum of physical property-based criteria tailored for specific ultimate objectives. The preeminent adhesive attributes of bitumen, coupled with its impermeability, thermoplasticity, strength, versatility, and recyclability, render it an ideal substance for endeavours in engineering and construction. In asphalt mixtures, bitumen derived from petroleum is the predominant binder. According to the International Bitumen Emulsion Federation's estimations, 111 metric tonnes of bitumen were globally utilized in 2019 for constructing roads and pavements.

The primary approach employed in bitumen production involves the application of vacuum distillation to carefully selected blends of crude oil. This method involves the extraction of the non-distillable fraction, commonly referred to as vacuum residue, in technical terms. The essential process of bitumen synthesis involves separating lighter, lower-boiling-point fractions from crude oil, resulting in a substance distinguished by higher boiling points, increased molecular weight, and reduced volatility. The inherent properties of bitumen are predominantly influenced by the specific crude oils utilized in the manufacturing process. Immediate adjustments are effected through a blending or refining process to adhere to prescribed grade standards. Furthermore, bitumen is susceptible to subsequent processing, which facilitates modifications in its physical properties to conform to specified requirements.

The asphalt mixture is the predominant material employed for the construction and upkeep of road pavements, projected to reach 276.9 million metric tons in the EU and 500 million metric tons in the USA in 2020. Asphalt surfacing is estimated to cover over 90% of Europe's 5.2 million kilometres of paved roads and highways [24]. Canada has approximately 415,000 kilometres of road infrastructure, with asphalt roads comprising roughly 90% of the total paved road network [10]. An asphalt mixture is typically created by combining bitumen, filler, and mineral aggregates at temperatures exceeding 100 °C. A robust and enduring mineral framework is established through the compaction of aggregates. The cohesive liquid bitumen binds the totals, while the filler fills gaps between aggregates, reducing overall bitumen consumption. The porosity, rigidity, and stability of the asphalt mixture are influenced by bitumen viscosity, filler quantity, and aggregate [11].

Crude oil is the primary source of bituminous binder; however, its pricing exhibits considerable variability and has experienced a significant increase in recent years. The escalating costs of crude oil are affecting the expenditure associated with asphalt mixtures. Additionally, using such materials contributes substantially to carbon dioxide (CO_2) emissions, a greenhouse gas implicated in augmenting the greenhouse effect [12]. Moreover, technological advancements in oil refineries have reduced the quantity and quality of asphalt, resulting in increased asphalt costs and necessitating modifications. In response to mounting expenses and environmental apprehensions, engineers actively seek sustainable and renewable alternatives to replace petroleum-derived binders or enhance binder performance [28]. Within the realm of road construction, a discernible shift is underway towards adopting novel materials and technologies. Scientists have initiated investigations into the properties of specific substances that exhibit both chemical and physical compatibility with bitumen [13].

As the International Renewable Energy Agency (IRENA) highlights, the current challenge involves a global energy transformation aimed at ceasing fossil fuel production by 2050. Despite its widespread and satisfactory application in the road materials sector, contemporary economic and environmental considerations have instigated the pursuit and advancement of increasingly high-performance materials sourced from renewable origins to create durable road pavements. Scientists have turned their attention to utilizing lignin in road pavement applications. When separated from biomass, the carbon within lignin is isolated and used to modify bitumen binders. Including lignin in bitumen enables carbon neutrality, as any CO_2 generated during bitumen production and transportation is absorbed by the biomass, thereby preventing an increase in atmospheric CO_2 levels [14].

Lignin, an abundantly available biopolymer originating from wood and paper industry byproducts, is among the most prevalent biopolymers on Earth. As the second-largest plant polymer on Earth, lignin follows cellulose in prevalence. Approximately 98% of lignin is derived from the papermaking and pulping industries, primarily employed for combustion heating or power generation. Conversely, only 2% of lignin finds application in more valuable contexts, notably in the production of surfactants, adhesives, and dispersants. Given its hydrocarbon composition, lignin manifests as a three-dimensional, heavily cross-linked macromolecule, sharing chemical similarities with bitumen. Lignin and bitumen are fundamentally hydrocarbon compounds, predominantly comprised of carbon, hydrogen, and oxygen at their core [15] [16].

Various studies have investigated the utilization of lignin as a modifier for bitumen. In two separate investigations, varying concentrations of 2%, 4%, 5%, 6%, 8%, and 10% of lignin were introduced to bitumen, revealing that due to lignin's low carbonyl index, its incorporation into bitumen binders could potentially enhance their resistance to aging, consequently mitigating the degradation of bitumen's fatigue life.

When the addition of lignin to bitumen exceeds 10%, it is commonly considered a bitumen extender. In such instances, lignin can replace over 10% of the bitumen content, diminishing reliance on non-renewable bitumen resources. Numerous research endeavours have explored the utilization of substantial amounts of lignin as an extender in asphaltic materials. One investigation, for instance, examined asphalt mixtures incorporating 0%, 5%, 10%, 20%, and 40% of lignin-containing waste. The findings from this study suggested that the optimal percentage for lignin incorporation as a bitumen extender was 20%. Additionally, an alternative perspective acknowledges lignin as a promising antioxidant in asphalt materials, with optimal efficacy observed at a singular dosage of 10% by weight. Another study elucidates the fundamental mechanisms underlying the anti-aging properties of bitumen based on first principles [17].

Within the domain of pavement engineering, lignin finds application in three primary categories of pavement materials: asphalt, asphalt mixture, and road base soil, apart from its role as a bitumen modifier. In the realm of asphalt, lignin serves as a coupling agent, emulsifier, extender, antioxidant, and modifier. Additionally, lignin can be introduced into asphalt mixtures. Lignin stabilizes road-based soils, offering potential applications in soil improvement.

Concerning rheological properties, the addition of lignin alters bitumen rheology, as demonstrated by dynamic shear rheology (DSR) tests on lignin-modified bitumen. The outcomes reveal a significant increase in the storage modulus of bitumen with a lesser impact on the loss modulus. This incorporation enhances high-temperature deformation resistance, potentially elevating the high-temperature grade of bitumen binders. However, this enhancement may increase the risk of low-temperature cracking or lead to a decline in fatigue performance. Some researchers argue that lignin's impact on the low-temperature properties of bitumen is negligible. The adjustability of lignin's molecular weight and functional groups through various processes can significantly enhance its low-temperature properties.

Regarding aging resistance, thermogravimetric testing (TG) was employed to compare bitumen binder specimens before and after short-term aging. The findings indicate that lignin-modified bitumen exhibits superior thermal stability compared to virgin bitumen, retarding short-term thermal and oxygen aging. Changes in the complex shear modulus aging index support verification of lignin's ability to improve aging resistance. Fourier infrared spectroscopy (FTIR) tests on bitumen specimens after thermal oxygen aging reveal that lignin-modified bitumen has a lower carbonyl index after aging, showing better aging resistance than unmodified bitumen. Similar results from weathering aging, followed by infrared spectroscopy tests, confirm lignin's potential as an anti-aging agent for bitumen [18].

2.1 Asphalt Binder Grading Systems

Asphalt binders are typically classified using shorthand grading systems based on their physical properties, which have evolved from simple to complex methods for characterizing them. Currently, the Superpave performance grading (PG) system is widely adopted or being considered for adoption by most state agencies [19]

Penetration Grading**:**

Penetration grade is determined through softening point and penetration tests. Penetration-grade bitumen exhibits a thermoplastic characteristic, allowing it to soften in elevated temperatures and harden in lower temperatures. This special temperature/consistency relationship is vital while deciding the execution parameters, for example, the adhesion, rheology, durability, and application temperatures of bitumen [20]

The graded bitumen should have the following characteristics:

- Penetration depth of 100 g
- Penetration depth at 25°C (77°F) with a 100g needle
- Flash point temperature
- Ductility at 25°C (77°F)
- Solubility in trichloroethylene
- Thin film oven test (considering short-term aging during hot aggregate mixing)
- Retained penetration
- Ductility at 25°C (77°F).
- Penetration depth using a 100g needle at 25°C (77°F)
- Temperature of flash point
- Flexibility at 25°C (77°F)
- Ability to dissolve in trichloroethylene

- Evaluation through a thin film oven test (addresses short-term aging during hot aggregate mixing)
- Maintained penetration
- Flexibility at 25°C (77°F).

The core principle of penetration grading is based on the relationship between asphalt viscosity and needle penetration depth. This depth, though only roughly linked, correlates with the performance of the asphalt binder. As such, "soft" asphalt binders with higher penetration values are used in cold climates, while "hard" binders with lower penetration values are favoured in hotter conditions.

<u>Viscosity Grading</u>:

Viscosity grading has replaced the empirical penetration test as the primary method for characterizing asphalt binders. The specified traits for graded bitumen include:

- Viscosity at 60°C (140°F)
- Viscosity at 135°C (275°F)
- Penetration depth using a 100g needle applied for 5 seconds at 25°C (77°F)
- Flash point temperature
- Ductility at 25°C (77°F)
- Solubility in trichloroethylene
- Evaluation via thin-film oven test (addressing short-term aging):
- Viscosity at 60°C (140°F)
- Ductility at 25°C (77°F).

Viscosity grading can be conducted on original (as-supplied) asphalt binder samples, referred to as AC grading, or on aged residue samples, referred to as AR grading. The AR grading system aims to replicate the properties of asphalt binders after a typical hot-mix asphalt (HMA) manufacturing process, offering a more representative behaviour of asphalt binders in HMA pavements. In contrast, AC grading characterizes asphalt binders based on the properties they possess before undergoing the HMA process [21].

Viscosity is measured in poise. The lower the poise number, the lower the Viscosity and the more manageable the substance flows.

Performance Grading (PG)

Performance grading is done to determine the deformation of asphalt binders at different temperatures. The binders are graded, and the Temperature is classified in a positive and negative range. The favorable Temperature determines that asphalt has its highest physical properties at that Temperature, and the low Temperature determines that the asphalt binder has its lowest physical properties. Dynamic shear rheometer, bending beam rheometer, and direct tension were used to determine the actual range of PG grade [22]. The available grades of the Performance Grading system are shown along with their significant specifications in Figure 1 [23]

Performance Grade	PG 46			PG 52							PG 58					PG 64						PG 70						PG 76					PG 82				
	34	40	46	10	16	22	28	34	40	46	16	22	28	34	40	10	16	22	28	34	40	10	16	22	28	34	40	10	16	22	28	34	10	16	22	28	34
Average 7-day Max Pavement Design, Temperature. °C	< 46			< 52							< 58					< 64						< 70						< 76					< 82				
Min Pavement Design, Temperature. °C	-34	-40	-46	-10	-16	-22	-28	-34	-40	-46	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-10	-16	-22	-28	-34
ORIGINAL BINDER																																					
Flash Point Temp,T48, Min °C	230																																				
Viscosity, ASTM D 4402: Max.3 Pa's, Test Temp. °C	135																																				
Dynamic Shear,TP 5 Min,1Kpa G*/sin8 Test Temp @ 10 rad/s.°C	46			52							58					64						70						76					82				
ROLLING THIN FILM OVEN RESIDUE (T 240)																																					
Mass loss,Max, percent	1.00																																				
Dynamic Shear,TP 5 Min,2.20 Kpa G*/sin8 Test Temp @ 10 rad/s.°C	46			52							58					64						70						76					82				
PRESSURE AGING VESSEL RESIDUE (PP 1)																																					
PAV, Aging Temperature °C	90			90							100					100						100 (110)						100 (110)					100 (110)				
Dynamic Shear,TP 5 Min,5000 Kpa G*/sin8 Test Temp @ 10 rad/s.°C	10	7	4	25	22	19	16	13	10	7	25	22	19	16	13	31	28	25	22	19	16	34	31	28	25	22	19	37	34	31	28	25	40	37	34	31	28
Physical Hardening	Report																																				
Creep Stiffness, TP 1 Determine the critical cracking temperature as described in PP 42	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24
Direct Tension, TP 3 Determine the critical cracking temperature as described in PP 42	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24

Figure 1:Performance Grading Chart

Dynamic Shear Rheometer (DSR)

The Dynamic Shear Rheometer (DSR) is used to evaluate the viscoelastic behavior of asphalt binders across a range of temperatures and loading frequencies. It measures the complex shear modulus (G*) and phase angle (δ), which provide insights into the material's stiffness and elasticity. These parameters are crucial for assessing rutting resistance at high temperatures and fatigue cracking at intermediate temperatures. For unaged, RTFO-aged, and PAV-aged binders, DSR testing serves as the foundation for defining the Performance Grade (PG) and ensuring the binder can resist permanent deformation and fatigue-related distresses under traffic loads . The test's ability to simulate field-like loading conditions makes it indispensable for modern binder evaluation [24].

Bending Beam Rheometer (BBR)

The Bending Beam Rheometer (BBR) test evaluates the low-temperature performance of asphalt binders by measuring their stiffness (S) and creep rate (m-value). These values indicate the binder's ability to resist thermal cracking when exposed to cold climates. In particular, the m-value reflects the rate at which a binder can relax stress over time. The test is conducted on PAV-aged samples to simulate long-term field aging. Binders that are too stiff or lack stress relaxation capabilities are prone to cracking during freeze–thaw cycles. The BBR test is a critical component of the PG grading system for cold-climate applications like those in Ontario [25].

Rotational Viscometer (RV)

The Rotational Viscometer (RV) test is integral to characterizing asphalt binder under the Superpave system, particularly for evaluating viscosity at elevated temperatures. It determines the binder's resistance to flow at 135°C and 165°C, which correspond to mixing and compaction temperatures during hot mix asphalt production. These temperatures are essential for ensuring that the binder can be pumped and adequately coat aggregates during construction. Asphalt binders that are too viscous can impede proper mixing, while excessively low viscosity may cause flushing or bleeding. The RV test provides a precise and reproducible method to assess the binder's workability and is a critical specification for both virgin and modified binders [26].

Mathematical Models for AC Binders

The Viscosity of the asphalt binder at interest temperature can be determined from the ASTM viscosity-temperature relationship defined by the Equation for any aging condition of an AC binder [27].

The Equation is as follows:

$$\log\log \eta = A + VTS \log T_R \quad (1)$$

Equation 1: ASTM Viscosity-Temperature Relationship for Asphalt Cement (AC) Binders

where η = viscosity, cP, TR = temperature, degree Rankine, A = regression intercept, VTS = regression slope (viscosity-temperature susceptibility parameter)

This relationship applies not only to virgin asphalt cement binders but also to a wide range of modified binders, provided that the percentages of modifications are not excessively high (less than 2% - 3%). Although this Equation commonly develops mixing and compaction temperatures with data from viscosity measurements at 60℃ and 135 degrees Celsius, it can be extended to lower temperatures using ring and ball softening point and penetration data. Studies conducted initially by Shell and subsequently verified by Mirza and Witczak indicate that the ring and ball softening point aligns with a viscosity of 13,000 poise for the majority of unmodified asphalt binders. Penetrations from tests using 100 g, 5-sec loading can be converted to Viscosity using the following Equation [2]:

$$\log \eta = 10.5012 - 2.2601\log(Pen) + 0.00389(\log(Pen))^2 \quad (2)$$

Equation 2:Empirical Relationship Between Penetration Value and Viscosity for Asphalt Binders

where η = viscosity, P, Pen = measured penetration

Thus, the Viscosity of the binder over a wide range of temperatures can be determined from equations using a combination of penetration, ring, and ball softening points, and cinematic and absolute viscosity measurements routinely measured to ensure compliance with viscosity grading. Mirza and Witczak have also developed equations that change the viscosity-temperature relation of the original binder for short-term aging during mixing and compaction and long-term in-situ aging. These equations consider the binder's aging potential, the pavement temperature, and the service time.

2.2 Design Guides

2.2.1 Mechanistic Empirical Guide

ME Design is an empirical, traditional method based on a history of data or experience that yields relations between pavement performance and traffic, materials, climate, and other factors. It is relatively simple and thus used for small roads or those with lower traffic volumes [28].

ME Design in Ontario, Canada

Ontario currently uses the ME Design in pavement construction because of its reliability, simplicity, and ability to adapt to local conditions. It is a method with a long history of use, thus allowing accumulated knowledge and experience. Besides, ME Design agrees well with the Canadian climate and materials.

Mix Design Methods: Marshall and Superpave

Mix design methods are foundational to evaluating the structural performance and durability of asphalt mixtures. They aim to identify the optimal bitumen content that ensures sufficient strength, durability, and workability under varying service conditions. Two widely adopted mix design methodologies in pavement engineering are the Marshall Method and the Superpave Gyratory Compactor (SGC) Method. Both provide quantifiable performance indicators under standardized laboratory conditions and are widely used by transportation agencies and researchers worldwide.

The Marshall Mix Design Method involves compacting asphalt mixtures using a static hammer and evaluating the mixture's strength and deformation through Marshall Stability and Flow values. These parameters respectively represent the maximum load the specimen can sustain and the plastic deformation it undergoes. The method also considers volumetric properties like air voids, voids in mineral aggregate (VMA), and voids filled with asphalt (VFA) to ensure structural integrity and resistance to rutting or fatigue. Although developed in the 1940s, the Marshall method remains widely used for dense-graded mixtures, particularly in countries like Canada, due to its simplicity and reliability [29].

In contrast, the Superpave Mix Design system—developed under the Strategic Highway Research Program (SHRP)—focuses on performance-based properties and uses the Superpave Gyratory Compactor (SGC) to compact asphalt mixtures. The SGC applies vertical pressure and gyratory motion at a fixed angle to simulate field compaction and traffic loading. This process better replicates real-world conditions compared to the impact method of Marshall compaction. Parameters such as design gyrations (N_design), air void content, VMA, and VFA are evaluated to ensure long-term performance against rutting, fatigue cracking, and thermal cracking. The Superpave method is fully integrated with the Performance Grading (PG) system for binders and aligns with the requirements of the Mechanistic-Empirical Pavement Design Guide (MEPDG), making it a vital component of modern pavement design practices [30].

2.2.2 Mechanistic-Empirical Pavement Design Guide

Mechanistic-Empirical Pavement Design Guide to provide the most reliable and standard method based on mechanistic-empirical (M-E) principles so that pavement design engineers have a specific guide in making decisions while designing and analyzing new and restored roads. This guide can also calculate specific responses (stresses, strains, and deflections) to develop additional damage on pavement over time. The procedure relates empirically to the cumulative damage to pavement distress observed. The difference between MEPDG and other guides is that MEPDG predicts multiple performance indicators and links up with materials, structural Design, construction, climate, traffic, and pavement management systems [31].

Overview of the MEPDG Design Procedure

Any design involving the MEPDG process is mainly based on iteration. The engineer/designer uses the trial-and-error method until they have the relatively same value as the design performance criteria values of MEPDG. Therefore, the designer has the option to choose different design features and materials to meet the criteria of MEPDG. In the beginning, the designer takes the condition of the site road (i.e., traffic, climate, subgrade, existing pavement condition for rehabilitation) into consideration. The outputs of pavement distress and smoothness, not layer thicknesses, are received from this practice. The M- E approach enables the Design to be fully optimized and ensures that specific distress types are limited to values lower than the failure criteria during most of the pavement's design life. These steps are more broadly elaborated in the flow chart.

Significance and use of the MEPDG

The MEPDG is a massive alteration in how pavement design is carried out. Mechanistic relates to evaluating the design engineering ideals that contribute to a logical development process with three essential aspects: (1) The theory is used to forecast the critical response of pavements, which includes strains, stresses, and deflection, as a component of traffic and climatic loading. (2) Material characterization treatments that support and comply with the selected theory. (3) Field observed distress relationships between the critical pavement response parameter (empirical part).

The MEPDG offers a homogenous and extensive set of analytical and Design guidelines for new and rehabilitated pavements for both flexible and rigid pavements. The MEPDG uses general design parameters for all pavement types for traffic, materials, subgrade, climate, and reliability, which can be used to create alternative designs using various materials and construction protocols. The structure (layer materials and thickness) of new (including lane reconstruction) and rehabilitated pavements are being recommended, along with protocols for selecting pavement layer thickness, rehabilitation treatments, subfloor drainage, foundation improvement strategies, and other design features.

MEPDG's output is predicted distress and IRI (smoothness) at the carefully chosen reliability level. It is, therefore, not a direct thickness design practice but an analysis method that the designer can use in an iterative mode. Particularly, the MEPDG is used to assess a test design (combination of layer types, layer thickness, and Design features) at a specified level of reliability for a set of site conditions and failure criteria .

MEPDG General Design Approach

The MEPDG design approach consists of three key several steps. Stage 1 is the determination of input values for the design of the test. Strategies to be considered in the design cycle are identified during this phase. A crucial step in this process is the analysis of the foundations. In case of new pavements, the foundation analysis or site investigation comprises of determining resilient modulus, assessment of the shrink-swell capability of high-plasticity soils, frost heave-thaw diminishing capacity of frost-sensitive soils and drainage problems. Analysis of the foundations or evaluation of pavements for rehabilitation of projects and initiatives includes recommendations for assessment in the structure of pavement conditions to examine the various sorts of distress and actual reasons which aids in these distresses. The mechanism concentrates on calculating the strength of existing pavement layers and foundations by means of back-calculation and non-destructive deflection basin tests. Quantifying or estimating the damaged modulus conditions of existing structural layers can be conducted with the help of deflection basin. Moreover, the method also include recommendations for the use of survey to sort out the conditions of pavements, survey for drainage and ground penetration radar (GPA) data to measure the pavement layers in its in-place conditions (damaged modulus values).

In stage 1 of the design approach, the material, traffic and climate characterization process are included. The characterization of the materials is an important aspect in this design procedure, and modulus is the vital layer property required for all layers of the pavement. Resilient modulus is required for all unbound paving layers and foundations, while dynamic modulus is necessary for all Hot-Mix Asphalt (HMA) layers and elastic modulus is required for all Portland Cement Concrete (PCC) or chemically stabilized layers.

Characterization of traffic is the evaluation of the axle-load distributions adhered to the structure of the pavement. Equivalent Single Axle Loads (ESAL) and the development of load factor equivalence are not required in MEPDG. In addition to standard single, tandem, tridem, and quad axle loading, the MEPDG procedures permit special axle configurations to allow for specialized analysis. Another considerable enhancement in the design of the pavement embedded in the MEPDG is the integrated application of climatic effects on pavement materials, reaction and distress. Tested effects are projected using the Integrated Climate Model (ICM), a powerful tool for simulating the impacts of climate, which is used to model temperature and moisture in each pavement layer and the base (foundation). The climatic model primarily considers hourly environmental climate statistics, including temperatures, precipitation, wind speed, cloud cover, and relative humidity, from weather stations across the United States to estimate pavement layer temperatures and humidity. The temperature and moisture forecast for the pavement layer from the ICM is determined hourly and utilized in various approaches to evaluate the material properties of the foundations and pavement layers throughout the design lifespan. Stage 2 of the planning process is the structural assessment and forecasting of selected performance and smoothness indicators. The analytical approach is an iterative one that commences with the selection of the primary trial design. The designer can generate initial test designs, acquired from an existing design protocol or a general catalogue. The test segment is progressively analyzed with the pavement response and model of distress. The results of the analysis encompass material properties, accumulated damage, distress, and smoothness over time, among other substantial process-specific forecasts. If the test design does not meet or exceed the specified reliability level, modifications are made, and the investigation is repeated until a fair and reasonable result is achieved.

Stage 3 of the procedure encompasses the activities necessary to evaluate the structurally feasible alternatives. Critiques of engineering and life-cycle cost analysis of these alternatives are included in these activities.

Hierarchical Input Levels

The hierarchical input level in the MEPDG is a system used to categorize the designer's input parameter knowledge. There are three levels to determine the input values for almost all material and traffic parameters. The upcoming list discusses each level.

• Input level 1: The input parameter is either site-specific or project-specific, directly measured. This level represents the greatest knowledge of the input parameter for a particular project but has the highest cost of testing and data collection to determine the input value. Level 1 should be used for pavement designs with unusual location features, materials or conditions outside the inferential space used to develop the correlations and defaults for input levels 2 and 3.

•Input Level 2: The input parameter of correlations or regression equations is estimated. In other words, the input value is calculated using other site - specific data or measurement parameters that are less expensive. Input level 2 can also represent regional measured values that are not specified to the project.

•Input Level 3: The input parameter is based on the default or "best estimated "values. Level 3 inputs are based on global or regional default values — the median value from a similar data group. This input level has the least knowledge of the input parameter for the project but has the lowest cost of testing and data collection.

Introduction to Hierarchical Input Levels

This hierarchical input structure enables state agencies and users with minimal M - E experience to use the method with little initial investment. The MEPDG hierarchical approach is used in the traffic, material and condition of the existing input parameters of the pavement. Usually one of the three input levels is used to estimate the input values. The highest input level available for pavement sections was used to calibrate the MEPDG and to determine the standard error of each prediction model in section 5 of MEPDG .

Purpose of the Hierarchical Input Levels

The hierarchical input concept or approach allows the designer to obtain the inputs for a design project based on the criticality of the project and the resources available. The hierarchical input structure enables the user to use the MEPDG only with limited experience in M - E design procedures and standard test equipment for measuring material properties. On the other hand, it allows an experienced user to measure many inputs for a project design - build type or for the forensic evaluation of an existing pavement. The accuracy of the design of the MEPDG depends both on the confidence of the designer's inputs and on the accuracy of the prediction models. Where data for calibration was available at each of the three input levels, such as thermal cracking, significantly increases the accuracy of the performance forecast. The original purpose of the model calibration effort was to do the same for all foreseen problems. This was not possible due to the lack of sufficient data for the development of error estimates at each hierarchical level. Except for transverse HMA or thermal cracking, the input levels for the global calibration were kept constant, so that the same standard error was used for all three input levels. Individual agencies will be able to improve the prediction performance accuracy through local calibration by taking into account the reliability (level 1–3) of the input data expected in their individual calibration process and eventual design process. However, it should be noted that the use of more reliable data inputs (Level 1–3) results in more accurate predictions using the models, although the global calibration could not adjust the models for higher levels of inputs .

Recommended Input Parameters and Values; Limited or No Testing Capabilities for HMA (Input Levels 2 or 3)

Dynamic Modulus, E* (New HMA Layers)

No dynamic modulus, E*, laboratory testing required :

- Use E* predictive equation; either the NCHRP 1-37A model based on viscosity or the 1-40D G* model. Both predictive equations are included in the aid screens for software. The inputs are gradation, viscosity of the bitumen or dynamic shear module and phase angle, frequency of loading, air vacuum content and efficient volume content. Input variables can be obtained from testing laboratory-prepared mixture and asphalt samples or from historical records of the agency.
- Use the software's default A-VTS values based on the grade of asphalt binder (PG, viscosity or penetration grades) as shown below.

$$\log\log \eta = A + VTS\,(\log T_R) \qquad (3)$$

Equation 3:Asphalt Binder Viscosity–Temperature Susceptibility Relationship (ASTM Standard)

$\log\log \eta = A + VTS\,(\log T_R)$

where: η = Viscosity, cP; T_R = Temperature, Rankine; and A and VTS are the intercept and slope resulting from a regression of the asphalt viscosity-temperature susceptibility relationship, respectively.

Dynamic Modulus, E* (Existing HMA Layers)

No dynamic modulus, E*, laboratory testing required :

- As described above, use the E* predictive equation. The inputs are gradation, viscosity of the bitumen or dynamic shear module and phase angle, frequency of loading, air vacuum content and efficient volume content. Variables of input can be obtained by testing the core and asphalt extracted from field samples or historical records of the agency.
- Use default A-VTS values based on age-hardened asphalt binder grade (PG, or viscosity, or penetration grades).
- Determine existing pavement condition rating (excellent, good, fair, poor, very poor); calculate the modulus from deflection basins.

2.3 Rheological Properties of Asphalt Cement Binder

The rheological properties of the Asphalt Cement (AC) binder, such as consistency, stiffness, age hardening, and temperature and time susceptibility, significantly affect the performance of the pavement.

Age Hardening: Primary significant hardening in the Asphalt Cement (AC) binder occurs in the pug mill or drum mixer, where the heated aggregate is mixed with hot asphalt cement [32]. The thin films of Asphalt Cement (AC) binder are usually exposed to air at temperatures ranging from 135°C to 163°C (275°F to 325°F). There are some substantial rheological changes, like a reduction in penetration or an increase in binder viscosity. The loss of more volatile segments and air oxidation mainly cause these alterations. This age-hardening continues but at a much slower rate while the Hot Mix Asphalt (HMA) is being processed by surge or storage silo, transported and compacted to the pavement site. This part of aging is often called "short-term aging." The age-hardening process continues, for its service life is much slower when the pavement is opened to traffic. This is often referred to as "long-term aging".

Oxidation, Volatilization, Polymerization, Thixotropy, Syneresis, and Separation are responsible for the age hardening of asphalt binder. Because asphalt cement consists of organic molecules, it reacts with oxygen in the atmosphere, leading to alterations in the structure and composition of the asphalt molecules. This causes oxidative or age hardening, making the asphalt cement more fragile. Oxidative hardening takes place slower but can accelerate in hot climates. Asphalt pavements compacted improperly usually have high air voids that allow more oxidative hardening. In practice, there is a significant amount of oxidative hardening before asphalt is placed, especially in a hot mixing facility. The process where the lighter constituents of asphalt cement evaporate is termed volatilization and is influenced by Temperature. Nevertheless, this is not a contributing factor in long-term in-service aging. Apart from polymerization, thixotropy, syneresis, and separation, Traxler recommended some of the supplementary factors such as effect due to light and water, aggregate chemical reaction, microbiological degradation, and absorption of elements containing heavy asphalts on the surface of aggregates [32, 33].

Penetration: Penetration is the practical measurement of the Asphalt Cement (AC) binding consistency obtained by a standard penetration test. Penetration at 77°F was broadly used in asphalt cement specifications because there is currently no simple method for determining viscosity at 77°F or lower Temperature. Penetration at 77°F usually gives the Asphalt Cement (AC) binder consistency of almost the average annual service temperature. Thus, it has a particular relationship with the overall performance of Hot Mix Asphalt (HMA) pavements.

Ductility: The ductility of a pavement binder is measured by the distance it elongates before it breaks when two ends of a briquette sample are separated at a predefined speed and Temperature. This test is an empirical way of obtaining the fracture characteristics of a binder. Due to its experimental nature and poor reproducibility, the importance of the ductility test as a means of Asphalt Cement (AC) binder quality control is controversial [34].

Viscosity: Shear rate is the ratio of the shear stress to shear strain rate, the viscosity of a binder at any given temperature. At elevated temperatures like 275°F, the Asphalt Cement (AC) binder behaves like a Newtonian liquid, maintaining a consistent ratio of shear stress to shear strain rate. However, Asphalt Binder (A.C.C) operates as a non-Newtonian liquid at lower temperatures, where this ratio no longer remains constant. As a fundamental measure of consistency in absolute units, viscosity typically remains unchanged despite test setup or sample geometry modifications. Asphalt Binder (AC) behaves like a non-Newtonian liquid, where the ratio is no longer constant at low temperatures. Viscosity is a fundamental consistency measurement in absolute units that is generally unaffected by alterations in the test configuration or geometry of the sample [35]. The viscosity of the Asphalt Cement (AC) binder at 140°F has an impact on the performance of the Hot Mix Asphalt (HMA) pavements in hot summer days when the surface temperature of the pavement is close to 140°F [38]. The low viscosity of 140°F can lead to flushing and rutting if other factors (variables) are not taken into account. It was also observed that the aging of Hot Mix Asphalt (HMA) pavement leads to a gradual increase in viscosity over time.

Stiffness refers to the relationship between stress and strain as a function of loading time and Temperature, Which is represented by the Stiffness Modulus or Stiffness of the Asphalt Cement (AC) binder. This relationship is also referred to as the binder's rheological behaviour. In numerous applications of Hot Mix Asphalt (HMA), its stiffness characteristics are crucial for assessing the mix's behaviour and determining the pavement's performance. Generally, for a highway pavement surface course, increased binding stiffness at high service temperatures (nearly 140°F) is preferred to avoid rutting, while decreased binder stiffness is ideal at low service temperatures to prevent cracking due to low-temperature shrinkage [35].

Shear Susceptibility: Most Asphalt Cements (AC) binders exhibit non-Newtonian or viscoelastic flow behaviour at low temperatures. The viscosity increases as the rate of shear increases; therefore, their viscosity is highly reliant on the shear rate and vice versa. The rate of viscosity change with the shear rate is called shear susceptibility, which is sensitive to the shear rate and is viewed as an inherent property of asphalt cement. The rate at which shear susceptibility increases compared to the rise in viscosity at 77°F appears to be a key factor influencing the performance of Hot Mix Asphalt (HMA) pavement. It was reported that a relatively lower gain in shear susceptibility, corresponding to a rise in viscosity, was correlated with better pavement performance.

Temperature susceptibility: The consistency of the Asphalt cement binder varies with Temperature as it is a thermoplastic material. This rate of change is known as temperature susceptibility. Asphalt cement binders with high-temperature susceptibility are not usually desirable because their viscosity can be very low at the hot Mix Asphalt (HMA) compaction temperature, causing tender mix and compaction problems. However, the viscosity at the lowest service temperature can be too high, leading to low-temperature cracking. Three common measurements of this behaviour of AC binders are the Penetration Index (PI), Pen-Vis Number (PVN), and Viscosity-Temperature Susceptibility (VTS).

2.3.1 Aging Simulation Tests: RTFOT and PAV

Asphalt cement binders are subject to both short-term and long-term aging, which affects their rheological behaviour significantly. To simulate these effects in a controlled laboratory environment, two standardized tests are employed: the Rolling Thin Film Oven Test (RTFOT) and the Pressure Aging Vessel (PAV). The RTFOT simulates short-term aging, which occurs during the mixing, transport, and placement of hot-mix asphalt. It involves exposing a thin film of binder to heated air at 163°C for 85 minutes, inducing oxidative hardening and volatilization similar to what occurs during plant and paving operations.

To simulate long-term field aging, the Pressure Aging Vessel (PAV) is used on RTFOT-aged samples. The binder is subjected to a pressure of 2.1 MPa at 100–110°C for 20 hours. This accelerated aging procedure approximates five to ten years of in-service aging, taking into account extended oxidative exposure at moderate temperatures. PAV-aged binders are typically used for testing long-term properties using the Dynamic Shear Rheometer (DSR) and the Bending Beam Rheometer (BBR), which help assess fatigue resistance and thermal cracking susceptibility over a pavement's lifespan. Together, the RTFOT and PAV tests form a critical part of performance grading and binder durability characterization in the Superpave system [36].

2.4 Performance Testing of Asphalt Mixtures

Performance testing of asphalt mixtures is crucial for evaluating how pavement materials behave under real-world loading, climatic, and service conditions. Unlike binder-specific tests that focus on properties such as viscosity or modulus, performance tests evaluate the mechanical strength, fatigue life, and durability of the entire asphalt mixture. These tests are critical in validating mix designs and ensuring that the mixture can meet the long-term structural and functional requirements of a pavement system.

One of the most commonly used performance tests is the Indirect Tensile Strength (ITS) test, which determines the tensile capacity of cylindrical asphalt specimens. The ITS test is particularly valuable for assessing moisture susceptibility and cracking potential, as higher tensile strength typically correlates with better resistance to low-temperature cracking and fatigue [49]. The test involves applying a compressive load diametrically across the specimen and calculating the tensile strength based on the failure load and dimensions.

Another critical test is the Four-Point Bending Beam Fatigue Test, which evaluates the fatigue performance of asphalt mixtures under repeated flexural loading. This test simulates the real-life bending stresses that pavement layers experience due to vehicular loads. The beam specimen is subjected to controlled cyclic loading, and its stiffness is monitored over time. The number of cycles to failure is used to estimate the fatigue life of the pavement material [37].

The Asphalt Mixture Performance Tester (AMPT) is a more advanced device that performs multiple performance tests, including dynamic modulus (E*), flow number, and cyclic fatigue tests. The AMPT provides critical inputs for mechanistic-empirical design models by characterizing the stiffness, permanent deformation, and fatigue resistance of asphalt mixtures under various temperatures and loading frequencies. The dynamic modulus test, in particular, is central to the Mechanistic-Empirical Pavement Design Guide (MEPDG) and is used to model the pavement's response to traffic over time [38].

Collectively, these performance tests provide a robust framework for assessing the expected in-service behavior of asphalt mixtures, making them essential tools in modern pavement engineering and mix design validation.

2.5 Classification of Lignin Types:

There are several classifications of lignin, primarily based on their source, extraction methods, and subsequent purification processes. The key types include:

a. Kraft Lignin:

The production of Kraft lignin involves treating wood chips with an alkaline solution containing sodium hydroxide and sodium sulfide. This byproduct results from the Kraft pulping process, commonly used in the paper and pulp industries. Its structural complexity and high sulfur content define it. The broad range of molecular weights and the frequent presence of impurities in kraft lignin restrict its direct application without further purification.

b. Organosolv Lignin:

Organosolv lignin is produced using a comparatively eco-friendly method that breaks down the lignin-carbohydrate matrix in plant materials using organic solvents like ethanol, methanol, or acetone combined with an acid or catalyst. This process produces more pure lignin and has fewer impurities, making it more suited for various industrial uses. There are certain advantages to using organosolv lignin over kraft lignin. As a result, the former has a lower glassy transition temperature and is thus thermally easier to treat. It is an appealing thermal fuel because it has a greater fluidity index and can be fed more readily into the oven and boiler combustion chambers. Finally, because Organosolv lignin includes less ash than kraft lignin, it results in cleaner burning [39].

c. Lignosulfonates:

A byproduct of sulfite pulping is lignosulfonates, produced when sulfurous acid or its salts are applied to wood chips in an acidic environment. Because of its sulfonic acid groups, this particular form of lignin has unique characteristics and is soluble in water. Lignosulfonates are used as binders in animal feed pellets and as an ingredient in concrete and dispersants, among other applications.

To understand how different lignin types influence asphalt performance, it is important to distinguish between sulfur-containing and sulfur-free lignins. These two categories differ significantly in terms of chemical structure, environmental impact, availability, and effects on binder behavior. The comparison below summarizes key characteristics of each type, based on literature including Gaudenzi et al. (2023) in Table 1 [40].

Table 1:Comparison of sulfur-free and sulfur-containing lignins with respect to their chemical characteristics, environmental impact, and effects on asphalt binder performance (adapted from Gaudenzi et al., 2023).

Criteria	Sulfur-Free Lignin	Sulfur-Containing Lignin
Examples	Organosolv, Soda, Enzymatic Hydrolysis Lignin	Kraft, Lignosulfonate, Klason

Criteria	Sulfur-Free Lignin	Sulfur-Containing Lignin
Sulfur Content	0%	1–8%
Environmental Impact	Environmentally friendly; no sulfur emissions or odors	Potential for sulfur dioxide emissions and odor during mixing
Purity & Structure	Higher purity, more chemically defined	Less pure; may contain inorganic impurities
Production Source	Derived from green chemistry and biofuel processes	Byproducts of traditional pulp and paper mills
Cost & Availability	Higher cost; limited industrial availability	Lower cost; widely available (especially Kraft lignin)
Mixing Behavior	Better compatibility and less foaming	May pose challenges due to moisture or sulfur interaction
Performance in Asphalt	Enhanced aging resistance and oxidative stability	Increased stiffness, but may reduce fatigue resistance
Applications	Targeted for high-performance, sustainable pavements	Widely used in current LMA research due to supply volume
Antioxidant Properties	High (due to phenolic groups)	Present, but less documented

In addition to chemical composition, the physical form of lignin—powder versus pellet—plays a critical role in its behavior when blended with bitumen. Table 2 presents a comparative analysis based on FPInnovations' laboratory studies, highlighting differences in processability, rheological performance, and field applicability [41]

Table 2:As demonstrated in laboratory studies conducted by FPInnovations (2021), the form of lignin—powder versus pellet—significantly affects mixing behavior, viscosity, and storage stability.

Criteria	Lignin Powder	Lignin Pellets
Form	Fine particulate material (after grinding)	Solid, compressed granules (HDI or LDI)
Processing Requirement	Requires **grinding to fine powder** for stable mixing	Requires **high shear mixing (HSM)** to incorporate into bitumen

Criteria	Lignin Powder	Lignin Pellets
Mixer Type	Low-shear mixer (850 rpm) is sufficient if fully powdered	Only high-shear mixer (5000–7000 rpm) can effectively incorporate pellets
Content Used in Tests	20% by binder mass	10% by binder mass
Storage Stability	Improved with fine powder; poor with coarse or unground lignin	Unstable even after HSM; phase separation observed
Impact on PG Grade	Increases both high and low PG grades (e.g., PG 58-28 → PG 64-22)	No PG grade shift; stiffness changes minimal
Effect on DSR & BBR	Significant improvement in stiffness (DSR), low-temp stiffness (BBR)	Slight increase in stiffness; better low-temp relaxation due to oil content
Viscosity (RV)	Increased, especially at 135 °C	Slightly decreased, especially at 135 °C
MSCR – Jnr3.2	Lower values → better rutting resistance	Similar to virgin binder (no significant improvement)
Asphalt Mix Impact	Higher air voids; reduced compactability	Higher air voids but within spec (4–7%)
Application Challenges	Needs consistent fine grinding	Needs longer mixing time and high-speed blending
Additional Notes	Grinding stage critical to ensure homogeneity	Pellet formulation may include oils that affect rheology and compactability

Characteristics of Different Lignin Types:

Each type of lignin exhibits distinct characteristics, making them suitable for specific applications. The lignin variants exhibit significant variations in their chemical composition, which influences their solubility, chemical reactions, and interactions with other substances. Differences in their molecular structures are critical in defining their efficacy, mainly when applied as additives in various industries. The presence of contaminants in kraft lignin usually makes it less suitable for direct usage without further purifying procedures than organosolv lignin. Lignin accounts for up to 30% of lignocellulosic biomass by weight and is a renewable source of aromatic compounds. However, because of its natural complexity and excellent stability, it has yet to be adequately exploited. As a result, sustained and active efforts should be made to understand better the chemical structure and composition of lignin to create more efficient and environmentally friendly degrading procedures. [42]

Suitability for Asphalt Modification:

Selecting the right kind of lignin to maximize the qualities of asphalt requires balancing several factors. Because of their natural chemical structure, some lignin variations are more compatible with asphalt binders, which improves uniformity and overall performance. The procedures used for extraction and purification significantly influence the readiness of lignin for modification in asphalt; cleaner extraction processes may require less subsequent cleansing. Furthermore, the various effects of each type of lignin on the essential characteristics of asphalt, such as viscosity, stiffness, and resistance to aging and fatigue, highlight the necessity of closely evaluating their additive performance in asphalt compositions.

Material Making

Material Making of Lignin: Extraction, Purification, and Quality Parameters

Lignin, a complex biopolymer abundantly found in plant cell walls, is extracted as a byproduct from various industrial processes. This report examines the material-making process of lignin, encompassing extraction methods, purification techniques, and key parameters that impact the quality of lignin-based materials.

Extraction Methods:

a. Kraft Process:

The Kraft pulping process involves treating wood chips with sodium hydroxide (NaOH) and sodium sulphide (Na2S) under high temperatures and pressure. This approach effectively retrieves lignin from biomass sources, primarily wood, which has extensive applications in the paper and pulp sectors.

Four Kraft pulp mills in Quebec could convert residues and produce up to 150,000 tonnes annually. Although almost exclusively used for generating energy by pulp mills currently in Canada, numerous products are produced with commercial-grade lignin, including plywood glues, bioplastics, resins, carbon fibre composites, lignin-based gasoline and diesel, and dispersants in textile dyes. [43]

b. Organosolv Process:

The organosol process utilizes organic solvents such as ethanol, methanol, or acetone, along with acids or catalysts, to break down the lignin-carbohydrate matrix in plant materials. This method offers a more environmentally friendly extraction approach, yielding lignin with lower impurity levels than the Kraft process.

c. Sulphite Process:

The sulphite process involves treating wood chips with sulphurous acid or sulphite salts under acidic conditions. This approach generates lignin in the form of lignosulfonates, water-soluble derivatives valued across multiple industries for their distinctive characteristics. These properties render them useful as dispersants, concrete additives, and binders for animal feed.

Purification Techniques:

The process of refining lignin involves several stages to attain higher purity. Larger contaminants are removed from the pulping fluid by isolating lignin using filtering or centrifugation techniques. The lignin is precipitated by adjusting the pH of the solution with acids or bases, making it easier to separate from the other ingredients. The lignin solution can be purified using methods such as dialysis or ultrafiltration, which help eliminate smaller molecules and contaminants, thereby increasing the solution's overall purity. According to studies, it is possible to improve the antioxidant capacity of alkaline lignin samples by utilizing secondary intensification processes, such as ultrafiltration, differential precipitation, or purification procedures, to eliminate hemicellulose impurities. [44]

Parameters Affecting Quality:

Several critical elements strongly influence the quality of lignin. The extraction and purifying techniques used extensively affect the purity of lignin; for example, because the extraction procedure differs between Kraft and organosolv lignin, Kraft lignin frequently contains more significant amounts of impurities. Furthermore, lignin's molecular weight and structure are essential in its performance in various applications; more considerable molecular weight variations may require further processing to improve solubility and functionality. Moreover, the chemical makeup and functional groups of lignin determine its reactivity, compatibility with other materials, and potential uses. Proper drying and storage conditions are essential to maintain the quality of lignin-based products, as moisture content management has a significant impact on their stability and shelf life.

2.6 Applications of different quantities of lignin with asphalt

A characteristic bitumen mixer (IKA RW16 basic overhead stirrer) and manually shaking the mixing container were used to mix 0%, 10%, 20%, 30%, and 50% lignin by weight with virgin bitumen. The Temperature in the mixer was kept constant at 170 °C while various mixing speed combinations were evaluated, and mixing time was assessed. The temperature remained at 170 °C while the container was mechanically shaken for sixty minutes. Storage stability tests were conducted to evaluate potential changes in the distribution of lignin within the lignin-bitumen blend over time, influenced by environmental factors such as Temperature, humidity, and light. This also helps quantify the mixture's homogeneity. The test procedure (LC 25-003) involved conditioning tubes partially packed with bitumen at 163 °C for 48 hours, followed by freezing for 3 hours. Following the procedure mentioned above, thin bitumen discs were cut at the top and bottom of every single tube, and their softening points (as measured by AASHTO T53) were determined. Finally, the bitumen's storage stability is associated with the variation in softening levels between the top and bottom discs. The bitumen is considered stable if the difference in softening temperatures is less than 3 °C.

Table 3: Variables used for evaluating mixing parameters and storage stability of lignin-modified bitumen (Adapted from Gaudenzi, Zofka, & Osei, 2023)

	Lignin% % by weight	Mixing Temperature (^{0}C)	Duration (minutes)	Mix Speeds

SGC air void	0, 10, 20, 30, 50	170	15,30,60	1000 rpm
analysis	20	170	60	Manual shaking
Stability test	0, 10, 20, 30, 50	140,170	15,30,60	1000 rpm
	20	170	60	Manual shaking

The stability of the 10% and 20% lignin samples corresponded to that of the virgin bitumen, and the mixing temperature had minimal effect. The 10% and 20% lignin combinations have a combined storage stability of less than 0.8° C[45]. In comparison, the 50% lignin produced a paste with minimal workability at 140° C but was more thoroughly incorporated at 170° C with significantly higher workability and stability during storage at 0.4° C. This study demonstrated that wet mixing (adding lignin directly to bitumen) yields outstanding homogeneity at 10% and 20% bitumen as substitute rates, but raising the mixing temperature may be required for 30% and higher substitution rates.

The mixing temperature was found to have the most significant impact on mixing success among the three mixing factors (Temperature, duration, and speed). Both wet and dry mixes containing 10% lignin had good mixability. Wet mixing 20% lignin produced relatively little amalgamated lignin; however, dry mixing 20% had less. Wet mixing 50% lignin generated a paste at 140° C, whereas 170° C produced a more consistent mixture that included lignin and bitumen. Because the appropriate lignin content has yet to be determined, it was judged premature to calculate the mixing temperature using conventional approaches. The results of this mix as shown in Table 3 experiment demonstrate that 10% to 20% dry lignin disperses efficiently into bitumen when mixed with a standard mixer at 170° C for 15 to 30 minutes. A high-shear mixer is not required. The efficient operation of hand mixing shows that adding lignin into a hot bitumen tanker and blending the mixture with the transport motion may be viable.1 The efficient operation of dry mixing shows that lignin might be added to the pugmill after the aggregate heating step, much like any filler, streamlining the process of adding lignin to the asphalt commercial level.

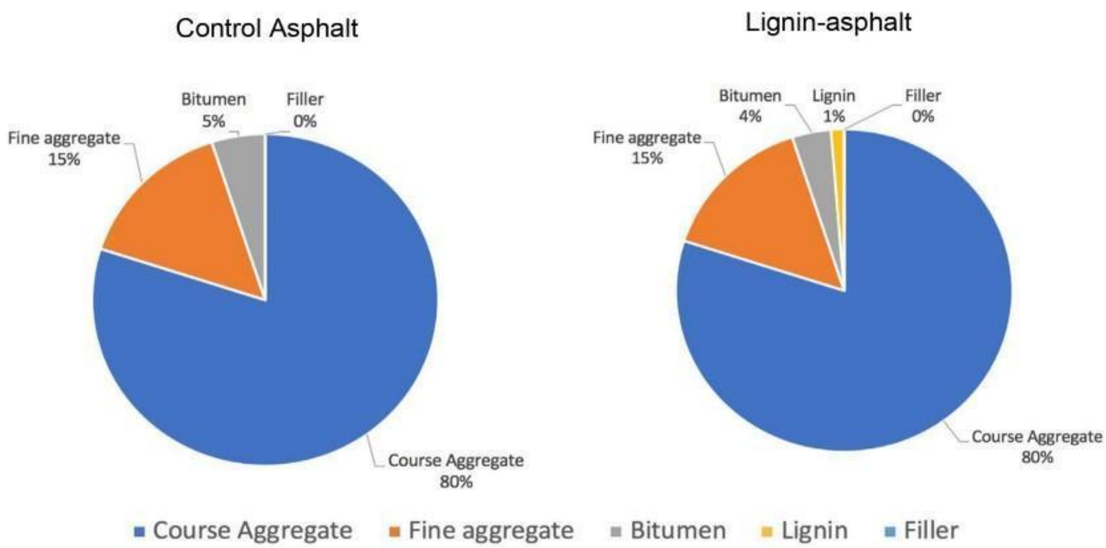


Figure 2:Lignin Asphalt Percentage

The Equations of Rheological Behaviours

- Average percent recovery,

$$R\% = \varepsilon_m - \varepsilon_{nr} / \varepsilon_p \quad (3)$$

Equation 4:Average percent recovery

- Irreversible creep compliance, $J_{nr} = \varepsilon_{nr} / \sigma$

$$J_{nr} = \varepsilon_{nr} / \sigma \quad (4)$$

Equation 5:Irreversible creep compliance

where εm /εnr /is the maximum/non-recoverable/percentage strain; σ is the stress level. "Irreversible creep compliance" most likely refers to the percentage of creep compliance that cannot be restored once the prescribed stress has been eliminated. It indicates the residual deformation or strain in the material after the tension has been eliminated. This irreversible ingredient is critical for comprehending material long-term deformation properties [46].

$$\log(a(T)) = -C1\Delta T / C2 + \Delta T \quad (5)$$

Equation 6:Time–Temperature Superposition Shift Factor Equation

$$\log(\xi) = \log(f) + \log(a(T)) \quad (6)$$

Equation 7:Reduced Frequency in Rheological Analysis

$$\log(G*) = \delta + \alpha /1 + e^{\beta+\gamma \log(\xi)} \quad (7)$$

Equation 8:Generalized Logistic Sigmoidal Function for Complex Modulus

$$\log(G*) = \delta + \alpha\ 1 + e\ \beta+\gamma(\log(f)+ -C1\ \Delta T\ C2+\Delta T) \quad (8)$$

Equation 9:Master Curve Representation Combining Shift Factor and Sigmoidal Parameters

$$\log(G*) = \delta + \alpha / 1 + e^{\beta+\gamma(\log(f)+ -C1\ \Delta T\ C2+\Delta T)} \quad (9)$$

Equation 10:Alternate Sigmoidal Master Curve for Complex Modulus

where β and γ are the shape parameters of the Equation; α, δ is the span of G* values and the minimum modulus value, respectively [47].

- Asphalt (oil) absorption rate (OA) [48]

$$m_3 - m_2 - m_1/m_1 \quad (10)$$

Equation 11: Asphlat absorption rate equation

where,

$m_{1}=$ Take Basalt fibre (BF) and Lignin Fiber out according to the Fiber Mix ratio (FMR), the total weight.

$m_{2}=$ Measure the weight of the clean basket

m_3 = Measure the weight of the basket (with asphalt and fibre)

- Cantabro loss

$$m_1 - m_2 / m_2 \times 100 \quad (11)$$

Equation 12: Cantabro loss equation

where m_1 is the weight of the initial sample and m_2 is the weight after the test. [49]

- Dynamic Stability (DS.)

$$(t_1 - t_2) \times N / d_1 - d_2 \quad (12)$$

Equation 13:Dynamic Stability equation

where t_1 and t_2 correspond to the time at 45 min and 60 min, respectively; d_1 and d_2 are rut depth at time t_1 and t_2; N is the number of cycles of wheel passes over the sample per minute. [50]

- Tensile Strength Ratio (TSR)=

$$R_{T2} / R_{T1} \times 100 \quad (13)$$

Equation 14:Tensile Strength Ratio equation

TSR is the tensile strength ratio (%), and R_{T1} and R_{T2} are the average tensile strength of fresh and frozen-thawed samples, respectively (MPa). [51]

$$\sigma_p = P/A \tag{14}$$

Equation 15:Uniaxial Penetration Stress Calculation

$$R_\tau = f_\tau \times \sigma \tag{15}$$

Equation 16:Calculation of Uniaxial Penetration Strength

where σ_p is the uniaxial penetration stress, P is the maximum load, A is the cross-section area of the load plunger, R_τ is the uniaxial penetration strength, and f_τ is the correlation coefficient (0.35). Uniaxial penetration stress aids in classifying materials in terms of mechanical characteristics, flow behaviour, and deformation response. The elements are critical in various sectors, including polymer development, road construction and comprehending the behaviour of materials under multiple circumstances.[52]

$$R_B = 3LP_{B/}\ 2bh^2 \tag{16}$$

Equation 17:Flexural Strength (Modulus of Rupture) in Bending Beam Test

$$\varepsilon_B = 6hd\ /L^2 \tag{17}$$

Equation 18:Maximum Bending Tensile Strain in Beam Flexure Test

Where RB is the flexural strength, L, b, and h are the specimen's length, width, and height, PB is the maximum load during the test, εB is the maximum bending tensile strain, and d is the most considerable deflection of the middle of the specimen [53].

The boundary of the systematic Control Asphalt

The asphalt mixture plant is the system's core, where essential components such as aggregates, bitumen, and additives are blended in precise amounts to create asphalt mixtures. The system surround contains systems responsible for providing raw materials to the mixing plant, such as aggregates and bitumen. Conveyor systems, storage silos, and pumps are examples of these. The system also includes quality control methods such as testing equipment and processes to guarantee that the asphalt fulfills the established requirements and specifications. Boundaries within the system are meant to monitor and manage environmental variables like dust and contaminants, and security protocols. This assures that regulations are adhered to and fosters a safe working environment . This is illustrated in figure 3[54]

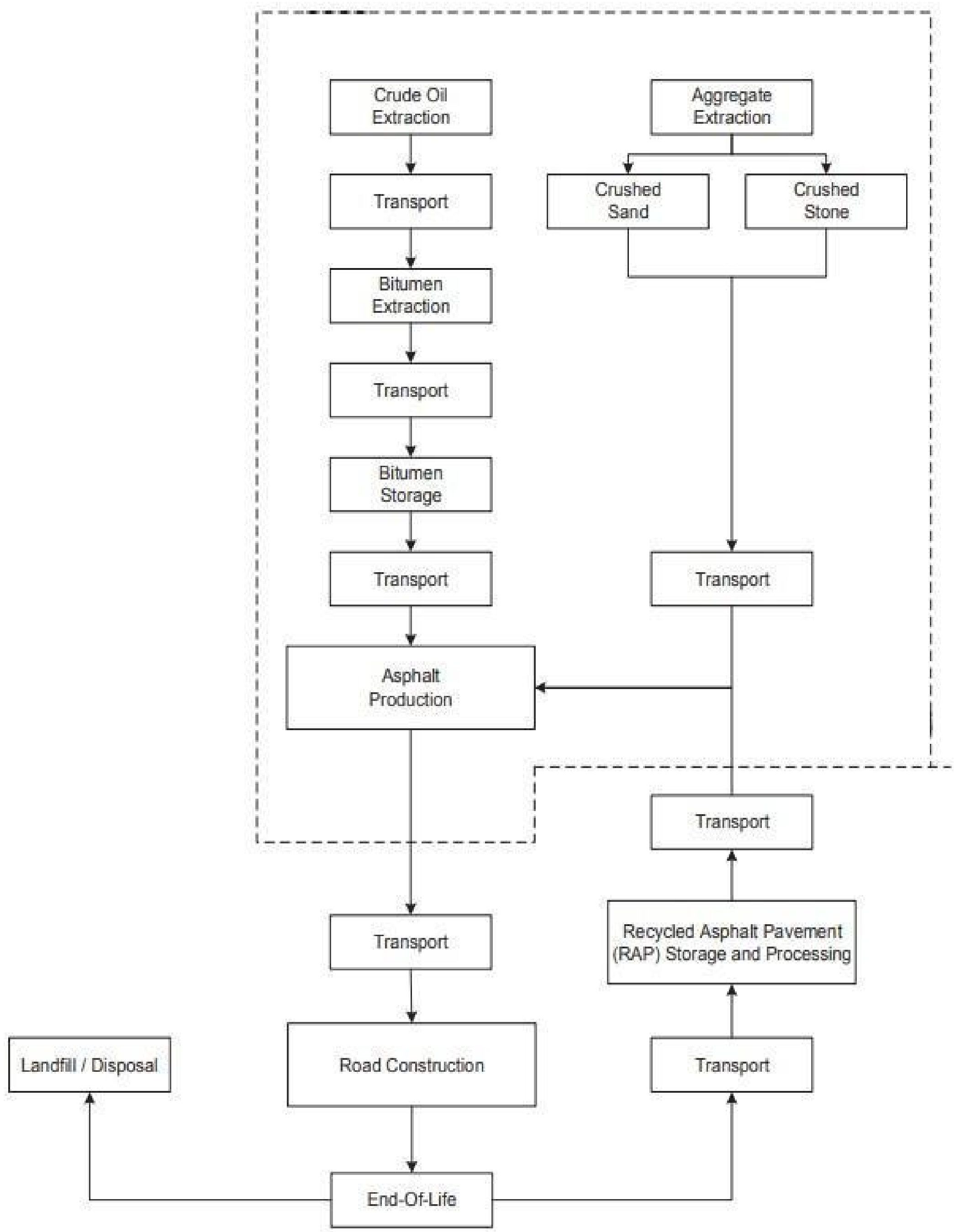


Figure 3:System Boundary of Control Asphalt. Adapted from Bradley (2023).

The boundary of the systematic Lignin Asphalt

Identify and define the raw material sources, such as lignin and other components, that contribute to the formation of "Lignin Asphalt." This may involve combining, heating, or other chemical processes to blend lignin with asphalt or other materials. Establish the criteria and methods for quality control and testing during manufacturing to ensure the intended qualities and performance of Lignin Asphalt.

This encompasses the movement of raw materials to the manufacturing site and the distribution of the final product to end consumers or construction sites. Define the circumstances and settings under which it is meant to be used, such as road construction, pavement applications, or additional applicable purposes. Evaluate and record the potential environmental and social implications associated with the entire life cycle of Lignin Asphalt, including the extraction procedure, production, consumption, and disposal [55].

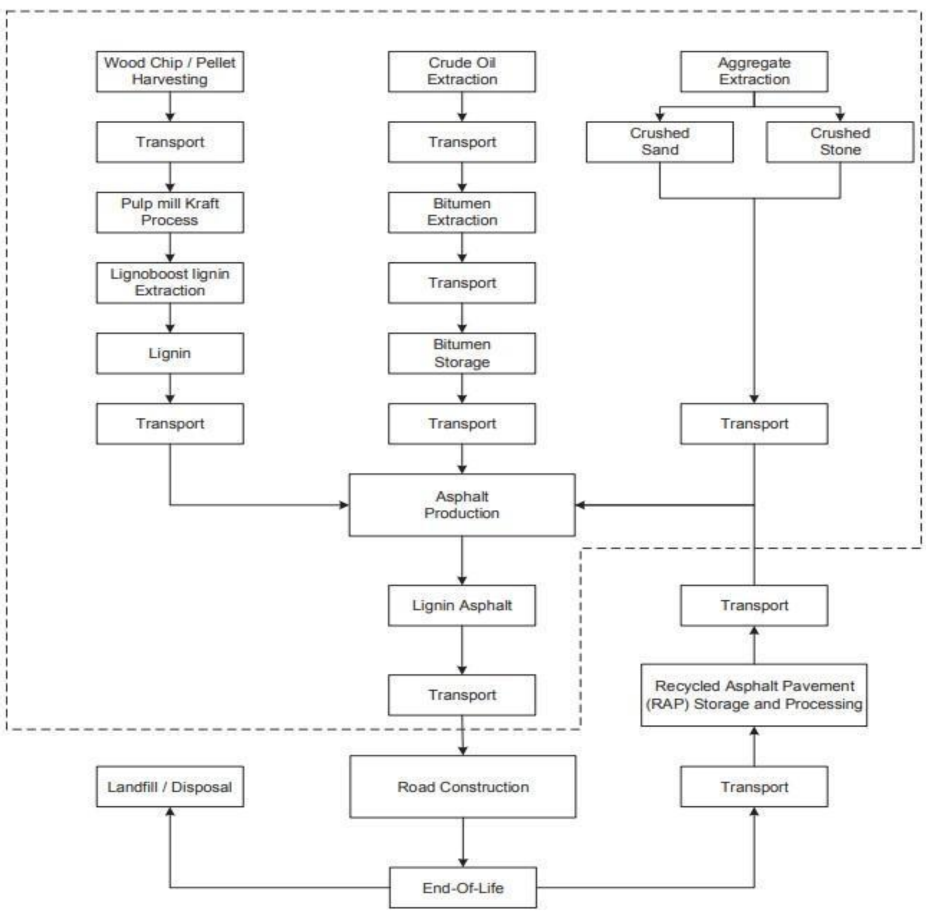


Figure 4: System Boundary of Lignin Asphalt. Adapted from Bradley (2023).

2.6 Mixing of Asphalt and Bitumen: Rheological Challenges and Solutions:

Integrating bitumen and asphalt in road construction involves a complex mixing process critical to achieving durable, high-performance pavements. Cracking due to applied stresses was likely to occur at low temperatures (below 0 °C), and thermally induced shrinkage was also possible. Rutting from persistent deformation is a critical failure concern in typical asphalts at higher temperatures (over 50 °C) [56].

This report examines the methodologies of asphalt-bitumen mixing, highlighting the rheological challenges encountered during this process and proposing potential solutions. Asphalt preparation involves several methods, each with its unique approach and challenges. To create a thorough blend that promotes strong adhesion between aggregate and bitumen, producing a tough and long-lasting asphalt mixture, hot mix asphalt (HMA) involves heating aggregates and bitumen to high temperatures. Compared to conventional Hot-Mix Asphalt (HMA), Warm Mix Asphalt (WMA) utilizes additives or technology to reduce mixing temperatures. This has advantages for the environment, but it also presents challenges for controlling rheological qualities at lower temperatures. Conversely, Cold Mix Asphalt (CMA) uses cutback bitumen or emulsified bitumen for mixing at room temperature, eliminating the need for heating aggregate or bitumen. However, obtaining the appropriate rheological characteristics without heating remains a significant obstacle in manufacturing CMA, underscoring the need for innovative solutions.

Rheological Challenges Assumed:

It considers several variables closely to manage the difficulties of bitumen and asphalt mixing. The viscosity of bitumen is highly sensitive to temperature changes, which significantly impact its flow and coating properties. Reliability depends on maintaining a constant bitumen viscosity during the mixing operation at specific temperatures. It is also essential to apply appropriate shear forces, as this enables the homogeneity necessary for a uniformly mixed asphalt binder. Crucial are the maintenance of structural integrity and the avoidance of aggregate agglomeration. Rheological issues, including poor aggregate coating and clustering, increase the likelihood of weaker asphalt surfaces deteriorating prematurely, underscoring the importance of paying close attention to details throughout the mixing process.

Effect of Lignin Modifier

Softening Point and Penetration:

Figure 5 demonstrates the impact of the penetration test (a) and softening point test (b). The bar chart indicates that the introduction of lignin resulted in decreased penetration values, signifying higher stiffness. Specifically, regardless of lignin type, LMA reduced the penetration value of

Pen60/70, while the penetration values of KLA and CLA decreased from 64 to 58 and 57 mm, respectively. This aligns with prior research [57], which indicates that a 5 wt.% addition of lignin reduces penetration. Conversely, applying lignin resulted in higher softening points, consistent with previous studies [57]. The softening point values of KLA and CLA were 2.1 °C and 1.4 °C higher than those of Pen60/70, respectively. In summary, LMA exhibited lower penetration values and higher softening points, showcasing superior performance at elevated service temperatures. KLA demonstrated behaviour similar to CLA, while KL showed a slight improvement in high-temperature performance compared to CLA [63].

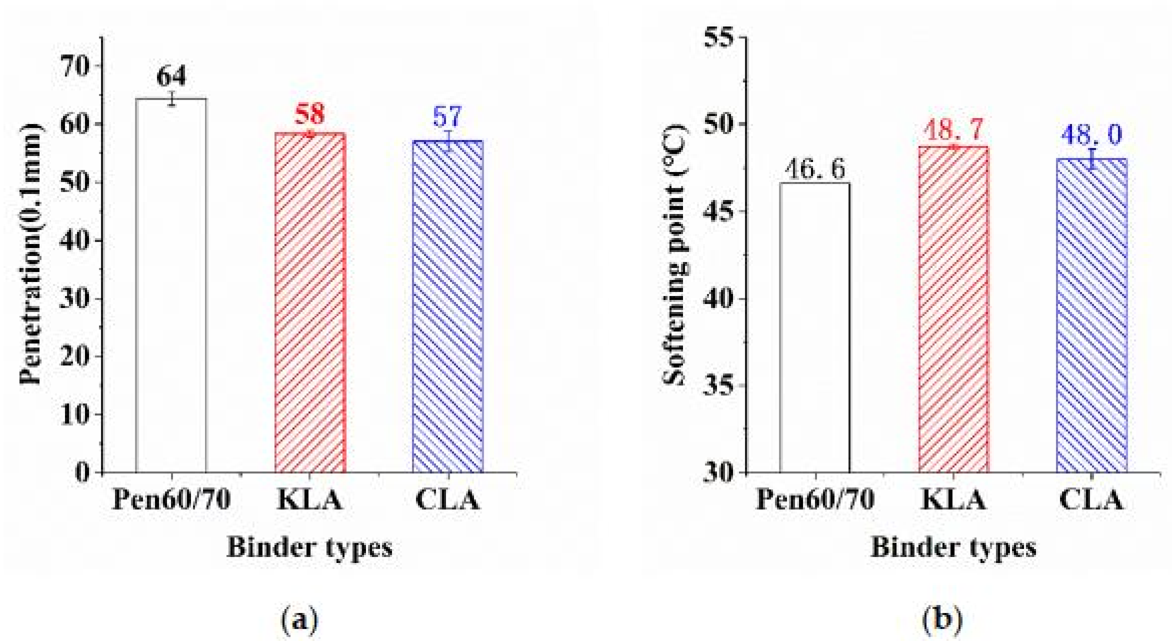


Figure 5:Results analysis (a) penetration and (b) softening point

Workability:

Figure 6 illustrates the viscosity values of the modified binder with five wt.% of lignin at temperatures of 135 ◦C and 160 ◦C. Viscosity, a crucial parameter for assessing the mixability and workability of bitumen binders, is vital in ensuring proper liquidity for construction purposes. As anticipated, the viscosity of test samples increased as the Temperature decreased. At 135 ◦C, the viscosity of Pen60/70, KLA, and CLA was 384.5, 487.5, and 443.8 cp, respectively, all meeting the AASHTO specification requirement of 3000 cp. This indicates sufficient fluidity for pumping during construction. Consistent with prior studies, the addition of lignin increased the viscosity of asphalt binders. At 160 ◦C, Pen60/70, KLA, and CLA viscosities were 134, 302.5, and 230.5 cp, respectively. Notably, LMA exhibited higher viscosity at all temperatures than Pen60/70, with KLA having the highest viscosity value, 2.2 times larger than Pen60/70, and CLA with a viscosity value of 1.7 times larger than Pen60/70 at 160 ◦C.

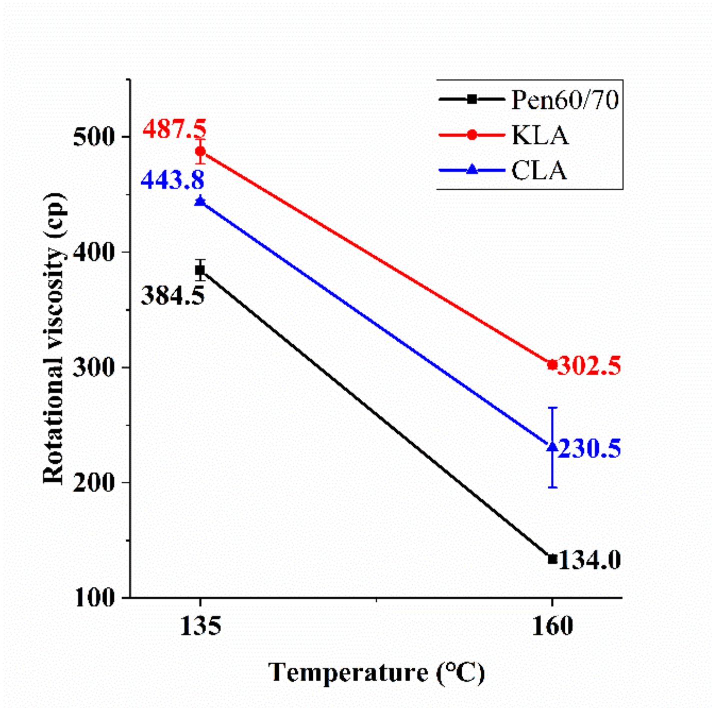
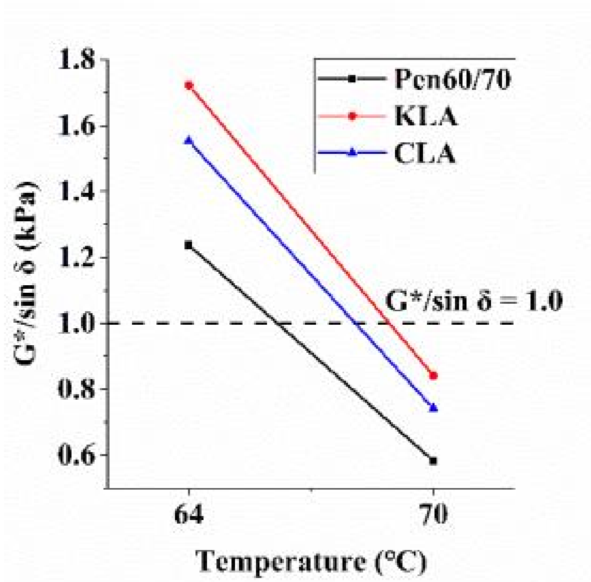


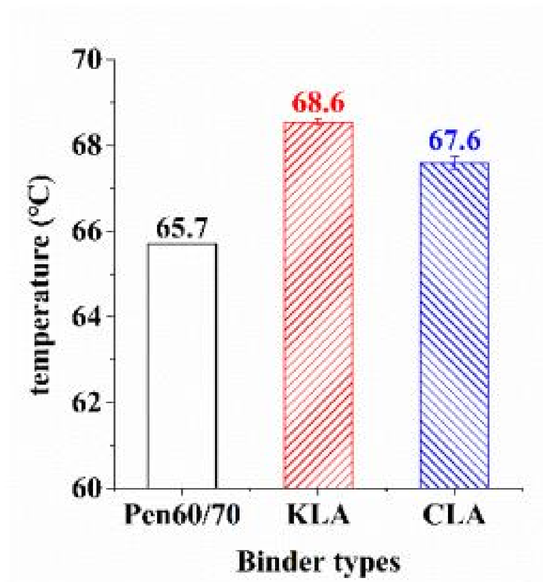
Figure 6:Rotational viscosity test results

Rutting Resistance

The rutting resistance of bitumen binders is assessed using the parameter G*/sinδ, where higher values indicate better resistance. Unaged and RTFO-aged, virgin, and modified bitumen binders were tested, and Figures 3a and 3c display testing temperature and corresponding G*/sinδ values. Figure 7 shows final failure temperatures. Lignin application improved the rutting factor and failure temperature for Pen60/70, with KLA exhibiting the highest improvement at 68 ◦C. G*/sinδ values of RTFO-aged binders increased with lignin addition at 58 ◦C, 64 ◦C, and 70 ◦C, consistent with past studies [59]. The MSCR test analyzed recovery and non-recovery at stress levels of 0.1 kPa and 3.2 kPa, showing moderate improvement in rutting resistance for lignin-modified binders. CLA exhibited more elastic behaviour, while KLA showed higher deformation recovery. Results align with the softening point test and G*/sinδ, consistent with previous research conducted by Arafat [58].



(a)



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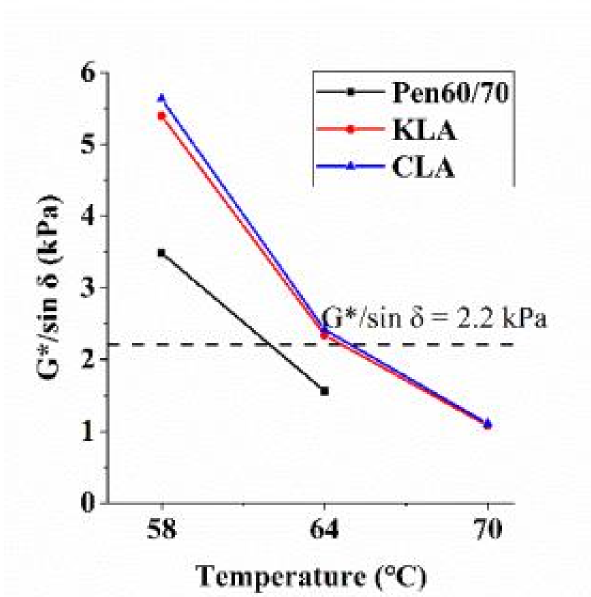
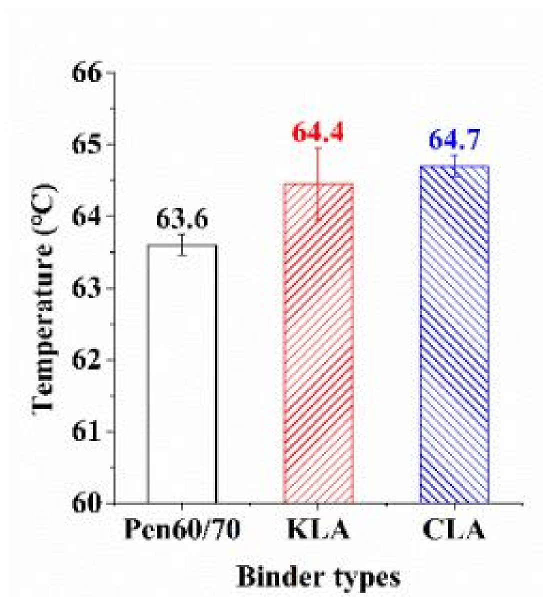


(c)



(d)

Figure 7:Superpave rutting parameter test results: (a) rutting parameter (unaged); (b) failure

temperature (unaged); (c) rutting parameter (RTFO-aged) and (d) failure temperature (RTFO-aged).

Table 4:MSCR test results (The numbers after " " " " """"""" ±" are standard deviations)

Binder Types	% Recovery		J_{nr}		
	0.1 kPa (kPa^{-1})	3.2 kPa (kPa^{-1})	0.1 kPa (kPa^{-1})	3.2 kPa (kPa^{-1})	$J_{nr\text{-}diff}$
Pen60/70	0.400 ± 0.350	0.000 ± 0.000	2.578 ± 0.070	2.766 ± 0.056	7.300 ± 0.700
KLA	1.350 ± 0.050	0.100 ± 0.000	2.366 ± 0.041	2.552 ± 0.046	7.850 ± 0.050
CLA	1.050 ± 0.050	0.200 ± 0.000	2.025 ± 0.011	2.180 ± 0.010	7.650 ± 0.050

Fatigue Resistance

The fatigue resistance of bitumen binders was assessed through the LAS test, using G*sinδ as the evaluation indicator. Figure 4a illustrates the relationship between test temperatures and G*sinδ values, showing an increase in G*sinδ values for all binders as the test temperature decreases. KLA exhibited the highest G*sinδ values at 31 °C and 28 °C [59]. Figure 9 displays the corresponding failure temperatures, with KLA having the highest value (30.6 °C), followed by CLA (30.5 °C), and the virgin asphalt without lignin showing the lowest failure temperature. While adding lignin hurt fatigue performance, the effect was insignificant. At 31 ◦C, adding KL and CL increased the G*sinδ value by 12.7% and 11.4%, respectively. At 28 ◦C, the increase was 21.1% for KL and 11.2% for CL. However, KL and CL-modified asphalt showed higher failure temperatures than the virgin binder, indicating poorer fatigue resistance.

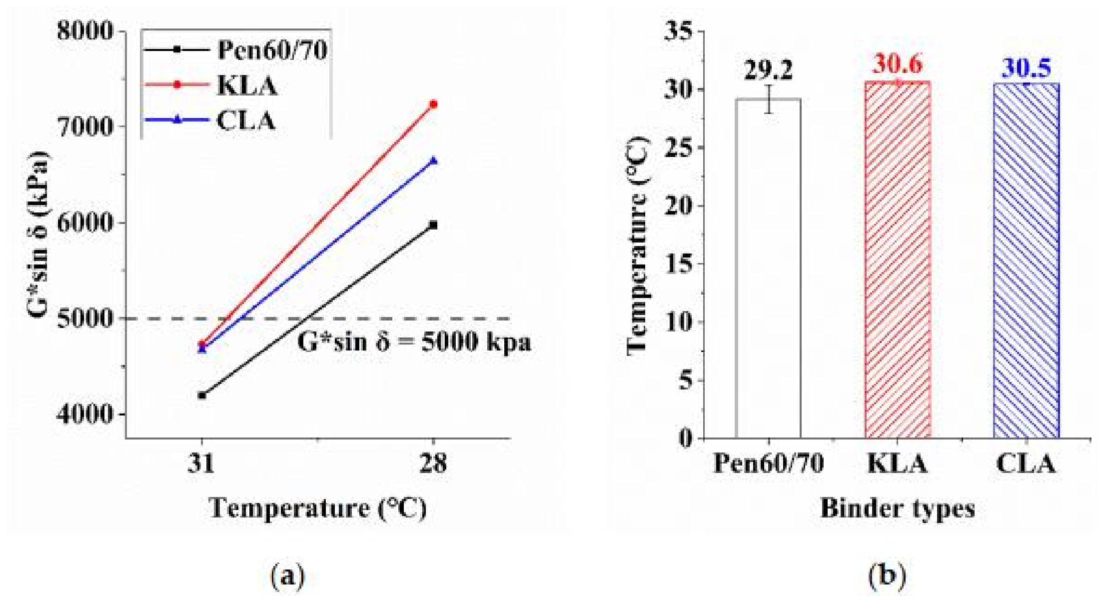


Figure 8: Results analysis: (a) Fatigue parameter (PAV-aged) and (b) failure temperatures (PAV-aged).

The LAS test results using PAV-aged samples in Figure 5 indicate that higher strain levels reduce fatigue life for all binders, and the application of lignin further decreases Nf values [60]. KLA exhibited the smallest Nf values at both strain levels, followed by CLA. The reduction in Nf values for KLA was 11.65% at 2.5% strain and 32.04% at 5% strain, while CLA showed decreases of 6.05% and 8.68% at the respective strain levels. These findings indicate that the inclusion of lignin modifiers reduced the fatigue life of the binders, which is consistent with previous studies. Additionally, CL exhibited better fatigue resistance compared to KL.

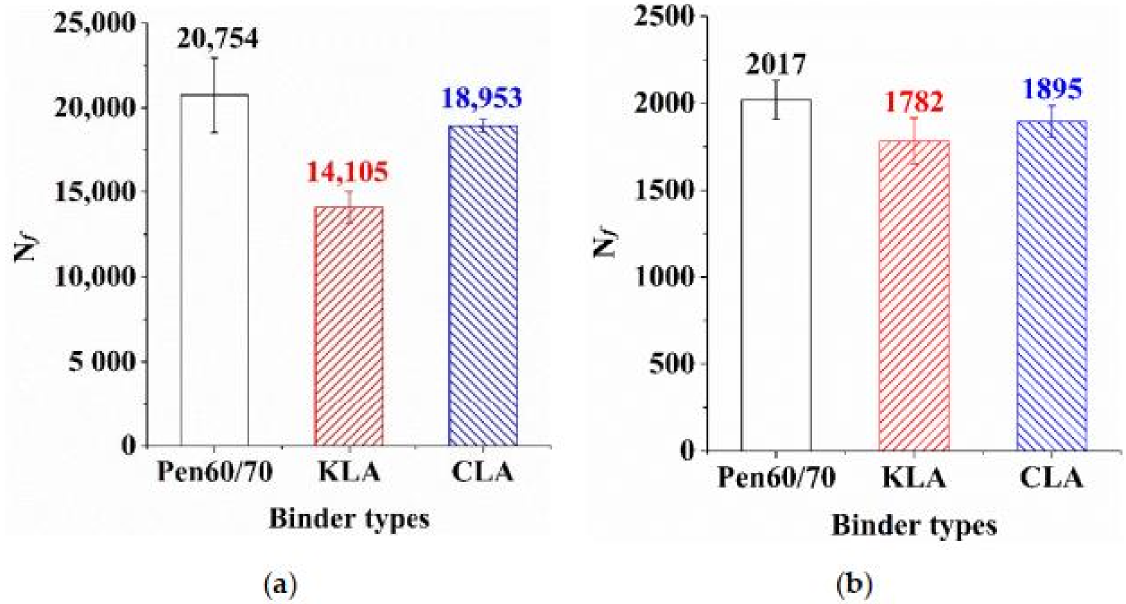


Figure 9:LAS test results analysis: (a) strain value 2.5% and (b) strain value 5.0%.

Low-Temperature Performance

The BBR test assessed the low-temperature properties of test binders at −6, −12, and −18 ◦C, focusing on stiffness and creep rate (m-value), presented in Table 4. Lower stiffness and higher m-value indicate better low-temperature performance. All binders exhibited stiffness less than 300 MPa and m-values greater than 0.3 at −6 and −12 ◦C, meeting AASHTO T313 specifications. LMA binders, compared to Pen60/70, demonstrated lower stiffness and higher m-value. Compared to KLA, CLA showed a slightly larger m-value and more minor stiffness except at −18 ◦C. Overall, LMA exhibited slightly better low-temperature performance than the raw binder, with CLA having slightly superior properties to KLA. These BBR test results contrast with some prior studies suggesting minimal adverse effects of lignin on thermal cracking potential but align with others indicating higher resistance to thermal cracking at low temperatures in lignin binders.

Table 5:BBR test results

Binder Types	−6 °C		−12 °C		−18 °C	
	Stiffness (MPa)	m-Value ($\times 10^{-2}$)	Stiffness (MPa)	m-Value ($\times 10^{-2}$)	Stiffness (MPa)	m-Value ($\times 10^{-2}$)
Pen60/70	156	34	284	31	441	21
KLA	142	35.9	233	32.5	369	24.5
CLA	132	37.1	226	38	378	29

2.7 Tests conducted on Lignin Modified Asphalt

The section explores the standard characterization tests used for virgin asphalt binders and explains how these tests have been modified to evaluate lignin-modified asphalt. The tests, which include rheological and viscosity measurements and aging simulations, are crucial for determining how lignin affects binder behavior under different thermal and mechanical conditions. The section provides comparative insights between lignin-modified binders and their unmodified counterparts to assess the feasibility of lignin as a partial bitumen substitute in meeting performance specifications.

2.7.1 Rheological tests conducted

Dynamic Shear Rheometer

The Dynamic Shear Rheometer (DSR) is one of the most widely used tools for evaluating the high-temperature rheological behaviour of asphalt binders, including those modified with lignin. This test measures the complex shear modulus ($|G^*|$) and phase angle (δ), which define the binder's resistance to permanent deformation under loading. The parameter $|G^*|/\sin \delta$ is used to evaluate rutting resistance, with higher values indicating better performance. In the case of lignin modification, several studies have shown that incorporating Kraft lignin increases the complex modulus and decreases the phase angle, thus improving the $|G^*|/\sin \delta$ ratio. For example, Rezazad Gohari et al. (2023) examined binders modified with 5%, 10%, and 20% lignin and found a consistent increase in stiffness with higher lignin content. The research also compared mixing techniques, and mechanically blended samples were found to be stiffer than those produced using high-shear mixing, likely due to increased oxidation from greater air exposure during blending. These results indicate that the amount of lignin and the method of incorporation significantly affect the binder's high-temperature performance.The specific effects on base binder grade, lignin type (Kraft vs. sulphur-free), and regional specifications may differ, but the overall trend from the studies indicates that lignin improves the high-temperature rutting resistance of asphalt binders when tested using DSR.[61]

The FPI study show that the Dynamic Shear Rheometer (DSR), following AASHTO T315 standards, evaluates the high-temperature rheological performance of asphalt binders. In the study conducted by FPInnovations (2021), bitumen of grades PG 58-28 and PG 52-34 was modified with varying lignin contents (10%, 20%, and 30% by mass of binder) and tested using the DSR to determine the rutting resistance parameter ($|G^*|/\sin\delta$) [62].

The results indicated a clear and consistent increase in $|G^*|/\sin\delta$ with increasing lignin content for both binder types. For PG 58-28, the parameter increased by approximately 15% at 10% lignin, 60% at 20%, and 150% at 30%. Similarly, for PG 52-34, the values rose by about 40% at 10% lignin and 100% at 20% lignin. This demonstrated that lignin improves the stiffness and elastic response of the binder at high temperatures. Furthermore, the effect was more pronounced in the softer grade binder (PG 52-34), indicating that lignin has a strong potential to raise the high-temperature PG grade of softer base binders. These findings suggest that lignin significantly improves rutting resistance, particularly under high-temperature conditions [62]

Bending Beam Rheometer

The Bending Beam Rheometer (BBR) test assesses the low-temperature performance of asphalt binders, specifically their stiffness (S-value) and ability to relax stresses over time (m-value), as per AASHTO T313 standards. These values are critical indicators of a binder's susceptibility to thermal cracking in cold climates.

FPInnovations (2021) tested BBR on PG 58-28 and PG 52-34 binders modified with Kraft lignin at 10%, 20%, and 30% substitution levels. The results showed that increasing lignin content led to a consistent increase in stiffness and reduction in m-value, indicating that lignin modification reduced the binder's ability to dissipate thermal stresses over time. For PG 58-28, all lignin-modified samples failed to meet the minimum m-value requirement of 0.300, especially at higher dosages. The impact was even more pronounced for PG 52-34 binders, where 20% and 30% lignin content resulted in excessively stiff behavior, likely to induce thermal cracking in colder regions.

These findings suggest that although lignin improves high-temperature performance, it may negatively impact low-temperature flexibility. Therefore, optimal lignin content must be balanced to meet the requirements of rutting resistance and cold-climate cracking.

Rotational Viscometer

The Rotational Viscometer (RV) test is a standard method to evaluate the viscosity of asphalt binders at elevated temperatures, typically 135°C and 165°C. These temperatures represent the conditions during mixing and compaction in the field. The viscosity values help determine the binder’s workability and pumpability in asphalt plants and during construction.

In the FPInnovations (2021) study, the RV test was conducted on PG 58-28 and PG 52-34 binders modified with varying levels of Kraft lignin (10%, 20%, and 30%). The results showed a consistent increase in viscosity with increasing lignin content, particularly at 135°C. For instance, at 135°C, viscosity values rose sharply as lignin levels increased from 10% to 30%, indicating reduced workability. The viscosity increase at 165°C was less significant which implies that higher temperatures could potentially counteract the stiffening effect caused by lignin. The results show that lignin improves high-temperature stiffness but higher mixing temperatures might be required to achieve workable construction properties.

2.7.2 Mix Design Testing

Marshall Test

The Marshall Stability and Flow test is widely used to evaluate the strength and plasticity of asphalt mixtures. Stability refers to the maximum load a compacted specimen can withstand, while flow indicates the deformation it undergoes under loading. These parameters are crucial for assessing hot mix asphalt's structural capacity and flexibility, especially in modified binders[29].

In the FPInnovations (2021) study, Marshall tested dense-graded asphalt mixtures prepared with 10%, 20%, and 30% Kraft lignin. The results revealed a non-linear increase in Marshall Stability with rising lignin content, reaching a peak at 20% lignin. Beyond this point (i.e., at 30%), stability values plateaued or slightly declined, suggesting a threshold beyond which additional lignin does not improve structural resistance. The Flow values showed a general decrease with increasing lignin content, indicating stiffer and more brittle mixtures. These results suggest that 20% lignin provides an optimal balance between mixture strength and workability, while higher contents may introduce brittleness and reduce flexibility[28].

Super Pave Gyratory

FPInnovations (2021) extended their study of lignin-modified asphalt to mixture-level analysis by evaluating the compatibility and mechanical performance of Hot Mix Asphalt (HMA) produced with varying lignin contents. Superpave Gyratory Compactor (SGC) tests revealed that lignin content above 5% significantly increased air voids and failed to meet Québec's LC-4202 compaction standards. Moisture sensitivity tests indicated improved water resistance with lignin, while rutting tests showed enhanced deformation resistance. Indirect Tensile Strength Ratio (ITSR) and Thermal Stress Restrained Specimen Test (TSRST) results also suggested no negative impact on low-temperature cracking for mixes with 5% lignin. These findings suggest that low-dose lignin modification may enhance HMA durability under wet and high-stress conditions.

2.7.3 Aging Tests

The Rolling Thin Film Oven

The Rolling Thin Film Oven (RTFO) test is a standardized method to simulate the short-term aging of asphalt binders during mixing, transportation, and laying operations. It involves heating the binder in rotating glass containers at 163°C while exposing it to airflow, mimicking the oxidative and thermal effects encountered during the early stages of pavement construction. The primary outcome of the RTFO test is the mass loss, which reflects binder volatility and oxidation tendency during high-temperature processing.

In the FPInnovations (2021) study, RTFO testing was conducted on PG 58-28 and PG 52-34 binders modified with 10%, 20%, and 30% Kraft lignin. The test results showed a slight reduction in mass loss for lignin-modified binders compared to their unmodified counterparts. This suggests that lignin may help improve binder thermal stability during production by lowering the evaporation of lighter volatile components. These findings imply that lignin contributes positively to preserving binder content and minimizing premature aging during plant and field handling.

Pressure Aging Vessel

The Pressure Aging Vessel (PAV) test simulates the long-term aging of asphalt binders in service. Following AASHTO R28, this test subjects previously RTFO-aged binder to elevated pressure (2.1 MPa) and temperature (typically 100–110°C) over 20 hours. The objective is to replicate oxidative hardening that occurs over 5 to 10 years of pavement life.

In the FPInnovations (2021) study, the PAV test was applied to PG 58-28 and PG 52-34 binders modified with 10%, 20%, and 30% Kraft lignin. The test did not directly report changes in chemical composition. However, the aging behaviour was inferred through follow-up rheological tests, particularly by observing how binder stiffness evolved after long-term oxidation. The application of PAV helped assess how lignin influences the durability and resistance of the binder to oxidative degradation. The results suggest that higher lignin contents tend to increase stiffness after aging, reflecting a greater susceptibility to long-term oxidative hardening, especially at dosages above 20%.

2.7.4 Performance Tests

The Indirect Tensile Strength (ITS) test is commonly used to evaluate the tensile properties of asphalt mixtures. It can be seen that the material's ability to resist cracking under tensile loading is particularly relevant for assessing the moisture damage resistance and cohesive strength of hot mix asphalt (HMA).

The literature review by Gaudenzi et al. (2023) presents various studies that utilized the ITS test to investigate the effects of lignin on asphalt mixture strength. The research findings demonstrated that moderate lignin additions, between 10–20%, resulted in improved ITS values, indicating better tensile strength and bonding within the mix. This improvement was attributed to lignin's rigid and polar structure, which promotes better adhesion between the binder and aggregates. However, some studies have reported a decrease in ITS at higher lignin contents (above 30%), likely due to excessive stiffness and reduced ductility, which can contribute to brittleness and increased cracking potential. These findings highlight the importance of optimizing lignin dosage to strike a balance between strength and flexibility [62].

Chapter 3: Methodology

The methodology employed in this research project comprises five stages. The first stage delves into a comprehensive review of relevant literature. The following stage provides a detailed description of data analysis, where the properties of the bitumen and lignin-modified asphalt mixtures are presented, as well as the supporting information gathered from FPInnovations. The next stage is divided into two parts: one part presents information related to the tests considered for the practical approach, which depends on the availability of resources, equipment, and time. The practical tests include Marshall Stability and Superpave Gyratory Compaction, focusing on evaluating compaction, volumetric properties, and structural performance. The other part represents an in-depth analysis for the theoretical portion, which reveals the rheological behaviour, aging characteristics (RTFO and PAV), and PG-grade shifts based on secondary data. It also discusses the types of lignin used (primarily kraft lignin), along with the advantages and disadvantages of using it as a binder modifier. These two approaches will be combined to form the final stage of the methodology, which presents the conclusion. This is clearly illustrated in the flow chart of Figure 12.

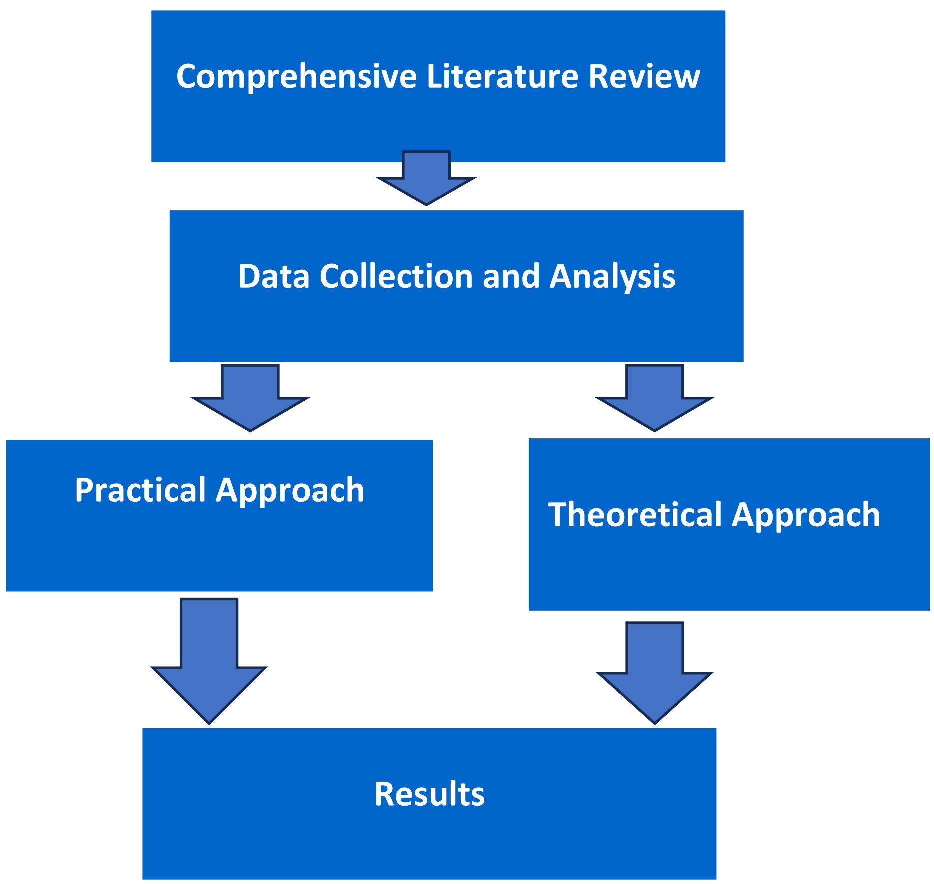


Figure 10:Methodology Flow chart

Comprehensive Literature Review

The research begins with an in-depth review of existing literature on asphalt binders, bitumen's role, and lignin's potential as a sustainable modifier or extender. This includes:

- Historical and modern applications of bitumen and asphalt.
- Challenges of bitumen include environmental impact, cost, and crude oil dependency.
- Types, properties, and extraction methods of lignin (e.g., Kraft lignin, sulphur-free lignin).
- Global and Canadian case studies on lignin-modified asphalt (LMA).
- Previous performance assessments and environmental evaluations of bio-binders.

This stage establishes the foundation and context for the research, helping identifying knowledge gaps and justifying the need to investigate lignin content variations in asphalt mixtures used in Thunder Bay.

Data Collection and Analysis

After the literature review, existing and experimental data are collected and organized for analytical processing. The data sources include:

Past studies and test reports from FPInnovations

To collect data from FPInnovation, one of the research leads of the company was contacted via official personnel and was met by the researcher. The research lead, after hearing the method and purpose behind the research, agreed to help after getting permission from his supervisor. The data was confidential and thus the data has not been officially published. Theoretical data provided by FPInnovations included rheological performance results from prior binder tests, specifically involving DSR and BBR evaluations, as well as RTFO and PAV aging simulations. These datasets were used to interpret stiffness changes and projected PG grade shifts for LMA samples at each substitution level. The tensile behavior assessment included additional literature-based data which included ITS values and stiffness moduli. The external resources provided context to the experimental results and reduced the effects of equipment constraints during binder-level testing.

Rheological Analysis

Purpose:

The evaluation of lignin-modified asphalt binders includes their flow behavior as well as elasticity and stiffness properties and temperature sensitivity. The reason behind this selection:

- Rheology determines how the binder behaves under varied loading and temperature conditions, which is essential for understanding rutting resistance at high temperatures and crack resistance at low temperatures.
- The use of Dynamic Shear Rheometer (DSR) and Rotational Viscometer helps assess workability and performance grade (PG) compatibility.
- Since lignin can stiffen the binder or change its viscoelastic properties, rheological testing reveals how well it meets Superpave specifications.

Aging Analysis

Purpose:

To understand how lignin-modified binders perform over time when exposed to oxidation, heat, and environmental stress. The reason behind this selection:

- Asphalt binders undergo short-term aging (during mixing and compaction) and long-term aging (in-service oxidation).
- Using RTFO (Rolling Thin Film Oven) and PAV (Pressure Aging Vessel), this analysis simulates aging and measures changes in stiffness and durability.
- It helps determine whether lignin improves or deteriorates the oxidative stability of the binder and whether it contributes to longer pavement life.

Indirect Tensile Strength (ITS) Analysis

Purpose:

To assess the cracking resistance and tensile strength of asphalt mixtures under tensile loading. The reason behind this selection:

- ITS provides insight into how well the asphalt mixture can resist thermal cracking and fatigue failure—two critical failure modes in cold climates, such as Ontario.
- It is a straightforward method for testing the cohesion between the binder and aggregate, which may change with the addition of lignin.
- Since lignin can affect binder stiffness and flexibility, ITS offers a mechanical performance perspective beyond binder rheology.

Variation levels of 10%, 20%, and 30% kraft lignin by weight of binder were selected based on findings from previous research, which identified these ranges as effective for assessing mechanical and volumetric performance. This range also enabled comparative evaluation across low, moderate, and high levels of bitumen replacement.

Although formal statistical methods such as ANOVA or t-tests were not applied due to limited sample size, comparative trend analysis was conducted across lignin content levels using standard thresholds from MTO and AASHTO specifications to support interpretive validity.

Experimental test data from Pioneer Construction's laboratory

The materials used for practical testing were sourced through a collaborative arrangement with Pioneer Construction and FPInnovations. PG52-34 bitumen was obtained from Pioneer Construction, while kraft lignin was supplied by FPInnovations. For each substitution level (0%, 10%, 20%, and 30%), three asphalt specimens were prepared using both the Marshall Mix Design and Superpave Gyratory Compactor (SGC) procedures—resulting in a total of 24 specimens (12 Marshall and 12 SGC). The total tests were conducted for 8 separate tests. For Marshall test for each substitution level (0%, 10%, 20%, and 30%) three specimens were used totalling 12 specimens out of 24. The same method was used for SGC procedures which also included 4 tests totalling in 12 specimens. All 24 specimen were used and non was wasted. These were tested for stability, flow, bulk relative density, air voids, and volumetric properties following MTO protocols.

Practical Approach– Mix Design Testing

This section outlines the experimental component of the research. Due to time and material constraints, the scope was focused on mix design testing to evaluate the influence of lignin substitution on asphalt mixture performance. Two standard procedures were used: the **Marshall Stability Test** and the **Superpave Gyratory Compactor (SGC)** method. These were applied to samples prepared with 0%, 10%, 20%, and 30% kraft lignin substitution by weight of binder. All tests used PG 52-34 asphalt as the base binder, sourced from Pioneer Construction, with lignin supplied by FPInnovations.

a. Mix Design Testing

The testing of mix design serves to determine how asphalt mixtures will behave under particular loading conditions and environmental factors. Two established methodologies were used in this study:

Marshall Stability Test: Conducted according to AASHTO T 245, this method assesses the load-bearing capacity and deformation resistance of the asphalt mixture. Key performance indicators include:

- **Marshall Stability** (maximum load capacity),
- **Flow** (deformation before failure),
- **Bulk Relative Density (BRD)**,
- **Air Voids**, and
- **Voids in Mineral Aggregate (VMA)**.
- Specimens were compacted with a standard number of blows and tested using MTO protocols. Three samples were prepared for each lignin percentage (a total of 12).

Superpave Gyratory Compactor (SGC): This test evaluates the compaction and volumetric behaviour of asphalt under simulated field conditions. Following AASHTO TP4 and MTO Category C traffic level requirements, specimens were compacted using:

- **N_initial = 7**,
- **N_design = 75**, and
- **N_max = 160** gyrations.

- Compaction curves and final air void content were analyzed to assess workability, mix stability, and compatibility. Like the Marshall method, three specimens were prepared per mix, totalling 12 SGC samples.

The primary goal of the practical testing was to determine how varying levels of kraft lignin (10%, 20%, 30%) influence the structural integrity, compaction behaviour, and volumetric performance of asphalt mixtures in cold-climate contexts such as Ontario. These results were analyzed against MTO specification thresholds and were later compared with theoretical findings from FPInnovations to evaluate consistency.

Listed below are the initial proposed tests which are characterised with ME Design for LMA for conformation:
Rheological Testing: Determining the viscosity, elastic behavior, and temperature susceptibility of lignin-modified asphalt using tests such as dynamic shear rheometer (DSR), bending beam rheometer (BBR), and rotational viscometer.

Performance Testing: Assessing the mechanical properties and performance of lignin-modified asphalt mixtures through tests like the indirect tensile strength test, resilient modulus test, Four-Point Bending Beam Fatigue Test, and Asphalt Mixture Performance Tester

Mix Design Testing: Assessing and ensuring the performance of asphalt mixtures under different conditions and criteria. Marshall Stability and Superpave Gyrator

Environmental Assessment: Evaluating the environmental impact and sustainability of lignin-modified asphalt, including its carbon footprint and potential for recycling, or reusing the modified asphalt materials.

Aging tests: Rolling Thin Film Oven Test (RTFOT) to simulate the short-term aging and the Pressure Aging Vessel (PAV) to simulate the long-term aging process

Theoretical Approach – Rheological, Performance, and Aging Assessment

In addition to the practical testing methods, a separate theoretical methodology was implemented to evaluate the rheological and aging behavior of lignin-modified asphalt binders. This approach relied on secondary data from FPInnovations and published literature to simulate expected performance under MTO PG specifications. Given the time constraints, equipment availability, and material resources, the study employed a theoretical evaluation of the rheological and aging behavior of LMA. The following subsection outlines the test categories and analytical focus.

a. Rheological Testing

This component focused on evaluating the viscosity, elastic behaviour, and temperature susceptibility of LMA binders. The following test methods and parameters were reviewed:

• Dynamic Shear Rheometer (DSR): Evaluates viscoelastic behavior for rutting and fatigue resistance. (AASHTO T 315)

• Bending Beam Rheometer (BBR): Assesses low-temperature flexibility and cracking resistance.

• Rotational Viscometer: Measures high-temperature viscosity for workability and pumpability.

b. Performance Testing

Additional performance parameters were evaluated through established theoretical protocols:

• Indirect Tensile Strength Test (IDT): Measures tensile strength and moisture susceptibility. (AASHTO TP 9)

• Four-Point Bending Beam Fatigue Test: Assesses fatigue life under repeated loading. (AASHTO T 321)

• Asphalt Mixture Performance Tester (AMPT): Evaluates modulus, flow number, rutting, and fatigue. (AASHTO T 378)

c. Aging Simulation

To predict long-term durability, the following aging tests were interpreted through secondary data:

• Rolling Thin Film Oven Test (RTFOT): Simulates short-term aging during mix placement.

• Pressure Aging Vessel (PAV): Simulates long-term field aging due to oxidation and environmental exposure.

This theoretical framework complements practical findings and supports the performance-based classification of LMA. It provides insight into long-term sustainability and adaptation to Ontario's PG specification system using MEPDG-compatible parameters.

Conclusion

The final phase of the methodology will synthesize findings from both the practical and theoretical components to evaluate the potential applicability of lignin-modified asphalt (LMA) in Ontario's pavement infrastructure. The mix design testing phase will provide measurable data on structural performance, including how varying lignin substitution levels influence compaction, air voids, and overall mix behavior. In parallel, theoretical insights drawn from secondary rheological and aging data will help interpret long-term binder performance, temperature susceptibility, and mechanical resilience under cold-climate service conditions.

This integrative step will be used to:

- Determine which lignin substitution levels may best balance mechanical strength and workability.
- Assess the extent to which LMA formulations align with Ontario Ministry of Transportation (MTO) PG specifications.
- Explore the feasibility of lignin as a partial alternative to petroleum-based bitumen in sustainable binder design.
- Identify areas requiring further investigation, particularly through lab-based rheological testing and extended field trials.

By combining these datasets, the methodology will support a data-informed interpretation of LMA performance and will guide the development of final recommendations and conclusions.

Chapter 4: Results and Analysis

This chapter presents the study's results, organized into two distinct sections: practical and theoretical. The first section outlines the **practical results** based on laboratory mix design testing using the Marshall Method and the Superpave Gyratory Compactor (SGC). These tests evaluated the performance of asphalt mixtures modified with 10%, 20%, and 30% kraft lignin as a partial replacement for conventional bitumen. Key parameters such as Marshall Stability, flow, air voids, VMA (Voids in Mineral Aggregate), and VFA (Voids Filled with Asphalt) were measured to assess the structural integrity and workability of the mixtures. The results offer insight into how varying lignin content influences the physical performance of asphalt mixtures in cold-climate conditions.

The second section describes the theoretical findings derived from a thorough analysis of the body of research and earlier experimental findings pertinent to lignin-modified asphalt. This analysis includes expected trends in rheological behavior, aging characteristics (e.g., from RTFO and PAV simulations), and performance grading outcomes. It also incorporates comparative findings from studies on lignin's chemical structure, its interaction with bitumen, and its long-term sustainability and environmental benefits.

Together, the practical and theoretical results fully present a full picture of the lignin's potential as an alternative or extender to petroleum-based asphalt binders.

4.1 Results

4.1.1 Practical Results

For this project, PG 52-34 asphalt binder was selected as the base binder for both the Marshall Stability testing and the Superpave Gyratory Compactor (SGC) testing. PG 52-34 is a soft-grade binder commonly used in cold climate regions such as Ontario, offering enhanced flexibility and low-temperature cracking resistance. The choice of PG 52-34 allows for the evaluation of the effects of lignin modification on a binder type representative of real-world applications in northern environments. The binder was modified with varying lignin replacement levels (0%, 10%, 20%, and 30%) to assess changes in the mechanical and volumetric properties of the asphalt mixtures through both Marshall and Superpave mix design procedures.

The Marshall Stability

The Marshall Stability test was conducted to evaluate the mechanical performance of asphalt mixtures modified with different percentages of lignin (0%, 10%, 20%, and 30%). Key performance indicators such as Bulk Relative Density (BRB), Maximum Relative Density (MRD), air voids (%), flow (mm), stability (kN), and voids in mineral aggregate (VMA) were measured. These values were summarized in table 3 below.

Table 6:Table generated from combined Marshall tests

Lignin (%)	0	10	20	30
BRB(g/cm³)	2.531	2.524	2.53	2.497
MRD(g/cm³)	2.66	2.662	2.67	2.681
Voids (%)	4.8	5.2	5.2	6.9
Flow (mm)	9.3	9.3	10.3	9.7
Stability (kN)	10632	10566	12689	11607
A.c. Content (%)	5	4.5	4	3.5
VMA(%)	16.1	16.3	16.1	17.2

Marshall Test Findings:

Bulk Relative Density (BRB):

The **Bulk Relative Density (BRB)** exhibited a slight decreasing trend with increasing lignin content, except at the 20% substitution level, where it remained relatively stable. This behaviour indicates that the incorporation of lignin, having a lower or different specific gravity compared to conventional asphalt binder, modifies the density of the compacted mix. The reduction in BRB suggests that lignin may introduce microstructural changes that affect the consolidation of the mixture under compaction. At 20% lignin, the BRB stabilizes, implying an optimal interaction between lignin and aggregate particles that maintains sufficient compaction. However, the notable drop in BRB at 30% lignin replacement highlights a potential disruption in mix uniformity and compaction efficiency. This could be due to the excessive presence of lignin, which interferes with the binder's ability to coat and bond the aggregates effectively, leading to increased internal voids and reduced structural integrity of the mix.

Maximum Relative Density (MRD):

The **Maximum Relative Density (MRD)** exhibited a gradual increase with rising lignin content, with a more pronounced uptick at a 30% substitution level. MRD represents the theoretical density of the mix, assuming no air voids are present. The observed increase suggests that lignin, when incorporated into the binder system, alters the overall mass-to-volume relationship of the mix in a way that increases this theoretical value. This may be attributed to lignin's fine particulate structure and potential to fill microvoids in the matrix or to interact more rigidly with the aggregate framework. At higher dosages, particularly 30%, lignin may be densifying the theoretical structure, even as actual compaction becomes more challenging, indicating a divergence between theoretical and real-world mix behaviour.

Air Voids (%):

The percentage of **air voids** rose steadily from 4.8% at 0% lignin to 6.9% at 30% lignin content. This metric is crucial as it reflects the void spaces left unfilled by bitumen within the compacted mixture. The gradual increase up to 20% substitution implies a modest reduction in compaction efficiency, possibly due to lignin interfering slightly with binder flow and aggregate coating. However, the sharp increase at 30% signals a compaction deficit, suggesting that excessive lignin disrupts binder viscosity or aggregate-binder adhesion, trapping more air in the structure. High air voids accelerate moisture intrusion and oxidation, ultimately reducing pavement durability and service life. Therefore, this trend underscores the importance of controlling lignin dosage to maintain acceptable void levels.

Flow (mm):

Flow values, which measure the mix's deformation at failure under load, increased slightly from 0% to 20% lignin, followed by a decrease at 30%. The initial increase indicates improved mixture ductility and flexibility, likely because lignin at moderate concentrations can slightly soften the binder or modify its viscoelastic behaviour. This enhances the mix's ability to accommodate load-induced strain without cracking. However, the reduction in flow at 30% suggests the mix becomes stiffer and less able to deform, likely due to the high lignin content disrupting the binder matrix or contributing to a more brittle structure. This stiffening reduces the pavement's ability to resist cracking under repeated loading or temperature-induced stress.

Stability (kN):

Marshall Stability values showed a nuanced response to lignin content: a slight dip at 10%, a peak at 20%, and a decline at 30%. Stability reflects the maximum load the specimen can withstand before failure and is a direct indicator of the mix's structural integrity. The initial decrease at 10% may be due to a slight weakening of the binder-aggregate bond caused by early lignin incorporation. At 20%, the peak stability suggests an optimal balance where lignin enhances stiffness and bonding without compromising flexibility. However, beyond this point—specifically at 30%—the decline suggests that excessive lignin introduces brittleness, reduces cohesion within the mix, and limits its load-bearing capacity. This reduction compromises both short-term strength and long-term pavement performance.

According to these results from the Marshall Stability tests, the optimal lignin replacement level in the binders is 20%. At this percentage, the mixture achieved the best balance of mechanical properties, including increased stability, acceptable air void content, and favorable flow characteristics. Specifically, 20% lignin replacement led to enhanced load-bearing capacity without significantly compromising compaction or flexibility. At lower lignin content (10%), there was a slight reduction in stability compared to the control mix (0% lignin), but the air voids and flow remained within acceptable limits, suggesting manageable impacts on performance. However, at higher lignin content (30%), the mixture exhibited increased air voids, reduced compaction efficiency, a decline in stability, and signs of brittleness. This indicates that excessive lignin in the binders can adversely affect the mechanical integrity and durability of the pavement.

Overall, the findings suggest that lignin can be successfully incorporated into asphalt mixtures up to a 20% replacement level without detrimental effects, while higher substitution rates may compromise the long-term performance and structural durability of the mix.

Superpave Gyratory

Superpave mix design testing was conducted using the Superpave Gyratory Compactor (SGC) to evaluate the compactability and volumetric properties of asphalt mixtures modified with lignin.

For this project, Category C was selected based on the Ministry of Transportation of Ontario (MTO) Superpave mix design specifications, which corresponds to a traffic level of 3 to <10 million ESALs (Equivalent Single Axle Loads). As per MTO guidelines, the specified design gyration parameters for Category C are taken from Table 5

$N_{initial}$ = 7 gyrations

N_{design} = 75 gyrations

N_{max} = 160 gyrations

These values determine the compaction effort applied during sample preparation to simulate real-world traffic loading over the pavement's service life. The SGC compacts cylindrical specimens by applying a constant vertical pressure and internal angle, rotating the mold to mimic field compaction conditions. Throughout the compaction process, parameters such as air void content, VMA (Voids in Mineral Aggregate), and VFA (Voids Filled with Asphalt) were monitored to assess the mix's workability and densification behavior.The guidelines used here are **AASHTO R 35** (*Standard Practice for Superpave Volumetric Design*) and **MTO LS-262** (*Superpave Method of Mix Design for Hot Mix Asphalt*), the designated compaction effort for Category C involves:

Table 7:Ontario Ministry of Transportation. (n.d.). MTO category and design ESALs chart for Superpave gyratory compactor

Design ESALs	**Category**	**N initial**	**N design**	**N max**
< 0.3 million	A	6	50	75
0.3 to < 3 million	B	7	75	115
3 to < 10 million	C	7	75	160
10 to < 30 million	D	8	100	205
≥ 30 million	E	9	125	305

These values simulate the field densification process over the pavement's service life. The SGC compacts cylindrical specimens by applying constant vertical pressure and internal angle of gyration, rotating the mould to replicate real-world field compaction. During this process, critical volumetric properties—such as **air void content**, **VMA (Voids in Mineral Aggregate)**, and **VFA**

(Voids Filled with Asphalt)—are monitored to evaluate the mix's workability and densification characteristics.

During the Superpave mixing process, notable challenges were observed, especially with the 30% lignin substitution level. The binder exhibited poor workability, with significant portions sticking to the mixing container, making uniform blending difficult. Additionally, the compacted molds at 30% substitution tended to crumble at the edges, indicating poor cohesion and suboptimal compaction. These issues suggest that higher lignin contents may negatively affect mixing uniformity and mechanical integrity, highlighting the need for optimized blending methods or potential use of chemical compatibilizers at elevated dosages. In contrast, the 10% and 20% blends showed better integration with the base binder and behaved more consistently during the compaction and molding process. The problems indicate that higher lignin contents could lead to poor mixing uniformity and mechanical integrity which requires either optimized blending techniques or higher chemical compatibilizer dosages. The 10% and 20% blends showed improved integration with the base binder and maintained consistent behavior throughout compaction and moulding operations.

The selected gyration levels ensure that the compacted mixtures meet the volumetric acceptance criteria outlined in **both AASHTO and MTO frameworks**, which provide consistent guidance for designing high-performance asphalt mixtures. Additionally, mix compactibility was interpreted using **Pioneer Engineering's internal evaluation thresholds**, which classify mixtures with **N_inital densities below 89%** as well compacted and those achieving **Ndesign 96% or higher** as optimally compacted. These benchmarks were adopted to supplement standard specifications with practical field-based experience.

This dual-guideline approach—merging MTO design specifications with AASHTO standards and local engineering practice—ensured a comprehensive and regionally relevant assessment of lignin-modified asphalt mixtures, enabling the identification of the most effective substitution level for performance-grade applications in cold-climate conditions.

Table 8:Combined SuperPave Results

Lignin Content (%)	**N_initial < 89**	**N_design**
0	90.7	95.7

10	90.2	95.3
20	89.3	96.1
30	89.1	95.3

According to the results presented in Table 6, 20% lignin substitution yielded the most favorable combination: a lower percentage of under-compacted specimens (<89%) and the highest compacted density (96.1%) at N_design. This aligns with MTO specifications, which require Superpave mixes to fall within specific air void and density thresholds for durability and field performance. A compacted density close to 96% at N_design is consistent with MTO's expectations for field-compacted pavements, as outlined in LS-262. Therefore, 20% lignin meets both the volumetric compliance criteria set by MTO and the optimal compaction guidelines recommended by Pioneer, making it the most suitable substitution level for practical field implementation.

These findings reinforce the conclusion that 20% lignin substitution strikes the best balance between workability, compaction quality, and compliance with Superpave specifications. The observed compaction behaviour mirrors the trends seen in the Marshall Stability tests, where 20% lignin also yielded the highest mechanical stability. The consistency across both compaction and mechanical tests strengthens the argument for adopting 20% as the optimal lignin dosage. Conversely, the difficulties observed at 30% substitution—such as binder segregation and mould crumbling—suggest potential risks for premature field failures, such as ravelling or cracking, if not addressed through enhanced mixing or additive strategies.

4.1.2 Theoretical Results

The section presents the study's theoretical findings, developed through an extensive review of published research, technical reports, and communications with industry partners such as FPInnovations (FPI). One of the stated objectives of this research was to assess the **anticipated behaviour of lignin-modified asphalt binders and mixtures** using theoretical and experimental approaches. This section fulfills that objective by establishing an informed baseline for expected performance trends before discussing the practical outcomes in Section 4.2.

The researcher contributed to both the scientific understanding and practical application of lignin-modified asphalt by evaluating its performance using a combination of laboratory mix design testing and secondary rheological and aging analysis. It offers specific insights into lignin's behaviour under Ontario PG specifications and supports the feasibility of 20% lignin substitution for cold climate performance-grade pavements, contributing to the sustainable development of bio-based asphalt materials.

Key performance indicators—including rutting resistance, aging susceptibility, and workability—are evaluated through a synthesis of data from Dynamic Shear Rheometer (DSR), Rolling Thin Film Oven Test (RTFOT), and Pressure Aging Vessel (PAV) experiments reported in the literature. To understand performance implications, a comparative analysis was performed across varying PG grades, lignin replacement levels, and mixing techniques. While the detailed methodology for this theoretical review was outlined in Chapter 3, the current section focuses on synthesizing the relevant outcomes and patterns observed. These findings offer a critical context for interpreting the experimental results presented in the following section and support the overall assessment of lignin's viability as a sustainable asphalt modifier.

Dynamic Shear Rheometer

As part of this study, theoretical rheological trends were further validated by referencing unpublished data directly received from FPInnovations (FPI). Their laboratory investigation focused on modifying PG 58-28 and PG 52-34 bitumen using Kraft lignin at varying proportions (10%, 20%, and 30%). The study utilized the Dynamic Shear Rheometer (DSR) to determine the complex modulus-to-phase angle ratio ($|G^*|/\sin\delta$), a primary measure of rutting resistance.

The DSR results from FPInnovations showed that the $|G|/sin\,\delta^*$ values—representing rutting resistance—**increased non-linearly with lignin content**. For **PG 58-28**, adding 30% lignin raised the rutting parameter by approximately **150%**, while 10% and 20% lignin increased it by about **15%** and **60%**, respectively. Even steeper gains were observed for the **softer binder PG 52-34**, with up to **100% improvement** at 20% lignin replacement. These results indicate that **lignin acts as a stiffening agent**, enhancing the binder's resistance to deformation under high-temperature loading conditions. This effect is particularly pronounced in lower-grade binders, where the base rheological properties are more susceptible to modification.

This trend supports the **theoretical prediction of high-temperature PG grade shifts**. Specifically, the elevated $|G^*|/\sin\delta$ values at higher lignin contents suggest that the binders could meet the performance criteria for higher temperature grades within the Superpave system. For example, a PG 52-34 binder enhanced with 20–30% lignin may demonstrate rheological behaviour comparable to PG 58-28 or higher, depending on the threshold values defined by AASHTO M320. Thus, these findings validate earlier assumptions (outlined in Chapter 3) that lignin incorporation improves high-temperature performance, aligning with performance-based binder grading criteria.

The theoretical findings match the observed high-temperature PG grading shift in similar experimental results [75]

Bending Beam Rheometer

The theoretical implications of lignin modification at low temperatures were evaluated using BBR data obtained from FPInnovations (2021), specifically from their Phase I laboratory study on Kraft lignin-modified bitumen. In this study, PG 58-28 and PG 52-34 binders modified with 10–30% Kraft lignin were subjected to PAV aging and tested using a BBR (AASHTO T313) to assess their thermal cracking potential.

Two critical parameters were measured:

- **Stiffness (S_{60}):** indicating the binder's resistance to deformation under thermal stress, and
- **m-value (m_{60}):** reflecting the binder's ability to **relax stress over time** (i.e., creep compliance), which is essential for preventing low-temperature cracking.

Table 9:Summary of BBR Results for Lignin-Modified Binders (after PAV aging)

Binder Type	Lignin (%)	Test Temp (°C)	S_{60} (MPa)	m_{60} (MPa/s)
PG 58-28	0	-24	429	0.275
		-18	216	0.346
	10	-24	561	0.249
		-18	281	0.312
	20	-18	384	0.288
		-12	167	0.361
	30	-18	413	0.277
		-12	221	0.346
	0	-30	570	0.258

PG 52-34		-24	263	0.333
	10	-24	357	0.296
		-18	157	0.363
	20	-24	434	0.283
		-18	212	0.352

As lignin content increased, **S-values rose**, and **m-values declined**, confirming the binder's increasing stiffness and decreasing ability to relax thermal stresses. Notably, the **m-value dropped below the AASHTO threshold of 0.300** for PG 58-28 with 10% lignin at -24°C, and for PG 52-34 at 20% lignin—indicating a **reduced tolerance to thermal contraction and higher cracking potential**.

- AASHTO M320 specifies that binders must exhibit **m_{60} ≥ 0.300** and **S_{60} ≤ 300 MPa** at the designated low-temperature grade.
- At **20% and 30% lignin**, several test points violate the m-value threshold, which may disqualify those formulations for use in **cold climate applications** such as Northern Ontario or Quebec.

This behavior is attributed to the **rigid, aromatic structure of Kraft lignin**, which increases stiffness and reduces flexibility at low temperatures, effectively making the binder **more brittle**.

Rotational Viscometer

The influence of lignin modification on binder **workability** was evaluated theoretically using viscosity data from **FPInnovations (2021)**, which applied the **Rotational Viscometer (RV) test** to assess the flow characteristics of binders modified with **Kraft lignin at 10%, 20%, and 30%** replacement levels. The RV test was conducted according to **AASHTO T316**, which measures viscosity at elevated temperatures under controlled shear conditions.

The binders evaluated were **PG 58-28 and PG 52-34**, and viscosity was recorded at two critical temperatures:

- **135°C**, which corresponds to the standard asphalt mixing temperature
- **165°C**, which corresponds to the typical compaction temperature

Table 10:Rotational Viscosity of Lignin-Modified Binders at 135°C and 165°C(Source: FPInnovations, 2021)

Binder Type	Lignin (%)	Viscosity @135°C (cP)	Viscosity @165°C (cP)
PG 58-28	0	484	210
	10	524	224
	20	603	251
	30	666	285
PG 52-34	0	432	190
	10	519	234
	20	631	272
	30	751	295

The data shows a **clear and consistent increase in viscosity** with higher lignin content, especially at **135°C**, the critical temperature for mixing. For instance:

- PG 52-34 increased from 432 cP (0% lignin) to 751 cP (30% lignin)—a 74% rise.
- PG 58-28 also showed a 38% increase from 484 cP to 666 cP at 30% lignin.

At **165°C**, viscosity also increased, but the effect was less pronounced, suggesting that elevated mixing or compaction temperatures can **partially offset lignin-induced stiffening**. For example, PG 52-34 with 30% lignin exhibited a viscosity of 295 cP at 165°C, compared to 190 cP for the unmodified binder—an increase of ~55%.

These results demonstrate that while **lignin improves high-temperature stiffness**, it can adversely affect **plant workability** and **mixing ease**, especially at standard processing temperatures.

Marshall Stability

The strength and deformation characteristics of lignin-modified asphalt mixtures were evaluated using **Marshall Stability and Flow tests**, as reported in the FPInnovations (2021) Phase I study. The mixtures were prepared using **PG 58-28 asphalt** modified with **10%, 20%, and 30% Kraft**

lignin, with an asphalt content of 5.4% and air voids of 4.5%, as per standard Marshall mix design procedures (AASHTO T245).

Marshall Stability reflects the **maximum load** a compacted asphalt specimen can withstand, while Flow indicates the **deformation (mm)** it undergoes at the point of failure. These parameters help assess the balance between strength and flexibility, both of which are critical for durable pavement performance.

Table 11: Marshall Stability and Flow Test Results (FPInnovations, 2021)

Lignin Content	**Marshall Stability (N)**	**Flow (mm)**
0% (Control)	10,632	9.3
10%	10,566	9.3
20%	12,689	10.3
30%	11,607	9.7

The data shows that **Marshall Stability increased significantly at 20% lignin**, rising from 10,632 N (control) to 12,689 N—a gain of nearly **19%**, indicating enhanced **load-bearing capacity** due to improved binder stiffness and better aggregate interlock. However, at **a 30% lignin content, stability dropped slightly to 11,607 N, suggesting that excessive lignin may induce brittleness** or reduce mix cohesiveness.

Flow values also changed with increasing lignin content. While the flow increased to 10.3 mm at 20% lignin, indicating moderate ductility, it decreased slightly to 9.7 mm at 30%, suggesting a **loss of deformation capacity** as the binder becomes more rigid. This consistent trend suggests a **strength-flexibility tradeoff**, especially concerning in cold climates where mixes must accommodate thermal contraction without cracking.

Superpave Gyratory

The theoretical evaluation of lignin-modified asphalt mixtures was supplemented by performance data from **FPInnovations (2021)**, which examined **Hot Mix Asphalt (HMA)** samples containing **0%, 5%, 10%, and 20% Kraft lignin** (by binder weight) using the **Superpave Gyratory**

Compactor (SGC). The goal was to assess the effect of lignin on **compaction characteristics**, **air voids**, and key performance indicators such as **moisture susceptibility** and **rutting resistance**.

SGC testing was conducted under standard conditions to simulate field compaction. The primary output of interest was the **air void content (Va)** at the design number of gyrations (N_design), which should fall between **3–5%** according to Superpave specifications (AASHTO R35).

Table 12:Air Voids at N_design (75 gyrations) for Various Lignin Contents

Lignin (%)	**Air Voids (%)**
0	4.5
5	4.8
10	5.5
20	6.3

The SGC data clearly show that only the **0% and 5% lignin mixtures** met the **Superpave air void requirement** of 3–5%. At **10% and 20% lignin**, the air voids exceeded acceptable limits, indicating that higher lignin content **negatively affects compactability**, likely due to increased binder stiffness. This confirms the **stiffening impact** of lignin, consistent with the viscosity and DSR results, and suggests that binder-aggregate coating and compaction behavior are both affected.

Despite these compaction challenges, **performance-related tests** provided valuable insights into lignin's potential benefits:

Table 13:Performance Metrics for Lignin-Modified HMA

Lignin (%)	**Air Voids (%)**
0	4.5
5	4.8
10	5.5

20	6.3

- ITSR (Indirect Tensile Strength Ratio) increased significantly at 5% lignin, improving resistance to moisture-induced damage.
- Rutting depth decreased with higher lignin content, indicating enhanced resistance to permanent deformation under load.

These findings imply that, while compaction may be more difficult at higher lignin levels, **mechanical durability and moisture resistance improve**, especially in **wet and high-traffic conditions**.

Rolling Thin Film Oven

The short-term aging behavior of lignin-modified binders was theoretically evaluated using **RTFO test results** provided by **FPInnovations (2021)**. This test simulates the oxidative and thermal aging that occurs during hot mixing and laydown operations in the field. Two Performance Grade (PG) binders—**PG 58-28** and **PG 52-34**—were modified with varying amounts of **Kraft lignin (10%, 20%, 30%)** and conditioned according to **AASHTO T240**.

Mass Loss Results:

The RTFO test measures **mass loss (% by weight)**, which is a proxy for binder **volatilization and short-term oxidative stability**. Lower mass loss indicates better thermal stability and resistance to hardening during mixing.

Table 14:RTFO Mass Loss (%) for Lignin-Modified Binders

Binder Type	**Lignin (%)**	**RTFO Mass Loss (%)**
PG 58-28	0	0.48
	10	0.38
	20	0.31
	30	0.29
PG 52-34	0	0.43
	10	0.36

	20	0.28

These results show a **consistent decrease in mass loss** with increasing lignin content, confirming that **lignin improves short-term thermal stability** by reducing binder volatility. This property is advantageous during plant mixing and transportation, as it minimizes binder degradation.

DSR Analysis After RTFO Aging:

To assess **rheological changes** following RTFO aging, **Dynamic Shear Rheometer (DSR)** testing was performed on the aged samples. The *rutting parameter (|G|/sin δ)** was calculated for each blend, providing insight into the **stiffening behavior of the binder** post-aging.

Table 15:DSR Rutting Parameter (|G*|/sin δ) after RTFO Aging

Binder Type	**Lignin (%)**	**Viscosity @135Â°C (cP)**	**Viscosity @165Â°C (cP)**
PG 58-28	**0**	**484**	**210**
PG 58-28	**10**	**524**	**224**
PG 58-28	**20**	**603**	**251**
PG 58-28	**30**	**666**	**285**
PG 52-34	**0**	**432**	**190**
PG 52-34	**10**	**519**	**234**
PG 52-34	**20**	**631**	**272**
PG 52-34	**30**	**751**	**295**

Pressure Aging Vessel

The long-term oxidative aging behavior of lignin-modified asphalt binders was assessed using data from **FPInnovations (2021)**, which employed the **Pressure Aging Vessel (PAV)** in accordance with **AASHTO R28**. This procedure simulates **5 to 10 years** of in-service aging by exposing **RTFO-aged binders** to elevated temperature (100°C) and pressure (2.1 MPa) over 20 hours. After PAV aging, the binders were tested using the **Dynamic Shear Rheometer (DSR)** to evaluate their **fatigue resistance**, measured through the *|G|·sin δ** parameter—where lower values indicate better resistance to fatigue cracking.

According to **AASHTO M320**, the binder passes the fatigue criterion if *|G|·sin δ ≤ 5000 kPa** after PAV aging.

Table 16:DSR Fatigue Parameter (|G*|·sin δ) After PAV Aging

Binder Type	**Lignin (%)**	**Test Temp (°C)**	**|G*|·sin δ (kPa)**	**Pass/Fail**
PG 58-28	**0**	**19**	**3210**	✓
PG 58-28	**10**	**19**	**3556**	✓
PG 58-28	**20**	**19**	**4954**	✓
PG 58-28	**30**	**19**	**5197**	✗
PG 52-34	**0**	**16**	**2780**	✓
PG 52-34	**10**	**16**	**3842**	✓
PG 52-34	**20**	**16**	**5082**	✗
PG 52-34	**30**	**16**	**5624**	✗

The results clearly show that *|G|·sin δ increases** with higher lignin content for both binder grades, reflecting a **rise in stiffness** after simulated aging. While this stiffening enhances resistance to permanent deformation, it **negatively affects fatigue resistance**:

- **PG 58-28** remained within the 5000 kPa limit up to **20% lignin**, but exceeded the threshold at **30%**.
- **PG 52-34**, being a softer binder, exceeded the fatigue limit at both **20% and 30%** lignin, indicating greater susceptibility to embrittlement with lignin addition.

These findings **confirm theoretical expectations** that lignin's rigid aromatic structure, while beneficial for rutting resistance, also contributes to **reduced relaxation capability and increased brittleness** under oxidative aging. The shift in the **fatigue cracking threshold** is a critical performance trade-off, especially in cold or fatigue-prone pavement conditions.

Indirect Tensile Strength

Theoretical expectations regarding the tensile performance of lignin-modified asphalt mixtures are supported by Indirect Tensile Strength (ITS) test results compiled in the meta-analysis by Gaudenzi et al. (2023). ITS is a critical indicator of a mixture's resistance to cracking under tensile stress, particularly in cold climates or under repeated traffic loading. The ITS test evaluates the cohesion of the asphalt matrix and the quality of aggregate-binder bonding, both of which contribute to long-term pavement durability.

According to the compiled data, incorporating 10–20% lignin (by binder weight) consistently resulted in significant increases in ITS values, typically in the range of 15–30% compared to unmodified controls. These improvements suggest stronger aggregate-binder interaction, likely due to the presence of polar functional groups in lignin (e.g., hydroxyl and carboxyl groups), which enhance adhesion and help resist moisture-induced debonding.

Table 17:Summary of ITS Values from Studies Referenced in Gaudenzi et al. (2023)

Lignin Content	**ITS (MPa)**	**% Increase vs. Control**
0% (Control)	0.92–1.00	—
10%	1.08–1.21	+17% to +25%
20%	1.14–1.29	+20% to +30%

30%	0.95–1.05	±0% to +10% (inconsistent)

While 10–20% lignin improved tensile strength and durability, substitution levels ≥30% showed inconsistent or declining ITS values. This trend is theoretically supported by the expectation that excess lignin stiffens the binder matrix, reducing its ability to absorb energy or deform plastically under stress. This reduction in ductility may lead to premature micro-cracking, especially in cold or fatigue-prone environments.

Thus, optimal tensile performance is achieved by balancing the increased stiffness (beneficial for strength) with retained flexibility (necessary for crack resistance).

4.2 Analysis

The initial research design included performing a complete set of characterization tests on conventional and lignin-modified asphalt binders. The study scope needed to be adjusted because of practical limitations which included restricted access to advanced laboratory equipment and time constraints and resource limitations.. The research used a hybrid methodology which combined experimental mix design testing with theoretical analysis supported by secondary data from credible sources including FPInnovations. The modified approach allowed researchers to finish their work within existing constraints while creating a base for upcoming performance assessments and application-oriented guidelines. The experimental component consisted of mix design testing only but the results showed a strong match with theoretical expectations and existing published data. The laboratory findings gain increased validity because of the strong convergence between the results and the theoretical expectations and existing published data despite the limited testing framework.

The efficient mixing of asphalt binder and aggregates during pavement construction depends primarily on rheological binder properties. Bio-based modifiers, including Kraft lignin, present a promising method to improve asphalt stiffness, elasticity, and deformation resistance across various temperatures. Theoretical findings from FPInnovations show that the incorporation of lignin significantly increases rutting resistance ($|G^*|/\sin\delta$) following RTFO aging, particularly at 20–30% substitution levels. However, the same study reported that fatigue resistance ($|G^*|\cdot\sin\delta$)

declines with higher lignin dosages after PAV aging. This highlights the importance of dosage optimization to prevent premature embrittlement under prolonged loading conditions.

Rotational Viscometer (RV) tests further indicated that workability declines at standard mixing temperatures (135°C) as lignin content increases, though this effect is moderated at 165°C. BBR test results revealed that low-temperature flexibility deteriorates beyond 20% lignin, failing to meet the minimum m-value required for cold climates. As a result, a substitution range of 10–20% lignin is recommended to strike a balance between high-temperature stability and low-temperature flexibility. At a 20% lignin content, the PG grading shifted from PG 52-34 to PG 58–28, indicating enhanced thermal performance. However, no further improvement was observed beyond this threshold. The PG shift was estimated using secondary rheological data from FPInnovations and supporting literature because direct PG grading tests (e.g., DSR and BBR) could not be conducted due to equipment limitations. These included RTFO and PAV aging results, which demonstrated an upward shift in high-temperature PG classification with increasing lignin content, particularly at the 20% substitution level. While experimental confirmation was not possible, the theoretical findings exhibited consistent trends, suggesting enhanced rutting resistance and thermal stability.

The Life Cycle Assessment (LCA) results from industry studies, including Khandewal and CHAPLIN-XL, indicate that partial replacement of fossil-based bitumen with lignin can decrease greenhouse gas (GHG) emissions by 25% to 70% based on production inputs and system boundaries. This aligns with the Sustainable Development Goals and demonstrates lignin as a green alternative in asphalt binder modification. The careful selection of mixing equipment, quality control protocols, and advanced rheological testing (e.g., DSR, BBR, MSCR) are essential in achieving consistent and durable binder performance.

The literature review showed that performance-based testing methods such as fatigue, rutting, and thermal cracking evaluations are not consistently applied and lack standardization across studies despite the growing interest in lignin-modified asphalt. The selection of characterization methods depends on the researchers' objectives, available resources, and the specific limitations of each study. The evaluation methods for LMA show the need for standardized procedures to get reliable results. Theoretical findings in this research provided essential background information for understanding the experimental results. The experimental results confirmed the theoretical predictions about increased stiffness and rutting resistance at 20% lignin content which was supported by rheological and aging data. The experimental trends observed in the data match the theoretical expectations which enable the use of secondary data to expand the analytical scope of the study.

The final objective of this research was to evaluate whether both conventional and lignin-modified binders conform to Ontario's cold climate Performance Grading (PG) specifications. This was investigated by preparing asphalt binder samples with 10%, 20%, and 30% Kraft lignin substitutions and assessing their behavior through a combination of laboratory testing, secondary data analysis, and theoretical modeling. A logical future step—pending full-scale implementation—would be to incorporate these performance data into Mechanistic-Empirical Pavement Design Guide (MEPDG) models to support adoption in performance-based design frameworks. Overall, the integration of theoretical and experimental findings enhances the analytical robustness of this study. The combined findings create a fundamental reference point for lignin-modified asphalt mechanical stability and PG compatibility and mixing characteristics which validate the technical and environmental findings of Chapter 5.

Chapter 5: Summary, Conclusions and Recommendations

5.1 Summary

This study examined the mechanical and performance behaviour of asphalt binders modified with varying percentages of kraft lignin (10%, 20%, and 30%) using a dual approach: practical laboratory testing and theoretical analysis. The research was motivated. The study was motivated by a recognized research gap in the limited empirical data available on the performance of lignin-modified asphalt under cold-climate conditions, particularly in Canada. This gap is especially relevant, as the increasing scarcity of bitumen derived from petroleum and the environmental benefits of bio-based alternatives, such as lignin, have heightened the need for sustainable binder substitutes in pavement engineering.

To address this need, the study investigated whether lignin-modified asphalt binders (LMA) could meet the performance and durability expectations of Ontario's cold-climate pavement infrastructure, as specified by Performance Grade (PG) standards. Practical testing focused on mix design evaluation using the Marshall Stability Test and the Superpave Gyratory Compactor method. These methods were applied to determine the structural and volumetric performance of asphalt mixtures under different lignin dosages, using PG52-34 as the base binder. Key metrics such as stability, flow, bulk density, and air voids were measured. Among the variations, the 20% lignin mix showed favourable results in terms of compaction and structural integrity without significant adverse effects.

To complement the practical results, the theoretical component was based on data from FPInnovations and prior literature to assess rheological behaviour, aging resistance, and temperature sensitivity. Though in-lab DSR, BBR, and aging tests were not feasible, secondary data were used to simulate long-term binder performance and PG shifts. It also allowed theoretical evaluation using MEPDG-related parameters such as dynamic modulus and fatigue life.

Overall, the combined methodology provided a structured and integrated understanding of how kraft lignin can serve as a partial substitute for bitumen in asphalt binder formulations. It confirmed the feasibility of specific lignin dosages from both mechanical and environmental perspectives, providing a knowledge base for identifying the most promising substitution levels. These findings provide a critical foundation for future full-scale testing, long-term validation, and the development of standardized guidelines for the use of bio-binders in cold-climate pavement applications, such as those found in Thunder Bay.

5.2 Conclusions

Based on the combined analysis of practical and theoretical assessments, the following conclusions are drawn in alignment with the research objectives:

- The mix design results from the Marshall Stability and Superpave Gyratory Compactor tests suggest that a 20% kraft lignin substitution offers the most favourable balance in compaction, stability, and air void levels. While this supports 20% as a potentially optimal substitution level, 10% also displayed viable properties, whereas 30% showed reduced performance due to higher air voids and possible workability issues.
- Prior literature had identified 20% lignin as a promising threshold for balancing mechanical performance and workability. However, further rheological and field-based performance testing is necessary to validate and refine this level under diverse conditions.
- Theoretical data from FPInnovations and published studies indicate that lignin addition may increase the binder's high-temperature PG rating (e.g., from PG 52-34 to PG 58-34), enhancing rutting resistance. These findings were inferred from external DSR and RTFO/PAV datasets rather than direct laboratory testing.
- Although in-lab aging and fatigue tests were not performed due to equipment limitations, the reviewed RTFO and PAV data suggest that kraft lignin may improve oxidative aging resistance, particularly at moderate substitution levels, without significantly compromising low-temperature flexibility.
- While the results are based on laboratory mix design and secondary theoretical data, observed improvements in stability and compaction at 20% lignin suggest practical viability for cold-climate regions such as Ontario.
- The dual methodology—integrating practical testing with theoretical modeling—enabled a structured assessment of PG-related behaviour and potential alignment with MEPDG input requirements, despite the absence of in-house DSR and BBR testing.

- Theoretical analysis of rheological and aging data indicates improved stiffness and rutting resistance at 20% lignin substitution, suggesting a projected PG grade shift compatible with Ontario's specifications.
- The research shows that lignin-modified binders at 20% substitution meet PG 52-34 or PG 58-34 standards which makes them suitable for use in MEPDG-based pavement design frameworks.

While these findings do not yet justify immediate implementation of lignin-modified asphalt in large-scale projects, they provide a strong technical basis for further rheological validation and field performance studies. Kraft lignin shows potential as a sustainable and technically compatible partial bitumen substitute for cold-climate pavement applications.

5.3 Recommendations

While this research offers valuable preliminary insights into the potential of kraft lignin as a bitumen substitute in cold-climate asphalt applications, several avenues remain open for further study to strengthen and expand upon these findings: Expanded Testing Scope: Future studies should include in-lab rheological testing (DSR, BBR, RV) and mechanical performance evaluations (IDT, AMPT, Fatigue testing) to validate theoretical findings and simulate real-world field performance:

- Expanded Testing Scope: Although this study relied on mix design testing due to time and resource limitations, future research should incorporate laboratory-based rheological evaluations (such as DSR, BBR, and RV) and mechanical performance tests (IDT, AMPT, Fatigue testing). This would help confirm the theoretical projections made in this study and enable better simulation of long-term field performance.
- Field Trials in Cold Regions: This thesis provides a good sense of the laboratory behaviour of LMA mixtures; however, constructing test pavement sections in Thunder Bay or similar climates would allow researchers to monitor long-term durability, oxidative aging resistance, and rutting behaviour in situ, critical for validating lab-based predictions.
- MEPDG Input Calibration: Although MEPDG-compatible parameters were inferred through secondary data, future studies should include complete mechanical testing to generate accurate modulus and fatigue life values. This would help refine MEPDG inputs for lignin-modified binders and improve design precision within the Ontario MTO framework.

- Exploration of Material Variants: This study focused on kraft lignin and PG52-34 bitumen. Expanding the scope to compare sulphur-free lignin, hydrolyzed lignin, and alternative PG binders would help evaluate how different material combinations influence performance and PG behaviour.
- Clarification of PG Targets: This work utilized the PG52-34 binder as a baseline. While theoretical projections suggested a potential PG shift, further investigation is needed to confirm if a new PG target (such as PG58-40) is appropriate. Any future recommendations should be supported by direct rheological testing.
- Improved Blending Techniques: This research highlighted some mixing challenges at higher lignin contents. Future work could explore pre-treatment methods, such as chemical compatibilizers or pre-blending processes, to enhance uniform dispersion and improve workability across a broader range of substitutions.
- Cost and Construction Feasibility: Although this study primarily focused on technical performance, future work should also include a cost-benefit analysis of lignin substitution. Discussions with industry experts, including Fred Hakala and Allan Bradley, suggest that cost savings may be achievable at moderate substitution levels due to reduced demand for binders. However, real-world implementation would require a comprehensive evaluation of material procurement, blending compatibility, and potential adjustments to compaction or paving processes.

References

1. Brown, E. R., & Mallick, R. B. (1995). Bituminous pavement materials and construction. McGraw-Hill.
2. Roberts, F. L., Kandhal, P. S., Brown, E. R., Lee, D. Y., & Kennedy, T. W. (1996). Hot mix asphalt materials, mixture design, and construction (2nd ed.). National Asphalt Pavement Association Research and Education Foundation.
3. Huang, Y. H. (2004). Pavement analysis and design (2nd ed.). Pearson Prentice Hall.
4. Dey, T., & Mallick, R. B. (2021). Lignin as an asphalt binder modifier: State of the art and future direction. Transportation Research Record, 2675(8), 673–685.
5. Qiao, X., Huang, J., & Xue, L. (2016). Investigation of lignin-modified asphalt binder: Preparation, performance and interaction mechanism. Fuel Processing Technology, 148, 463–471.
6. Norgbey, E., Hofko, B., & Grothe, H. (2020). Influence of lignin modification on mechanical and chemical performance of bituminous binders. Construction and Building Materials, 260, 119818.
7. Van Vliet, T., Wiegman, D., & Kuiper, P. (2016). Performance of lignin-modified asphalt mixtures in cold climates. Journal of Materials in Civil Engineering, 28(12), 04016214.
8. Lesueur, D. (2011). The colloidal structure of bitumen: Consequences on the rheology and on the mechanisms of bitumen modification. Advances in Colloid and Interface Science, 145(1-2), 42–82.
9. Wang, H., Liu, X., Apostolidis, P., & Scarpas, T. (2018). Review of warm mix asphalt with bio-based additives. Journal of Cleaner Production, 177, 302–313.Thives, L. P., & Ghisi, E. (2017). Asphalt mixtures emission and energy consumption: A review. Renewable and Sustainable Energy Reviews, 72, 473-484
10. Hassan, M. A., Hassan, A. A. A., & Mohamed, A. A. A. (2021). A novel performance-based method to design asphalt mixtures. Construction and Building Materials, p. 288, 123305. doi:10.1016/j.conbuildmat.2021.123305

11. MDPI. (2020, May 11). A novel approach for kinematic analysis and simulation of a 3-DOF parallel manipulator using screw theory. Applied Sciences, 10(9), 3324. https://www.mdpi.com/2076-3417/10/9/3324

12. Zhang, Y., Wang, X., Ji, G., Fan, Z., Guo, Y., Gao, W., & Xin, L. (2020). Mechanical Performance Characterization of Lignin-Modified Asphalt Mixture. Applied Sciences, 10(9), 3324. https://doi.org/10.3390/app10093324

13. Kalampokis, S., Papamoschou, M., Kalama, D. M., Pappa, C. P., Manthos, E., & Triantafyllidis, K. S. (2022). Investigation of the Characteristic Properties of Lignin-Modified Bitumen. CivilEng, 3(3), 734–747. https://doi.org/10.3390/civileng3030042

14. Batista, K., Padilha, R., Castro, T., Silva, C., Araújo, M., Leite, L., Pasa, V., & Lins, V. (2018). High-temperature, low-temperature and weathering aging performance of lignin-modified asphalt binders. Industrial Crops and Products, 111, 107-116. https://doi.org/10.1016/j.indcrop.2017.10.010

15. Gaudenzi, E., Cardone, F., Lu, X. et al. Performance assessment of asphalt mixtures produced with a bio-binder containing 30% of lignin. Mater Struct 55, 221 (2022). https://doi.org/10.1617/s11527-022-02057-

16. Gaudenzi, E., Cardone, F., Lu, X., & Canestrari, F. (2023). The use of lignin for sustainable asphalt pavements: A literature review. Construction and Building Materials, 362, 129773. https://doi.org/10.1016/j.conbuildmat.2022.129773

17. Wu, J., Liu, Q., Wang, C., Wu, W., & Han, W. (2021). Investigation of lignin as an alternative extender of bitumen for asphalt pavements. Journal of Cleaner Production, 283, 124663. https://doi.org/10.1016/j.jclepro.2020.124663.

18. Su, Y., Tang, S., Cai, M., Nie, Y., Hu, B., Wu, S., & Cheng, C. (2023). Thermal oxidative aging mechanism of lignin-modified bitumen. Construction and Building Materials, 363, 129863. https://doi.org/10.1016/j.conbuildmat.2022.129863

19. Website of Pavement Interactive: https://pavementinteractive.org/reference-desk/materials/asphalt/. Accessed on April 25, 2025

20. Website of Infinity Galaxy: http://www.infinityexport.org/en/article/163/penetrationgrade-bitumen.aspx. Accessed on September 15, 2018

21. Mamlouk, M. S. and Zaniewski, J. P., ―Materials for Civil and Construction engineers‖, Addison-Wesley, Menlo Park, California, 1999.

22. Asphalt Institute, Performance Graded Asphalt Binder Specification and Testing‖, SuperPave Series No. 1 (SP-1), Asphalt Institute, Lexington, Kentucky, 1996.

23. Empowering Roads, Fuelling Growth: Vivasvanna Exports Pvt Ltd. (2024, October 14). Superpave Technology Bitumen - Empowering Roads, fuelling Growth: Vivasvanna Exports Pvt Ltd. https://vivasvannaexports.com/products/superpave-technology-bitumen/

24. Pavement Interactive. (n.d.). Dynamic shear rheometer. Pavement Interactive. Retrieved April 22, 2025, from https://pavementinteractive.org/reference-desk/testing/binder-tests/dynamic-shear-rheometer/

25. Al-Falahat, W., Carret, J.-C., & Carter, A. (2021). Lignin-modified asphalt: Laboratory study – Phase 1 (Final Report). FPInnovations and LCMB-ÉTS. Internal report provided through direct communication.

26. Sydney PROD. (2019, April). Ontario’s default parameters for AASHTOWare Pavement Me Design Interim Report 2019. https://www.library.mto.gov.on.ca/SydneyPLUS/Sydney/Portal/default.aspx?component=AAAAIY&record=89a3febc-f471-4f73-8f3b-4a5c52068874

27. Website of Pavement Interactive: https://pavementinteractive.org/reference-desk/design/mix-design/marshall-mix-design/. Accessed on April 25, 2025.

28. Website of Pavement Interactive: https://pavementinteractive.org/reference-desk/design/mix-design/superpave-mix-design/. Accessed on April 25, 2025.

29. AASHTO (2008). Mechanistic-Empirical Pavement Design Guide United States of America, USA: American Association of State Highway and Transportation Officials.

30. Vallerga, B.A., Monismith, C.L. and Grantham, K., A Study of Some Factors Influencing the Weathering of Paving Asphalts‖, Proceedings AAPT, Vol. 26, 1957.36.

31. Traxler, R.N., “Durability of Asphalt Cements”, Proceedings AAPT, Vol. 32, 1963.

32. Asphalt Binder Testing Capabilities. https://rowancreates.org/facilities/rucom/asphalt-binder-testing-capabilities.html

33. Pavement Interactive. (n.d.). Indirect tensile strength test. Retrieved April 25, 2025, from https://pavementinteractive.org/reference-desk/testing/asphalt-tests/indirect-tensile-strength-test/

34. Pavement Interactive. (n.d.). Fatigue test: Four-point bending beam. Retrieved April 25, 2025, from https://pavementinteractive.org/reference-desk/testing/asphalt-tests/fatigue-test-four-point-bending/

35. Pavement Interactive. (n.d.). Asphalt Mixture Performance Tester (AMPT). Retrieved April 25, 2025, from https://pavementinteractive.org

36. De la Torre, M. J., Moral, A., Hernández, M. D., Cabeza, E., & Tijero, A. (2013). Organosolv lignin for biofuel. Industrial Crops and Products, 45, 58-63. https://doi.org/10.1016/j.indcrop.2012.12.002

37. Kalampokis, S., Papamoschou, M., Kalama, D. M., Pappa, C. P., Manthos, E., & Triantafyllidis, K. S. (2020). Investigation of the Characteristic Properties of Lignin-Modified Bitumen. In E. Chailleux, A. A. A. Molenaar, & H. Di Benedetto (Eds.), RILEM International Symposium on Bituminous Materials (pp. 1563-1569). Springer. https://link.springer.com/chapter/10.1007/978-3-030-40663-9_2

38. FPInnovations. (2021). Lignin-modified asphalt binder: Laboratory evaluation of rheological properties. FPInnovations Internal Report. Provided via direct communication

39. Roberts, F. L., Knadhal, P. S., Brown, E. R. and Lee, D.Y., Kennedy, T. W., ―Hot Mix Asphalt Materials, Mixture Design, and Construction‖, NAPA Education Foundation, Lanham, Maryland, 1996.

40. Finn, F. N., Factors Involved in the Design of Asphaltic Pavement Surfaces‖, H.R.B., NCHRP Report 39, 1967.

41. Kandhal, P.S. and Wenger, M.E.,Asphalt Properties concerning Pavement Performance‖, TRB, Transportation Research Record 544, 1975.

42. FPInnovations. (2021). Lignin-modified asphalt binder: Laboratory evaluation of rheological properties. FPInnovations Internal Report. Provided via direct communication.

43. Norgbey, E.; Huang, J.; Hirsch, V.; Liu, W.J.; Wang, M.; Ripke, O.; Li, Y.; Takyi Annan, G.E.; Ewusi-Mensah, D.; Wang, X.; et al. We are unravelling the efficient use of waste lignin as a bitumen modifier for sustainable roads. Constr. Build. Mater. 2020, 230, 116957.

44. Bradley, A. H., & Thiam, P. (2021). Development of a Lignin-modified Bitumen for Canadian Asphalt Pavements. Trid.trb.org. https://trid.trb.org/view/1887378

45. Gaudenzi, R., Zofka, A., & Osei, A. (2023). Performance evaluation of bio-modified asphalt binders and mixtures: An experimental study. Transportation Research Record: Journal of the Transportation Research Board, 2677(2), 85–97. https://doi.org/10.1177/03611981231160908

46. García, A., González Alriols, M., Spigno, G., & Labidi, J. (2012). Lignin is a natural radical scavenger. Effect of the obtaining and purification processes on the antioxidant behaviour of lignin. Biochemical Engineering Journal, 67, 173-185. https://doi.org/10.1016/j.bej.2012.06.013

47. Zhang, Y., Wang, X., Ji, G., Fan, Z., Guo, Y., Gao, W., & Xin, L. (2020). Mechanical performance characterization of lignin-modified asphalt mixture. Applied Sciences, 10(9), 3324.

48. Xu, C., Wang, D., Zhang, S., Guo, E., Luo, H., Zhang, Z., & Yu, H. (2021). Effect of lignin modifier on the engineering performance of bituminous binder and mixture. Polymers, 13(7), 1083.

49. Arafat, S.; Kumar, N.; Wasiuddin, N.M.; Owhe, E.O.; Lynam, J.G. Sustainable lignin enhances asphalt binder oxidative aging and mix properties. J. Clean. Prod. 2019, 217, 456–468

50. Kou, C., Wu, X., Xiao, P., Liu, Y., & Wu, Z. (2020). Physical, rheological, and morphological properties of asphalt reinforced by basalt fibre and lignin fibre. Materials, 13(11), 2520.

51. Tokede, O. O., Whittaker, A., Mankaa, R., & Traverso, M. (2020). Life cycle assessment of asphalt variants in infrastructures: The case of lignin in Australian road pavements. Structures, 25, 190-199. https://doi.org/10.1016/j.istruc.2020.02.026.

52. Batista, K.B.; Padilha, RPL; Castro, T.O.; Silva, CFSC; Araújo, MFAS; Leite, L.F.M.; Pasa, VMD; Lins, V.F.C. High temperature, low-temperature and weathering aging performance of lignin modified asphalt binders. Ind. Crop. Prod. 2018, 111,107–116.

53. Norgbey, E.; Huang, J.; Hirsch, V.; Liu, W.J.; Wang, M.; Ripke, O.; Li, Y.; Takyi Annan, G.E.; Ewusi-Mensah, D.; Wang, X.; et al. We are unravelling the efficient use of waste lignin as a bitumen modifier for sustainable roads. Constr. Build. Mater. 2020, 230, 116957.

54. Xu C.; Wang D.; Zhang S.; Guo E.; Luo E; Zhang Z.; Yu H. Effect of Lignin Modifier on Engineering Performance of Bituminous Binder and Mixture. Polymers 2021, 13, 1083.

55. Xu, G.; Wang, H.; Zhu, H. Rheological properties and anti-aging performance of asphalt binder modified with wood lignin. Constr. Build. Mater. 2017, 151, 801–808.

56. Arafat, S.; Kumar, N.; Wasiuddin, N.M.; Owhe, E.O.; Lynam, J.G. Sustainable lignin enhances asphalt binder oxidative aging and mix properties. J. Clean. Prod. 2019, 217, 456–468.

57. Yu, H.; Zhu, Z.; Leng, Z.; Wu, C.; Zhang, Z.; Wang, D.; Oeser, M. Effect of mixing sequence on asphalt mixtures containing waste tire rubber and warm mix surfactants. J. Cleaner Product. 2019, 246, 119008.

58. Wang, D.; Cai, Z.; Zhang, Z.; Xu, X.; Yu, H. Laboratory Investigation of Lignocellulosic Biomass as Performance Improver for Bituminous Materials. Polymers 2019, 11, 1253.

59. Rezazad Gohari, S., Quayyum, S. A., & Hossain, M. (2023). Effects of mixing protocol on the chemo-thermal characteristics of lignin-modified bitumen. Construction and Building Materials, 376, 131184. https://doi.org/10.1016/j.conbuildmat.2023.131184

60. Feaster, T. C. (2018). Characterization of Field-Produced HMA Mixtures from Nevada for Mechanistic-Empirical Pavement Design. https://core.ac.uk/download/304651780.pdf

61. Pavement Interactive. (n.d.). Rotational viscometer. Pavement Interactive. Retrieved April 22, 2025, from https://pavementinteractive.org/reference-desk/testing/binder-tests/rotational-viscometer/

62. FPInnovations. (2021). *Kraft lignin for asphalt binder modification – Internal testing results summary* [Unpublished internal report]. FPInnovations.

Appendices

	
Figure 13: Pan and Scale used for measuring Marshall Samples	Figure 11:Samples made for BRD

Figure 12:Samples made for MRD

Figure 13:Kraft Lignin provided by FPInnovations

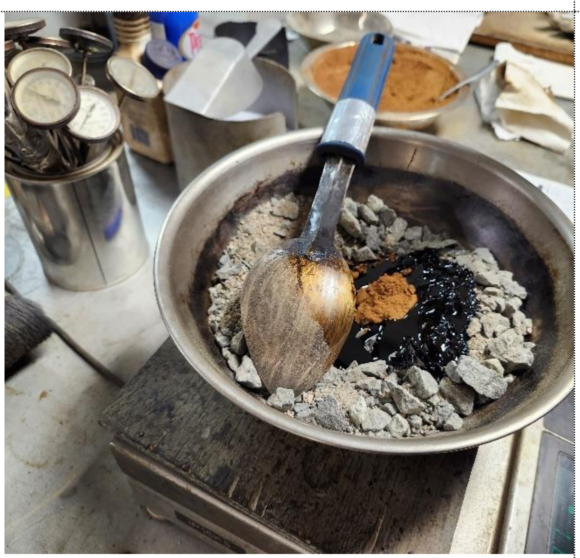


Figure 14:Initial mixing of bitumen, aggregate, and lignin additive for sample preparation

Figure 15:– Mold being prepared for heating

Figure 16:Thermometer inserted to monitor mixture temperature before compaction

Figure 17:– Compact Marshall specimens labeled for identification based on lignin content.

Figure 18:Vacuum saturation of compacted specimens as part of moisture conditioning .

Figure 19:Samples placed in a water bath for temperature conditioning

Figure 20:Combined aggregate blend after gradation adjustment, ready for mixing with binder.

Figure 21: Pouring the bitumen and lignin blend into the mixing bucket for preparation

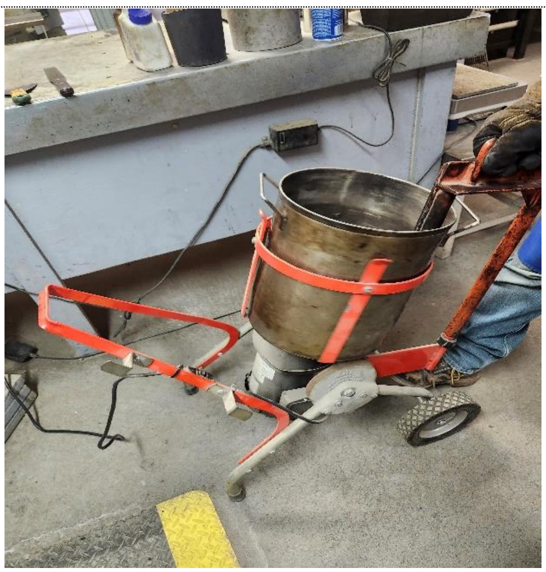


Figure 22: Mechanical mixing is ongoing

Figure 23Observation of the homogeneous mixing process of aggregate, bitumen, and lignin

Figure 24: Batched mixtures prepared in trays for short-term aging or compaction.

Figure 25: Splitting of the hot-mix asphalt using a mechanical splitter to create consistent sample batches.

Figure 26:Weighing samples after splitting

Figure 27: Flattening of hot mix asphalt during tray loading for short-term aging.

Figure 28: Gyratory compactor used to compact specimens at N_design gyrations under controlled pressure and angle.

Figure 29: Cooling samples after heating

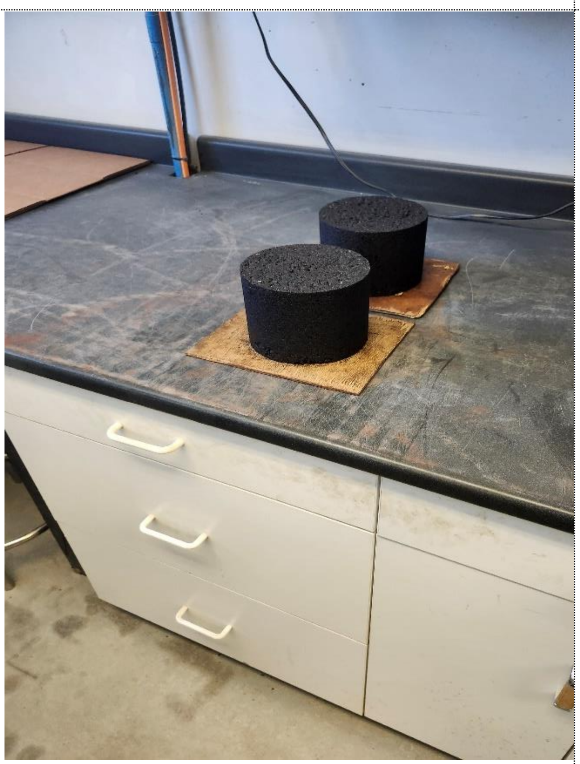


Figure 30: Cooling off molds

Figure 31MTO reference table

Figure 32: Gyrator Compactor display

Figure 33: Labeling the molds

Figure 34: Vacuum saturation of compacted specimens as part of moisture conditioning

Data Sheets

MARSHALL - WORKSHEET (LS-262, LS-263, LS-264)

PIONEER CONSTRUCTION

Date:	Jan 28/2025	Test Lab:	Thunder Bay Lab	Reviewed by:	Tyler McCoy

A	Mass of Compacted Specimen in Air	1296.9	1297.7	1300.1	
B	Surface Dry Mass of Specimen After Immersion in Water	1297.7	1298.6	1300.9	
C	Mass of Compacted Specimen in Water	783.6	787.9	786.9	
D	Volume = **B - C**	514.1	510.7	514.0	
E	Bulk Relative Density = **A/D**	2.523	2.541	2.529	
E_1	Water Temperature = (25 ° C) BRD Corrected for Temperature	2.523	2.541	2.529	2.531
	Beaker No.	1.0		2.0	
F	Mass of Beaker and Mixture in Air	2210.3		2212.9	
G	Mass of Beaker in Air	632.8		634.8	
H	Mass of Mixture in Air = **F-G**	1577.5		1578.1	
I	*Surface Dry Mass of Mixture in Air*				
J	Mass of Beaker and Mixture in water	1536.3		1540.1	
K	Mass of Beaker in Water	552.8		554.6	
L	Mass of Mixture in Water = **J - K**	983.5		985.5	
M	Volume =**H -L**	594.0		592.6	
N	*S.S.D Volume = **I - L***				
O	Maximum Relative Density = **H / M**	2.656		2.663	
O_1	Water Temperature = (25 ° C) MRD Corrected for Temperature	2.656		2.663	2.660
P	*S.S.D Maximum Relative Density = **H / N***				
Q	Percent Voids in Mixture = **$(O_1 - E_1)/O_1$*100**				4.8
R	*S.S.D % Voids in Mixture = **$(P - E_1)/P$*100***				

Contract No.:			Date Sampled:		
Dry	Medium Dry	Medium	Medium Rich	Rich	Surface Flooded

Remarks: **HL-4 Virgin Mix**

Stability and Flow Determination

Briquette Number	0	Test Lab:	0	Average
Time (seconds)				
a-Value (number)				
b - Dial Reading	233	266	269	
Flow a-b/100				
Flow (0.1)	10.0	9.0	9.0	9.3
Stability (N)	9723	11028	11146	
Volume Correction	1.00	1.00	1.00	
Corrected Stability	9723	11028	11146	10632

Gsb value from Mix Design	Gsb	2.865
Asphalt Cement Content value from corresponding Extraction Test	A.C. Content	5.00
	VMA	16.1

$$VMA = 100 - \left[E_1 \frac{(100 - \%A.C)}{Gsb}\right]$$

Volume	Correction
457 - 470	1.19
471 - 482	1.14
483 - 495	1.09
496 - 508	1.04
509 - 522	1.00
523 - 535	0.96
536 - 546	0.93
547 - 559	0.89

Temp.	Correction
20	1.001162
21	1.000950
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467
28	0.999187
29	0.998898
30	0.998599

Date Approved: October 19, 2022

Revision: 1

F2.2.07

"More Information Available Upon Request"

PIONEER CONSTRUCTION

MARSHALL - WORKSHEET (LS-262, LS-263, LS-264)

Date:	Jan 28/2025	Test Lab:	Thunder Bay Lab	Reviewed by:	Tyler McCoy

A	Mass of Compacted Specimen in Air	1292.6	1299.6	1300.7	
B	Surface Dry Mass of Specimen After Immersion in Water	1294.4	1301.2	1302.1	
C	Mass of Compacted Specimen in Water	783.3	785.8	786.4	
D	Volume = **B - C**	511.1	515.4	515.7	
E	Bulk Relative Density = **A/D**	2.529	2.522	2.522	
E_1	Water Temperature = (25 °C) BRD Corrected for Temperature	2.529	2.522	2.522	2.524
	Beaker No.	1.0		2.0	
F	Mass of Beaker and Mixture in Air	2210.8		2212.6	
G	Mass of Beaker in Air	632.8		634.8	
H	Mass of Mixture in Air = **F-G**	1578.0		1577.8	
I	*Surface Dry Mass of Mixture in Air*				
J	Mass of Beaker and Mixture in water	1537.5		1540.0	
K	Mass of Beaker in Water	552.8		554.6	
L	Mass of Mixture in Water = **J - K**	984.7		985.4	
M	Volume = **H -L**	593.3		592.4	
N	*S.S.D Volume* = ***I - L***				
O	Maximum Relative Density = **H / M**	2.660		2.663	
O_1	Water Temperature = (25 °C) MRD Corrected for Temperature	2.660		2.663	2.662
P	*S.S.D Maximum Relative Density* = ***H / N***				
Q	Percent Voids in Mixture = **$(O_1 - E_1)/O_1*100$**				5.2
R	*S.S.D % Voids in Mixture* = ***$(P - E_1)/P*100$***				

Contract No.:			Date Sampled:		
Dry	Medium Dry	Medium	Medium Rich	Rich	Surface Flooded

Remarks: HL-4 10% Lignin Mix

Stability and Flow Determination

Briquette Number	0	Test Lab:	0	Average
Time (seconds)				
a-Value (number)				
b - Dial Reading	226	270	267	
Flow **a-b/100**				
Flow (0.1)	8.0	11.0	9.0	9.3
Stability (N)	9446	11186	11067	
Volume Correction	1.00	1.00	1.00	
Corrected Stability	9446	11186	11067	10566

Gsb value from Mix Design	Gsb	2.865
Asphalt Cement Content value from corresponding Extraction Test	A.C. Content	5.00
	VMA	16.3

$$VMA = 100 - \left[E_1\frac{(100 - \%A.C)}{Gsb}\right]$$

Volume	Correction
457 - 470	1.19
471 - 482	1.14
483 - 495	1.09
496 - 508	1.04
509 - 522	1.00
523 - 535	0.96
536 - 546	0.93
547 - 559	0.89

Temp.	Correction
20	1.001162
21	1.000950
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467
28	0.999187
29	0.998898
30	0.998599

F2.2.07

Date Approved: October 19, 2022

Revision: 1

"More Information Available Upon Request"

PIONEER CONSTRUCTION

MARSHALL - WORKSHEET (LS-262, LS-263, LS-264)

Date:	Feb 4/2025	Test Lab:	Thunder Bay Lab	Reviewed by:	Tyler McCoy

A	Mass of Compacted Specimen in Air	1300.3	1300.2	1300.9	
B	Surface Dry Mass of Specimen After Immersion in Water	1301.5	1301.9	1302.5	
C	Mass of Compacted Specimen in Water	788.3	787.7	787.7	
D	Volume = **B - C**	513.2	514.2	514.8	
E	Bulk Relative Density = **A/D**	2.534	2.529	2.527	
E_1	Water Temperature = (25 °C) BRD Corrected for Temperature	2.534	2.529	2.527	2.530
	Beaker No.	1.0		2.0	
F	Mass of Beaker and Mixture in Air	2210.8		2212.8	
G	Mass of Beaker in Air	632.8		634.8	
H	Mass of Mixture in Air = **F-G**	1578.0		1578.0	
I	*Surface Dry Mass of Mixture in Air*				
J	Mass of Beaker and Mixture in water	1539.0		1542.5	
K	Mass of Beaker in Water	552.8		554.6	
L	Mass of Mixture in Water = **J - K**	986.2		987.9	
M	Volume =**H -L**	591.8		590.1	
N	*S.S.D Volume* = ***I - L***				
O	Maximum Relative Density = **H / M**	2.666		2.674	
O_1	Water Temperature = (25 °C) MRD Corrected for Temperature	2.666		2.674	2.670
P	*S.S.D Maximum Relative Density* = ***H / N***				
Q	Percent Voids in Mixture = **$(O_1 - E_1)/O_1*100$**				5.2
R	*S.S.D % Voids in Mixture* = ***$(P - E_1)/P*100$***				

Contract No.:			Date Sampled:		
Dry	Medium Dry	Medium	Medium Rich	Rich	Surface Flooded

Remarks: HL-4 20% Lignin Mix

Stability and Flow Determination

Briquette Number	0	Test Lab:	0	Average
Time (seconds)				
a-Value (number)				
b - Dial Reading	294	314	315	
Flow a-b/100				
Flow (0.1)	10.0	11.0	10.0	10.3
Stability (N)	12135	12945	12986	
Volume Correction	1.00	1.00	1.00	
Corrected Stability	12135	12945	12986	12689

Gsb value from Mix Design	Gsb	2.865
Asphalt Cement Content value from corresponding Extraction Test	A.C. Content	5.00
	VMA	16.1

$$VMA = 100 - [E_1 \frac{(100 - \% A.C)}{Gsb}]$$

Volume	Correction
457 - 470	1.19
471 - 482	1.14
483 - 495	1.09
496 - 508	1.04
509 - 522	1.00
523 - 535	0.96
536 - 546	0.93
547 - 559	0.89

Temp.	Correction
20	1.001162
21	1.000950
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467
28	0.999187
29	0.998898
30	0.998599

Date Approved: October 19, 2022

Revision: 1

F2.2.07

"More Information Available Upon Request"

MARSHALL - WORKSHEET (LS-262, LS-263, LS-264)

Date:	Feb 4/2025	Test Lab:	Thunder Bay Lab	Reviewed by:	Tyler McCoy

A	Mass of Compacted Specimen in Air	1301.5	1300.4	1299.2	
B	Surface Dry Mass of Specimen After Immersion in Water	1302.8	1306.6	1301.3	
C	Mass of Compacted Specimen in Water	784.7	782.9	781.0	
D	Volume = **B - C**	518.1	523.7	520.3	
E	Bulk Relative Density = **A/D**	2.512	2.483	2.497	
E_1	Water Temperature = (25 °C) BRD Corrected for Temperature	2.512	2.483	2.497	2.497
	Beaker No.	1.0		2.0	
F	Mass of Beaker and Mixture in Air	2210.6		2212.5	
G	Mass of Beaker in Air	632.8		634.8	
H	Mass of Mixture in Air = **F-G**	1577.8		1577.7	
I	*Surface Dry Mass of Mixture in Air*				
J	Mass of Beaker and Mixture in water	1541.4		1544.4	
K	Mass of Beaker in Water	552.8		554.6	
L	Mass of Mixture in Water = **J - K**	988.6		989.8	
M	Volume =**H -L**	589.2		587.9	
N	*S.S.D Volume = **I - L***				
O	Maximum Relative Density = **H / M**	2.678		2.684	
O_1	Water Temperature = (25 °C) MRD Corrected for Temperature	2.678		2.684	2.681
P	*S.S.D Maximum Relative Density = **H / N***				
Q	Percent Voids in Mixture = $(O_1 - E_1)/O_1*100$				6.9
R	*S.S.D % Voids in Mixture = $(P - E_1)/P*100$*				

Contract No.: | Date Sampled:

Dry	Medium Dry	Medium	Medium Rich	Rich	Surface Flooded

Remarks: **HL-4 30% Lignin Mix**

Stability and Flow Determination

Briquette Number	0	Test Lab:	0	Average
Time (seconds)				
a-Value (number)				
b - Dial Reading	331	265	256	
Flow a-b/100				
Flow (0.1)	10.0	9.0	10.0	9.7
Stability (N)	13640	10988	10632	
Volume Correction	1.00	0.96	1.00	
Corrected Stability	13640	10549	10632	11607

Gsb value from Mix Design	Gsb	2.865
Asphalt Cement Content value from corresponding Extraction Test	A.C. Content	5.00
	VMA	17.2

$$VMA = 100 - [E_1 \frac{(100 - \% A.C)}{Gsb}]$$

Volume	Correction
457 - 470	1.19
471 - 482	1.14
483 - 495	1.09
496 - 508	1.04
509 - 522	1.00
523 - 535	0.96
536 - 546	0.93
547 - 559	0.89

Temp.	Correction
20	1.001162
21	1.000950
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467
28	0.999187
29	0.998898
30	0.998599

Date Approved: October 19, 2022 Revision: 1 F2.2.07

"More Information Available Upon Request"

Superpave - WORKSHEET

		N_{design} 1	N_{design} 2	N_{max} 3	N_{max} 4
A	Mass of Compacted Specimen in Air	5055.0	5069.4		
B	Surface Dry Mass of Specimen After Immersion in Water	5055.6	5070.5		
C	Mass of Compacted Specimen in Water	3114.0	3123.3		
D	Volume = **B - C**	1941.6	1947.2	0.0	0
E	Bulk Specific Gravity of Mix = **A/D**	2.604	2.603	#DIV/0!	#DIV/0!
	Water Temperature = 25 ° C Gmb Corrected for Temperature	2.604	2.603	#DIV/0!	#DIV/0!
E_1	Average	2.603		#DIV/0!	
	Beaker No.	1.0	2.0		
F	Mass of Beaker and Mixture in Air	2169.1	2191.7		
G	Mass of Beaker in Air	632.8	634.8		
H	Mass of Mixture in Air = **F-G**	1536.3	1556.9		
J	Mass of Beaker and Mixture in water	1511.9	1527.4		
K	Mass of Beaker in Water	552.8	554.6		
L	Mass of Mixture in Water = **J - K**	959.1	972.8		
M	Volume =**H -L**	577.2	584.1		
O	Maximum Specific Gravity of Mix = **H / M**	2.662	2.665		
	Water Temperature= 25 °C Gmm Corrected for Water Temperature	2.662	2.665		
O_1	Average	2.664			
Q	Percent Voids in Mixture = **$(O_1 - E_1)/O_1$*100**	2.3		#DIV/0!	

Sample No.:		Mix Type:	SP 12.5mm Virgin
Contract No.		Date Sampled:	Feb 18/2025
Lot:		Sublot:	
Test Lab:	Thunder Bay Lab		
Remarks:			

G_{mb} Heights @

Specimen No	1	2	3	4
$N_{initial}$	120.5	121.2		
N_{design}	111.9	112.4		
N_{max}				
C=	0.929	0.927	#####	#####

G_{mb} @ $N_{initial}$

Specimen No	1	2	3	4
	2.419	2.413	#####	#####

Percent G_{mm} @

Specimen No	1	2	Average	Specification
$N_{initial}$	90.8	90.6	90.7	≤ 89
N_{design}	97.7	97.7	97.7	96.0
N_{max}	####	#####	#DIV/0!	≤ 98

Volumetric Properties

P_{mm}	100	
P_b	5.00	
G_b	1.017	
G_{se}	2.912	
G_{sb}	2.881	
P_{ba}	0.38	
P_s	95.0	
P_{be}	4.64	
DP	#RE	0.6-1.2
VMA	14.2	14
VFA	83.7	65-75

Reviewed by: ____________

More Information Available Upon Request

Temp.	Correction
20	1.001162
21	1.00095
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467

Effective Specific Gravity of Aggregate (Gse) $G_{se} = \frac{P_{mm} - P_b}{\frac{P_{mm}}{G_{mm}} - \frac{P_b}{G_b}}$

Dust Proportion (DP) $DP = \frac{P_{0.075}}{P_{be}}$

C Factor (C) $C = \frac{\text{Height @ } N_{design}}{\text{Height @ } N_{initial}}$

Percent Asphalt Absorbed by mass of Aggregate (Pba) $P_{ba} = 100x \frac{G_{se} - G_{sb}}{G_{sb}G_{se}} x G_b$

Void in Mineral Aggregate (VMA) $VMA = 100 - \left(G_{mb} \frac{(100 - P_b)}{G_{sb}}\right)$

G_{mb} @ $N_{initial}$ = C x(G_{mb} @ N_{design})

Percent Effective Asphalt Content of Mix (Pbe) $P_{be} = P_b - \frac{P_{ba}}{100} x P_s$

Filled with Asphalt (VFA) $VFA = 100x \frac{(VMA - Air Voids)}{VMA}$

$\% G_{mm} = \frac{G_{mb}}{G_{mm}} x 100$

Date Approved: January 3, 2022 Revision: 2 2.2.34

Superpave - WORKSHEET

		N_{design} 1	N_{design} 2	N_{max} 3	N_{max} 4
A	Mass of Compacted Specimen in Air	5071.0	5070.0		
B	Surface Dry Mass of Specimen After Immersion in Water	5073.2	5073.2		
C	Mass of Compacted Specimen in Water	3113.4	3112.7		
D	Volume = **B - C**	1959.8	1960.5	0.0	0
E	Bulk Specific Gravity of Mix = **A/D**	2.588	2.586	#DIV/0!	#DIV/0!
	Water Temperature = 25 ° C Gmb Corrected for Temperature	2.588	2.586	#DIV/0!	#DIV/0!
E_1	Average	2.587		#DIV/0!	

	Beaker No.	1.0	2.0		
F	Mass of Beaker and Mixture in Air	2174.5	2182.7		
G	Mass of Beaker in Air	632.8	634.8		
H	Mass of Mixture in Air = **F-G**	1541.7	1547.9		
J	Mass of Beaker and Mixture in water	1516.0	1521.9		
K	Mass of Beaker in Water	552.8	554.6		
L	Mass of Mixture in Water = **J - K**	963.2	967.3		
M	Volume =**H -L**	578.5	580.6		
O	Maximum Specific Gravity of Mix = **H / M**	2.665	2.666		
	Water Temperature= 25 °C Gmm Corrected for Water Temperature	2.665	2.666		
O_1	Average	2.666			
Q	Percent Voids in Mixture = **(O_1 - E_1)/O_1*100**	3.0		#DIV/0!	

Sample No.:		Mix Type:	SP 12.5mm (10%)
Contract No.		Date Sampled:	Feb 13/2025
Lot:	Sublot:		
Test Lab:	Thunder Bay Lab		
Remarks:			

G_{mb} Heights @

Specimen No	1	2	3	4
$N_{initial}$	121.5	122.1		
N_{design}	113.0	113.5		
N_{max}				
C=	0.930	0.930	#####	#####

G_{mb} @ $N_{initial}$

Specimen No	1	2	3	4
	2.406	2.405	#####	#####

Percent G_{mm} @

Specimen No	1	2	Average	Specification
$N_{initial}$	90.3	90.2	90.2	≤ 89
N_{design}	97.1	97.0	97.0	96.0
N_{max}	####	#####	#DIV/0!	≤ 98

Volumetric Properties

P_{mm}	100	
P_b	5.00	
G_b	1.017	
G_{se}	2.914	
G_{sb}	2.881	
P_{ba}	0.40	
P_s	95.0	
P_{be}	4.62	
DP	#RE	0.6-1.2
VMA	14.7	14
VFA	79.6	65-75

Reviewed by:

More Information Available Upon Request

Temp.	Correction
20	1.001162
21	1.00095
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467

Effective Specific Gravity of Aggregate (Gse) $G_{se} = \frac{P_{mm} - P_b}{\frac{P_{mm}}{G_{mm}} - \frac{P_b}{G_b}}$

Dust Proportion (DP) $DP = \frac{P_{0.075}}{P_{be}}$

C Factor (C) $C = \frac{\text{Height @ } N_{design}}{\text{Height @ } N_{initial}}$

Percent Asphalt Absorbed by mass of Aggregate (Pba) $P_{ba} = 100x \frac{G_{se} - G_{sb}}{G_{sb}G_{se}} x\, G_b$

Void in Mineral Aggregate (VMA) $VMA = 100 - \left(G_{mb} \frac{(100 - P_b)}{G_{sb}}\right)$

G_{mb} @ $N_{initial}$ = C x(G_{mb} @ N_{design})

Percent Effective Asphalt Content of Mix (Pbe) $P_{be} = P_b - \frac{P_{ba}}{100} x\, P_s$

Filled with Asphalt (VFA) $VFA = 100x \frac{(VMA - Air\ Voids)}{VMA}$

$\% G_{mm} = \frac{G_{mb}}{G_{mm}} x\, 100$

Date Approved: January 3, 2022 Revision: 2 2.2.34

Superpave - WORKSHEET

		N_{design} 1	N_{design} 2	N_{max} 3	N_{max} 4
A	Mass of Compacted Specimen in Air	5073.5	5073.5		
B	Surface Dry Mass of Specimen After Immersion in Water	5077.8	5080.6		
C	Mass of Compacted Specimen in Water	3104.0	3104.9		
D	Volume = **B - C**	1973.8	1975.7	0.0	0
E	Bulk Specific Gravity of Mix = **A/D**	2.570	2.568	#DIV/0!	#DIV/0!
	Water Temperature = 25 ° C Gmb Corrected for Temperature	2.570	2.568	#DIV/0!	#DIV/0!
E_1	Average	2.569		#DIV/0!	

	Beaker No.	3.0	4.0		
F	Mass of Beaker and Mixture in Air	2201.9	2213.7		
G	Mass of Beaker in Air	661.3	664.6		
H	Mass of Mixture in Air = **F-G**	1540.6	1549.1		
J	Mass of Beaker and Mixture in water	1541.3	1551.0		
K	Mass of Beaker in Water	577.6	580.3		
L	Mass of Mixture in Water = **J - K**	963.7	970.7		
M	Volume =**H -L**	576.9	578.4		
O	Maximum Specific Gravity of Mix = **H / M**	2.670	2.678		
	Water Temperature= 25 °C Gmm Corrected for Water Temperature	2.670	2.678		
O_1	Average	2.674			
Q	Percent Voids in Mixture = **$(O_1 - E_1)/O_1$*100**	3.9		#DIV/0!	

Sample No.:		Mix Type:	SP 12.5mm (20%)
Contract No.		Date Sampled:	Feb 13/2025
Lot:	Sublot:		
Test Lab:	Thunder Bay Lab		
Remarks:			

G_{mb} Heights @

Specimen No	1	2	3	4
$N_{initial}$	121.5	122.1		
N_{design}	113.0	113.5		
N_{max}				
C=	0.930	0.930	#####	#####

G_{mb} @ $N_{initial}$

Specimen No	1	2	3	4
	2.390	2.388	#####	#####

Percent G_{mm} @

Specimen No	1	2	Average	Specification
$N_{initial}$	89.4	89.3	89.3	≤ 89
N_{design}	96.1	96.0	96.1	96.0
N_{max}	####	#####	#DIV/0!	≤ 98

Volumetric Properties

P_{mm}	100	
P_b	5.00	
G_b	1.017	
G_{se}	2.925	
G_{sb}	2.881	
P_{ba}	0.53	
P_s	95.0	
P_{be}	4.50	
DP	#RE	0.6-1.2
VMA	15.3	14
VFA	74.5	65-75

Reviewed by: ____________

More Information Available Upon Request

Temp.	Correction
20	1.001162
21	1.00095
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467

Effective Specific Gravity of Aggregate (Gse) $G_{se} = \frac{P_{mm} - P_b}{\frac{P_{mm}}{G_{mm}} - \frac{P_b}{G_b}}$

Dust Proportion (DP) $DP = \frac{P_{0.075}}{P_{be}}$

C Factor (C) $C = \frac{\text{Height @ } N_{design}}{\text{Height @ } N_{initial}}$

Percent Asphalt Absorped by mass of Aggregate (Pba) $P_{ba} = 100x \frac{G_{se} - G_{sb}}{G_{sb}G_{se}} x\ G_b$

Void in Mineral Aggregate (VMA) $VMA = 100 - \left(G_{mb} \frac{(100 - P_b)}{G_{sb}}\right)$

G_{mb} @ $N_{initial}$ = C x(G_{mb} @ N_{design})

Percent Effective Asphalt Content of Mix (Pbe) $P_{be} = P_b - \frac{P_{ba}}{100} x\ P_s$

Filled with Asphalt (VFA) $VFA = 100x \frac{(VMA - \text{Air Voids})}{VMA}$

$\% G_{mm} = \frac{G_{mb}}{G_{mm}} x\ 100$

Date Approved: January 3, 2022 Revision: 2 2.2.34

Superpave - WORKSHEET

		N_{design} 1	N_{design} 2	N_{max} 3	N_{max} 4
A	Mass of Compacted Specimen in Air	5070.5	5070.5		
B	Surface Dry Mass of Specimen After Immersion in Water	5077.4	5078.7		
C	Mass of Compacted Specimen in Water	3092.1	3101.3		
D	Volume = **B - C**	1985.3	1977.4	0.0	0
E	Bulk Specific Gravity of Mix = **A/D**	2.554	2.564	#DIV/0!	#DIV/0!
	Water Temperature = 25 ° C Gmb Corrected for Temperature	2.554	2.564	#DIV/0!	#DIV/0!
E_1	Average	2.559		#DIV/0!	
	Beaker No.	3.0	4.0		
F	Mass of Beaker and Mixture in Air	2215.0	2191.8		
G	Mass of Beaker in Air	661.3	664.6		
H	Mass of Mixture in Air = **F-G**	1553.7	1527.2		
J	Mass of Beaker and Mixture in water	1552.2	1539.2		
K	Mass of Beaker in Water	577.6	580.3		
L	Mass of Mixture in Water = **J - K**	974.6	958.9		
M	Volume =**H -L**	579.1	568.3		
O	Maximum Specific Gravity of Mix = **H / M**	2.683	2.687		
	Water Temperature= 25 °C Gmm Corrected for Water Temperature	2.683	2.687		
O_1	Average	2.685			
Q	Percent Voids in Mixture = **$(O_1 - E_1)/O_1$*100**	4.7		#DIV/0!	

Sample No.:		Mix Type:	SP 12.5mm (30%)
Contract No.		Date Sampled:	Feb 18/2025
Lot:	Sublot:		
Test Lab:	Thunder Bay Lab		
Remarks:			

G_{mb} Heights @

Specimen No	1	2	3	4
$N_{initial}$	123.7	123.5		
N_{design}	114.7	114.5		
N_{max}				
C=	0.927	0.927	#####	#####

G_{mb} @ $N_{initial}$

Specimen No	1	2	3	4
	2.368	2.377	#####	#####

Percent G_{mm} @

Specimen No	1	2	Average	Specification
$N_{initial}$	88.2	88.5	88.4	≤ 89
N_{design}	95.1	95.5	95.3	96.0
N_{max}	####	#####	#DIV/0!	≤ 98

Volumetric Properties

P_{mm}	100	
P_b	5.00	
G_b	1.017	
G_{se}	2.939	
G_{sb}	2.881	
P_{ba}	0.70	
P_s	95.0	
P_{be}	4.34	
DP	#RE	0.6-1.2
VMA	15.6	14
VFA	69.9	65-75

Reviewed by: ____________________

More Information Available Upon Request

Temp.	Correction
20	1.001162
21	1.00095
22	1.000728
23	1.000495
24	1.000253
25	1.000000
26	0.999738
27	0.999467

Effective Specific Gravity of Aggregate (Gse) $G_{se} = \frac{P_{mm} - P_b}{\frac{P_{mm}}{G_{mm}} - \frac{P_b}{G_b}}$

Dust Proportion (DP) $DP = \frac{P_{0.075}}{P_{be}}$

C Factor (C) $C = \frac{\text{Height @ } N_{design}}{\text{Height @ } N_{initial}}$

Percent Asphalt Absorpted by mass of Aggregate (Pba) $P_{ba} = 100x \frac{G_{se} - G_{sb}}{G_{sb}G_{se}} \times G_b$

Void in Mineral Aggregate (VMA) $VMA = 100 - \left(G_{mb} \frac{(100 - P_b)}{G_{sb}}\right)$

G_{mb} @ $N_{initial}$ = C x(G_{mb} @ N_{design})

Percent Effective Asphalt Content of Mix (Pbe) $P_{be} = P_b - \frac{P_{ba}}{100} \times P_s$

Filled with Asphalt (VFA) $VFA = 100x \frac{(VMA - \text{Air Voids})}{VMA}$

$\% G_{mm} = \frac{G_{mb}}{G_{mm}} \times 100$

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