Asphaltenes-Modified Binders for High Modulus Asphalt Concrete Applications

by

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Abstract

Asphalt pavements are the most common type of pavements in North America. Increasing traffic demand, harsh weather conditions, and the tendency for infrastructure operators to reduce the cost (and, therefore, the frequency) of maintenance are the major reasons for asphalt performance improvement strategies.

High modulus asphalt concrete (HMAC) is an asphalt mixture of high quality designed to have a high strength, good fatigue life, and excellent rutting resistance. However, despite its superior performance, the application of HMAC is limited in cold climates due to its low flexibility and stress relaxation capacity. HMAC gains its superior mechanical performances from its key elements, which are a hard grade binder in conjunction with a strong continuous mineral skeleton. Polymer modified binders are asphalt binders commonly used in HMAC applications, however, the high cost of polymer modified binders as well as the high tendency of phase separation in these binders are the main concerns limiting their applications. On the other hand, sustainability and reduction in cost are the two main factors, which recently lead to incorporation of waste materials into modification of asphalt binders. Asphaltenes are considered as a waste of minimal value with minimal applications in the industry, with a relatively high rate of production in refineries, with the potential to be used in asphalt binder modification applications.

The current study mainly investigates the application of asphalt binders from different sources modified with asphaltenes for application as high modulus base courses in cold climates. Furthermore, different hard grade asphalt binders, with potential to be used in HMAC applications, are also studied and compared. Furthermore, in order to achieve a deeper understanding of Alberta oilsands asphalt binders, effects of distillation temperature and source of these binders are investigated on their performance at different temperatures. For this purpose, one crude oil binder and four asphalt binders from different Alberta oilsands bitumen sources were studied. Witczak regression model was used to determine the minimum performance grade to meet the dynamic modulus requirement for HMAC. Asphaltenes were used at different percentages for modification of different asphalt binders. A rolling thin film oven (RTFO), and a pressure aging vessel (PAV) were used to simulate different phases of aging which occurs during asphalt pavement service life.

Rheological properties of asphalt binders at high and intermediate temperatures were studied using a dynamic shear rheometer (DSR), while at low temperatures a bending beam rheometer (BBR) was used. The effect of asphaltenes modification on the viscosity of the binders was studied using a rotational viscometer. Also, the chemical composition of the asphaltenes-modified binders was determined by SARA analysis. Comparing common hard grade binders with asphaltenes-modified ones revealed that at in regions with cold climates, asphaltenes-modified binder would perform better. Results from rheological analysis indicated that the improving effects of asphaltenes on the performance of binders at high temperatures were relatively higher than its impact at low temperatures. Additionally, it was determined that binders from Alberta oilsands modified with asphaltenes could achieve the performance grade requirements for HMAC applications. Furthermore, from Alberta oilsands binders investigations, it was found that in spite of crude oil asphalts that require modification to achieve performance grade requirements for high modulus asphalt applications, using the right distillation temperature and source, oilsands bitumens were capable of achieving the binder requirements for HMAC applications without further modification.

Preface

This thesis is an original work conducted by Amirhossein Ghasemirad and under the supervision of Dr. Leila Hashemian, and co-supervision of Dr. Alireza Bayat.

Chapter 3 of this manuscript is submitted and published as "Ghasemirad, A., Bala, N., & Hashemian, L. (2020). High-Temperature Performance Evaluation of Asphaltenes-Modified Asphalt Binders. Molecules, 25(15), 3326".

A modified version of chapter 4, "Asphaltenes-Modified Binders for High Modulus Asphalt Concrete Applications", has been submitted to Transportation Research Board (TRB) conference that will be held in early 2021 in the United States, and also Transportation Research Record (TRR) journal.

A simple version of chapter 4, "Asphaltenes-Modified Binders for High Modulus Asphalt Concrete Applications", has been submitted to Canadian Technical Asphalt Association (CTAA) conference, which is going to be held in late 2020 in Canada.

Chapter 6, "High and Low Temperature Rheological Evaluation of Oilsands Asphalts Distilled at Different Temperatures", has been submitted to Canadian Journal of Civil Engineering (CJCE), and at this moment is under review.

I was responsible for most part of data collection, analysis, original draft preparation, software, and validation. Leila Hashemian was responsible for conceptualization, resources, supervision, and project administration, and assisted in data validation, and manuscript edits and reviews. Nura Bala assisted in data collection and validation, writing the manuscript, and manuscript edits and reviews.

To the memory of innocent hearts & souls lost in the tragedy of Flight PS752.

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Chapter 1. Introduction

1.1 Introduction

Currently as highway transportation is the most widely mode of transportation, pavements are now an essential asset for each country's development plan (1). Pavements can be categorized as asphaltic (or flexible) pavements, concrete (or rigid) pavements, and composite pavements (2, 3). Flexible pavements generally consist of an asphalt concrete layer placed over a base and/or a subbase layer supported by compacted soil, called subgrade (3). Rigid pavements typically consist of a portlant cement layer over the subgrade with or without a base layer (3). Composite pavements on the other hand happen usually in pavement rehabilitation, when a layer of portlant concrete is placed over a damaged asphalt concrete layer, or vice versa (3). According to survey in 2016, asphalt paved roads are the most common type of pavements in North America, composing about almost 94% of roads in the United States (4). It's also found that according to Canada's core public infrastructure survey in 2016, almost 50% of all roads in Canada were located in Ontario and Alberta, while Alberta (28.4%) and Saskatchewan (23.9%) accounted for the largest share of highways (5).

Pavements, especially asphalt pavements rather than concrete ones, are layered structures, with every layer functioning to decrease the stress within the load-bearing capacity of the layer underneath with an inferior quality (6). Figure 1-1 shows the conventional cross section of a flexible pavement (6). From top to bottom, the layers of a pavement structure are known as surface, binder, base, subbase, and subgrade; however, based on necessity and economy some of these courses might be discarded (3).

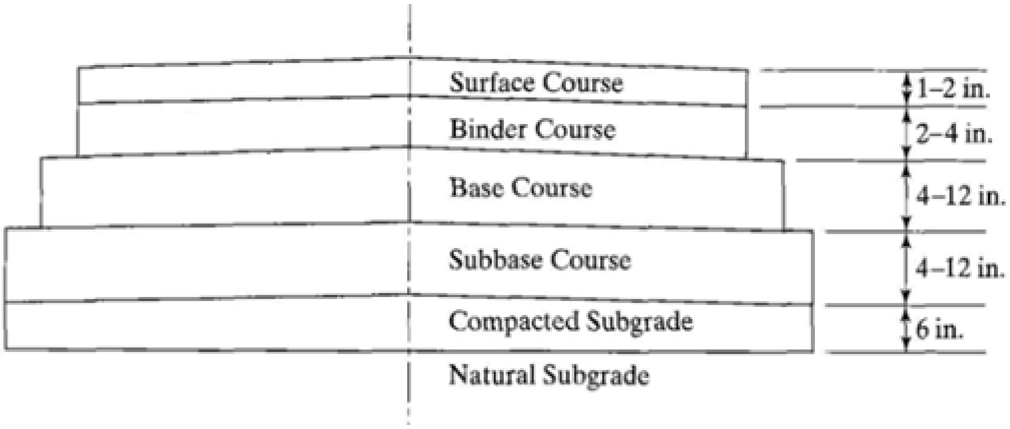


Figure 1-1. Typical Cross Section of a Conventional Flexible Pavement (6)

As can be seen in Figure 1-1 surface course (also known as wearing course) is the top layer in pavement structure. The asphalt mixture used in this layer is typically dense graded hot mix asphalt (HMA) (6). Moreover, this layer must be resistant against the distortion under traffic loading, waterproof to protect the whole system from moisture damage, and for the purpose of safety, it should provide a smooth and skid-resistant riding surface (6).

According to Figure 1-1, binder course is the layer placed below the surface course. There are mainly two reasons to use this layer in pavement structure. First, the wearing course is usually too thick to be compacted in a single layer; second, as this layer is not directly subjected to traffic loading or the destructive effects caused by water and oxidative hardening of asphalt binder, comparing to wearing course it has less binder content, and larger aggregates of lower quality (6).

Under the surface or binder course, the base course would be placed as shown in Figure 1-1. This layer is the primary load-carrying layer and hence most structurally important course in full-depth flexible pavement (7). The main responsibility of this layer is to dampen the stress transferring to subbase and subgrade from traffic loading. This layer should be resistant against permanent deformation, fatigue cracking caused by repeated loading, and thermal cracking when being exposed to low temperatures or intense temperature fluctuations (6). Typically base course has a dense graded aggregate structure, which can be composed of crushed stone, crushed slag, or other untreated or stabilized materials (8). When the pavement is going to experience heavy traffic loads, the base layer is usually hot mix asphalt (6).

The layer of material under the base course would be called the subbase course. The main reason to have two granular courses under the surface (or binder) course is to have a more economical design; comparing to base course, the aggregates used in subbase course are usually cheaper and of a lower quality that would be readily found locally (6).

Asphalt mixtures are mainly composed of asphalt binder, aggregates, and filler (1). In recent years, with increasing traffic loads and intense climatic conditions, addition of modifiers to asphalt binders/mixtures has become more conventional in order to prevent premature distresses in asphalt pavements (9, 10). Asphalt binder is a hydrocarbon product, which is produced in crude oil or oilsands refineries mainly through fractional distillation (11). After separation of lighter fractions (like liquid petroleum gas, gasoline, aviation fuel, kerosene, etc.), the heaviest fraction taken from crude oil distillation, which is a complex mixture of high molecular weight hydrocarbons, is processed further to obtain bitumen or asphalt binder (11).

Rehabilitation projects with depth constrains in urban areas back in 1980s made French engineers seek pavement materials with a higher modulus than conventional hot mix asphalts, in order to produce thinner layers but with the same service life (12). These efforts led to the introduction of high modulus asphalt concrete (HMAC), or Enrobé à Module Élevé (EME) in French (13). Although HMAC has been used in construction of surface courses (14), the main application of this type of mixture remains in base courses, where the tensile stress in pavement structure reaches its maximum state (15).

The key elements of HMAC mixes to ensure their superior performance, are hard grade binders combined with strong continuous dense-graded mineral skeletons (16). Using hard grade binder provides the mixture with high resistance to permanent deformations, while large content of asphalt binder and small content of air voids (closed structure) assure workability, fatigue durability, and water resistance (13, 14, 17, 18).

Hard binders used in production of HMAC mixes, also referred to as high modulus asphalt binders (HMAB), can be categorized into three different classes: hard grade binders, binders modified with natural asphalt, and polymer-modified binders (14, 17, 19, 20). The main concern about the application of straight run hard grade binders and those modified with natural asphalt is their poor performance at lower temperatures (21, 22). In case of polymer-modified binders, the final high cost on one hand, and poor storage stability on the other hand, limit the application of these binders in the base course, as the thickest layer of a pavement structure (23-25).

Unlike conventional mixture design methods which are based on volumetric properties, HMAC mix design is a performance-based design (26). In this regards, HMAC mixtures should be tested to ensure they meet several performance criteria including dynamic modulus, workability, durability, rutting resistance, and fatigue life (27).

There are several measures for the asphalt mixture stiffness, including dynamic modulus, flexural stiffness, creep compliance, relaxation modulus, and resilient modulus (28). Among these measures, dynamic modulus has a vast record of laboratory data for the test's input and output variables by different researchers over a considerable time, which makes the dynamic modulus prediction models more reliable (29). One of them main requirements for a high modulus asphalt concrete mixture, as the name implies, is for its dynamic modulus to be greater than 14,000 MPa at a loading frequency of 10 Hz, and a temperature of 15°C (27, 30). Difficulty to obtain dynamic modulus measurements in laboratory at extreme temperature and frequency conditions made researchers to develop predictive models using available mixture and binder data (28). These models proved to be useful in places with limited access to expensive laboratory facilities as well (31). These efforts lead to development of predictive regression models and more recently models based on artificial neural network (29, 32).

Sustainability and reduction in cost are the two main factors, which lead to incorporation of waste materials into modification of asphalt binders (33, 34). In HMAC area also there have been some efforts to use waste polymer in production of high modulus asphalt concrete, which yielded comparable results to those ones modified with commercial polymer modifiers (35).

According to polarity, asphalt binder can be divided into two general chemical groups of asphaltenes and maltenes (36), with asphaltenes being the most polar fraction of asphalt binder with high molecular weight (23, 37). Asphalt binder is a viscoelastic material, and polar fraction has been shown to be associated with the elastic part of binder's behavior, while non-polar fraction accounts for the viscous part (36). More asphaltenes content in asphalt binder could cause a decrease in penetration and an increase in softening point, which reflect an increase in asphalt stiffness (38).

Asphaltenes are considered as a waste of minimal value with minimal applications in the industry, with a relatively high rate of production in oil refineries. In northern Alberta facilities it's assumed that asphaltenes are produced at a rate of 17.5% of asphalts (39). Some efforts have been made to make use of asphaltenes (40), but they proved to be neither economical nor environmental-friendly (41). A more practical and sustainable solution seems to be necessary to create value out of this material.

1.2 Objectives

The aim of this research is to investigate and compare the effects of modification of asphalt binders with asphaltenes for high modulus asphalt concrete (HMAC) applications. Other specific objectives of this research are as follows:

- To investigate the application of asphaltenes, a waste material obtained from deasphalting operations in Alberta oilsands refineries, for improving the performance of high modulus base course in cold climates.
- To determine the rheological properties of hard grade asphalt binders, which are commonly used in HMAC base courses.
- To evaluates the rheological properties and chemical structure of different sources of asphalt binders from Alberta oilsands and compare their performance with those of a control crude oil binder.
- To investigate the potential of Alberta oilsands binders modified with asphaltenes for application in high modulus asphalt concrete base courses.

1.3 Methodology

In order to achieve the objectives of this study, different binders were collected from different sources. The base binders used in this study can be categorized into three groups: hard grade binders, straight-run binders from crude oil refineries, and binders from Alberta oilsands.

The 1999 Witczak model was used in this study to estimate the minimum performance grade (PG) requirement for the asphalt binder to be used in a HMAC mixture to meet the minimum dynamic modulus specification requirement for this type of mixture.

To modify asphalt binders with asphaltenes a high shear mixer a high shear mixer from Silverson Co., USA (Model L5M-A) was used to incorporate asphaltenes in the asphalt binder. To avoid aging during the modification process, the mixing temperature was controlled at 140±5°C using a hot plate. The required amount of asphaltenes powder was added to the heated binder, and mixed at a speed of 2000 rpm for 60 minutes to achieve a uniform dispersion in the mixture.

Short-term and long-term aging of asphalt binders were simulated in the laboratory using a rolling thin film oven (RTFO) and pressure aging vessel (PAV).

Dynamic mechanical analysis (DMA) was performed on the modified/unmodified asphalt binder using a dynamic shear rheometer (DSR) (Smartpave, Model 102, Anton Paar, Co., Ltd., Austria) to characterize the viscoelastic behaviour of the asphalt binders at high and intermediate temperatures. The rheological properties of the asphalt binders at low temperatures were evaluated using a bending beam rheometer (BBR) (Cannon Instrument Company®, USA). A Brookfield rotational viscometer was used to measure the viscosity of the asphalt binders at higher temperatures.

In order to study the chemical structure of asphalt binders, SARA (saturates, asphaltenes, resins, and aromatics) analysis was performed. This analysis consists of two main steps; solvent dissolution to separate asphaltenes from maltenes, followed by gravity-driven chromatography to separate different fractions of maltenes.

To verify the results from the 1999 Witczak model for the minimum asphalt binder performance grading requirements, dynamic modulus test was conducted according using a Universal Testing Machine (IPC global UTM-100).

Figure 1-2 shows the flowchart followed in this study in order to achieve the mentioned.

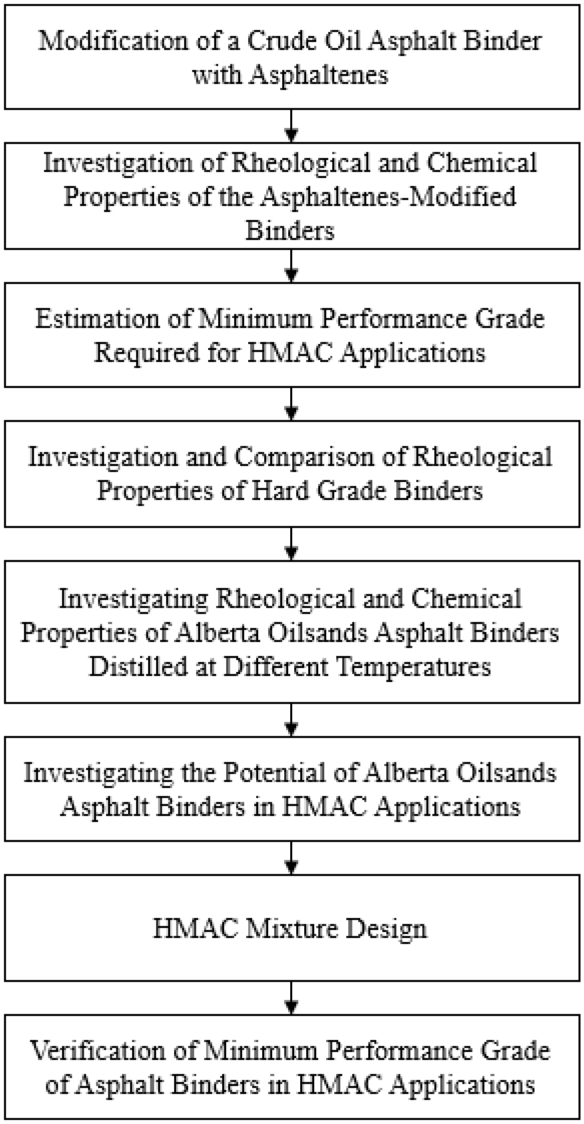


Figure 1-2. Flowchart of Different Steps Taken in This Research

1.4 Thesis Structure

This thesis is written in seven chapters with the following description for each chapter:

Chapter 1 – Introduction: In this chapter, a brief description of the whole research is presented, along with the objectives and the methodology followed towards achieving the objectives.

Chapter 2 – Literature Review: This chapter presents a comprehensive review on the background of high modulus asphalt concrete, its key elements and mixture design, asphalt binder chemical composition and their impacts on asphalt binder properties, asphaltenes and their use as modifiers in asphalt binder, and the background of different dynamic modulus prediction models.

Chapter 3 – High-Temperature Performance Evaluation of Asphaltenes-Modified Asphalt Binders: In this chapter, different contents of asphaltenes are added to a crude oil asphalt binder and the resulting changes in different properties of asphalt binder were investigated.

Chapter 4 – Asphaltenes-Modified Binders for High Modulus Asphalt Concrete Applications: This chapter studies asphaltenes-modified binders with different types of base binders either from crude oil or Alberta oilsands refineries. Also, the potential of these modified binders for high modulus asphalt concrete applications is verified using dynamic modulus test.

Chapter 5 – Hard Grade Asphalt Binders: This chapter tries to investigate and compare the rheological properties of hard grade binders from different sources, with penetration grades that make them potential candidates to be used for high modulus asphalt concrete applications. No further modification is applied on the binders from this chapter.

Chapter 6 – High and Low Temperature Rheological Evaluation of Oilsands Asphalts Distilled at Different Temperatures: In this chapter effects of distillation temperature on performance of asphalt binders from Alberta oilsands is investigated and compared with performance of commercially available crude oil source asphalts.

Chapter 7 – Summary and Conclusions: This chapter summarizes the idea, objectives, and scopes of this thesis. The most highlighted conclusions from all chapters, as well as the ideas for future research in this field are listed in this chapter.

Chapter 2. Literature Review

2.1 High Modulus Asphalt Concrete (HMAC)

Conventional asphalt binders can perform satisfactorily in asphalt pavements in majority of traffic and weather conditions (11). However, in some situations due to factors such as increasing traffic loads and weights, considerable temperature variations in some regions, and also the tendency to use thinner pavement layers and reduce the maintenance frequency, the performance limits of conventional asphalt binders have been reached (11). One approach to tackle these challenges is to modify asphalt binders with appropriate modifier (25, 42, 43).

The introduction of hard grade binders in asphalt industry of France back in 1960s provided the grounds for development of a new type of asphalt mixture (14). It was during 1980s that Laboratoire Central des Ponts et Chaussées (LCPC) in France, in cooperation with road enterprises introduced high modulus asphalt concrete (HMAC), or Enrobé à Module Élevé (EME) in French (13, 44). In the 1990s, the French developed a standard for EME mixtures (26). This standard considers two classes for EME mixtures. Class 1 is a low fatigue resistance mixture, while Class 2 has a higher fatigue resistant compared to the first class, with the main difference between these two classes being the binder content of the mixtures (27, 30).

As the name implies, high modulus asphalt concrete is a type of asphalt mixture whose main feature is high modulus or stiffness at intermediate temperatures (17). HMAC is designed to obtain an asphalt mixture with high mechanical performance, including high elastic stiffness, high deformation resistance and good fatigue resistance, with adequate workability, durability, and impermeability (18, 19, 45). Also in using HMAC in base courses, the thickness of the asphalt layers reduce up to 30% (18, 21, 46, 47), which would bring economic and environmental benefits by saving in quantity of raw materials used (14, 15, 44). On the other hand, the main concern about high modulus asphalt concrete application in pavements is their high potential to crack in cold climates (21, 48).

HMAC was initially used in France for reinforcement and rehabilitation projects where thinner layers were required due to depth restrictions (12). This limitation existed especially in urban areas where the excavation depth was limited due to frequent underground facilities and curbs (16, 30). Nowadays, high modulus base courses are widely used in many countries for different construction or rehabilitation applications such as heavily trafficked main routes, major urban roads, runways and taxiways, and long life pavement with life times more than 40 years (18, 49-52).

In pavement structures, the layers beneath surface course are subjected to the highest level of tensile stresses (15, 44, 53). The main application of high modulus asphalt mixtures is in binder and base layers (14, 30, 47). Although HMAC has been used in wearing courses as well (14, 47), in regions with rather cold climates it's been suggested not to use high modulus asphalt concrete in surface courses, as not only it may be prone to thermal cracking, but also regarding the lower air void contents of HMAC it may not provide enough surface texture with safe skid resistance (12, 18).

HMAC notable stiffness and load spreading ability (19, 54) minimizes the tensile strain at the bottom of the asphalt layer, and the compressive strain on top of the subgrade, even though the thickness of surface courses are generally thinner with HMAC base course (15, 55), which in return reduces the frequency and severity of structural distresses (56).

HMAC gains its superior mechanical performances from its constituents. The use of a hard grade binder in conjunction with a strong continuous mineral skeleton builds up a layer highly resistant to permanent deformation with a high capacity to absorb the loads transmitted by the road traffic (18, 22, 49, 57). To compensate the adverse effects of hard grade binder, larger content of binder and small content of air voids (closed structure) assure workability, fatigue durability, and water resistance in high modulus asphalt mixtures (13, 52).

The binders used in HMAC mixtures, also referred to as high modulus asphalt binder (HMAB), fall into three different categories. Traditional straight-run hard grade asphalt binder, asphalt binders modified with natural asphalt, and polymer-modified asphalt binders (14, 19, 20). Straight-run hard grade binders, usually with penetration grades up to 30, which have been widely used in base courses in Europe (12, 13, 45, 46, 53, 58, 59), have the main concern of poor performance at low temperatures (21, 22). This concern specially exists with air-blown hard grade binders (12). Natural asphalt, which mainly consists of lake asphalt and rock asphalt, is a highly viscous material that cannot be directly used as asphalt binder (17, 47, 60). Instead, to produce required penetration grades with higher modulus values, they are typically mixed with base binder (17, 20, 61, 62). Polyolefin and styrene butadiene styrene (SBS) are the most common polymers used in modification of asphalt binders for production of HMAC (17, 20, 47, 56, 63). Although presenting a higher fatigue and low temperature resistance, in comparison to hard grade binders, and with those modified with natural asphalt, polymer modified HMAB provide less dynamic modulus, higher shear strength, and rutting resistance (12, 60, 62). However, the high cost of polymer modified binders and using them in the thickest layers of a pavement on one hand (46, 64), and the high possibility of phase separation in polymer-modified binders on the other hand limit their application in HMAC base course (23-25).

2.2 High Modulus Asphalt Concrete Mixture Design

Unlike Marshall and Superpave mixture designs, which are mainly driven based on volumetric properties, HMAC mix design is mostly counted as a performance-based design (26). Performance-based mixture designs have been developed to evaluate the performance of asphalt mixtures under the real loading and environmental conditions that the mixture experience in the field (30). For designing a high modulus asphalt concrete mixture, a suitable aggregate gradation is developed using different aggregate fractions. Then asphalt binder content is calculated based on a minimum richness factor. Using this trial mixture design, Superpave gyratory specimens are compacted and tested to evaluate several performance aspects including workability, dynamic modulus, durability, rutting resistance, and fatigue life (27).

HMAC mixture design starts with development of a suitable well-graded aggregate gradation from different fractions. Then binder content is chosen based on richness factor and aggregate properties. Superpave gyratory compactor (SGC) is then used to compact trial sample to be tested for different specifications. Different performance tests can be done on the designed mixture to ensure about its satisfying performance, among which dynamic modulus test is one of the most important tests (27, 44).

2.2.1 Design Grading Formulation

Regarding the higher asphalt binder content in HMAC mixtures, this type of asphalt mix requires higher voids in the mineral aggregate (VMA) values to accommodate the binder (30). Accordingly, using fully crushed aggregate aid in increasing the VMA due to their surface area and texture (27).

In regards of aggregate gradation in high modulus asphalt concrete, different countries and regions, including France (26), South Africa (18), United Kingdom (45), and Australia (65) have developed different gradation envelopes to standardize the mixture design procedure. Some researchers have been developing their own gradations which might or might not fit into some standard envelopes (16, 30, 48), while some researchers have been using some techniques to optimize aggregate gradations in order to improve the performance (16). Different gradations used by different countries and researchers, as well as maximum density curve are shown in Figure 2-1.

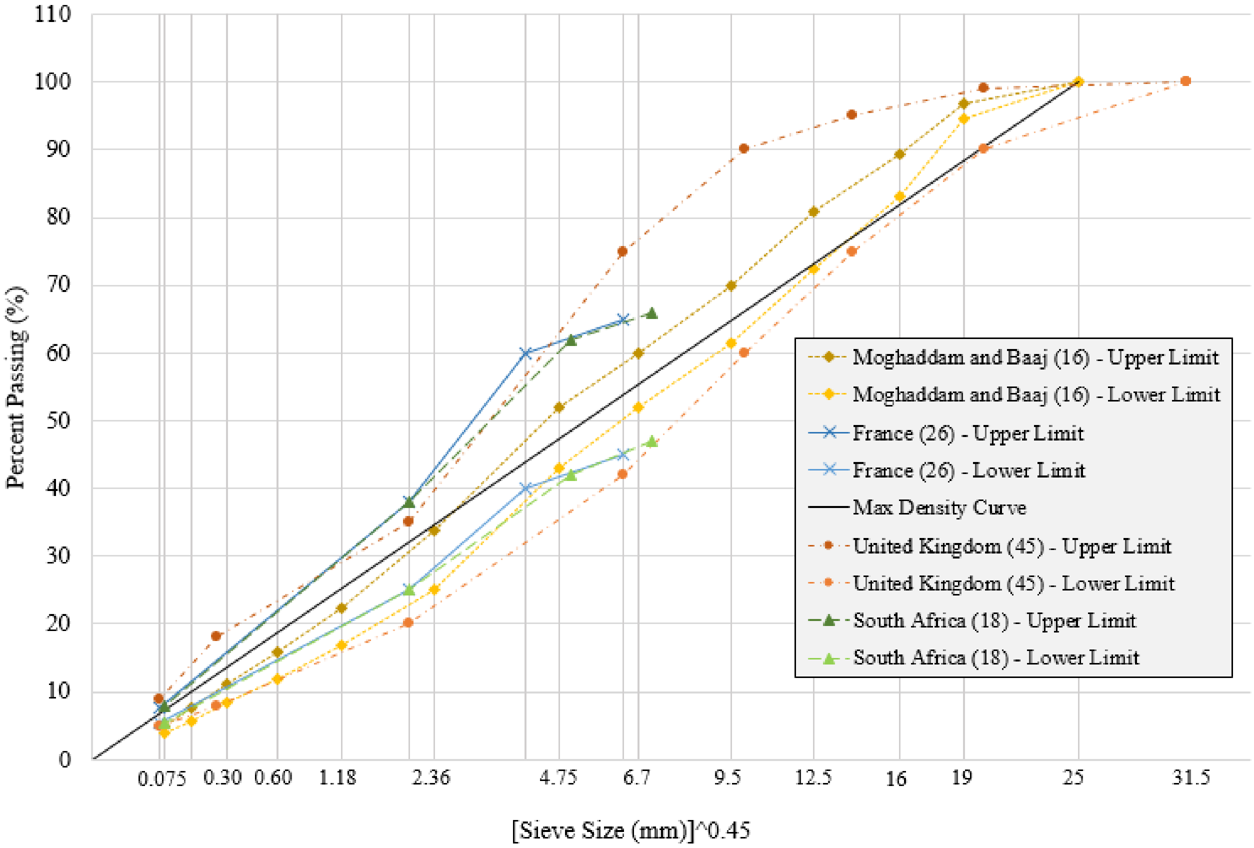


Figure 2-1. Different Gradation Envelopes for HMAC Mixtures

2.2.2 Binder Content Selection

Unlike what happens in common mix design procedures like Marshall or Superpave, in case of HMAC, the asphalt binder content is not estimated according to volumetric properties, but by calculating a richness factor, K (18). As a key design parameter used in the French asphalt mix design method, K factor is a proportional value related to the thickness of the binder film coating the aggregate (30). This factor is calculated through the following steps (27):

1- Calculate the specific surface area of the aggregate ($\sum$) using Equation 2-1:

$$100\Sigma = 0.25G + 2.3S + 12s + 150f \tag{2-1}$$

where G is the proportion of aggregate retained on and above the 6.3 mm sieve, S is the proportion of aggregate retained between the 0.25 mm and 6.3 mm sieves, s is the proportion of aggregate retained between 0.063 and 0.25 mm sieves, and f is the percent passing the 0.063 mm sieve.

2- Calculate a correction coefficient (α) for the relative density of the aggregate (RDA) using Equation 2-2:

$$\alpha = \frac{2.65}{RDA} \tag{2-2}$$

3- Calculate the binder content of the mixture by mass of total aggregate (TL_{est}) using Equation 2-3:

$$TL_{est} = K\alpha\sqrt[5]{\Sigma} \tag{2-3}$$

4- Calculate the percent binder by mass of total mixture (P_b) using Equation 2-4:

$$TL_{est} = \frac{100P_b}{100 - P_b} \tag{2-4}$$

Typical values for minimum binder contents and target richness factors, according to the density of aggregates and class of HMAC are presented in Table 2-1.

Table 2-1. Richness Factor Values for HMAC Mixtures (27)

	HMAC Class		
	Class 1	**Class 2**	
D (mm)	10, 14, 20	10, 14	20
$P_{b\,min};\ \rho = 2.65\ g/cm^3$	3.8	5.1	5.0
$P_{b\,min};\ \rho = 2.75\ g/cm^3$	3.8	4.9	4.9
Richness factor, K	2.5	3.4	3.4

The French use the maximum stone size D, with the requirement that 100% of aggregate passes the sieve at 2D, 98-100% passes at 1.4 D and 85-98% passes at D (27).

2.2.3 Compact Gyratory Specimens

When the trial aggregate gradation and asphalt binder contents are determined, the final phase of mixture design is to compact trial Superpave gyratory compactor (SGC) samples to conduct a series of performance tests to ensure the mixture will be durable in the field. Table 2-2 shows the five tests with their corresponding standards around which the French suite of tests revolves and their equivalents in U.S. standards from a study conducted in South Africa (26, 27).

Table 2-2. French Performance Tests and Their Equivalent U.S Tests (26, 27)

Parameter	**French Test Method**	**Selected U.S. Equivalent**
Workability	EN 12697-31: Gyratory Compactor	ASTM D6926: Superpave Gyratory Compactor
Durability	EN 12697-12: Duriez Test	ASTM D4867: Modified Lottman Test
Permanent Deformation	EN 12697-22: Wheel Tracker	AASHTO 320-03: Superpave Shear Tester
Dynamic Modulus	EN 12697-26: Flexural Beam	AASHTO TP 62: Dynamic Modulus
Fatigue Test	EN 12697-24: Prism Test	AASHTO T 321: Beam Fatigue

2.2.4 Dynamic Modulus Performance Test

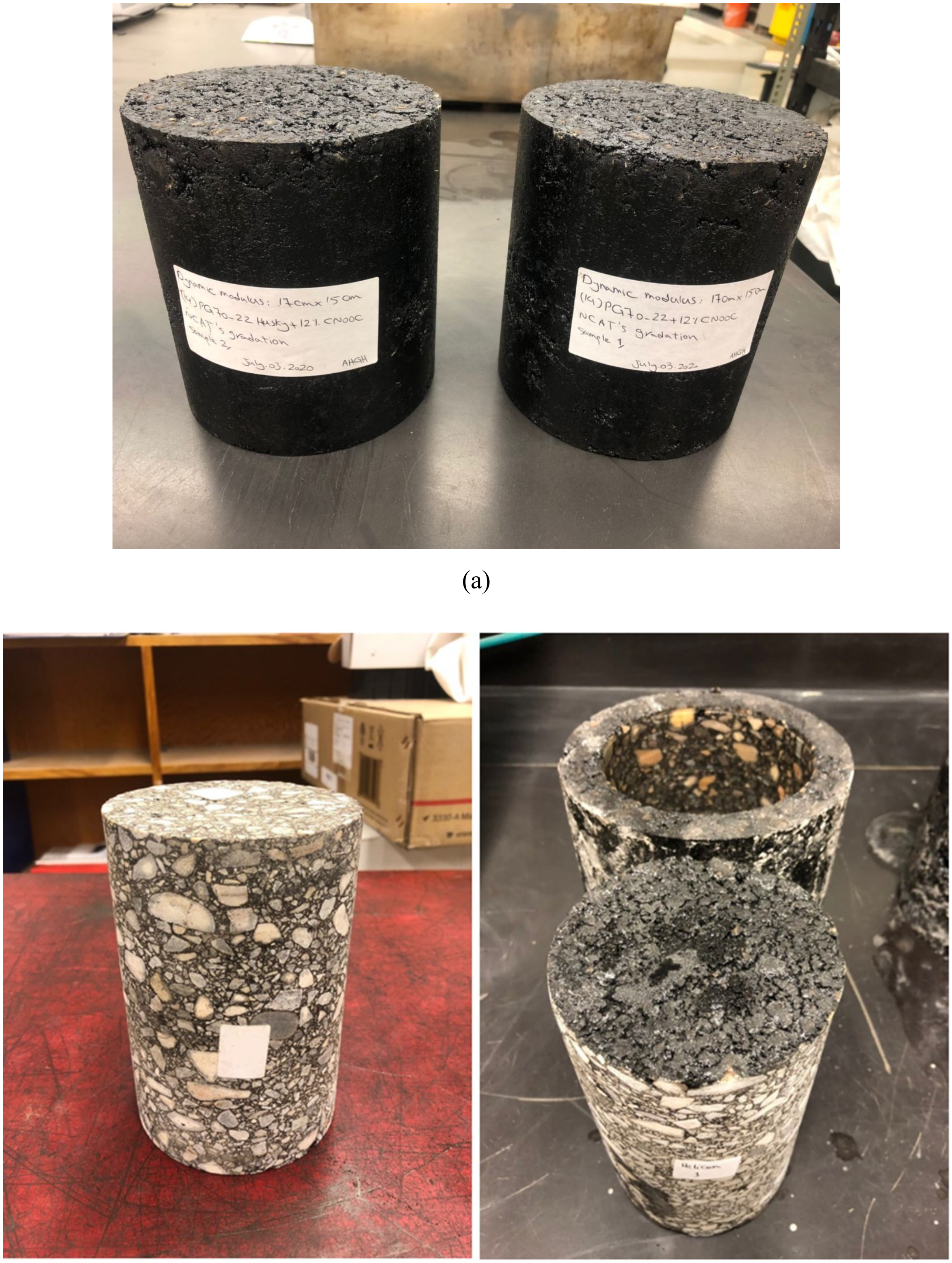
A Universal Testing Machine (IPC global UTM-100), as shown in Figure 2-2, is used to perform dynamic modulus test on asphalt samples according to AASHTO T378 (66). In the dynamic modulus test procedure, UTM applies a controlled sinusoidal (haversine) compressive stress to cylindrical samples of asphalt mixture at different temperatures and frequencies (66). The applied stresses and resulting axial strains are measured as a function of time and used to calculate different parameters including dynamic modulus (66). The absolute value of the complex modulus is calculated by dividing the peak-to-peak stress by the peak-to-peak strain for a material subjected to a sinusoidal loading (66). According to AASHTO R87 (67) dynamic modulus master curve can be developed testing an asphalt mixture sample at specific temperatures and frequencies. In the MEPDG, the dynamic modulus master curve is used to characterize the temperature and frequency dependent behavior of asphalt concrete (68). The master curve can be constructed for an arbitrarily selected reference temperature by horizontally shifting the test results obtained at various temperatures and loading frequencies according to the time–temperature superposition principle (TTSP). According to TTSP, the effects of time and temperature are related; the behavior at high temperatures over short time periods is equivalent to what occurs at lower temperatures and longer times (69).

Figure 2-2. Universal Testing Machine (UTM)

Superpave gyratory compactor (SGC) (shown in Figure 2-3) is used to compact dynamic modulus samples (150 mm diameter by 170 mm height). The SGC samples were then cored and cut to produce 100 mm diameter by 150 mm height cylindrical specimens using a coring device and an electric saw. Loose asphalt mixture in SGC mold is shown in Figure 2-3(b), while compacted SGC samples and the cored and cut samples are shown in Figure 2-4(a) and Figure 2-4(b), respectively.

(a) (b)

Figure 2-3. Syperpave Gyratory Compactor: (a) SGC; (b) Loose Mixture in SGC Mold Prior to Compaction



(a)

(b)

(c)

Figure 2-4. Dynamic Modulus Samples: (a) SGC Samples (170 mm×150 mm); (b) Cored and Cut Sample (150 mm×100 mm); (c) SGC Sample and Cored Sample Together

Dynamic modulus test setup in UTM is shown in Figure 2-5. As can be ssen in this figure, in dynamic modulus test setup linear variable displacement transducers (LVDTs) to measure the displacements. LVDTs are generally employed to measure the midspan vertical deflection of the samples in the different performance tests (70).

Figure 2-5. Dynamic Modulus Test Setup in UTM

2.2.5 Dynamic Modulus Criteria

Different countries have used different test methods along with different standards to measure the dynamic modulus of HMAC mixtures.

Table 2-3. HMAC Dynamic Modulus Criteria in Different Countries

Country	Test	Standard Method	HMAC Performance Requirements	
			Class 1	Class 2
France (26)	Stiffness, Two point bending flexural modulus 15°C, 10 Hz	EN 12697-26	≥ 14 GPa	≥ 14 GPa
South Africa (27)	Stiffness, Dynamic modulus test at 15°C, 10 Hz	AASHTO TP 62	≥ 14 GPa	≥ 14 GPa
United Kingdom (45)	Stiffness, Indirect Tensile Stiffness Modulus	DD 213: BSI 1996	N/A	≥ 5.5 GPa
Australia (71)	Stiffness, Four-point bending flexural modulus 15°C, 10 Hz	AGPT/T274	N/A	≥ 14 GPa

As it can be seen in Table 2-3, although different countries use different standards and test methods to measure modulus of HMAC mixtures, most of them agree on the criteria of minimum modulus of 14 GPa at a loading frequency of 10 Hz and a temperature of 15°C (16, 26, 27, 45, 71).

2.3 High Modulus Asphalt Concrete: Pros and Cons

Since emergence of HMAC in the 1980s, due to its advantages over conventional hot mix asphalt, this type of mixture has been considered an effective solution in construction of road and airport pavements (12, 54). These benefits include high durability, superior rutting performance, and good fatigue resistance (72).

A strong continuous mineral skeleton combined with a hard binder, impart a high capacity to absorb the loads transmitted by the traffic to HMAC mixes (57). These characteristics provide them with a high modulus, which reduces the appearance of structural distresses by minimizing the tensile strain at the bottom of the asphalt layer, and the compressive strain on top of the subgrade (56, 73). It was observed from test sections that the tensile strain values at the bottom of asphalt layer in pavements with HMAC base courses were lower than those of the conventional mix sections, although the surface course thicknesses of the HMAM sections were thinner than those of the conventional sections (56).

With all these superior advantages, the use of HMAC allows the construction of roads with major structural strengthening, which can lead to a reduction up to 30% in the thickness of the pavement, while the fatigue life remains unchanged (18, 21, 46, 47). This reduction result in a considerable saving in usage of raw materials including asphalt binder and aggregates (14, 15, 44).

Despite its superior performance at high and intermediate temperatures, due to their low flexibility and low stress relaxation capacity at low temperatures, HMAC application is limited in cold climates (21, 46, 48). It was revealed that pavements with high modulus asphalt bases are averagely 2.45 times more possible to be in a group of cracked pavements than pavements with conventional asphalt concrete base courses (21). This concern specially exists with traditional straight-run hard grade asphalt binders and air-blown hard grade binders (12, 21, 74).

Due to its different structure, the mechanism of low-temperature cracking is different in pavements with HMAC base courses compared to the ones with conventional bases. According to the theory of development of low-temperature cracking, transverse cracks are initiated in the point where the thermal stress is higher than tension strength of asphalt mix (74). In case of pavements with conventional base courses, in cold seasons the low-temperature cracks are likely to start in from the top of surface course, while in pavements with HMAC bases the base course usually cracks first at low temperatures due to its high stiffness (21, 74). Subsequently despite the fact that lower minimum temperature occurs in wearing course, the low-temperature crack in the HMAC base propagate upwards and finally appears on the surface course (21, 74).

2.4 Dynamic Modulus Prediction Models

Difficulty to obtain dynamic modulus measurements in laboratory at extreme temperatures and frequency conditions made researchers to develop predictive models using available mixture and binder data (28). These models proved to be useful in places with limited access to expensive laboratory facilities as well (31). These efforts lead to development of predictive regression models and more recently models based on artificial neural network (29, 32). Based on the results from 205 mixtures with 2,750 data points, including unmodified and modified asphalt binders, Andrei et al. (75) revised the original regression predictive model from Witczak, which was then incorporated into early versions of Mechanistic-Empirical Pavement Design Guide (MEPDG) (0.7 to 0.9) (31, 76). This model is based on a nonlinear regression analysis that includes mixture volumetric properties, aggregate gradation, and binder viscosity. This model has the disadvantage of using conventional viscosity (η) to characterize the binder, rather than rheological properties which are common these days in Superpave performance grading. Later, Bari (77), revised this model and replaced viscosity with rheological specifications of binder, including dynamic shear modulus and phase angle, using 7400 data points from 346 hot mix asphalt mixes. In 2007, and under the NCHRP 1-40D project, Bari and Witczak model was again modified, to have a better goodness of fit, less bias, and higher accuracy (31, 78, 79). This model was then incorporated in MEPDG 1.0 (28, 78).

However, regardless of the nature of the predictive model, none of them are universal and their accuracy drops when other gradation and binder variations not included within the database are being used to predict the dynamic modulus values, or when temperature, frequency, or E^* are at their extreme values (78, 80). Depending on the conditions, they might over- or underestimate the E^* values, yet most of the times with a reasonable accuracy (81-83).

For the purpose of pavement design, and according to the available data, MEPDG has adopted a hierarchical input level for the traffic and material characterization. Level 1 requires the actual measurement of dynamic modulus via laboratory testing. In level 2, regression predictive models are used to estimate the dynamic modulus based on rheological or viscosity test results on asphalt binder in laboratory. Level 3 also uses the same predictive model as level 2; however this level provides default values suggested by the designer based on past experience and engineering judgment when laboratory test results on asphalt binder are not available (68).

Witczak models are among the most common ones used for predicting dynamic modulus of asphalt mixtures. The Equations for the 1999, 2005, and 2007 Witczak regression models are shown in Equations 2-5, 2-6, and 2-7, respectively (31, 68, 77).

$$\begin{aligned}\log E^* &= 3.750063 + 0.02932\rho_{200} - 0.001767(\rho_{200})^2 - 0.002841\rho_4 - 0.058097V_a \\ &- 0.802208\left(\frac{V_{beff}}{V_{beff} + V_a}\right) \\ &+ \frac{3.871977 - 0.0021\rho_4 + 0.003958\rho_{38} - 0.000017(\rho_{38})^2 + 0.005470\rho_{34}}{1 + \exp(-0.603313 - 0.313351\log f - 0.393532\log\eta)}\end{aligned} \tag{2-5}$$

$$\begin{aligned}
&\log E^* \\
&= -0.349 \\
&+ 0.754(|G_b^*|)^{-0.0052}\begin{bmatrix} 6.65 - 0.032\rho_{200} + 0.0027(\rho_{200})^2 + 0.011\rho_4 - 0.0001(\rho_4)^2 \\ +0.006\rho_{38} - 0.00014(\rho_{38})^2 - 0.08V_a - 1.06\left(\dfrac{V_{beff}}{V_{beff} + V_a}\right) \end{bmatrix} \\
&+ \frac{2.56 + 0.03V_a + 0.71\left(\dfrac{V_{beff}}{V_{beff} + V_a}\right) + 0.012\rho_{38} - 0.0001(\rho_{38})^2 - 0.01\rho_{34}}{1 + \exp(-0.7814 - 0.5785\, log|G_b^*| + 0.8834\, log\, \delta_b)}
\end{aligned} \tag{2-6}$$

$$\begin{aligned}
&\log E^* \\
&= 0.02 \\
&+ 0.758(|G_b^*|)^{-0.0009}\begin{bmatrix} 6.8232 - 0.03274\rho_{200} + 0.00431(\rho_{200})^2 + 0.0104\rho_4 - 0.00012(\rho_4)^2 \\ +0.00678\rho_{38} - 0.00016(\rho_{38})^2 - 0.0796V_a - 1.1689\left(\dfrac{V_{beff}}{V_{beff} + V_a}\right) \end{bmatrix} \\
&+ \frac{1.437 + 0.03313V_a + 0.6926\left(\dfrac{V_{beff}}{V_{beff} + V_a}\right) + 0.00891\rho_{38} - 0.00007(\rho_{38})^2 - 0.0081\rho_{34}}{1 + \exp\left(-4.5868 - 0.8176\, log\left|G_b^*\right| + 3.2738\, log\, \delta_b\right)}
\end{aligned} \tag{2-7}$$

where:

E^*= dynamic modulus of mixture (psi);

η= viscosity of binder (10^6 poise);

f= loading frequency (Hz);

V_a= air voids (% by volume of the mixture);

V_{beff}= effective binder (% by volume of the mixture);

ρ_{34}= cumulative percentage retained on 3/4 inch (or 19 mm) sieve;

ρ_{38}= cumulative percentage retained on 3/8 inch (or 9.5 mm) sieve;

ρ_4= cumulative percentage retained on No. 4 (or 4,75 mm) sieve;

ρ_{200}= percentage passing No. 200 (or 0.075 mm) sieve;

$|G_b^*|$= dynamic shear modulus of binder (psi);

δ_b= phase angle of the binder associated with $|G_b^*|$ (degree).

In Witczak 2005 and 2007 models, G_b^* and δ_b should be measured at the loading frequency of f_s, according to Equation 2-8:

$$f_s = f_c/2\pi \tag{2-8}$$

where:

f_s = loading frequency of a dynamic loading in shear mode, as used in the complex shear modulus test of asphalt binders (Hz);

f_c = loading frequency of a dynamic loading in compression mode, as used in the complex dynamic modulus test of asphalt concrete mixtures (Hz).

Also the following regression model was developed for the mixtures in the data base which didn't have the G_b^* and δ_b measurements (68, 84):

$$\eta = \left(\frac{G_b^*}{10}\right)\left(\frac{1}{\sin\delta_b}\right)^{4.8628} \tag{2-9}$$

$$\log\log\eta = A + VTS\log T_R \tag{2-10}$$

$$\log\log(\eta_{f_s,T}) = A' + VTS'\log T_R \tag{2-11}$$

$$A' = 0.9699 f_s^{-0.0527} A \tag{2-12}$$

$$VTS' = 0.9668 f_s^{-0.0575} VTS \tag{2-13}$$

$$\begin{aligned}\delta_b = 90 &+ (-7.3146 - 2.6162VTS')\log\left(f_s.\eta_{f_s,T}\right) \\ &+ (0.1124 + 0.2029VTS')\left[\log\left(f_s.\eta_{f_s,T}\right)\right]^2\end{aligned} \tag{2-14}$$

$$|G_b^*| = 0.0051\, f_s\, \eta_{f_s,T} (\sin \delta_b)^{7.1542-0.4929 f_s+0.0211 f_s^2} \tag{2-15}$$

where:

η= binder viscosity (cP, centipoise);

T_R= testing temperature (Rankine);

A= regression intercept;

VTS= regression slope of the viscosity-temperature relationship;

A'= adjusted A for loading frequency;

VTS'= adjusted VTS for loading frequency;

$\eta_{f_s,T}$= binder viscosity as a function of both loading frequency (f_s) and temperature (T_R), (cP) (for calculated $\eta_{f_s,T} > 3 \times 10^{12}$ cP, use $\eta_{f_s,T} = 3 \times 10^{12}$ cP);

δ_b= binder phase angle (deg);

$|G_b^*|$= binder complex shear modulus (Pa) (for calculated $|G_b^*|$ >1 GPa, use $|G_b^*|$= 1 GPa).

Accuracy of Witczak models depend on several factors including temperature, and they may over- or underestimate the dynamic modulus values at different conditions (76). However, several studies comparing the accuracy of different models have shown that the highest level of accuracy and least biased E* estimates is yielded using Witczak 1999 model along with MEPDG Level-3 binder inputs (default A-VTS values based on the binder grade) (79, 85, 86).

2.5 Aging Behavior of Asphalt Binder

A considerable distinction of the Superpave binder specification is testing binder in conditions which simulate the critical stages during binder’s life, which are (69):

- during transport, storage, and handling,
- during mix production and construction, and
- after long periods of serving in field

Aging the binder in a rolling thin film oven (RTFO) simulates the second stage, during mix production and construction (87). This oven exposes films of binder to heat and air according to, and approximates the exposure of asphalt to these elements during hot mixing and handling (88). This device is shown in Figure 2-6.

Figure 2-6. Rolling Thin Film Oven (RTFO)

The third stage of binder aging occurs after a long period in a pavement. This stage is simulated by use of a pressure aging vessel (PAV), shown in (69). This test exposes binder samples to heat and pressure in accordance with AASHTO R28 in order to simulate, in a matter of hours, years of in-service aging in a pavement (89).

Figure 2-7. Pressure Aging Vessel (PAV)

2.6 High Temperature Properties of Asphalt Binder

To characterize viscoelastic behavior of asphalt binders at high temperatures, a dynamic shear rheometer (DSR) is used, according to AASHTO T315 (90). Rutting, or permanent deformation is the major distress that happens to asphalt pavements at high temperatures (91). DSR is shown in Figure 2-8.

Figure 2-8. Dynamic Shear Rheometer (DSR)

DSR basically applies sinusoidal shear loadings to samples, and measures the stress and resulted strain response, as shown in Figure 2-9. Using these data, this device is able to calculate the complex shear modulus (G^*), and phase angle (δ) of viscoelastic materials. G^* is the ratio of maximum applied shear stress to the maximum value of strain, and δ is the time lag between shear stress and shear strain response. Complex shear modulus is a representative of material's stiffness, and phase angle is an indicator of how elastic or viscous the material behaves. According to Superpave, different combinations of these two parameters are indicators of asphalt binder performance at different range of temperatures (69).

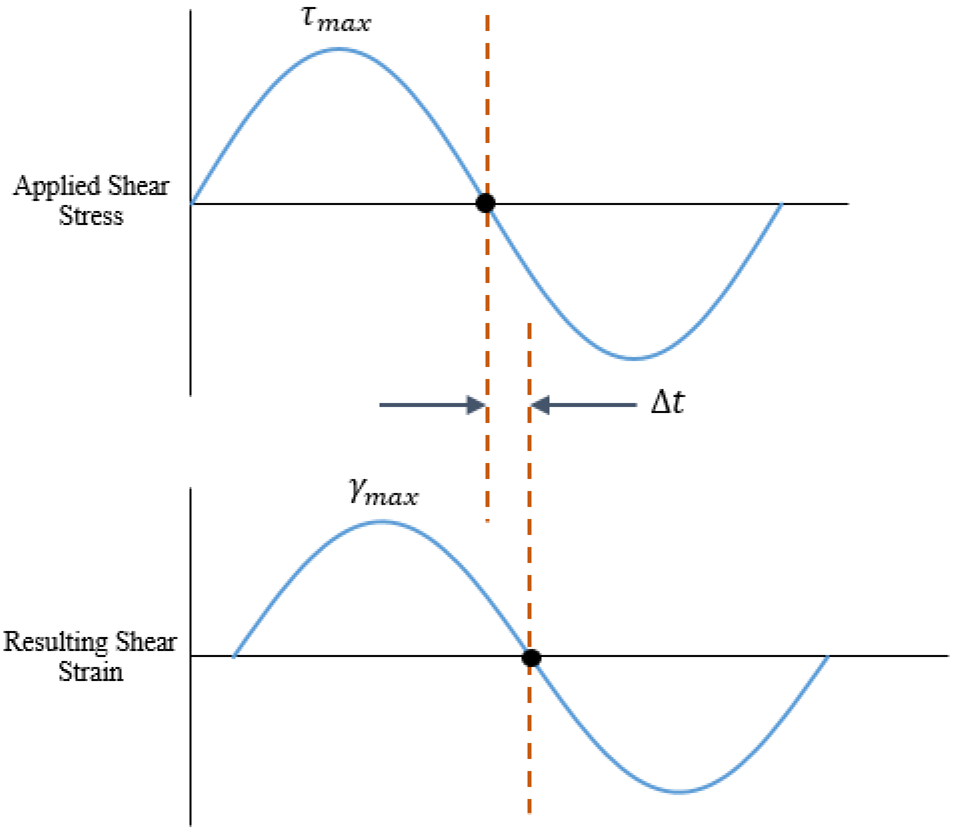


Figure 2-9. Measured Shear Stress and Strain Responses by DSR (69)

The G^* is made up of an elastic part (or storage modulus), which represents the temporary deformation in asphalt binder, and a viscous part (or loss modulus), which represents the permanent deformation in asphalt binder as shown in Figure 2-10. δ is the angle made with the horizontal axis, which indicates the relative amounts of temporary and permanent deformation.

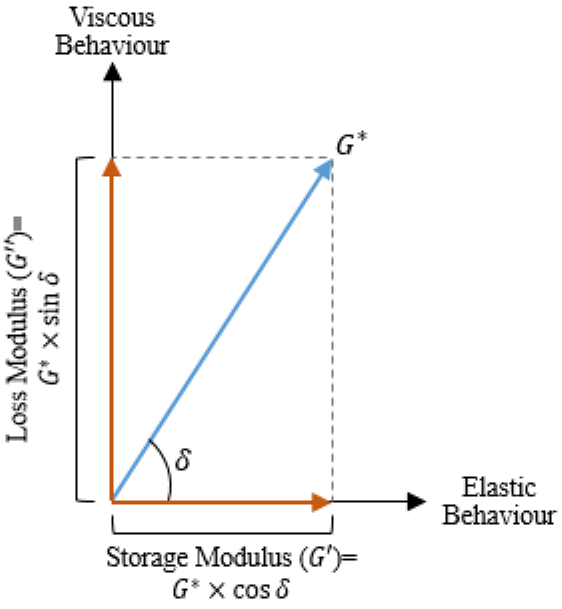


Figure 2-10. Illustration of the Elastic and Viscous Parts of Complex Shear Modulus (69)

At high temperatures, more stiffness and elasticity are desirable for a better performance (25). Superpave introduced a combination of these two parameters as an indicator of binder's resistance against rutting at elevated temperatures as $G^*/\sin\delta$ (69). The temperatures at which the parameter $G^*/\sin\delta$ falls below 1.0 kPa or 2.2 kPa at a loading frequency of 10 rad/s are recorded as the failure temperatures for unaged and RTFO-aged binder, respectively. However, the continuous high PG grade is the minimum of these two temperatures (92).

Rotational viscosity is used to evaluate high temperature workability of binders. A rotational coaxial cylinder viscometer, such as the Brookfield apparatus is used to measure this parameter (69). Brookfield viscometer is shown in Figure 2-11. As shown schematically in Figure 2-12, in a rotational viscometer (RV) the torque required to rotate a spindle in the asphalt binder sample at a specific speed and temperature is measured. This torque is then converted to viscosity of the sample (69).

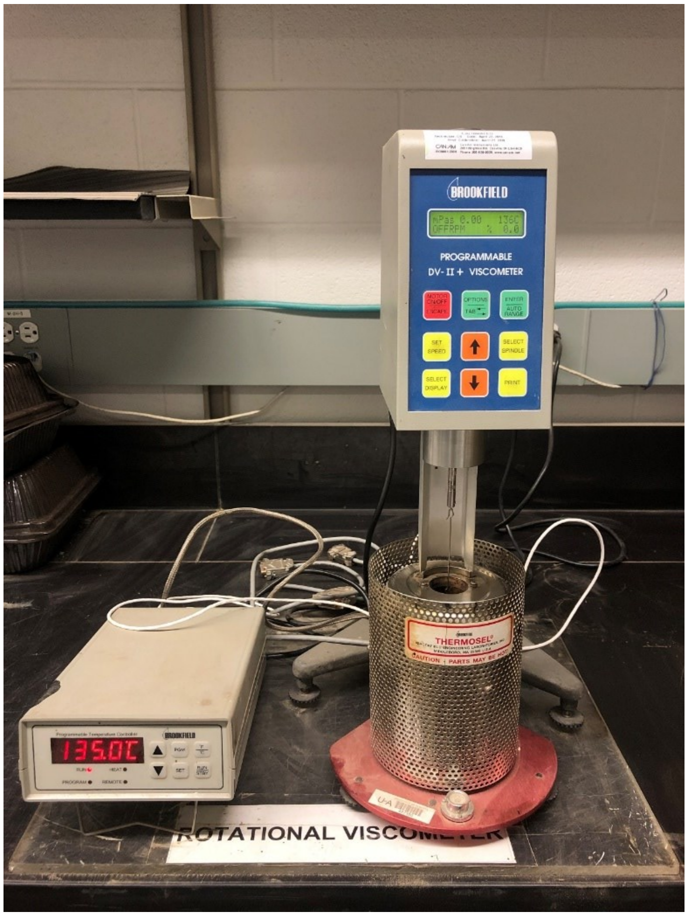


Figure 2-11. Brookfield Rotational Viscometer

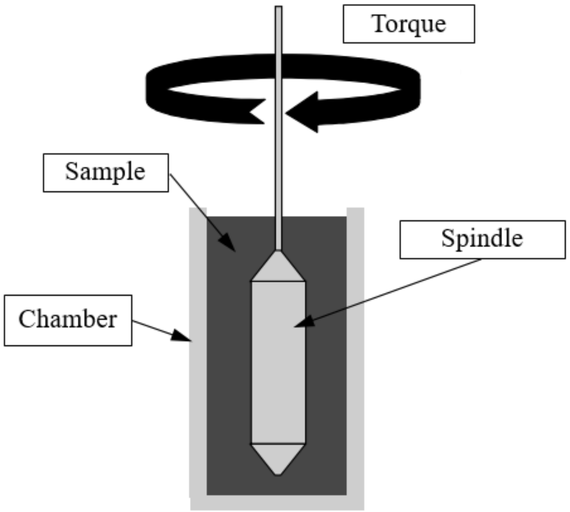


Figure 2-12. Schematic Figure of Rotational Viscometer (69)

High temperature binder viscosity is measured to ensure that the asphalt is fluid enough when pumping and mixing. According to the AASHTO M320 (92) binder specification, the viscosity of a binder must not exceed 3 Pa-s when measured at 135° C.

2.7 Intermediate Temperature Properties of Asphalt Binder

At intermediate temperatures, asphalt pavements are mostly prone to fatigue cracking (93). For evaluating the performance of asphalt binders at intermediate temperatures, a DSR is used to test PAV-aged samples under sinusoidal shear loadings (69).

In terms of fatigue resistance, asphalt binders are desired to have less stiffness and higher elasticity, and the indicator that Superpave has introduced to quantify this property is $G^{*}.\sin\delta$ (69). According to AASHTO M320, the temperatures at which the parameter $G^{*}.\sin\delta$ achieves a value more than 5000 kPa at a loading frequency of 10 rad/s is recorded as the failure temperatures of asphalt binder in regards of fatigue resistance (84).

2.8 Low Temperature Properties of Asphalt Binder

A bending beam rheometer (BBR), as shown in Figure 2-13, is used to characterize the rheological properties of asphalt binder at low temperatures, according to AASHTO T313 (94). BBR evaluates two parameters: creep stiffness and m-value. Creep stiffness is a measure of how the asphalt resists constant loading and the m-value is a measure of how the asphalt stiffness changes as loads are applied (69). BBR uses beam theory to calculate the stiffness of a small asphalt beam (127 mm×12.7 mm×6.35 mm) by measuring its deflection while applying a creep load (69). The relationship between creep stiffness and m-value is shown in Figure 2-14.

Figure 2-13. Bending Beam Rheometer (BBR)

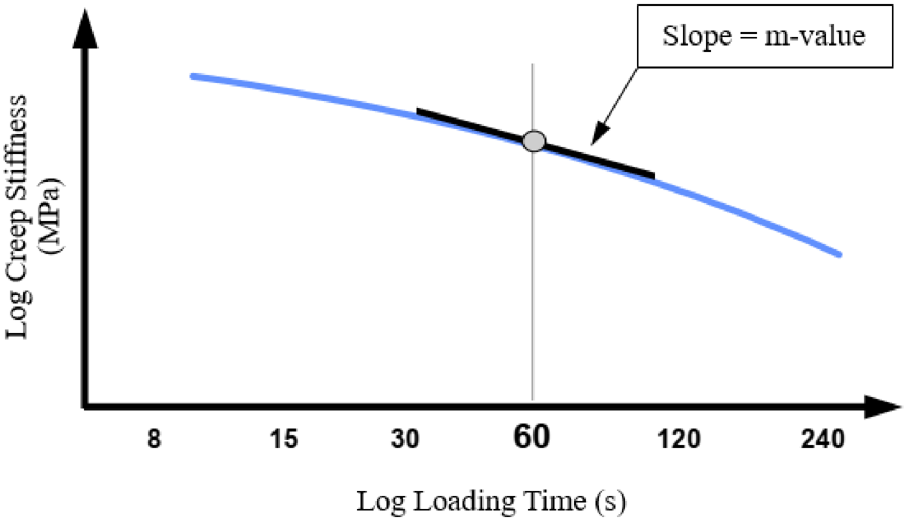


Figure 2-14. Relationship Between Creep Stiffness and m-value Measured by BBR (69)

To assess the thermal cracking resistance of asphalt binders at low temperature, the BBR test should be done on PAV-aged binder samples to measure creep stiffness and m-value at the loading time of 60 second, as the two deciding parameters in failure of the sample (94). There are two failure criteria in low temperature testing by BBR; first the temperature at which the stiffness exceeds 300 MPa, and second, the temperature at which m-values drops below 0.3. Occurrence of either of these two would be translated into failure of the sample, and the corresponding temperature would be interpreted to calculate the lowest temperature the binder can go through without major distress (69).

2.9 Chemical Composition of Asphalt Binder

As the chemical composition of asphalt binder considerably dictates its physical and rheological properties (95-97), an understanding of the chemical makeup of this component of asphalt pavements seems to be necessary to improve its performance (98). Factors affecting the chemical composition of asphalt binder, including source of the crude oil and refining process (99), provides its structure with such a complexity and diversity that makes it impossible to model the exact chemical composition and its relationship with the performance properties of asphalt binder (36, 98, 100). As an alternative it's common to categorize the chemical composition of asphalt binder based on different attributes, including molecular size, ionic character (acid, base, amphoteric) and polarity (36). Among these attributes, in pavement engineering, polarity has been used widely to link chemical makeup of asphalt binder to its rheological properties (101-103). Accordingly, asphalt binder can be divided into two general chemical groups of asphaltenes and maltenes (36), with asphaltenes being the most polar fraction of asphalt binder with high molecular weight (23, 37). Asphaltenes and maltenes can be separated using solvent extraction technique (104, 105). Using column chromatography, maltenes can be further divided into saturates, aromatics, and resins (95, 102, 103). The setup for SARA test is shown in Figure 2-15. While maltenes show a constant overall level of polarity, its sub-fractions are different in polarity, increasing from saturates' through aromatics' to resins' (98, 106). The separated fractions (Saturates, Aromatics, Resins, and Asphaltenes) are often referred to by their acronym SARA.

(a) (b)

Figure 2-15. SARA Test Setup: (a) Solvent Extraction; (b) Column Chromatography

At room temperature, saturates, aromatics, resins, and asphaltenes appear as a colorless or lightly colored liquid, a yellow to dark-red liquid, a dark-brown to black solid, and a black powder, respectively (98, 107). Figure 2-16 shows different constituents of asphalt binder and the methods to separate them from each other.

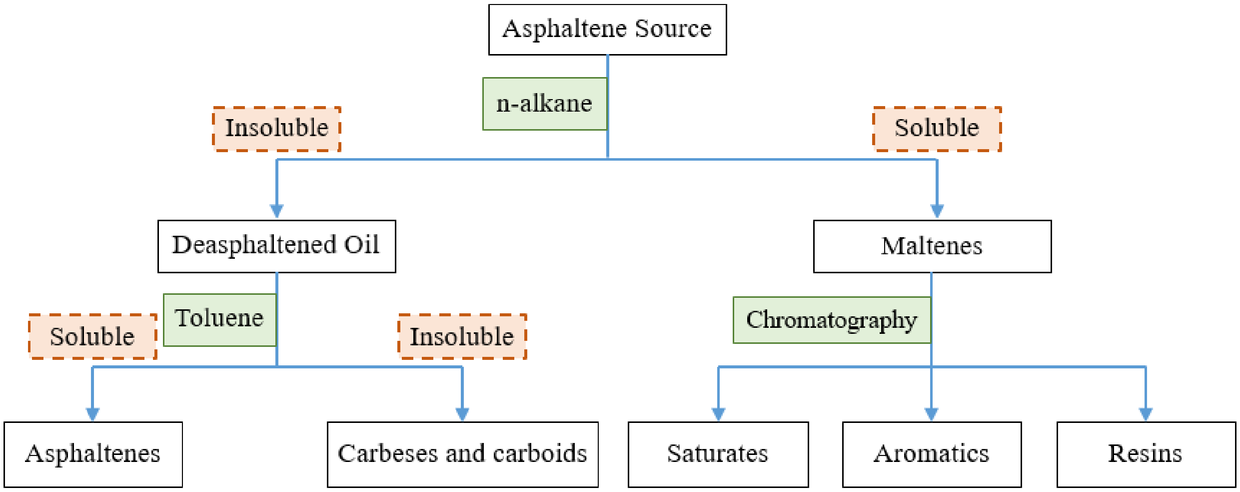


Figure 2-16. Different Chemical Constituents of Asphalt Binder (108)

Polarity and molecular weight of different fractions of asphalt binder are major factors contributing to its performance characteristics (97, 109, 110). Due to the similar level of polarity, asphaltenes and resins compose the polar fraction of asphalt binder, while saturates and aromatics form the non-polar part (98). Asphalt binder is a viscoelastic material, and polar fraction has been shown to be responsible of the elastic part of binder's behavior, while non-polar fraction is responsible for viscous part (36). That's the reason that increase in polar content cause dynamic modulus and viscosity to increase and phase angle, and creep compliance to decrease, and vice versa for increase in non-polar content of asphalt binder (36, 98, 110-112). Due to difference in polarity level between asphaltenes and saturates with resins and aromatics, the latter shows less effectiveness in imparting elastic and viscous characteristics to asphalt binder, respectively (98, 106).

In their molecular structures, resins have a polar head, which orients towards the polar surface of asphaltene particles, forming a micelle dispersed in maltenes phase (100, 102, 113, 114). The outer layer of these micelles, consisting of resins, reduces the difference in polarity between asphaltenes and surrounding maltenes, which then stabilizes polar asphaltenes phase into non-polar maltenes matrix (106, 115). Changes in pressure, temperature, composition, and shear rate might adversely affect the stability of asphaltenes particles, and initiate their precipitation and deposition (37, 116). This phenomenon in petroleum industry can be source of some problems during different phases of the production, transportation, and refining/processing (117). Instable asphaltenes particles separate from the maltene phase and start to precipitate (41, 114, 118). These highly polar particles could act as glue and mortar in oil reservoir wells, pipelines, and equipment, and make a barrier to the flow of oil (37, 115). This concern makes it necessary to check for instability of asphaltenes.

Colloidal index (CI) of asphalt binders is estimated to check for the stability of asphaltenes phase in maltene matrix. In this regard, colloidal index is defined as the ratio of the sum of the amounts of asphaltenes and saturates to the sum of the amounts of resins and aromatics, as shown in Equation 2-16 (101, 119).

$$CI = \frac{(Asphatlenes\ in\ wt\%) + (Saturates\ in\ wt\%)}{(Resins\ in\ wt\%) + (Aromatics\ in\ wt\%)} \tag{2-16}$$

Highly stable asphaltenes micelles have lower colloidal index values. For CI values more than 0.9, asphaltenes fraction tends to be unstable within the maltenes matrix; when CI has a value between 0.7 and 0.9, asphaltenes stability is uncertain, while for colloidal indices less than 0.7, the asphaltenes fraction is stable (103).

2.10 Asphaltenes

Asphaltenes, as the most responsible constituents in elastic behavior of asphalt binder (98, 110), are defined as the fraction that is insoluble in n-alkanes with low carbon number (n-hexane, n-heptane or n-pentane) but soluble in light aromatic hydrocarbons (toluene and benzene) (120, 121). The solubility-based definition brings a wide range of chemical compositions for asphaltenes, which mostly depend on its source and extraction method (41, 118, 122).

Asphaltenes have different rich sources including crude oil, asphaltite, oilsands, tar sand, and bituminous coal (123). Several methods are applied to extract asphaltenes from their source, from which solvent extraction, or solvent deasphalting, is vastly used (102, 104, 105, 124). In this method, which takes advantage of asphaltenes' solubility-base definition, n-alkanes, such as n-heptane or n-pentanare, are used as anti-solvents to precipitate asphaltenes particles (37, 102). To ensure fully precipitation of asphaltenes particles, a minimum anti-solvent volume of 40 times of oil's, and a contact time of at least 8 hours have been recommended (37, 104, 108).

In refineries, asphaltenes is considered as a waste of minimal value with insignificant applications in the industry, with a relatively high rate of production. In northern Alberta facilities it's estimated that asphaltenes are produced at a rate of 17.5% of bitumen's (39). Gasification is one technique to make use of asphaltenes, and convert them to gas fuel (40); however, this expensive technique is not environmental-friendly and produces considerable pollution (41). Refinery produced asphaltenes are shown in Figure 2-17 in two forms of solid and powdered.


(a) (b)

Figure 2-17. Asphaltenes: (a) Solid Form; (b) Powder Form after Passing through a #100 Sieve

Several studies have been conducted to investigate the effect of asphaltenes on the performance of asphalt binder (36, 105, 106). Two general methods of changing asphaltenes content in asphalt binders have been identified by these researches. In first method a mechanical stirrer is used at high temperatures for mixing asphalt binder with a material rich in asphaltenes, such as propane deasphalting tar (PDA) and natural bitumen (125), or reclaimed binder (38), or modifiers like polyphosphoric acid (PPA) (126). In second method, however, asphaltenes are extracted from an asphalt binder using solvent dissolution method, and then separated asphaltenes are added back to the asphalt binder in desirable percentages (36, 98, 105, 106, 124). Comparing to the first method, the second procedure has the advantage of asphaltenes content being adjustable, which has led to introduction of artificial bitumen concept (106, 124). For preparation of artificial bitumen, asphaltenes and maltenes are first separated using an n-alkane solvent (e.g. n-heptane), then a predefined concentrations of asphaltenes and maltenes were dissolved in toluene and mixed together. Toluene was then evaporated leaving re-dispersed asphaltenes in maltene phase, referred to as artificial bitumen (105). To the knowledge of author, the effects of addition of asphaltenes, a waste material produced in oil refineries, as a distinct modifier for the performance improvement of asphalt binder has not been investigated yet.

Previous investigations have shown that an increase in asphaltenes content, decreases the temperature susceptibility of asphalt binder (125, 126), and improves the asphalt resistance against aging (125). More asphaltenes content in asphalt binder caused a decrease in penetration and an increase in softening point, which reflect an increase in stiffness of asphalt (38). Adding even low contents of asphaltenes was found to have a considerable effect in creep compliance reduction, or equally, an increase in stiffness, which consequently affects mechanical properties of asphalt mixtures (98, 106). The abrupt change in creep compliance by addition of asphaltenes indicates fortifying the network formed by polar fractions (mainly asphaltenes) within the asphalt binder which gives elastic character to binder (105, 110). Furthermore, secondary intermolecular forces of attraction causes association between polar molecules (e.g. asphaltenes) which results in resistance against shear stresses (97). In return, a considerable increase in binder's viscosity with addition of asphaltenes is also observed (103, 111).

Chapter 3. High-Temperature Performance Evaluation of Asphaltenes-Modified Asphalt Binders

3.1 Introduction

Asphalt pavements are the most common type of pavement in North America, making them an important infrastructure asset. Asphalt mixture is composed of asphalt binder, aggregates, and fillers. In some cases additives or modifiers are also added to improve the mixture properties (10, 127). Asphalt binder is a hydrocarbon product produced in crude oil refineries, mainly through fractional distillation (11). After separation of lighter fractions (liquid petroleum gas, gasoline, aviation fuel, kerosene, etc.), the heaviest fraction taken from crude oil distillation, which is a complex mixture of high molecular weight hydrocarbons, is processed further to obtain asphalt binder (11). Although in the pavement industry asphalt binder is used as a binding agent for the aggregates, it also adds several characteristics to the asphalt mixture and plays an important role in determining distresses during the pavement's service life (128). Therefore, it is important to ensure that the asphalt binder used in the production of asphalt mixture performs well under different environmental conditions.

Increasing traffic demand, harsh weather conditions, and the tendency for infrastructure operators to reduce the cost (and, therefore, the frequency) of maintenance are among the major reasons for asphalt performance improvement strategies (11). This has motivated researchers to seek ways to modify conventional asphalt binders to improve their performance and extend their service life (10, 129). In this regard, polymers, which are one of the most common materials used in asphalt modification, have significantly enhanced asphalt mixtures in terms of performance (130). Despite the improvements recorded with these modifiers, some drawbacks are also associated with the use of polymers for asphalt binder modification. Perhaps the most notable disadvantage is that polymer-modified asphalt is much more expensive than conventional asphalt binder (64, 125). Also, phase separation may occur during the storage and application of polymer-modified asphalt binders (23, 25, 131).

Recently due to increasing public concerns about sustainability, and given the high cost of some modifiers, the application of waste materials to improve asphalt binder properties has gained more attention by researchers (33, 132). Asphaltenes is one of the waste materials obtained through the deasphaltation process in oilsands asphalt binder production. Despite its high rate of production, asphaltenes has generally been considered a waste with minimal value and few useful applications in industry (39, 41). However, there have been some efforts recently to make use of asphaltenes, including the development of a gasification technique to convert asphaltenes to gas fuel (40); however, gasification is an expensive process that generates a considerable amount of environmental pollution (41).

According to its polarity, asphalt binders can be classified by chemical composition into saturates, aromatics, resins, and asphaltenes—collectively referred to as ’SARA’ (95, 103). The polarity of asphalt binder compounds and their interactions, in turn, play an important role in influencing the rheological properties of the asphalt binder (109).

The polar fractions of asphalt binder—asphaltenes and resins—account for its elastic behavior, while the non-polar fractions—saturates and aromatics—are responsible for its viscous behavior (36, 98). Due to differences in polarity, asphaltenes particles may agglomerate and become unstable in the surrounding matrix of the remaining fractions (100, 102).

Two general methods of altering asphaltenes content in asphalt binders have been identified in the literature. The first is to use a mechanical stirrer to mix the asphalt binder with materials rich in asphaltenes (38, 126), while the second is to add the asphaltenes separated from the asphalt binder, by n-heptane extraction, back into the binder (98, 105). The second method has the advantage that the asphaltenes content is adjustable, which has made this procedure more popular and has led to the emergence of the concept of artificial bitumen (106).

Previous investigations have shown that the temperature susceptibility of an asphalt binder will decrease with an increase in asphaltenes content (125, 126). On the other hand, the addition of asphaltenes could improve the resistance of asphalt binders against aging (125). Furthermore, it has been shown that it is the asphaltenes content that is primarily responsible for the elastic component of an asphalt binder’s viscoelastic response (98, 105, 110). Higher asphaltenes content causes the asphalt stiffness to increase, which in return decreases the penetration and creep compliance (38, 98), and increases the binder’s viscosity (103). The abrupt change in asphalt binder properties resulting from the addition of asphaltenes is indicative of a fortifying of the network formed by the polar fractions (mainly asphaltenes) within the asphalt binder, a phenomenon which gives elastic characteristics to the binder (105, 110).

Based on a review of the existing literature, to the authors’ knowledge there has been no investigation of the effects of using asphaltenes produced as a waste material in crude oil refineries as a distinct modifier in performance improvement of asphalt binders. The present study thus aims to investigate the high-temperature performance of asphalt binders modified with different levels of asphaltenes content in specific reference to binders produced at oil refineries.

The main objective of this study is to investigate the rheological performance of asphalt binders modified with asphaltenes derived from Alberta oil refineries at high temperatures, as well as their chemical compositions.

3.2 Materials and Methods

3.2.1 Materials

3.2.1.1 Asphalt binder

The asphalt binder used for this study was a straight-run asphalt binder with a PG grading of 70-22, and Table 3-1 shows the specifications of this binder.

Table 3-1. Specifications of The PG 70-22 Binder (133)

Property	ASTM	Specification		Typical Value
		Minimum	Maximum	
Density @ 15°C, kg/L	D 70 (134)	---	---	1.0341
Penetration @ 25°C (100g, 5s), dmm	D 5 (135)	80	100	90
Flash Point (COC), °C	D 92 (136)	230	---	276
Ductility @ 25°C (5cm/min), cm	D 113 (137)	100	---	150+
Solubility in trichloroethylene, %	D 2042 (138)	99.5	---	99.9
Absolute viscosity @ 60°C, Pa.s	D 2171 (139)	150	---	183
Viscosity @ 135°C, Pa.s	D 4402 (140)	---	3.00	0.42
Mass loss, %	D 1754 (141)	---	1.0	0.37

3.2.1.2 Asphaltenes

Asphaltenes is a macro-polar structure that is found in solution within the oil matrix, and there are a few methods to extract asphaltenes from their sources, among which solvent dissolution, or solvent deasphalting, is the one that commonly used (102, 104). The solvent deasphalting process consists mainly of precipitation of asphaltenes by using n-alkanes as an anti-solvent (108). The asphaltenes used in this research was produced in northern Alberta facilities from SAGD produced Athabasca bitumen, by adding a non-polar solvent (e.g. typically NC3 to NC7) in enough quantity to the oil matrix, in order to disrupt the solubility of the asphaltenes and force them to precipitate.

Asphaltenes has been considered to be a waste with minimal value and few potential applications in industry. The asphaltenes used in this study was obtained in chunk form, as shown in Figure 3-1(a). In order to make the mixing process more effective and provide adequate surface area for mixing, the solid asphaltenes was ground into powder form before adding to asphalt binder, as shown in Figure 3-1(b). Asphaltenes particles passing through a #100 sieve with an opening size of 150 microns were used to modify the asphalt binder.

(a) (b)

Figure 3-1. Asphaltenes: (a) Solid form; (b) Powder Form After Passing Through a #100 Sieve

3.2.2 Methods

3.2.2.1 Preparation of asphaltenes-modified asphalt binders

All modified binders were prepared using a high shear mixer (L5M-A model, Silverson Co., USA). In order to prevent aging at elevated temperatures during mixing, a hot plate was used to maintain the temperature of the asphalt binder at 140±5°C while mixing. The desirable amount of asphaltenes was then added to the heated asphalt binder and mixed at a rotation speed of 2,000 rpm for 60 min in order to achieve a uniform blend.

In order to investigate the effects of asphaltenes across a wide range of asphaltenes content levels, asphaltenes-modified binders were prepared by adding 0% to 20% asphaltenes content (as a proportion of the weight of the asphalt binder) at increments of 3%, with the exception of the last mix, for which the asphaltenes content was increased from 18% to 20%.

3.2.2.2 Aging process and aging indices

In order to simulate the short-term aging that occurs in the asphalt binder during mixing in asphalt plants, a rolling thin film oven (RTFO) was used to age the asphalt binders in accordance with the AASHTO T240 standard (88). The impact of asphaltenes on the aging characteristics of asphalt binders was studied by estimating the aging indices. These indices, it should be noted, are defined as the ratio of a performance parameter of the aged binder to that of the unaged binder (142, 143). Two aging indices, complex modulus aging index (CAI) and phase angle aging index (PAI), were estimated in this study using Equation 3-1 and Equation 3-2, respectively (142):

$$CAI = \frac{aged\ complex\ modulus}{unaged\ complex\ modulus} \tag{3-1}$$

$$PAI = \frac{aged\ phase\ angle}{unaged\ phase\ angle} \tag{3-2}$$

where a higher value aging index indicates a more severe impact of aging on the properties of the asphalt binder (142, 144).

3.2.2.3 Dynamic shear rheometer (DSR)

To characterize the viscoelastic behavior of asphalt binders at high temperatures, samples were tested according to the AASHTO T315 standard (90) using a dynamic shear rheometer (DSR) (Smartpave 102 model by Anton Paar, Co., Ltd., Austria). DSR, it should be noted, applies sinusoidal shear loadings to samples and measures the stress and resulting strain response. Using these data, this device is able to calculate the complex shear modulus (G^*), and phase angle (δ) of viscoelastic materials, where G^* is the ratio of maximum applied shear stress to the maximum value of strain, and δ is the time lag between shear stress and shear strain response. The complex shear modulus, in turn, is representative of the material's stiffness, while the phase angle is an indicator of how elastic or viscous the material behaves. According to AASHTO M320 standard (92), at a loading frequency of 1.59 Hz, the temperatures at which the parameter G^*/sin(δ) drops below 1.0 kPa and 2.2 kPa, would be recorded as the binder's failure temperatures in regard to rutting resistance for unaged and RTFO-aged binders, respectively. The lower of these two temperatures is thus identified as the high PG grade of the binder.

3.2.2.4 Rotational viscosity

In this study, a Brookfield rotational viscometer was used to measure the viscosity of the asphalt binders at elevated temperatures in accordance with AASHTO T316 standard (145). In order to ensure adequate workability of the asphalt mixture, AASHTO M320 standard (92), which restricts the maximum viscosity of binders at 135°C to 3 Pa·s, was applied. The viscosities of the binders were investigated across a wide range of high temperatures (135°C to 185°C) in order to calculate the mixing and compaction temperature ranges of the asphalt binders. According to Superpave, compaction specimens require mixing and compaction under equiviscous temperature conditions corresponding to 170 ± 20 mPa·s and 280 ± 30 mPa·s, respectively.

3.2.2.5 SARA analysis

SARA analysis was conducted to separate different fractions of asphalt binders. The SARA test, it should be noted, is composed of two main techniques: solvent dissolution to separate asphaltenes from maltenes, and gravity-driven chromatography to separate different fractions of maltenes. In this process, asphaltenes is first separated from the sample according to ASTM D6560 standard (146) using n-heptane as the solvent. Saturates, aromatics, and resins fractions are then determined using the clay-gel adsorption chromatography method in accordance with ASTM D2007 standard (136).

In order to quantify the stability of the asphaltenes in the maltenes phase, the colloidal index (CI) was calculated for the asphalt binders using Equation 3-3 (147, 148).

$$CI = \frac{Asphaltenes\ in\ wt\% + Saturates\ in\ wt\%}{Resins\ in\ wt\% + Aromatics\ in\ wt\%} \tag{3-3}$$

where a lower value of CI represents a higher stability of asphaltenes micelles in the asphalt binder. In this regard, according to Lesueur (103), for CI values greater than 1.2, the asphaltenes fraction tends to be unstable within the maltenes matrix. When the CI value is between 0.7 and 1.2, meanwhile, the asphaltenes stability is uncertain, while, for CI indices less than 0.7, the asphaltenes fraction is stable.

3.3 Results and Discussion

The results and analysis from different tests are available in this section. All the results presented in different tables and figures are the average of at least 3 repetitions, and the maximum allowable standard deviation is 10%.

3.3.1 PG Grading Results

In order to determine the high PG grade of the binders, DSR testing was conducted on all unaged and RTFOT-aged control and asphaltenes-modified binders. The results for high PG grading of asphalt binders are presented in Table 3-2. It can be seen that a noticeable increase in the high PG grade of asphaltenes-modified binders was observed, as compared to the base binder; this increase indicates a considerable improvement in resistance against permanent deformation. It can be concluded from Table 3-2 that, on average, a one-interval increase in high PG of the asphalt binder can be expected because of addition of a 6% asphaltenes. This enhancement in PG grade is comparable with the improvement associated with some other well-known additives such as crumb rubber (25).

Table 3-2. High PG Grading Results for PG 70-22 Modified With Asphaltenes

Binder+asphaltenes, (% binder wt)	**High PG grade (Unaged Binder) °C**	**High PG grade (RTFO-Aged Binder) °C**	**Continuous high PG Grade °C**	**Standard high PG Grade °C**
0% Asph	70.5	71.1	70.5	70
3% Asph	73.9	75.1	73.9	70
6% Asph	76.5	78.2	76.5	76
9% Asph	81.3	81.1	81.1	76
12% Asph	82.9	84.6	82.9	82
15% Asph	85.9	87.6	85.9	82
18% Asph	88.0	90.3	88.0	88
20% Asph	89.9	92.6	89.9	88

The PG grade improvement of the binder at high temperatures corresponding to the addition of asphaltenes for both unaged and RTFO-aged binders is represented graphically in Figure 3-2. As can be seen in the figure, at both states of aging, the rate of improvement of the asphalt binder's performance at high temperatures is roughly proportional to the increase in asphaltenes content. On the other hand, the slope of the RTFO-aged plot is more inclined than that of the unaged one, suggesting that the contribution of asphaltenes to rutting resistance increases as the binder ages.

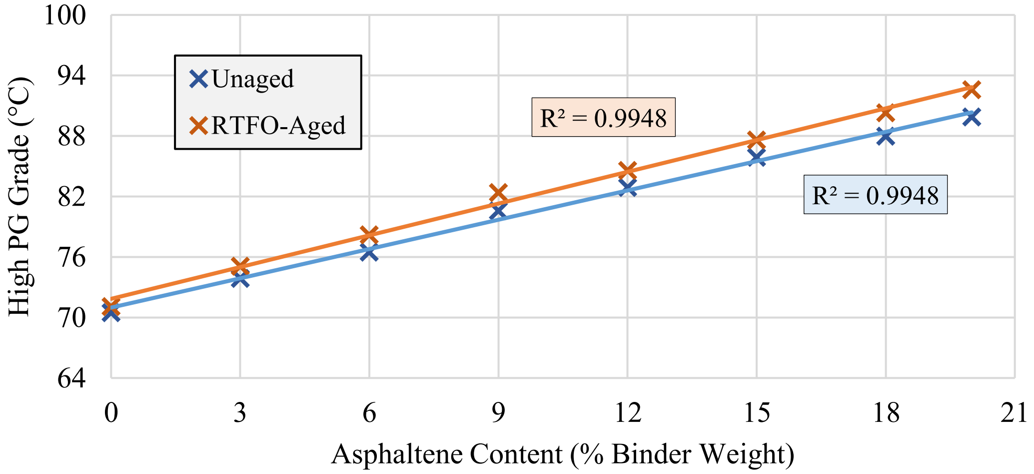
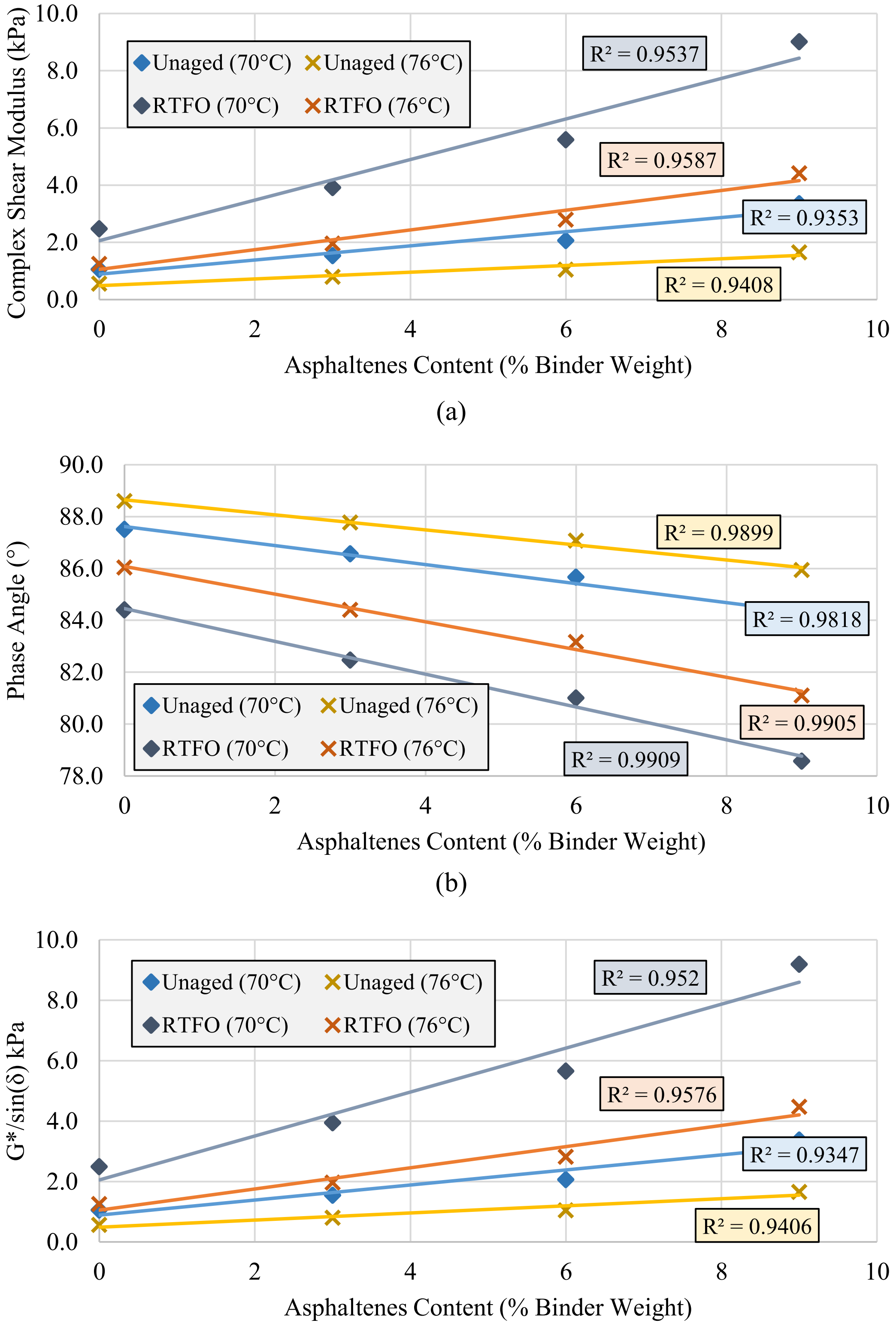


Figure 3-2. PG Grading Results for Unaged and RTFO-Aged Binders

3.3.2 Rheological Properties

The modified binders were divided into two groups: the first group included the binders containing 0%, 3%, 6% and 9% asphaltenes content, and the second category consisted of asphalt binders modified with 12%, 15%, 18% and 20% asphaltenes content (relative to the binder weight). Figure 3-3 and Figure 3-4 compare the rheological specifications of the asphalt binders in the first and second groups, respectively.

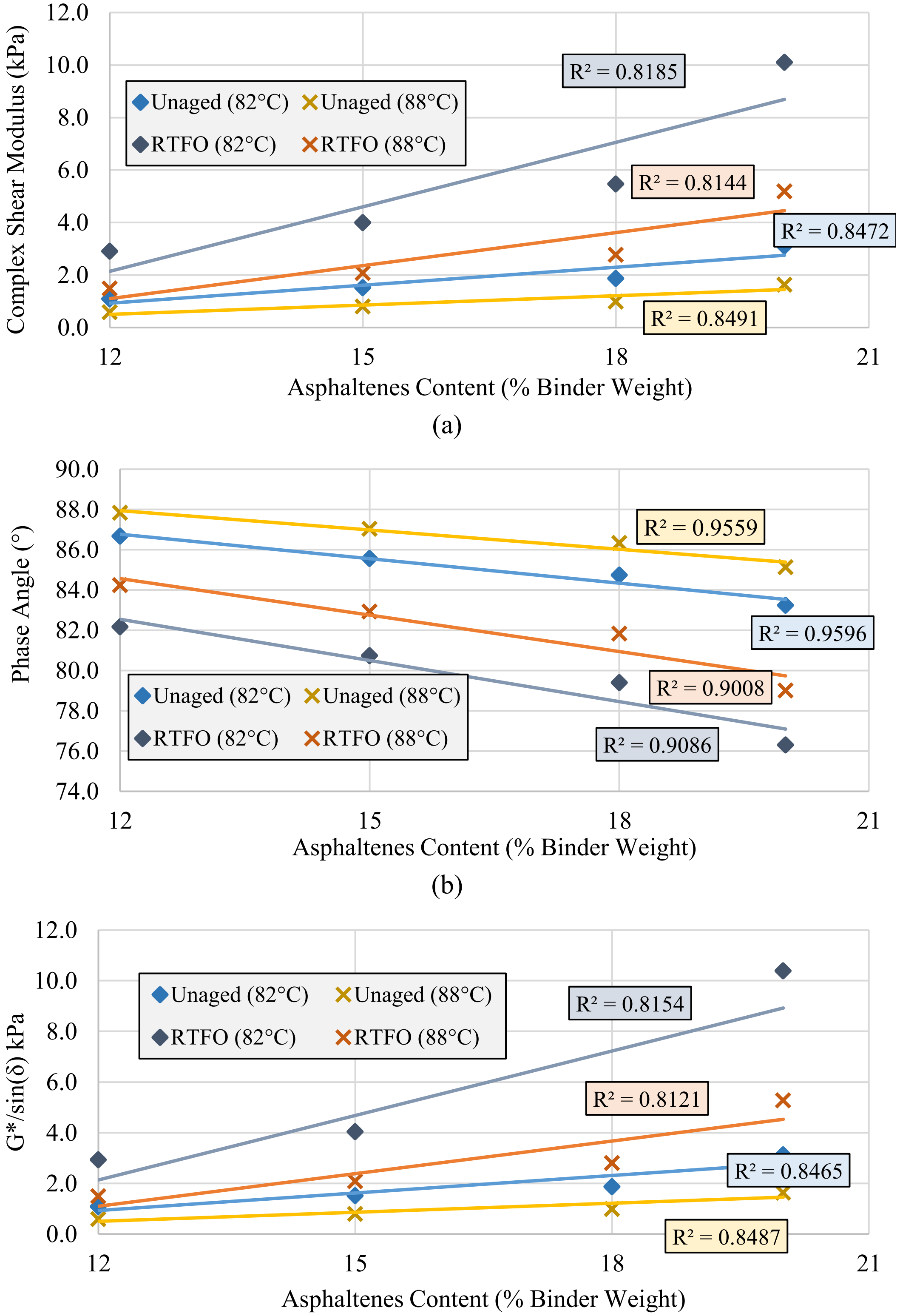


(a)

(b)

(c)

Figure 3-3. Comparison of Rheological Properties of the First Group of Modified Binders: (a) Complex Shear Modulus; (b) Phase Angle; (c) Rutting Factor



(a)

(b)

(c)

Figure 3-4. Comparison of Rheological Properties of the Second Group of Modified Binders: (a) Complex Shear Modulus; (b) Phase Angle; (c) Rutting Factor

From Figure 3-3 and Figure 3-4, it can be seen that increasing asphaltenes content was found to noticeably increase the complex shear modulus and decrease the phase angle, indicating an improvement in stiffness and elasticity of the asphalt binder. According to Figure 3-3(a) and Figure 3-4(a), the G^* plots are steeper in the RTFO-aged state than in the unaged state. Furthermore, this higher rate of improvement was found to be more significant at higher temperatures. In the case of phase angle (δ), in reference to Figure 3-3(b) and Figure 3-4(b), the rate of increase in elasticity in the aged state was found to be noticeably more than that in the unaged state. These observations reveal that the impact of asphaltenes on improving high-temperature performance parameters is more pronounced as the binder ages.

According to Figure 3-3(a, b) and Figure 3-4(a, b), at relatively lower temperatures, the differences between RTFO-aged rheological parameters and corresponding unaged parameters were found to be higher. From this observation it can inferred that, as the temperature decreases, asphaltenes shows a more significant impact in terms of increasing the asphalt binder's stiffness and elasticity due to aging. Given that higher values of stiffness and elasticity adversely affect the cracking resistance at relatively lower temperatures, it can be expected that the possibility of cracking would increase at lower temperatures with increased asphaltenes content.

As shown in Figure 3-3 and Figure 3-4, under the same aging condition, the rate of modulus change was found to increase as the temperature decreases, which implies that asphaltenes will have more impact on the shear modulus at lower temperatures. The relatively consistent slope in phase angle plots, however, reflects the similar impact of asphaltenes on this parameter at lower and higher temperatures.

The same trends as those observed in the complex modulus plots can be seen in the rutting factor (G^*/sin δ) charts shown in Figure 3-3(c) and Figure 3-4(c). These trends serve to confirm that, as the binder ages, the impact of asphaltenes on improving rutting resistance becomes more effective. Furthermore, the rutting resistance resulting from the addition of asphaltenes increases at a higher rate as the temperature decreases.

3.3.3 Aging Indices

The aging indices for the two groups of modified binders (at 70 °C and 82 °C) are presented in Figure 3-5(a) and Figure 3-5(b), respectively. These figures show that the phase angle aging index values for all modified binders remained consistently around 1.0 during aging, indicating that, regardless of asphaltenes content, aging has almost no effect on the phase angle and elasticity of the binder. However, in the case of complex modulus aging index, generally the addition of asphaltenes was found to increase the CAI at a similar rate for both groups of binders.

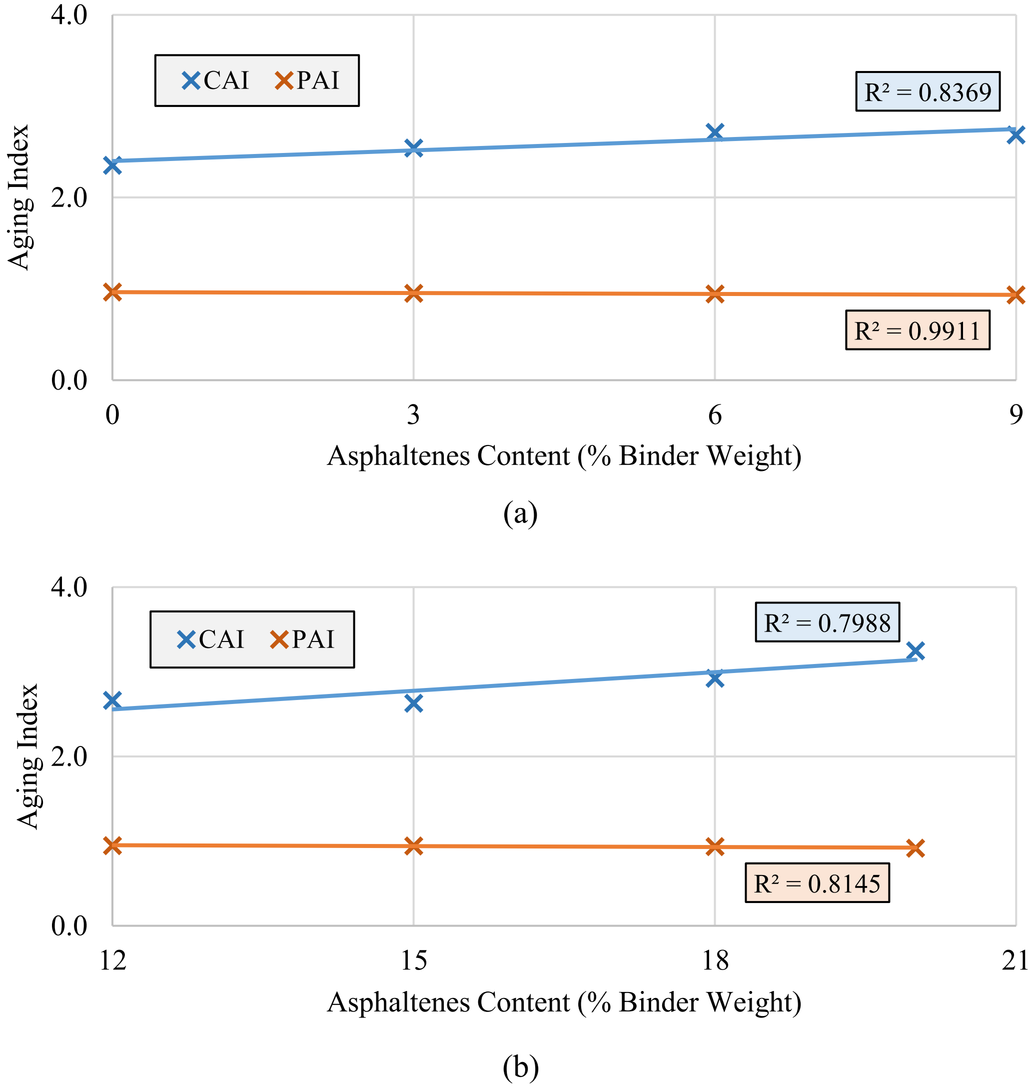


Figure 3-5. Aging Indices for Asphaltenes-Modified Binders: (a) Aging Indices of the First Group of Modified Binders At 70°C; (b) Aging Indices Of The Second Group Of Modified Binders At 82°C

3.3.4 High Temperature Viscosity

High-temperature viscosities of modified binders over a wide range of temperatures for different asphaltenes content are plotted and presented in Figure 3-6 and Figure 3-7 in relation to binder content and temperature, respectively. From Figure 3-6, it can be seen that, at all asphaltenes contents, the workability criterion of viscosity being less than 3 Pa·s at 135 °C, as specified in AASHTO M320 standard (92), was satisfied.

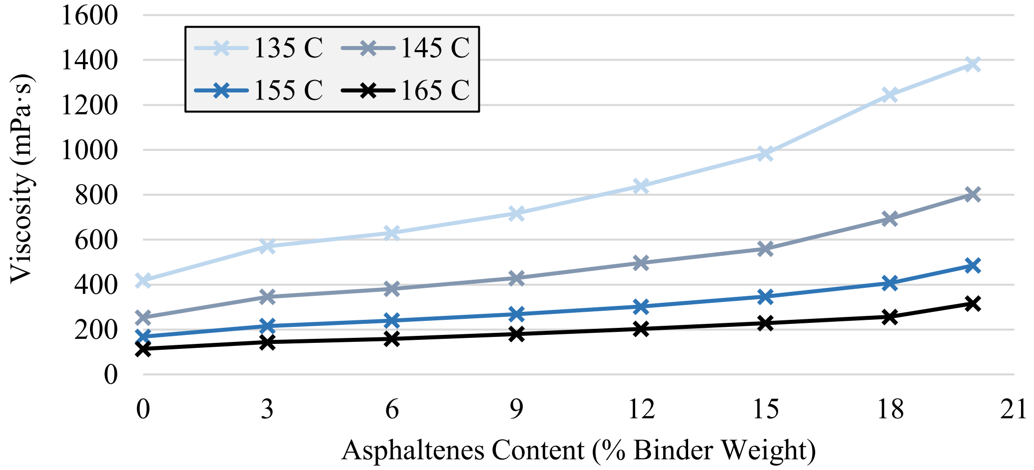


Figure 3-6. High-Temperature Viscosity of Asphaltenes-Modified Binders versus Asphaltenes Content

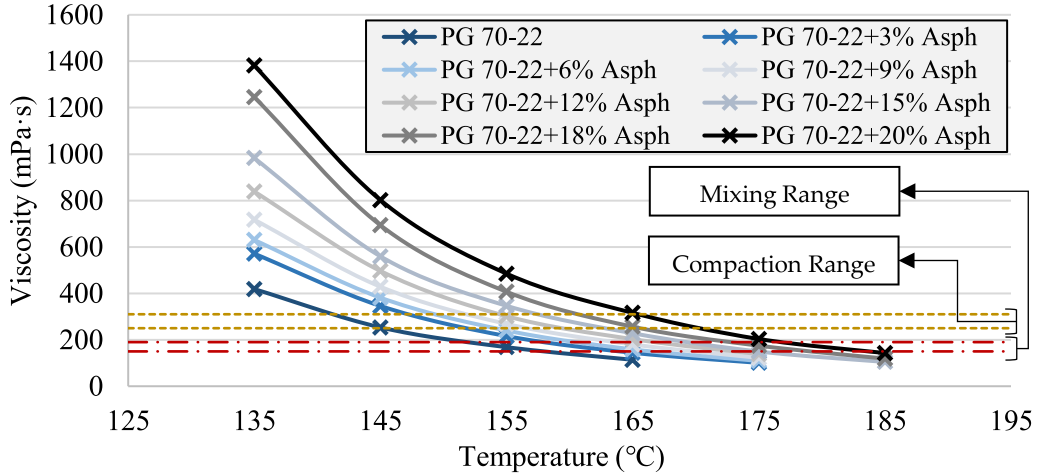


Figure 3-7. High-Temperature Viscosity of Asphaltenes-Modified Binders versus Temperature

As shown in Figure 3-6 and Figure 3-7, it was observed that, generally, the addition of asphaltenes increases the viscosity of the asphalt binder. Furthermore, it can be seen that the effect of asphaltenes on viscosity was found to be more evident at higher asphaltenes contents and at lower temperatures. The viscosities of different binders moreover, were found to exhibit a converging trend as the temperature increased, as seen in Figure 3-7. This shows that, as the temperature increased, the effect of asphaltenes content on the viscosity of the binder was found to decrease.

The data shown in Figure 3-7 was also used to estimate the mixing and compaction temperatures of the asphalt binders containing different asphaltenes contents, and the results are presented in Table 3-3. As shown in the table, it was found that an increase in the viscosity of the asphalt binders due to the addition of asphaltenes leads to an increase in the mixing and compaction temperature for the modified asphalt binders. Accordingly, the addition of every percent of asphaltenes content to the base binder PG 70-22 on average was found to increase the mixing and compaction temperatures by 1.4 °C and 1.3 °C, respectively.

Table 3-3. Mixing and Compaction Temperature Range of Base and Asphaltenes-Modified Binders

Binder+asphaltenes, (% binder wt)	Mixing Temperature Range (°C)	Compaction Temperature Range (°C)
0% Asph	152-158	141-145
3% Asph	158-164	147-152
6% Asph	161-167	149-154
9% Asph	164-170	152-157
12% Asph	167-173	154-160
15% Asph	169-175	158-163
18% Asph	173-179	161-166
20% Asph	177-184	165-170

3.3.5 SARA Analysis

In addition to the base binder PG 70-22, a few modified asphalt binders with higher asphaltenes contents were selected for SARA analysis because of their superior performance at high temperatures. Those selected were the asphalt binders with 12%, 18%, and 20% asphaltenes content. The asphaltenes was also tested for SARA content in order to determine its purity. The results of this analysis are summarized in Table 3-4.

As can be seen in Table 3-4, the addition of asphaltenes content was found to noticeably increase the polar fraction of the asphalt binder. Comparing the rheological and SARA analysis results, it can be concluded that the increase in polar fraction content causes the stiffness, elasticity, and viscosity of asphalt binder to increase.

Table 3-4. SARA Analysis Results for Select Asphalt Binders

Binder/ Material	Saturate	Aromatic	Resin	Asphaltenes	Polar Fraction	Non-polar Fraction	Colloidal Index
Asphaltenes	6.85	9.68	3.84	79.62	83.46	16.53	6.40
0% Asph	25.41	20.36	31.58	22.59	54.17	45.77	0.92
12% Asph	21.63	20.94	24.75	32.17	56.92	42.57	1.18
18% Asph	18.83	20.25	25.23	35.16	60.39	39.08	1.19
20% Asph	17.93	19.74	21.57	39.76	61.33	37.67	1.40

The CI values for the different asphalt binders were also calculated as presented in Table 3-4. It was observed that the addition of asphaltenes increases the instability of the asphaltenes particles in the maltenes matrix. Among the asphaltenes-modified binders, the one with 20% asphaltenes content tended to be the most unstable, with a CI value greater than 1.2 (103).

3.4 Conclusions

The effects of asphaltenes at different levels of content on the performance of asphalt binder PG 70-22 at high temperatures have been studied. Based on the analysis conducted in this study, the main conclusions drawn are presented as follows:

- The addition of asphaltenes increases the stiffness and elasticity of the asphalt binder, which in turn results in a considerable improvement in resistance against permanent deformation. On average, a 6% increase in asphaltenes content corresponds to a one-interval increase in high PG temperature grade of the asphalt binder.
- The effect of asphaltenes on high-temperature performance parameters, shear modulus, phase angle, and rutting factor is more pronounced as the binder ages.
- As the temperature decreases, the asphaltenes exhibits a stronger impact in terms of increasing the asphalt binder's stiffness and elasticity due to aging. Since higher values of stiffness and elasticity adversely affect the cracking resistance at lower temperatures, this indicates that the possibility of cracking would increase at lower temperatures with increased asphaltenes content.

- The use of asphaltenes to modify asphalt binder serves to increase the binder's high-temperature viscosity. The effect of asphaltenes on the binder viscosity, meanwhile, is more evident at higher levels of asphaltenes content and lower temperatures.
- Comparing the rheological and SARA analysis results, it can be concluded that the increase in polar fraction content due to the addition of asphaltenes, causes the stiffness, elasticity, and viscosity of the asphalt binder to increase.
- From the CI values, it can be concluded that the addition of asphaltenes increases the instability of the asphaltenes particles in the maltenes matrix, a phenomenon which is largely the result of polarity level differences between the asphaltenes and other fractions of the asphalt binder.

Chapter 4. Asphaltenes-Modified Binders for High Modulus Asphalt Concrete Applications

4.1 Introduction

During the 1980s, the Laboratoire Central des Ponts et Chaussées (LCPC) in France, in cooperation with road enterprises, introduced high modulus asphalt concrete (HMAC), or Enrobé à Module Élevé (EME) (13, 44). HMAC is designed to have high mechanical performance, including high elastic stiffness, high deformation resistance and good fatigue resistance, with satisfactory workability, durability, and impermeability (18, 19). HMAC mixes also have been used in development of long-life pavements with a design life of more than 40 years (149-151). However, the main concern about the application of HMAC in pavements is the high potential of cracking in cold climates (21).

The use of a hard grade binder in conjunction with a strong continuous mineral skeleton in HMAC mixtures gives a layer that is highly resistant to permanent deformation and has a high capacity to absorb transmitted traffic loads (18). To compensate for the adverse effects of using a hard grade binder, a larger binder content and smaller air void content (closed structure) assure workability, fatigue durability, and water resistance for HMAC mixes (13, 52).

HMAC mix design is mostly considered to be a performance-based design (26). HMAC mixtures should be tested to ensure that they meet several performance requirements, including workability, dynamic modulus, durability, rutting resistance, and fatigue life (27).

The difficulty of obtaining dynamic modulus measurements in the laboratory at extreme temperatures and frequency conditions drove the development of predictive models using available mixture and binder data (28, 152). Based on the results from 205 mixtures with 2,750 data points, including unmodified and modified asphalt binders, Andrei et al. (75) revised the original regression predictive model proposed by Witczak, which was then incorporated into early versions of Mechanistic-Empirical Pavement Design Guide (MEPDG 0.7 to 0.9) (31, 76). Later, Bari (77) also revised this model and replaced viscosity with rheological specifications of the binder. In 2007, the Bari/Witczak model was again modified, to have better fit, less bias, and higher accuracy (31, 79). This model was then incorporated in MEPDG 1.0 (28).

The binders used in HMAC mixtures are also referred to as high modulus asphalt binder (HMAB), and can be classified in three different categories. HMAB include traditional straight-run hard grade asphalt binders, asphalt binders modified with natural asphalt, and polymer-modified binders

(14, 19, 20). Straight-run hard grade binders, usually with penetration grades of up to 30, which have been widely used in base courses in Europe (13, 45, 46), have the drawback of poor performance at low temperatures (21, 22). This concern particularly applies to air-blown hard grade binders (12, 153). Natural asphalt, which mainly consists of lake asphalt and rock asphalt, is a highly viscous material and cannot be used directly as an asphalt binder in general (17, 47). Instead, to produce the required penetration grades, natural asphalts are typically used as additives to modify base binders (17, 20). In terms of polymer modified HMAB, polyolefin and styrene butadiene styrene (SBS) are the most common polymers used for binder modification (17, 20, 154). However, there are two factors that limit the application of polymer modified HMAB. The first is the high cost of polymer modified binders when used in the base layer, which is the thickest layer in the pavement structure (46, 64). Secondly, there is a high possibility of phase separation in polymer-modified binders, which limits their application as binders for HMAC base courses (25).

In refineries, asphaltenes are considered waste materials of minimal value with no other relevant applications in the industry, yet they have a high rate of production. In facilities in northern Alberta, it is assumed that asphaltenes account for 17.5% of the bitumen (39). One technique to make use of asphaltenes is gasification to convert them to gas fuel (40); however, this is expensive and is also not environmentally friendly, producing considerable pollution (41). A more practical and sustainable solution seems to be necessary to create value for this material.

In pavement engineering, it is common to categorize the chemical composition of asphalt binder based on polarity (102, 103). Accordingly, asphalt binders can be divided into two main types on the basis of their chemistry, asphaltenes and maltenes (36), with asphaltenes being the more polar fraction, with a high molecular weight (23, 37). Asphalt binder is a viscoelastic material, and the polar fraction has been shown to be associated with the elastic aspect of binder behaviour, while the non-polar fraction accounts for its viscous properties (36, 155). The addition of even low concentrations of asphaltenes was found to have a considerable effect in reduction of creep compliance, or stiffness increase (105, 106), which consequently affects mechanical properties of asphalt mixtures. Furthermore, intermolecular forces cause association between polar molecules such as asphaltenes, which results in resistance against shear stresses (97). A considerable increase in binder viscosity with addition of asphaltenes has also been observed (103, 111). The temperature susceptibility of asphalt binder also decreased with increase in asphaltenes content (125, 126), making it more resistant against cracking. Finally, the addition of asphaltenes could also improve the resistance of asphalt binder to aging (125).

4.2 Objective

The main objectives of this study are to investigate the potential application of asphaltenes to modify conventional asphalt binders for HMAC applications, to evaluate binders derived from Alberta oilsands for HMAC applications, and, finally, to compare the performance of asphalt binders from different sources. In this work, the performance of asphaltenes-modified binders is evaluated at different temperatures in order to determine whether these binders meet the requirements for use in HMAC.

4.3 Materials

4.3.1 Asphalt Binders

The asphalt binders used in this study include one straight-run asphalt binder with a performance grading (PG) of 70-22, and four asphalt binders (with different sources) derived from Alberta oilsands bitumens (referred to as Binders A, B, C, and D). The asphalt binders originating from Alberta oilsands were obtained through the distillation process of bitumen at 460°C. Table 4-1 shows the specifications for the PG 70-22 binder, which was used as a control sample.

Table 4-1. Specifications of The PG 70-22 Binder (133)

Property	**ASTM**	**Specification**		**Typical Value**
		Min	**Max**	
Density @ 15°C, kg/L	D 70 (134)	---	---	1.0341
Penetration @ 25°C (100 g, 5 s), dmm	D 5 (135)	80	100	90
Flash Point (COC), °C	D 92 (136)	230	---	276
Ductility @ 25°C (5 cm/min), cm	D 113 (137)	100	---	150+
Solubility in trichloroethylene, %	D 2042 (138)	99.5	---	99.9
Absolute viscosity @ 60°C, Pa·s	D 2171 (139)	150	---	183
Viscosity @ 135°C, Pa·s	D 4402 (140)	---	3.00	0.42
Mass loss, %	D 1754 (141)	---	1.0	0.37

4.3.2 Asphaltenes

Asphaltenes are macro-polar structures that are found in solution within oil matrices. There are a few different methods to extract asphaltenes: solvent dissolution, or solvent deasphalting, is the method most commonly used (102, 104). The solvent deasphalting process involves precipitation of asphaltenes by using n-alkanes as an anti-solvent (108). The asphaltenes used in this research were produced from Athabasca bitumen using steam assisted gravity drainage (SAGD) process. In this process a non-polar solvent is added to the oil matrix in order to disrupt the solubility of the asphaltenes and force them to precipitate.

The asphaltenes were obtained in chunk form, as shown in Figure 4-1(a). In order to increase the specific surface area of the asphaltenes particles to achieve better mixing, asphaltenes chunks were ground into powder and sieved (No. 100, 150 μm). The portion passing through this sieve (Figure 4-1(b)) was used for asphalt binder modification.


(a) (b)

Figure 4-1. Asphaltenes: (a) Solid Form; (b) Powder Form after Passing through a #100 Sieve

4.4 Methods

4.4.1 Estimation of Required Grade for Binder

The basic requirement for HMAC mixtures is that the dynamic modulus at a temperature of 15°C and a loading frequency of 10 Hz should be more than 14,000 MPa (27, 30). One of the most important contributing factors in satisfying such a condition is the performance grade of asphalt binder used (44). Several studies done to compare the accuracy of the different models have shown that the 1999 Witczak model, along with MEPDG Level 3 binder inputs, provided the highest accuracy for predicted dynamic modulus values (31, 79, 80, 85, 86). In this study, the 1999 Witczak regression model (Equation 5-1) was applied to determine the minimum performance grade of the asphalt binder necessary to meet the aforementioned dynamic modulus requirement (68).

$$
\begin{aligned}
&log\,E^* \\
&= 3.750063 + 0.02932\rho_{200} - 0.001767(\rho_{200})^2 - 0.002841\rho_4 - 0.058097V_a \\
&- 0.802208\left(\frac{V_{beff}}{V_{beff} + V_a}\right) \\
&+ \frac{3.871977 - 0.0021\rho_4 + 0.003958\rho_{38} - 0.000017(\rho_{38})^2 + 0.005470\rho_{34}}{1 + exp(-0.603313 - 0.313351\,log\,f - 0.393532\,log\,\eta)}
\end{aligned}
\tag{5-1}
$$

where $E^*$is the dynamic modulus of mixture (psi), η is the viscosity of binder (10^6 poise), f is the loading frequency (Hz); V_a is the air void content (% by volume of the mixture), V_{beff} is the effective binder (% by volume of the mixture), ρ_{34} is the cumulative percentage retained on 3/4 inch (or 19 mm) sieve, ρ_{38} is the cumulative percentage retained on 3/8 inch (or 9.5 mm) sieve, ρ_4 is the cumulative percentage retained on No. 4 (or 4.75 mm) sieve, ρ_{200} is the percentage passing a No. 200 (or 0.075 mm) sieve, $|G_b^*|$ is the dynamic shear modulus of binder (psi), and δ_b is the phase angle of the binder associated with $|G_b^*|$ (degree).

4.4.2 Aggregate Gradation

To assess the minimum binder performance grade needed to attain the minimum dynamic modulus requirements, an aggregate gradation, with a nominal maximum aggregate size (NMAS) of 19 mm was selected (30). Where required, volumetric parameters were used from the same reference. This gradation, combined with a hard grade binder, has proven to provide superior performance with regards to dynamic modulus values compared to others (16, 30, 48). The target gradation, as well as French envelopes and maximum density curve, are shown in Figure 4-2, while the corresponding volumetric properties are presented in Table 4-2.

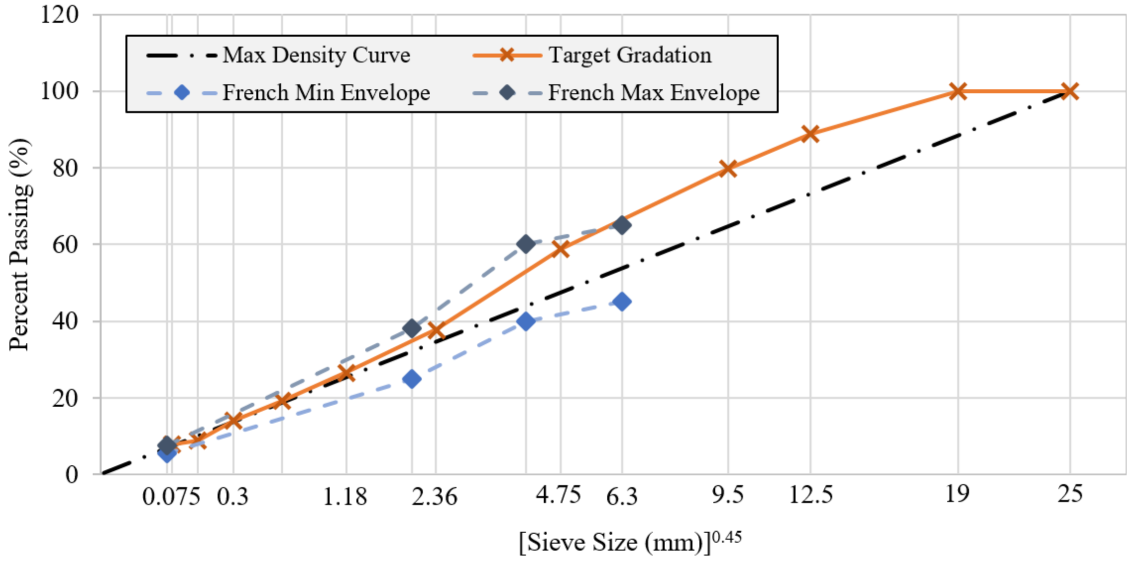


Figure 4-2. Aggregate Gradation (26, 30)

Table 4-2. Volumetric Properties of HMAC Mixes with NMAS of 19 mm (30)

Property	**Value**
Binder content	5.7
Binder grade	PG 88-16
Design air void (%)	1.5
VMA (%)	15.0
VFA (%)	90.0
Effective binder content by volume (%)	13.5
ρ_{34} (%)	0.0
ρ_{38} (%)	20.3
ρ_{4} (%)	41.1
ρ_{200} (%)	7.9

4.4.3 Modification of Asphalt Binders

A high shear mixer from Silverson Co., USA (Model L5M-A) was used to incorporate asphaltenes in the asphalt binder. To avoid aging during the modification process, the mixing temperature was controlled at 140±5°C using a hot plate. The required amount of asphaltenes powder was added to the heated binder, and mixed at a speed of 2000 rpm for 60 minutes to achieve a uniform mixture.

4.4.4 Aging Process and Aging Indices

Short-term and long-term aging of asphalt binders were simulated in the laboratory by using a rolling thin film oven (RTFO) and pressure aging vessel (PAV), according to the standards AASHTO T240 (88) and AASHTO R28-12 (89), respectively. Aging indices were used to study the effect of aging on the asphalt binders. In this study, the complex modulus aging index (CAI) and phase angle aging index (PAI) were estimated using Equation 5-2 and Equation 5-3, respectively (142). A higher value for either of these aging indices is indicator of a higher effect of aging on the properties of the asphalt binder (142, 144).

$$CAI = \frac{aged\ complex\ modulus}{unaged\ complex\ modulus} \tag{5-2}$$

$$PAI = \frac{aged\ phase\ angle}{unaged\ phase\ angle} \tag{5-3}$$

4.4.5 Rheological Tests

In accordance with AASHTO T315 (90), a dynamic shear rheometer (DSR) (Smartpave, Model 102, Anton Paar, Co., Ltd., Austria) was used to characterize the viscoelastic behaviour of the asphalt binders at high and intermediate temperatures. The rheological properties of the asphalt binders at low temperatures were evaluated using a bending beam rheometer (BBR) (Cannon Instrument Company®, USA), according to AASHTO T313 (94). A Brookfield rotational viscometer was used to measure the viscosity of the asphalt binders at elevated temperatures, using AASHTO T316 (145).

4.4.6 SARA Analysis

SARA (saturates, asphaltenes, resins, and aromatics) analysis consists of two main steps; solvent dissolution to separate asphaltenes from maltenes, followed by gravity-driven chromatography to separate different fractions of maltenes. For this purpose, asphaltenes are first separated from the sample according to ASTM D6560 (146) using n-heptane as the solvent. The saturated, aromatic and resin fractions are then determined using a clay-gel adsorption chromatography method, in accordance with ASTM D2007 (156).

The colloidal index (CI) of the asphalt binders was estimated according to Equation 5-4 to check for the stability of the asphaltenes phase in maltene matrix (101).

$$\text{CI} = \frac{(\text{Asphaltenes in wt\%}) + (\text{Saturates in wt\%})}{(\text{Resins in wt\%}) + (\text{Aromatics in wt\%})} \qquad (5\text{-}4)$$

Highly stable asphaltenes micelles have lower colloidal index values. For CI values more than 1.2, the asphaltenes fraction tends to be unstable within the maltene matrix. When the CI has a value between 0.7 and 1.2, the stability of the asphaltenes is uncertain, whereas for colloidal indices less than 0.7, the asphaltenes fraction is stable (103).

4.5 Results and Discussion

4.5.1 Required Grade for Binder

The dynamic modulus values for the four different PG grades of asphalt binders estimated using different versions of the 1999 Witczak model along with the corresponding MEPDG Level 3binder inputs, are presented in Table 4-3. It should be noted that the stiffest binder with available binder inputs in MEPDG Level 3 is PG 82-XX (68).

According to Table 4-3 the chosen gradation combined with PG 82-16 asphalt binder almost meets the minimum requirement of 14 GPa dynamic modulus value at a loading frequency of 10 Hz and a temperature of 15°C, as one of the main specifications of HMAC. In addition, this grade of asphalt binder is consistent with results of other studies on HMAC mixes (16, 20, 21, 30, 55). Accordingly, for further testing, target PG grades of PG 82-16 and PG 88-16 were used.

Table 4-3. Predicted E* Values for Different PG Grades

Predication Model	**Binders**	**Predicted E^* (GPa) according to mixture properties in Table 4-2**
Witczak 1999	PG 76-16	13.05
	PG 76-22	11.86
	PG 82-16	***13.79***
	PG 82-22	12.75

4.5.2 PG Grading Results

Table 4-4 shows the base binders and eligible modified binders that meet the minimum high standard PG grade of 82ºC, along with their PG grading results for low, intermediate, and high temperatures. It should be noted that due to its relatively poor performance at both high and low temperatures compared to the other asphalt binders, Binder D was not used in further tests.

According to Table 4-4, the minimum asphaltenes content to reach a high PG grade of 82ºC was highly dependent on the PG grading of the base binder. For the PG 70-22 binder and Binder B, at least 12% asphaltenes content (relative to the binder weight) was required to reach this target, while 6% asphaltenes content was required for Binder A. For Binder C, it was found that it already met the minimum requirement for HMAC applications, without any modification.

According to Table 4-4, regardless of binder type, on average, a 6% addition of asphaltenes causes the final high and intermediate PG grades to increase by approximately 6 ºC (corresponding to a one-interval increase in PG grade at high temperatures, and a two-interval increase in PG grade for intermediate temperatures). At low temperatures, however, the rate of increase in low PG grade is more dependent on asphalt binder source, compared to high temperature grades. On average, in the case of PG 70-22 and Binder A, a one-interval low PG grade increase (equal to 6°C) was found to result from every 10% addition of asphaltenes, while every 20% addition of asphaltenes to Binders B and C increased their low PG grade by one-interval. This indicates that the improvement in PG grade at high temperatures due to increased asphaltenes content has a higher rate than any negative effect (i.e. increase in low PG grade) at low temperatures.

Table 4-4. Performance Grading Results for Asphalt Binders

Binder+ asphaltenes, (% by wt of binder)	**High PG Grade**		**Int. Grade, PAV-Aged (ºC)**	**Low PG Grade, as Based on**		**True PG Grade (ºC)**	**Standard PG Grade (ºC)**
	Unaged (ºC)	**RTFO-Aged (ºC)**		**m-value (ºC)**	**Stiffness (ºC)**		
PG 70-22	70.5	71.1	19.2	-27.2	-27.1	70.5-27.1	70-22
PG 70-22 + 12% Asph.	82.9	84.6	29.0	-21.8	-23.3	82.9-21.8	82-16
PG 70-22 + 15% Asph.	85.9	87.6	31.5	-19.6	-22.1	85.9-19.6	82-16
PG 70-22 + 18% Asph.	88.0	90.3	34.5	-17.4	-21.1	88.0-17.4	88-16
PG 70-22 + 20% Asph.	89.9	92.6	35.9	-16.6	-20.3	89.9-16.6	88-16

Binder A	77.9	77.2	26.0	-24.1	-23.9	77.2-23.9	76-22
Binder A + 6% Asph.	84.5	84.2	30.3	-19.7	-21.3	84.2-19.7	82-16
Binder A + 9% Asph.	86.7	87.4	35.2	-17.8	-20.1	86.7-17.8	82-16
Binder B	70.1	69.0	17.7	-27.8	-26.6	69.0-26.6	64-22
Binder B + 12% Asph.	82.2	86.1	30.4	-21.2	-22.1	82.2-21.2	82-16
Binder B + 15% Asph.	84.4	85.6	30.8	-21.0	-21.9	84.4-21.0	82-16
Binder C	83.8	82.6	33.5	-19.1	-18.3	82.6-18.3	82-16
Binder C + 6% Asph.	88.2	88.5	37.1	-17.4	-16.6	88.2-16.6	88-16
Binder D	75.1	75.2	30.7	-20.7	-18.9	75.1-18.9	70-16

4.5.3 Dynamic Modulus Verification

The modified binders PG 70-22+12% Asph, Binder B+12% Asph, and Binder C+6% Asph were used to verify the results from the 1999 Witczak predicative model for dynamic modulus. The dynamic modulus test results at a frequency of 10 Hz and a temperature of 15°C along with the corresponding predicted dynamic modulus value are shown in Table 4-5.

Table 4-5. Actual and Predicted Dynamic Modulus Values for Selected Binders

Binder+ Asphaltenes, (% by wt of binder)	**Continuous PG Grade (ºC)**	**Standard PG Grade (ºC)**	**Dynamic Modulus (GPa)**	**1999 Witczak Model**	
				Binder PG Grade (ºC)	**Predicted Dynamic Modulus Value (GPa)**
PG 70-22 +12% Asph	82.9-21.8	82-16	14.02	PG 82-16	13.79
Binder B +12% Asph	82.2-21.2	82-16	14.01	PG 82-16	13.79
Binder C +6% Asph	88.2-16.6	88-16	18.64	NA	NA

According to Table 4-5, the requirement for dynamic modulus to be more than 14 GPa at a loading frequency of 10 Hz and a temperature of 15°C was achieved for all included asphaltenes-modified binders. Also, comparing the actual dynamic modulus value with predicted one in this table, 1999 Witczak model seems to be predicting the dynamic modulus values for HMAC mixes well.

Dynamic modulus master curves were also developed for the asphalt mixture samples containing the binders PG 70-22+12% Asph, Binder B+12% Asph, and Binder C+6% Asph, which is shown in Figure 4-3. According to this figure, dynamic modulus increases at higher frequencies, or equally lower temperatures, and vice versa. Also it can be observed that the harder binder (Binder C+6% Asph) has higher dynamic modulus values along different frequency ranges.

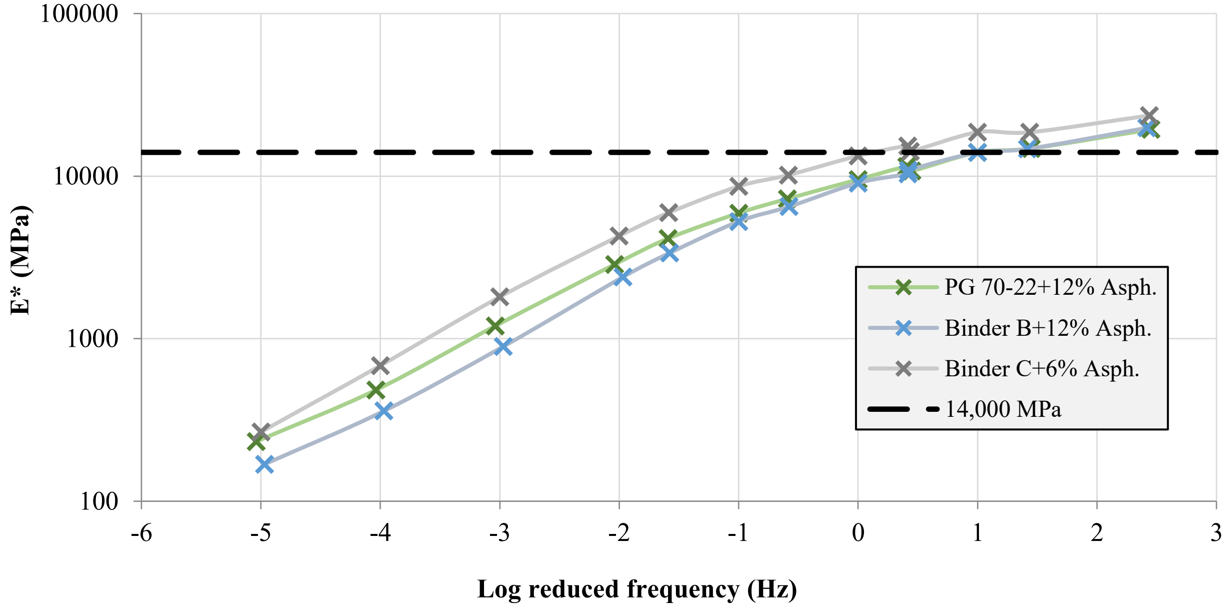


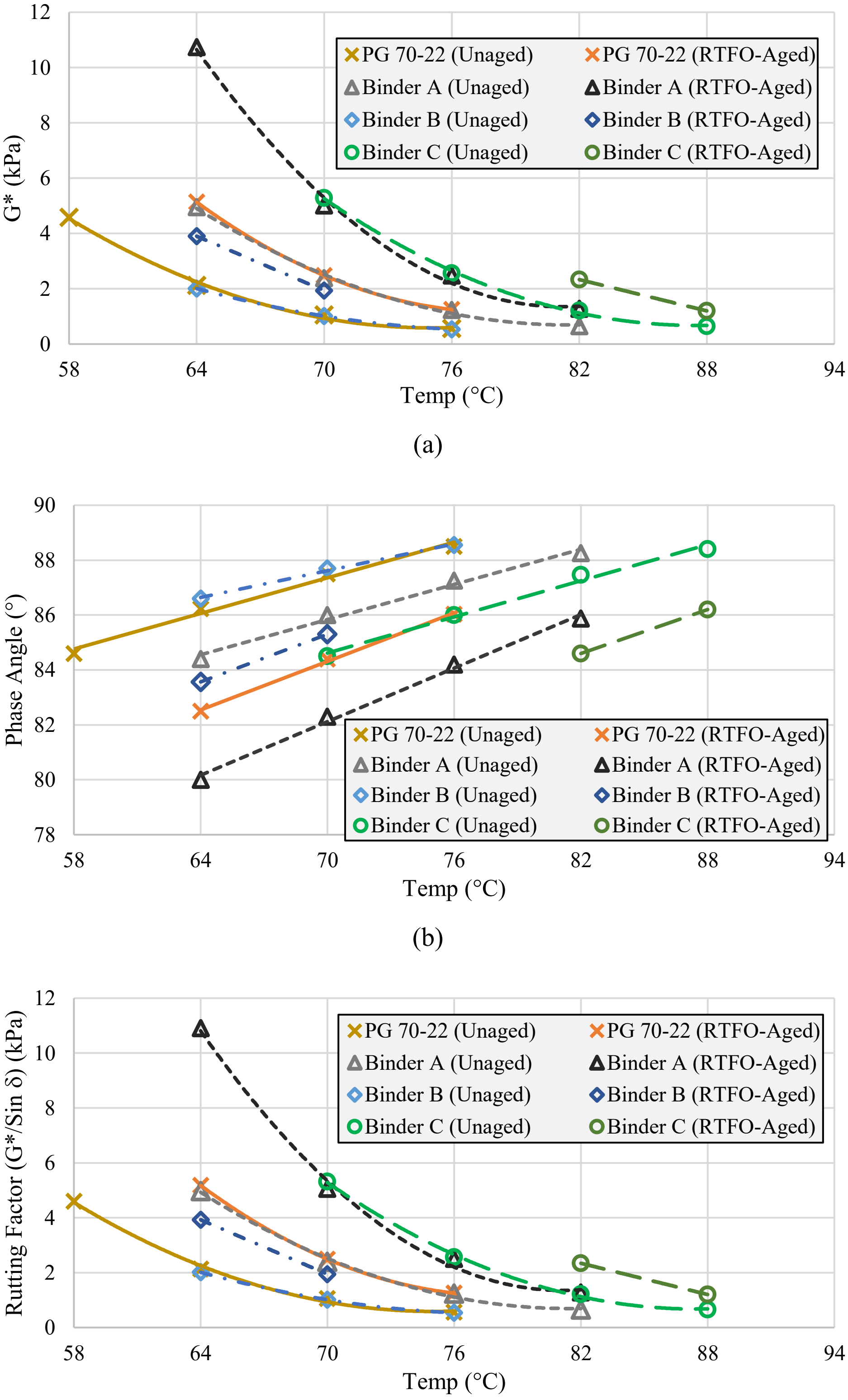
Figure 4-3. Dynamic Modulus Master Curve for Selected Asphaltenes-Modified Binders at the Reference Temperature of 15°C

4.5.4 High Temperature Performance

To compare the response of different binders, their rheological parameters, including complex shear modulus (G^*), phase angle (δ) and rutting parameter ($G^*/\sin\delta$) were evaluated, with the results shown in Figure 4-4. Regardless of the binder type, the phase angle increases with increasing temperature, while the shear modulus and rutting factor decrease as the temperature increases, indicating less elasticity and rutting resistance. However, the rate of these changes decreases at higher temperatures. In addition, aging seems to have a considerable effect on providing better resistance to permanent deformation, as indicated by the rheological testing.

Figure 4-5 shows the high temperature rheological properties of PG 70-22 for different asphaltenes contents. According to this figure, asphaltenes considerably enhance the high temperature performance of the asphalt binder by increasing the shear modulus and elasticity. In addition, a higher slope was observed for RFTO-aged binders compared to unaged binders, as shown in Figure 4-5(a-c). This indicates that as the binder ages, the improving effect of asphaltenes (in terms of increased rutting resistance) is more pronounced.

According to Figure 4-5, given the same aging conditions, the rates of change of both modulus and rutting resistance increase as the temperature decreases, which indicates that asphaltenes have more effect on increasing shear modulus and rutting resistance at lower temperatures. However, the consistent slope for the phase angle reflects that asphaltenes have a similar effect at low and high temperatures for this parameter.

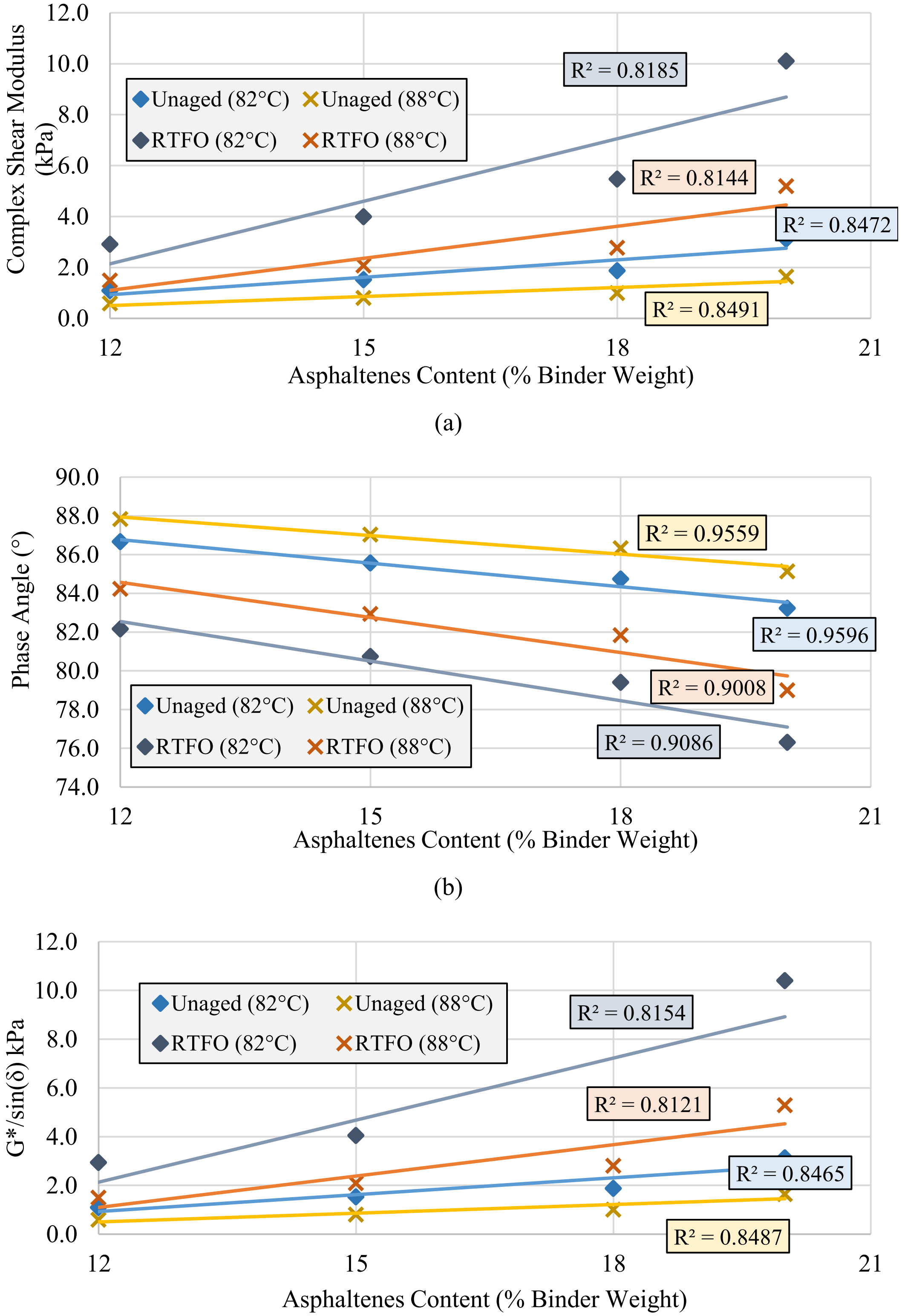


(a)

(b)

(c)

Figure 4-4. High Temperature Rheological Properties of Different Asphalt Binders versus Temperature: (a) Complex Shear Modulus; (b) Phase Angle; (c) Rutting Factor



(a)

(b)

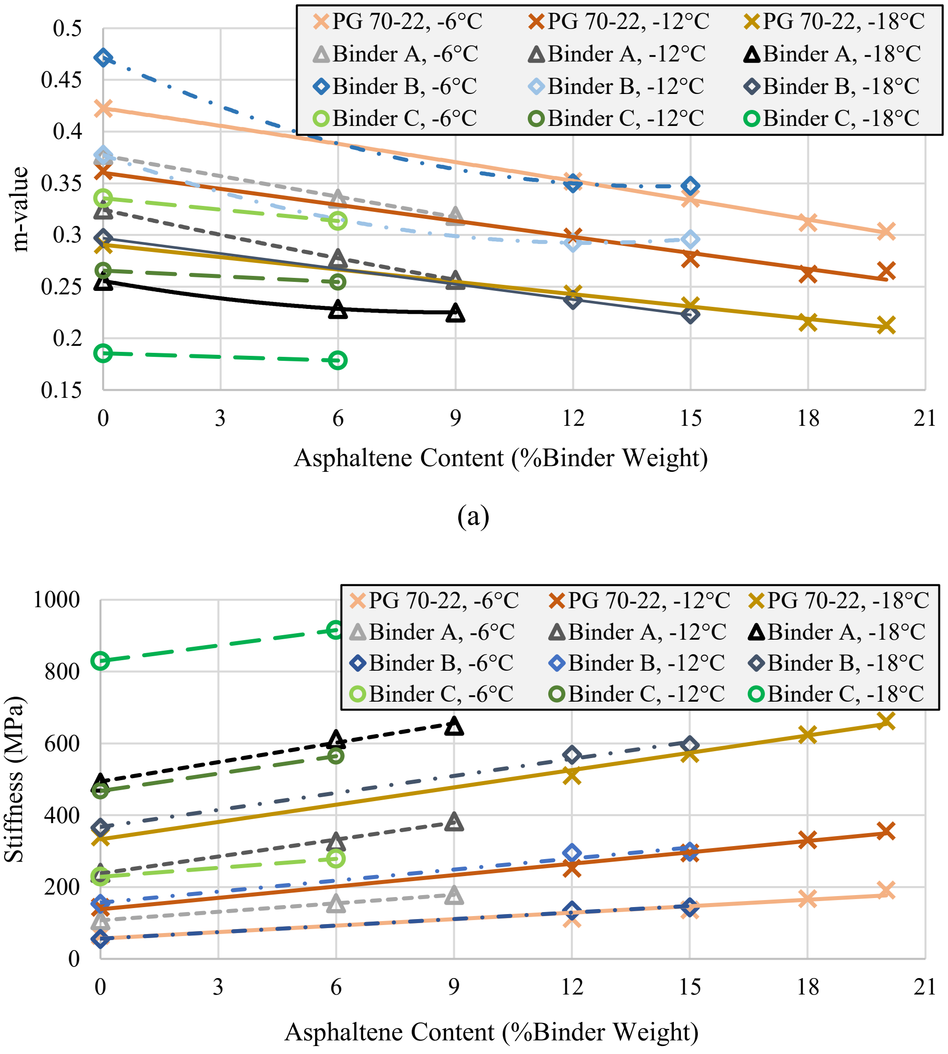
(c)

Figure 4-5. High Temperature Rheological Properties of PG 70-22 versus Asphaltenes Content: (a) Complex Shear Modulus; (b) Phase Angle; (c) Rutting Factor

4.5.5 Low Temperature Performance

The effect of asphaltenes content, temperature, and binder source on the low temperature properties of asphalt binders is shown in Figure 4-6. Generally, an increase in asphaltenes content and decrease in temperature adversely affect the low temperature performance by decreasing the m-value and increasing the stiffness. Also, the greater consistency in the trendlines included in Figure 4-6b compared to Figure 4-6a is an indicator of the greater dependency of m-value on binder source rather than stiffness.

According to Figure 4-6, for each binder, regardless of source, the rate of decrease in m-value and increase in stiffness relative to asphaltenes content seems to be consistent for different temperatures. This indicates that given the response of one binder at different asphaltenes content and a single temperature, its behavior at other temperatures may be predictable.



(a)

(b)

Figure 4-6. Low Temperature Rheological Properties of Asphalt Binders versus Asphaltenes Content: (a) m-Value; (b) Stiffness

4.5.6 High Temperature Viscosity

The viscosity of each asphalt binder over a wide range of high temperatures is shown in Figure 4-7. As observed, all the asphalt binders satisfy the maximum viscosity limitation of 3 Pa·s at 135ºC set in AASHTO M320 (92). Also, generally the addition of asphaltenes to the asphalt binder increases its viscosity at different temperatures. Regardless of the source of the asphalt binder, every 2% increase in asphaltenes content was found to increase the viscosity by approximately 0.1 Pa·s at 135ºC. The convergence of the viscosity curves for different asphalt binders at higher temperatures can also be observed in Figure 4-7. This indicates that the stiffening effect of asphaltenes gradually decreases at higher temperatures.

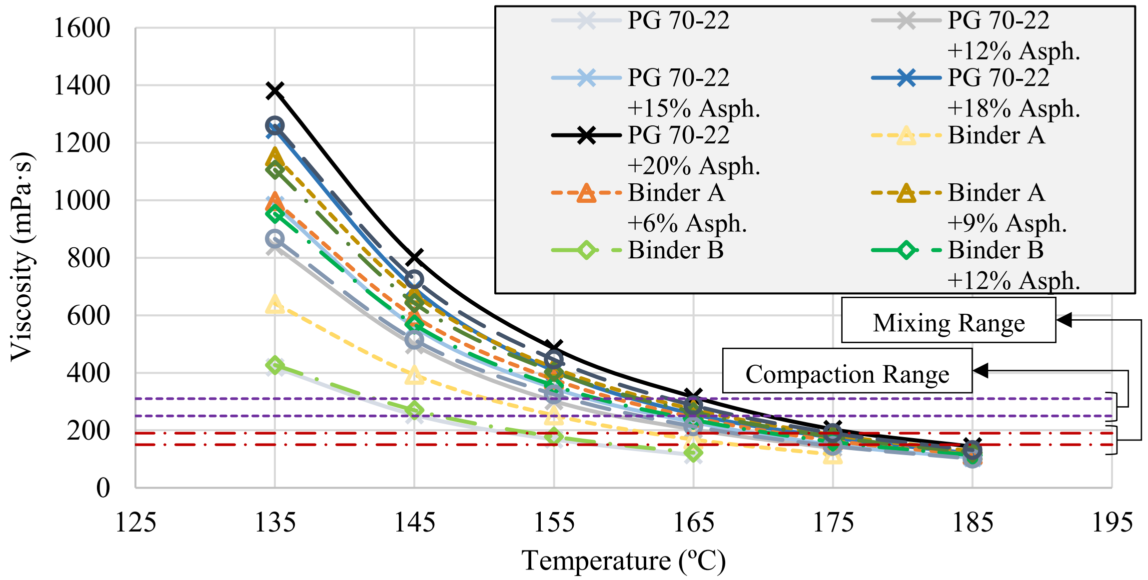


Figure 4-7. Viscosity of Asphalt Binders versus Temperature

The viscosity change for different asphalt binders plotted against asphaltenes content for a temperature range of 135ºC-165ºC is presented in Figure 4-8. According to this figure, the effect of asphaltenes content on viscosity is higher at lower temperatures and higher concentrations.

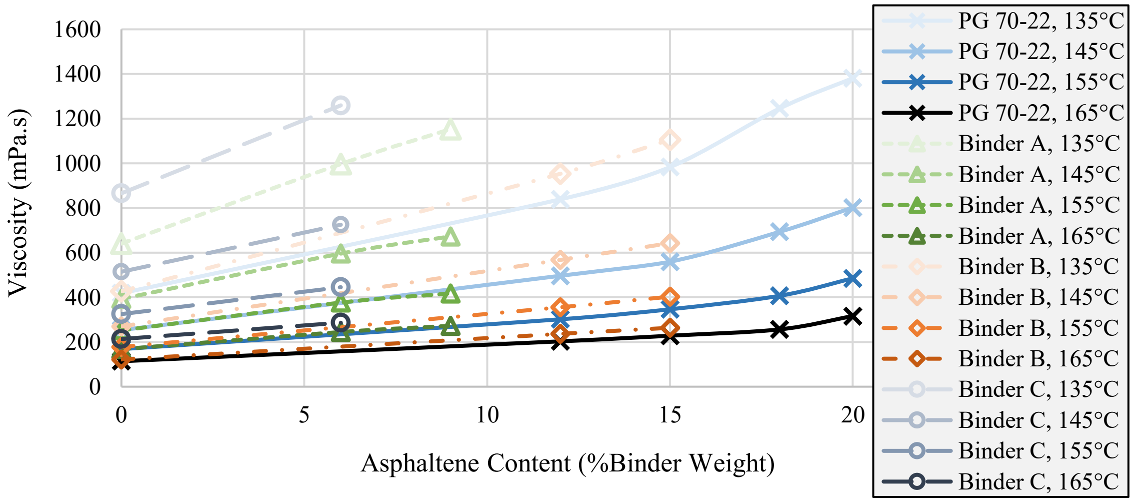


Figure 4-8. Viscosity of Asphaltenes-Modified Binders versus Asphaltenes Content

The results of viscosity measurements over a wide range of temperatures were used to estimate the mixing and compaction temperature ranges for the different asphalt binders (Figure 4-9). As expected, both mixing and compaction temperatures increase with the asphaltenes content, due to the increase in viscosity. According to Figure 4-9, on average, every 1% increase in asphaltenes content of the binder increases the corresponding mixing and compaction temperature ranges by approximately 1.3 ºC, regardless of the binder source.

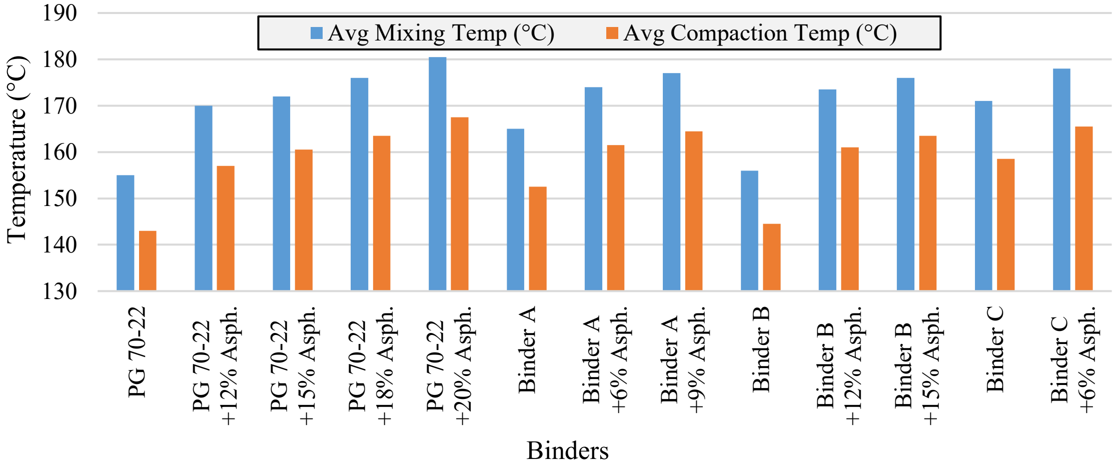
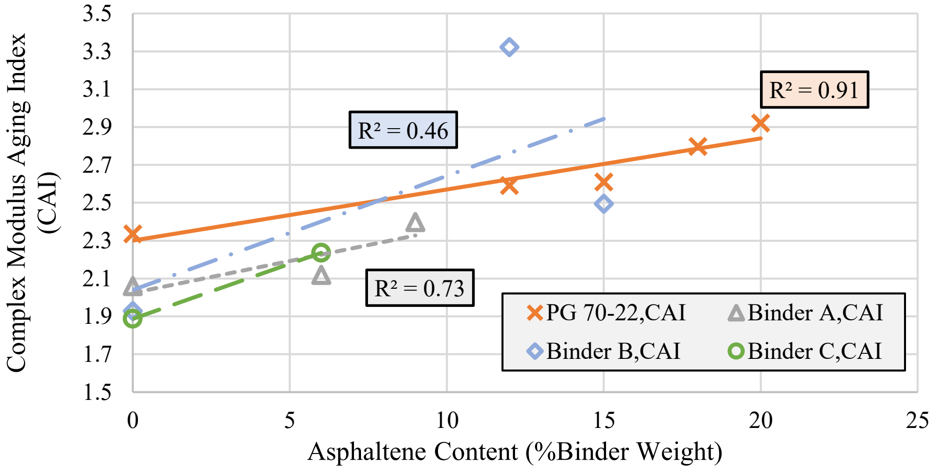


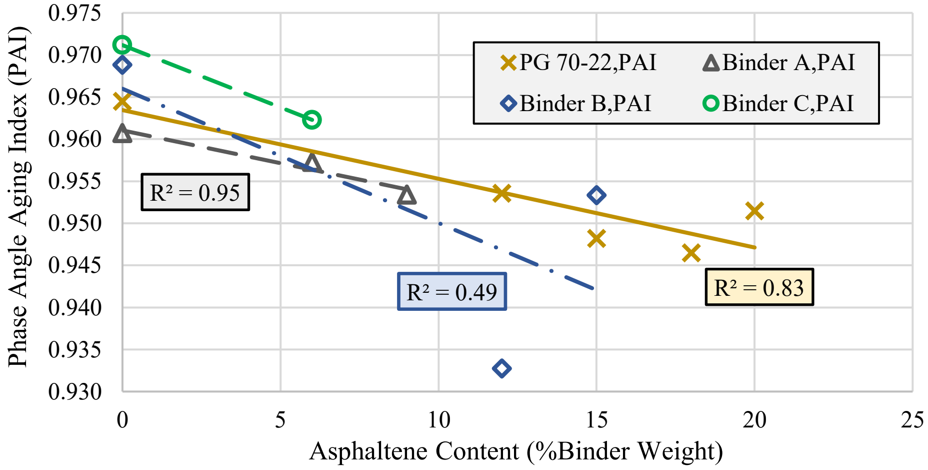
Figure 4-9. Average Mixing and Compaction Temperatures for Different Asphalt Binders

4.5.7 Aging Indices

Two aging indices, CAI and PAI, were estimated for different asphalt binders, as plotted in Figure 4-10. From this, it can be seen that the two indices are independent of each other. The PAI was found to be around 1.0 in all cases, and showed almost no change with either binder source or asphaltenes content. For the case of CAI, however, increasing the asphaltenes content results in an increase of the aging index. These observations indicate that aging does not result in a change in the phase angle, regardless of temperature, binder source or asphaltenes content. Also, according to Figure 4-10a, a higher asphaltenes content intensifies the effect of aging as observed by the changes in complex shear modulus. As can be seen in Figure 4-10a, binders derived from Alberta oilsands (A-C) generally show lower CAI values than PG 70-22 binder, which indicates that they have more stability in terms of aging.



(a)



(b)

Figure 4-10. Aging Indices of the First Group of Modified Binders at 70ºc: (a) Complex Modulus Aging Index; (b) Phase Angle Aging Index

4.5.8 SARA Analysis

All base binders, as well as the modified PG 70-22 binder samples were tested using SARA analysis. SARA analysis was also performed on the bulk asphaltenes used in this work to check its composition, with all results shown in Table 4-6.

Table 4-6. SARA Analysis Results for Chosen Asphalt Binders

Binder/ Material	Saturate	Aromatic	Resin	Asphaltenes	Polar Fraction	Non-polar Fraction	Colloidal Index
Bulk Asphaltenes	6.85	9.68	3.84	79.62	83.46	16.53	6.40
PG 70-22	25.41	20.36	31.58	22.59	54.17	45.77	0.92
PG 70-22 + 12% Asph	21.63	20.94	24.75	32.17	56.92	42.57	1.18
PG 70-22 + 18% Asph	18.83	20.25	25.23	35.16	60.39	39.08	1.19
PG 70-22 + 20% Asph	17.93	19.74	21.57	39.76	61.33	37.67	1.40
Binder A	11.87	33.60	33.50	21.01	54.51	45.47	0.49
Binder B	26.13	17.76	36.32	19.76	56.08	43.89	0.85
Binder C	19.42	23.65	38.52	18.37	56.89	43.07	0.61

According to this table, the addition of asphaltenes considerably increases the polar fraction content of asphalt binders. By comparing the different trends observed in the rheological properties of asphalt binders with the SARA analysis results, it can be observed that an increase in polar fraction content corresponds with an increase in the stiffness, elasticity, and viscosity of the binders.

The colloidal index (CI) of each material was also calculated and presented in Table 4-6. According to this table, the addition of asphaltenes causes CI to increase, indicating less stability of the asphaltenes phase in the surrounding maltene matrix. According to Lesueur (103), the only unstable asphalt binder in Table 4-6 is PG 70-22 modified with 20% asphaltenes, with CI greater than 1.2. Also, it was observed that asphalt binders derived from Alberta oilsands bitumen have lower CI values; signifying their higher stability for asphalt binder compared to the crude oil asphalt binder PG 70-22.

4.6 Conclusions

Five different asphalt binders sourced from different crude oils and Alberta oilsands bitumens were modified with different asphaltenes concentrations to assess their suitability for use in HMAC. The main conclusions of this study based on the test results and analysis are as follows:

- According to predictive 1999 Witczak model along with MEPDG Level 3 binder inputs, the most appropriate PG grading to meet the dynamic modulus requirements of the binders for use in HMAC were determined to be PG 82-16 and PG 88-16.
- Performing dynamic modulus test on PG 70-22+12% Asph, Binder B+12% Asph, Binder C+6% Asph, with continuous PG gradings of 82.9-21.8, 82.2-21.2, and 88.2-16.6, respectively, verified the predicted results from the 1999 Witczak model.
- On average, every 6% addition of asphaltenes causes an increase of one or two intervals in high and intermediate PG grades, respectively, regardless of the source of the binder. At low temperatures, every 10-20% increase in asphaltenes content corresponded to a low PG grade increase of one interval; however, this was found to depend on the binder type.
- An increase in asphaltenes content resulted in a greater improvement in asphalt binder properties at high temperatures. The positive effects at high temperatures were of greater magnitude than the negative effects observed at low temperatures.
- An increase in asphaltenes content adversely affects the low temperature performance of binders, decreasing the m-value and increasing the stiffness.
- Regardless of the source of the asphalt binder, every 2% increase in asphaltenes content was found to increase the binder viscosity at 135ºC by approximately 0.1 Pa·s. An increase in the average mixing and compaction temperatures by 2.6 ºC was also observed for every 2% increase in asphaltenes.
- Aging does not appear to have an effect on the phase angle, regardless of the temperature, binder source or asphaltenes content. However, in the case of the complex shear modulus, a higher asphaltenes content intensifies the effect of aging as observed by the changes in complex shear modulus.
- Binders sourced from Alberta oilsands bitumen have lower CI values, which signifies their higher stability for the asphalt binders compared to crude oil asphalt binder PG 70-22.
- By comparing different trends in the rheological properties of the various asphalt binders with SARA test results, it can be observed that an increase in polar fraction content correlates with an increase in the stiffness, elasticity, and viscosity of the asphalt binder.

Chapter 5. Hard Grade Asphalt Binders

5.1 Introduction

High modulus asphalt concrete has the general requirements to have dynamic modulus values more than 14 GPa at a loading frequency of 10 Hz and a temperature of 15°C (16). Asphalt binder and aggregates are the key elements of HMAC mixes to achieve this requirement (52). The binders used in HMAC mixtures, also referred to as high modulus asphalt binder (HMAB), fall into three different categories. Traditional straight-run hard grade asphalt binder, asphalt binders modified with natural asphalt, and polymer-modified ones (14, 19, 20). Hard-grade paving asphalts refer to asphalt binders having a penetration at 25°C (according to AASHTO T49 (157)) lower than 25 mm/10 (12).

It was until the early 1960s in France that the penetration grades of used paving asphalts were 80/100 and 180/220, which were produced by direct distillation and imported from Central America (12). With increasing traffic loads, the need for a higher rigidity to increase the rutting resistance of asphalt pavements (14), led to introduction of hard grade binders in France. Production of 40/50 and 60/70 PG asphalts started in 1966 and the first 20/30 PG appeared in 1968 (12). Air-blowing technique was used to further increase the rigidity of asphalt binders to have a higher resistance to permanent deformation in late 1960s (12). Although effective with respect to rutting resistance, using air-blown asphalt binder led to extensive cracking at the pavement surface (12).

Later in 1980s, hard grade binders were common to be used in high modulus asphalt concrete (HMAC) mixes that were first applied on rehabilitation projects with restricted excavation depth (12). This type of mixture was originally developed by French Road Institute or Laboratoire Central des Ponts et Chaussées (LCPC) in cooperation with road enterprises (13). High modulus asphalt concrete, or Enrobé a Module Élevé (EME) in French, was mainly used in construction of base courses or binder courses of pavement structures (27).

It is conventional in HMAC mixes to mention the asphalt binders according to their penetration grades (14, 15, 54, 72). However, it has been shown that grading asphalt binders according to the penetration depth of a 100 gr needle at 25°C, is not an accurate grading system, as the properties of different binders with the same penetration grade can vary considerably (12).

5.2 Objectives

The objective of this chapter is to investigate and compare the rheological properties of hard grade binders, with penetration grades that make them potential candidates to be used for HMAC applications.

5.3 Materials

In this section, rheological properties of 10 hard grade binders from different sources are studied. The PG 70-22 binder from crude oil was also tested as the control binder. One of these asphalt binders was produced using air-blowing technique (PG 76-28 air-blown), another one (EME Pen 20/30) was a common hard grade binder used in high modulus asphalt concrete in France, five other binders (referred to as Q8 binders) were produced with penetration grades ranging from 5/15 to 50/70, and three remaining ones were named Vacuum Tower, Cold Lake, and Pitch.

5.4 Methods

In accordance with AASHTO T315 (90), a dynamic shear rheometer (DSR) (Smartpave, Model 102, Anton Paar, Co., Ltd., Austria) was used to characterize the viscoelastic behavior of the asphalt binders at high and intermediate temperatures. The rheological properties of the asphalt binders at low temperatures were evaluated using a bending beam rheometer (BBR) (Cannon Instrument Company®, USA), according to AASHTO T313 (94). A Brookfield rotational viscometer was used to measure the viscosity of the asphalt binders at elevated temperatures, using AASHTO T316 (145). Asphalt binders were short-term and long-term aged using a rolling thing film oven (RTFO), and a pressure aging vessel (PAV), according to AASHTO T240 (88) and AASHTO R28 (89), respectively.

5.5 Results and Discussion

5.5.1 High Temperature PG Grading

According to section 4.5.1, the minimum PG grade of binder to meet the dynamic modulus criteria for HMAC mixes was estimated and verified in section 4.5.1, to be PG 82-16. Asphalt binders were tested at high temperatures to grade them according to AASHTO M320 (92), and the ones with high PG grades less than 82°C disqualified as a proper binder to be used in HMAC mixes, and excluded from further testing. The results of grading different asphalt binders at high temperatures are shown in Table 5-1.

Table 5-1. High PG Grading Results for Hard Grade Binders

Binder	**High PG Grade, °C**		**Continuous High PG Grade, °C**	**Standard High PG Grade, °C**	**Satisfied Minimum of 82°C?**
	Unaged Binder	**RTFO-Aged Binder**			
PG 70-22	70.5	71.1	70.5	70	No
PG 76-28 (Air-blown)	78.7	83.0	78.7	76	No
EME Pen 20/30[1]	84.0	83.7	83.7	82	Yes
Vacuum Tower	88.9	90.2	88.9	88	Yes
Cold Lake	86.1	85.9	85.9	82	Yes
Pitch	106.5	104.3	104.3	100	Yes
Q8 Pen 5/15[1]	96.5	94.3	94.3	94	Yes
Q8 Pen 15/25[1]	89.1	85.9	85.9	82	Yes
Q8 Pen 20/30[1]	84.4	82.4	82.4	82	Yes
Q8 Pen 35/50[1]	77.8	76.0	76.0	76	No
Q8 Pen 50/70[1]	73.4	71.7	71.7	70	No

1. Penetration grade of binder according to AASHTO T49 (157)

5.5.2 Intermediate and Low Temperature PG Grading

Table 5-2 shows the PG grading results for binders with minimum of high PG grade pf 82°C at low and intermediate temperatures, using DSR and BBR, respectively. This table also includes the final PG grading results for these binders.

Table 5-2. Final PG Grading Results for Qualified Hard Grade Binders

Binder	**High PG Grade, °C**	**Low PG Grade, °C**		**Continuous Int. PG Grade, °C**	**Continuous PG Grade, °C**	**Standard PG Grade, °C**
		m-value Criterion	**Stiffness Criterion**			
PG 70-22	70.5	-27.2	-27.1	19.2	70.5-27.1	70-22
EME Pen 20/30	83.7	-15.5	-19.7	35.7	83.7-15.5	82-10
Vacuum Tower	88.9	-9.7	-16.9	41.1	88.9-9.7	88-4
Cold Lake	85.9	-18.4	-20.0	33.5	85.9-18.4	82-16
Pitch	104.3	N/A		64.4	104.3-N/A	100-N/A
Q8 Pen 5/15	94.3	-8.7	-7.9	48.2	94.3-7.9	94-4
Q8 Pen 15/25	85.9	-15.0	-15.0	39.6	85.9-15.0	82-10
Q8 Pen 20/30	82.4	-16.5	-16.7	36.7	82.4-16.5	82-16

From Table 5-2, it can be seen that the low PG grade of the binders ranges between -4 to -22 , the binder PG 70-22 have the highest low temperature flexibility , while the Vacuum Tower and Q8 Pen 5/15 binders have the least performance at low temperature.

It is notable that in case of low temperature performance of Pitch binder, even at the test temperature of +12°C (which is representative of performance at +22°C) the binder did not pass the criteria for low PG grade. This indicates that Pitch binder is a very stiff and brittle binder and not proper to be used in asphalt construction in any region.

Comparing the low temperature performance of the studied hard grade binders, with that of asphaltenes-modifed binders (Table 4-4), it can be observed that with similar high PG grades, asphaltenes-modified binders fail at lower temperatures, which makes them better choice in regions with cold climates.

5.5.3 High Temperature Viscosity

To ensure that the workability of the binders at high temperatures is sufficient, AASHTO M320 (92) restricts the maximum viscosity of binders to 3 Pa·s at a temperature of 135°C. The viscosities of the binders measured at this specific temperature is presented in Table 5-3. According to this table, the only binder that does not pass the workability criterion is the Pitch binder, with which has a viscosity of 3.21 Pa·s at 135°C.

Table 5-3. Viscosities of Hard Grade Binders at 135°C

Binder	**Rotational viscosity at 135°C (Pa·s)**	**Less than 3 Pa·s?**
PG 70-22	0.42	Yes
EME Pen 20/30	0.79	Yes
Vacuum Tower	1.11	Yes
Cold Lake	0.97	Yes
Pitch	3.21	No
Q8 Pen 5/15	2.05	Yes
Q8 Pen 15/25	1.26	Yes
Q8 Pen 20/30	0.94	Yes

Using the viscosity readings over a wider range of high temperatures, the mixing and compaction temperatures ranges for the asphalt mixtures containing each binder is was estimated, and with the result is shown in Table 5-4. According to Superpave, asphalt samples require mixing and compaction under equiviscous temperature conditions corresponding to 170±20 mPa·s and 280± 30 mPa·s, respectively.

Table 5-4. Mixing and Compaction Temperature Range of Binders

Binder	**Mixing Temperature Range (°C)**	**Compaction Temperature Range (°C)**
PG 70-22	152-158	141-145
EME Pen 20/30	164-170	153-158
Vacuum Tower	172-178	160-165
Cold Lake	168-175	157-162
Pitch	184-189	174-178
Q8 Pen 5/15	184-191	172-177
Q8 Pen 15/25	176-182	163-169
Q8 Pen 20/30	169-175	157-163

5.6 Conclusions

Several hard grade binders with the potential to be used in high modulus asphalt concrete applications were studied and compared. According to the results the following conclusions can be drawn:

- Depending on the source and penetration grade of hard grade binders, they can be able to achieve the minimum performance grade requirement of a binder to be used in HMAC applications.
- Regarding the low temperature performance, with similar high PG grades, asphaltenes-modified binders fail at lower temperatures, which makes them better choices in regions with cold climates.

Chapter 6. High and Low Temperature Rheological Evaluation of Oilsands Asphalt Distilled at Different Temperatures

6.1 Introduction

The vast majority of roads throughout the world are made of flexible pavements consisting of asphalt binder and aggregates. Flexible pavements play an increasingly important role in terms of goods conveyance and movement of people; this is because of their numerous advantages, which include good performance during their service life, high driving comfort, safety, and easy maintenance (103, 158).

Asphaltic pavements are generally designed to withstand an enormous traffic load during their service life. Nevertheless, traffic loads, in addition to severe environmental conditions in terms of temperature, oxygen and ultraviolet light, alter the asphalt's binder properties, making it more vulnerable to distresses such as cracking and deformation (159). The onset of such distresses greatly decreases the performance as well as the service life of pavement (160, 161). Therefore, a thorough understanding of asphalt binder rheological properties will greatly assist in the selection of the appropriate asphalt binder for higher performance and longer service life through the minimisation of potential distresses.

Asphalt binder is a heterogenous and viscoelastic material in properties, and it is made of up of high molecular asphaltenes dispersed in maltenes or oily media of a lower molecular weight (103, 162, 163). The asphaltenes and maltenes present in asphalt binder determine its rheological linear viscoelastic (LVE) properties, which range from Newtonian behavior to non-Newtonian behaviour (164).

According to polarity, asphalt binder chemical composition is classified to saturates, aromatics, resins, and asphaltenes, abbreviated as SARA (95). Polarity of asphalt binder compounds and their interactions play an important role in rheological properties of asphalt binder (109).

Polar fractions of asphalt binder, including asphaltenes and resins, imparts elastic behavior to asphalt binder (98), while the non-polar part, including saturates and aromatics, is responsible for asphalt binder viscous behavior. Due to difference in polarity, asphaltenes particles might agglomerate and become instable in surrounding matrix composed of remaining fractions (102).

Asphalt binder is characterized by its rheological properties, which in turn depend heavily on temperature. These properties are governed by the physical and chemical interactions of the asphalt-respective SARA constituents (147, 165). At low temperatures, stress considerably increases in asphalt pavement and, if this stress reaches the material strength limit, cracking will develop on the pavement surface. Cracking of asphalt pavements due to low temperature is a major concern globally, especially in cold-temperature regions (147). Evaluation of asphalt's low-temperature properties is thus a key research focus at present (165, 166).

The price of oil globally has increased considerably in the past decades, and the cost of asphalt material at those times has also increased accordingly to almost double its cost during that decades. Furthermore, the demand for fuel in those past decades, as well as other petroleum products, surpasses the quantity obtained from existing crude oil sources (167). Hence, during that period, it becomes essential to identify alternative asphalt sources in addition to new methods of pavement construction to meet the asphalt demands at that time. In addition, as readily available light crude oil deposits are getting exhausted around the world during those decades, countries with alternative sources of crude increasingly exploits the heavier and less accessible oil sources that need additional processing (168). Most recently, there have been significant efforts around the world within the petroleum industry to utilize low-cost heavy oil fractions and to exploit additional petroleum products from heavy oil and oilsands (bituminous sand) sources (169).

Oilsands are among the largest naturally occurring deposits of crude oils globally. Oilsands contain crude oil suspended in an ore that is a combination of sand, silt/clay particles, heavy oils, minerals, and water. Due to the significant differences in composition and properties between oilsands and conventional petroleum, there is a considerable degree of complexity, as well as some additional refining steps, involved in converting the oilsands into liquid petroleum products. These additional procedures, which require different refining technologies, make it more difficult to process than conventional petroleum (170).

In the past, the behavior and grading of asphalt binders have been evaluated using conventional tests such as penetration and viscosity tests. These conventional tests do not specifically address field performance distress modes, as they only grade asphalt binders based on their consistency, and neither do they describe the full viscoelastic properties for rheological analysis (171). In current practice, to attain a detailed understanding of viscoelastic properties of asphalt binders at low, intermediate, and high temperatures, a fundamental rheological examination introduced by

Superpave is utilized. The Superpave technique determines the viscous and elastic behaviors of asphalt binders at varying temperatures and frequency ranges (172).

6.2 Research Objective

The main objective of the research presented in this paper was to investigate the low- and high-temperature rheological performance as well as PG grading of different oilsands bitumen binders derived from Alberta oilsands and distilled at different temperatures, and to compare them with various commercially available crude oil source asphalts.

6.3 Materials and Methodology

6.3.1 Materials

For this study, two commercially available crude oil performance-graded asphalt binders, PG 58-31 and PG 64-22 from Husky Energy, and three asphalt binders from three different sources of oilsands bitumen binders in Alberta, Canada, were used for the rheological investigation (the performance-grade asphalts were used as the control asphalts for comparison with oilsands asphalts). The crude oil asphalts used have the following properties: they appear as a black, viscous liquid with a softening point greater than 31°C, have a boiling range above 228°C, a flash point greater than 243°C, as well as a relative density of 1.02 to 1.04 at 15°C and 1 atmosphere, and a viscosity of 0.100 to 0.800 Pascal-seconds at 135°C.

The oilsands bitumens were obtained from three different sources of Alberta's oilsands and are accordingly referred to herein as Asphalt A, Asphalt B, and Asphalt C. The oilsands binders used were distilled at three different temperatures: 400°C, 430°C, and 460°C.

6.3.2 Methodology

6.3.2.1 Brookfield Rotational Viscosity

For this study, a Brookfield rotational viscometer (RV) was utilised for the measurement of high-temperature viscosity. An RV measures viscosity by determining the torque required in order to maintain a constant rotational speed of 20 revolutions per minute (RPM) for a submerged cylindrical spindle in an asphalt sample under a constant temperature. The amount of torque obtained is automatically converted by the RV equipment in to a viscosity value expressed in mPa.s. In our study the viscosity was measured at different temperatures in accordance with standard AASHTO T316 (145). Asphalt samples of approximately 10.5 grams were used for determining the viscosity values, and RV spindle number 27 with a varying rotational speed was employed.

6.3.2.2 Aging Process

Laboratory simulation of the short-term aging of asphalt that typically occurs during production and compaction of hot mix asphalt mixtures was carried out using a rolling thin film oven (RTFO) following standard AASHTO T240 (88). In this method, asphalt samples of approximately 35 grams are placed into RTFOT glass containers. After placing, the RTFO containers with asphalts samples are transferred to the RTFO carriage inside the oven. Each container is placed such that its top opening is directly facing a jet of air supply inside the oven. The oven is closed, and the temperature is maintained at 163°C with the carriage set at a rotating at speed of 15 rpm for the 85-minute aging process to take place.

For assessing the intermediate and low-temperature performance and grade of the asphalts, a sample from the residue of the RTFO aged was used to simulate long-term aging in the asphalts using a pressure-aging vessel (PAV) following AASHTO R28 (89). This simulated the aging of a binder during its service life (long-term aging). Approximately 50-gram samples of the RTFO residue were transferred into individual PAV pans and placed in a PAV sample rack. The samples were then subjected to a pressure of 2.1 MPa and constant temperature of 100°C for a duration of 20 hours. The residue in each pan was scraped into a single container and placed inside a vacuum oven set at 170°C for 30 minutes to degas the sample.

6.3.2.3 Dynamic Shear Rheometer (DSR) Test

To gain understanding of the intermediate- and high-temperature rheological properties of the asphalts, a DSR (SmartPave 102, Anton Paar, Austria) was used. DSR, it should be noted, is capable of determining rheological characteristics of asphalts under a condition of sinusoidal loading. A parallel plate and 8 mm spindle with 2 mm gap (or with a 25 mm spindle and 1 mm gap, depending on test temperature) was used for the rheological evaluation, in compliance with Superpave binder specifications and AASHTO T315 (90). The main rheological viscoelastic properties, expressed in terms of complex modulus (G^*) and phase angle (δ), were used to calculate the performance-related criteria (performance grade) of the asphalts. Three duplicate samples of each of the asphalts were tested, and the average of the three test samples determined for use in the analysis.

6.3.2.4 Bending Beam Rheometer (BBR)

A Cannon BBR rheometer was used to determine the cracking resistance of the asphalt at low temperatures in accordance with AASHTO M320 (92). In this method, two replicate asphalt samples are prepared for each test sample, where the parameters, creep stiffness (S), and creep rate (m-value) are used to determine the asphalt's low-temperature PG. (The low-temperature PG is understood to be the temperature at which both values of creep stiffness (S) are less than 300 MPa and both m-values are greater than 0.30 at 60 seconds of loading.) In this study, BBR test was carried out on PAV aged samples at four different temperatures, where the dimensions of the samples were 125 × 6.35 × 12.7 mm and the temperatures tested were −6, −12, −18 and −24 °C. Samples after preparation were conditioned for one hour prior to testing in an ethanol bath at the required test temperature. During the test, a load of 980 ± 50 mN was constantly applied on each beam sample, and the resulting deflection on the beams was estimated at loading times of 8, 15, 30, 60, 120, and 240 seconds. Using these time intervals, a plot of load and deflection against time was drawn; from the graph, the stiffness (S) and creep rate (m-values) were calculated at 60 seconds for each of the test samples.

6.3.2.5 SARA Fraction Separations

Separation of the asphalt samples into respective SARA constituents was carried out in accordance with ASTM standards. The asphaltene was separated in accordance with ASTM D6560 (156), while the remaining maltenes fractions were separated by a gravity-driven chromatography method in accordance with ASTM D2007 (156). The residues obtained after n-heptane precipitation called maltenes were further fractionated into saturate, aromatics, and resin after passing through a chromatographic column.

Colloidal instability index (CI), which is an indicator of asphaltene deposition in asphaltic material, is a significant factor in defining the stability of asphalt binders, where the SARA result is used to estimate the CI value. The CI is estimated as the ratio of the sum of saturates and asphaltenes to the ratio of corresponding sum of resins and aromatics, as shown in Equation 7-1.

$$\text{Colloidal Instability Index (CI)} = \frac{Asphaltene + Saturates}{Aromatic + \mathrm{Re}\sin s} \qquad (7\text{-}1)$$

6.4 Results and Discussion

6.4.1 Rotational Viscosity

Asphalt viscosity at high temperature is a significant property that describes the workability of the asphalt sample at high temperatures. Sufficient workability for asphalt binder is necessary to ensure the asphalt's ability to be pumped in the asphalt manufacturing plant, adequately coated with aggregate in hot mix asphalt, and placed and compacted easily (173). Figure 6-1 presents the viscosity over a wide range of temperatures for crude oil asphalts and oilsands bitumen binders distilled at 430°C, while Figure 6-2 presents the viscosity of the crude oil asphalts and oilsands bitumens binders distilled at 460°C.

As shown in Figure 6-1, in the present study the viscosity of the asphalt was found to decrease as the test temperature increased, irrespective of asphalt source. Moreover, it can be noted that Asphalt A was found to have the lowest viscosity, while PG 64-22 showed the highest viscosity at all test temperatures. Asphalt B and Asphalt C were found to have higher viscosities than that of PG 58-31 and lower than that of PG 64-22. The higher viscosity observed in PG 64-22 indicates better workability as compared to PG 58-31 and other oilsands bitumens binders.

As shown in Figure 6-2, a similar trend was also observed with respect to reduction in viscosity values as the testing temperatures increased, irrespective of asphalt source. However, it is apparent that the oilsands bitumen binders distilled at 460°C have greater viscosity values than the crude oil asphalt binders PG 58-31 and PG 64-22. Comparing the oilsands binders distilled at 430°C and 460°C, it can be seen that the asphalt binders produced at distillation temperature of 460°C were found to have the higher viscosity values, irrespective of binder source, as compared to those distilled at temperature of 430°C. This indicates that the binders produced at 430°C will have higher workability as compared to the binders distilled at the higher temperature.

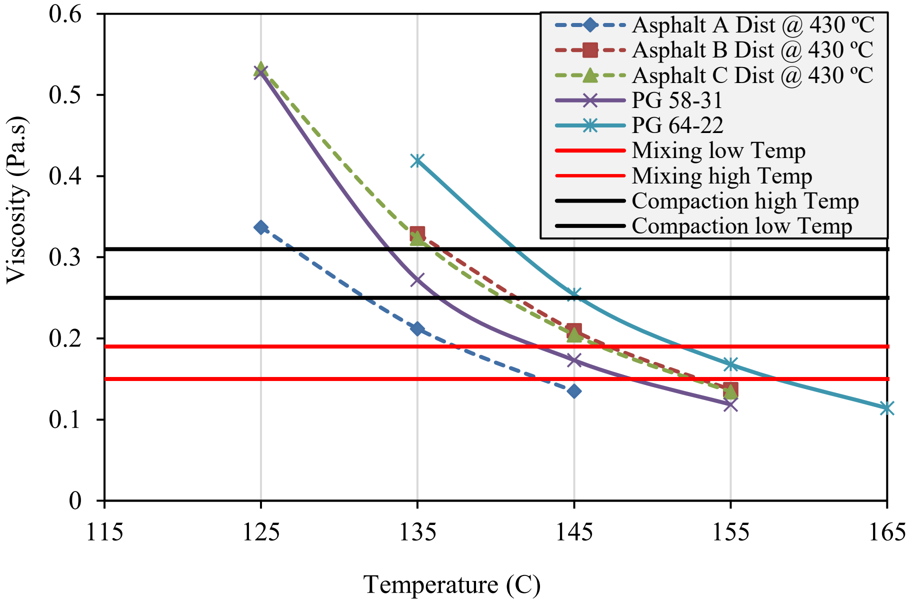


Figure 6-1. Viscosity Values of Crude Oil Asphalts and Oilsands Asphalts Distilled at 430°C

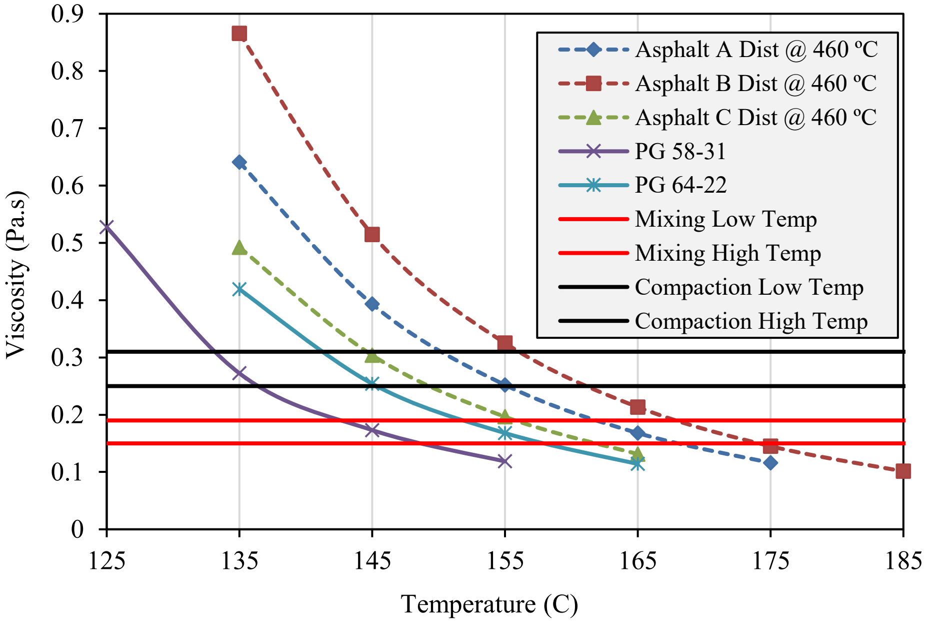


Figure 6-2. Viscosity Values of Crude Oil Asphalts and Oilsands Asphalts Distilled at 460°C

Table 6-1 presents the viscosities of the asphalts binders at 135°C for PG 58-31, PG 64-22 and oilsands bitumens binders distilled at 430°C and 460°C. In accordance with Strategic Highway Research Program (SHRP) binder specifications, the asphalt viscosity value should not exceed 3 Pa.s at a temperature of 135°C to ensure adequate workability. As presented in Table 6-1, all the viscosity values were found to be lower than 3 Pa.s at 135°C, regardless of asphalt binder source, and thus they were found to satisfy the SHRP specification. The oilsands asphalt binders distilled at 460°C were observed to have the highest viscosities at 135°C, indicating that they are stiffer than the binders distilled at 430°C and the other two asphalts tested (PG 64-22 and PG 58-31). As mentioned, it was also found that all the viscosity values were less than 3 Pa at 135°C, a notable finding considering that viscosities exceeding this threshold result in more energy consumption due to the higher mixing and compaction temperature required.

Table 6-1. Rotational Viscosities at 135°C

Distillation Temp °C	**Asphalt**	**Rotational Viscosity at 135 °C (Pa.s)**	**Less than 3 Pa·s?**
N/A	PG 52-31	0.27	Pass
	PG 64-22	0.42	Pass
430	Asphalt A	0.21	Pass
	Asphalt B	0.33	Pass
	Asphalt C	0.32	Pass
460	Asphalt A	0.64	Pass
	Asphalt B	0.87	Pass
	Asphalt C	0.49	Pass

6.4.2 Mixing and Compaction Temperatures

It is desirable to use low temperatures for both mixing and compaction of hot mixed asphalt mixtures in order to reduce energy consumption. Table 6-2 shows the mixing and compaction temperature ranges for the different asphalts under investigation in our study. For the oilsands bitumens, the increases in distillation temperature were found to result in an increase in the mixing and compaction temperatures of the asphalt samples. Asphalt A increased by 19°C and 21°C, while Asphalt B increased by 15°C and 14°C for mixing and compaction, respectively. However, the effect of distillation temperature on Asphalt C was insignificant compared to its effect on the other oilsands asphalt binders. Comparing PG 58-31 and PG 64-22 with the oilsands bitumens, it can be noted that oilsands bitumens distilled at 460°C have both the highest mixing and compaction temperatures, while those distilled at 430°C and PG 58-31 have lower (and similar) values of mixing and compaction temperatures. Additionally, PG 64-22 was found to have higher mixing and compaction temperatures slightly less than those of oilsands asphalts distilled at 460°C. The higher mixing and compaction temperatures found for oilsands bitumens distilled at 460°C and PG 64-22 is an indication of an increase in stiffness of the asphalt binders, which in turn corresponds to an increase in the energy requirement during construction.

Table 6-2. Mixing and Compaction Temperature Ranges

Distillation Temp (°C)	**Asphalt**	**Mixing Range (°C)**	**Compaction Range (°C)**
N/A	PG 58-31	143-148	133-137
	PG 64-22	152-158	141-145
430	Asphalt A	138-143	127-129
	Asphalt B	148-153	136-141
	Asphalt C	148-153	136-141
460	Asphalt A	162-168	150-155
	Asphalt B	168-174	156-161
	Asphalt C	156-161	145-149

6.4.3 Failure Temperature (High Temperature)

A failure temperature is a specific temperature whereby a G*/sin δ value for an asphalt sample is less than 1.0 kPa, where a high failure temperature is associated with a low rutting potential of asphaltic pavements (173). Figure 6-3 presents the failure temperatures for the different asphalt samples under investigation. It can be seen that PG 64-22 and the asphalt binders distilled at 460°C were found to have failure temperatures higher than 70°C, while PG 58-22 and the asphalt binders distilled at 430 °C were found to have failure temperatures lower than 65 °C, with the exception of Asphalt B and Asphalt C distilled at 430°C. The asphalts distilled at 430°C were all found to have failure temperature higher than 55°C but lower than 67°C, while the asphalts distilled at 400°C were found to have the lowest failure temperature among the asphalts studied (lower than 55°C). Additionally, for the oilsands asphalt binders, the failure temperature was observed to vary with the distillation temperatures, meaning that it increased along with an increase in distillation temperature. Asphalt B was found to have the highest failure temperature at all distillation temperature ranges, thus indicating Asphalt B is the stiffest of the oilsands bitumens binders under investigation.

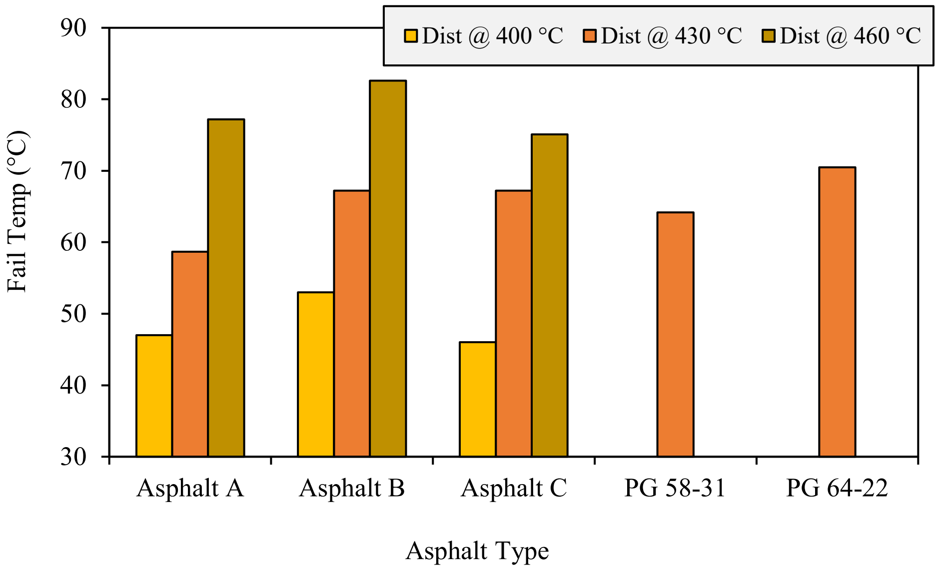


Figure 6-3. Failure Temperatures of Binders (at High Temperatures)

6.4.4 High Temperature Rutting Resistance (G*/Sin δ)

A high temperature specification G*/sin δ ratio of RTFOT aged asphalt binder is often used as the rutting resistance indicator for asphalt binders. For the aged RTFOT asphalt sample, Superpave criteria specified a minimum G*/sin δ value of 2.2 kPa at higher temperatures (92, 174). Generally, a stiffer asphalt binder provides a higher G*/sin δ ratio, which is indicative of a high rutting resistance potential. Figure 6-4 presents the plot of the G*/sin δ ratio at different temperatures for PG 58-31, PG 64-22, and the oilsands bitumens distilled at 430°C, while Figure 6-5 shows the G*/sin δ ratios for PG 58-31, PG 64-22, and the oilsands bitumens distilled at 460°C. Figure 6-4 shows that the G*/sin δ value decreases as the temperature increases, irrespective of asphalt binder source. All asphalt binders were found to have G*/sin δ ratios higher than 2.2 kPa at 64°C, with the exception of Asphalt A. Asphalts B and C distilled at 430°C were both found to have a G*/sin δ ratio higher than that of PG 58-31 and lower than that of PG 64-22. Moreover, PG 64-22 was found to have a higher G*/sin δ ratio than the oilsands bitumens distilled at 430°C had, indicating better resistance to rutting deformation than all other binders.

From Figure 6-5 it can be seen that the oilsands bitumens were found to have a higher G*/sin δ ratio than the PG 58-31 and PG 64-22 had. The oilsands bitumens were found to have G*/sin δ ratios higher than 2.2 kPa at 80°C, while those of PG 58-31 and PG 64-22 are higher than 2.2 kPa at 66°C and 74°C. This indicates higher rutting resistance potential for the oilsands asphalts distilled at 460°C. In addition, the asphalts distilled at 460°C were found to have higher G*/sin δ ratios than the oilsands asphalts distilled at 430°C had. The higher values of G*/sin δ observed for all asphalts binders distilled at 460°C in comparison with the asphalts distilled at 430°C is indicative of their superior high-temperature resistance properties (175). The results show that higher distillation temperatures serve to increase the asphalt's stiffness, which in return increases the rutting parameter (G*/sin δ), thus enhancing the pavement's resistance to rutting deformation at high temperatures. It can also be seen that the oilsands bitumens distilled at higher temperatures will have greater rutting resistance compared to the crude oil asphalt binders.

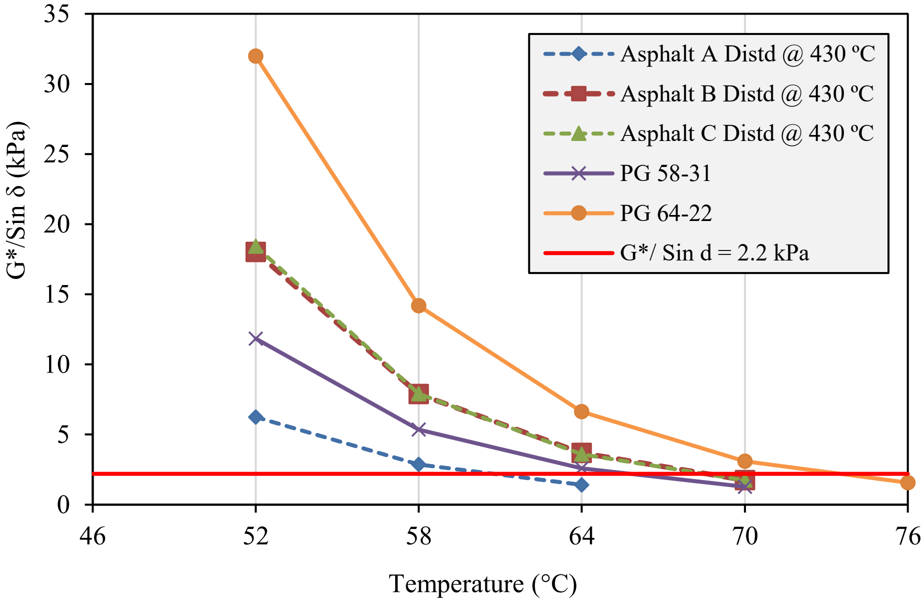


Figure 6-4. Rutting Resistance (G*/Sin δ) of PG 58-31, PG 64-22 and Asphalts Distilled at 430°C

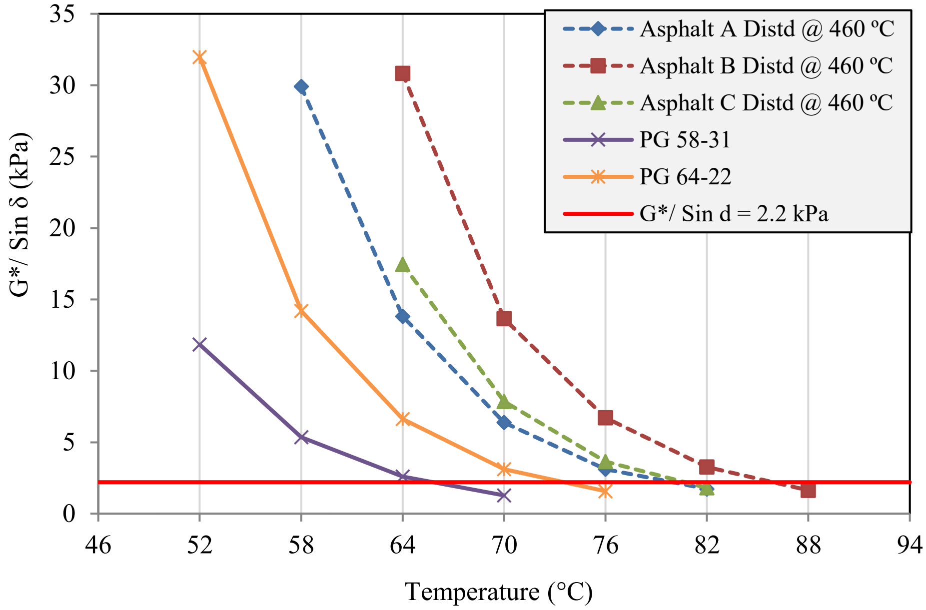


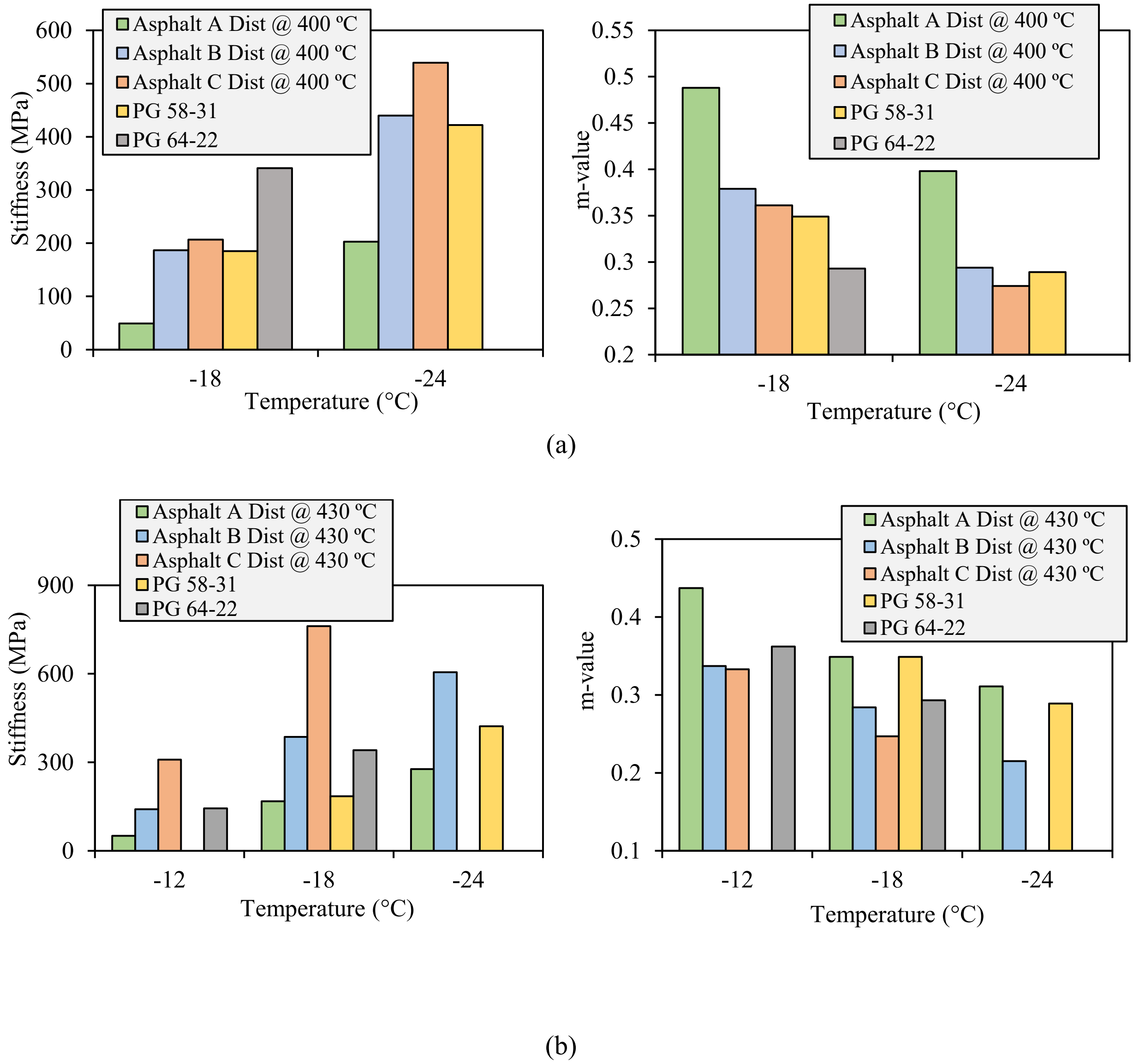
Figure 6-5. Rutting resistance (G*/sin δ) of PG 58-31, PG 64-22 and asphalts distilled at 460°C

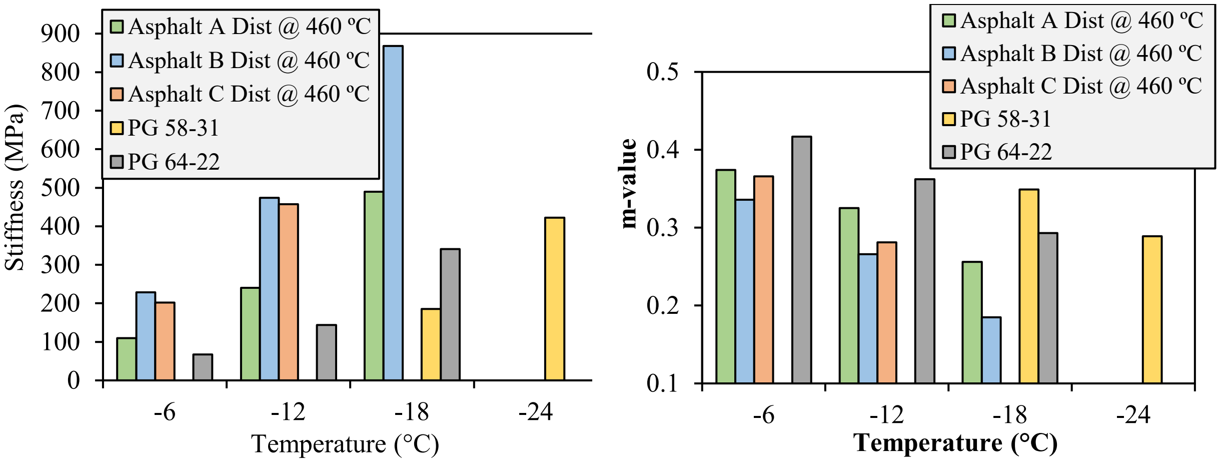
6.4.5 Creep Stiffness (Low-temperature Cracking)

A BBR test was conducted to determine the low-temperature performance of the different asphalt binders. The creep stiffness and m-value, which are the parameters considered for estimating asphalt low temperature cracking resistance, were obtained at different test temperatures. The results for the different asphalts under study are presented in Figure 6-6, where it can be seen that the low-temperature properties of the different asphalts are dependent on the distillation temperature as well as the asphalt source.

Figure 6-6(a) shows a comparison of the creep stiffness and m-value of the asphalts distilled at 460°C with those of PG 58-31 and PG 64-22. It can be observed that, at the low temperature of −18°C, which corresponds to actual field temperatures of −28°C, all the asphalts, with the exception of PG 64-22, were found to fall below the required creep stiffness of 300 MPa and m-value of 0.300, while at −24°C all the asphalts distilled at 400°C were found to fall short of both the stiffness and m-value requirements.

Figure 6-6(b) shows that the oilsands asphalts distilled at 430°C were found to exert a significant effect on the reduction of low-temperature stiffness of the samples; in other words, the samples were found to satisfy the stiffness criteria at −12°C but not at −18°C (i.e., a one-step decrease compared to asphalts distilled at 400°C). Similarly, as shown in Figure 6-6(c), the asphalts distilled at 460°C were also found to exert a more significant effect on the reduction of low-temperature stiffness of the asphalt samples: with the exception of Asphalt A which both passed stiffness and m-value criteria's at −6°C as well as −12°C and failed at −18°C, they were all found to satisfy the stiffness criteria at −6°C but not at −12°C (i.e., a two-step decrease as compared to asphalts distilled at 400°C). Evidently, the lower the creep stiffness value is, the greater the low-temperature flexibility will be, and the lower the capacity of the asphalt binder to disperse stresses will be. Moreover, a decrease in distillation temperature was found to correspond to a significant decrease in creep stiffness, where the asphalts distilled at 400°C showed the lowest creep stiffness (and thus the greatest resistance to low-temperature cracking).





(c)

Figure 6-6. Stiffness and m-values of PG 58-31, PG 64-22 and Oilsands Asphalts (a) Distilled at 400°C, (b) Distilled at 430°C, (c) Distilled at 460°C

6.4.6 Performance Grading Analysis

The continuous high and low PG temperatures of different asphalt binders are presented Table 6-3. For the oilsands bitumens, the PG of particular asphalt was found to vary with changes in distillation temperature. It is notable that the high-temperature PG was found to increase significantly with an increase in the distillation temperature of the asphalt binder, while, in contrast, for low temperatures, a reduction in low PG temperature corresponding to an increase in distillation temperature was observed. This indicates that higher distillation temperatures result in reductions in the low-temperature performance properties of asphalt binders. For all of the oilsands bitumens under study, a 30°C increase in distillation temperature was found to result in a one-interval (−6°C) decrease in standard low-temperature grade. For high temperatures, meanwhile, a 30°C increase in distillation temperature was found to result in a two-interval (12°C) increase in standard high-temperature grade.

For the crude oil bitumens, it is seen that the original standard PG of the binders changed as a result of the analysis, where the high-temperature PG of both PG 58-31 and PG 64-22 were found to increase by 6°C, while the low-temperature performance of PG 58-31 decreased slightly to −28°C, and for PG 64-22 no change was observed. Notably, the asphalts distilled at 460°C and crude oil asphalts PG 58-31 and PG 64-22 were found to have higher high PG service temperatures than the other asphalts under study, indicating a better resistance to permanent deformation in high temperature conditions. The overall results indicate that oilsands bitumens have the best high- and low-temperature PG service temperatures among the asphalts investigated. Also, compared to crude oil binders, the oilsands bitumen binders have the best high- and low-temperature PG service temperatures.

Table 6-3. PG Properties of the Asphalt Binders

Dist. Temp °C	**Asphalt**	**T_{conts} High °C**	**T_{conts} Intm °C**	**T_{conts} Low °C**	**Stdrd High Grade °C**	**Stdrd Low Grade °C**	**PG Grade**
	PG 58-31	64.2	16.3	-31.5	64	-28	64-28
	PG 64-22	70.5	19.2	-27.1	70	-22	70-22
400	Asphalt A	47	5.9	-37	46	-34	46-34
	Asphalt B	53	14.5	-32	52	-28	52-28
	Asphalt C	46	15.2	-37	46	-34	46-34
430	Asphalt A	58.7	6.7	-33.8	58	-28	58-28
	Asphalt B	67.2	19.8	-26.2	64	-22	64-22
	Asphalt C	67.2	23.1	-22.6	64	-22	64-22
460	Asphalt A	77.2	26	-23.9	76	-22	76-22
	Asphalt B	82.6	33.5	-18.3	82	-16	82-16
	Asphalt C	75.1	30.7	-18.9	70	-16	70-16

T_{conts}: Continues temperature, Stdrd: Standard, Intm: Intermidiate

6.4.7 SARA Analysis

SARA fraction analysis provides insight into the role that each fraction plays in determining the physical and chemical properties of asphalts, as the interactions between these fractions govern the rheological properties of asphalt binders. Figure 6-7, Figure 6-8, and Figure 6-9 show the results of the SARA analysis comparing oilsands bitumens A, B, and C with PG 58-31 and PG 64-22.

In terms of composition, it can be seen that saturates account for approximately 25% and 28% crude oil bitumens PG 64-22 and PG 58-31, respectively, while, for oilsands bitumens distilled at different temperatures, the saturate content was found to be generally lower, ranging from 11% to 25%. Similarly, asphaltenes were found to account for approximately 19% of the content of oilsands bitumens, while for PG 58-31 the proportion was 16%; PG 64-22, meanwhile, was found to have the highest asphaltene content at 22%. With respect to resin, PG 64-22 was found to have the lowest content at 26%, while the oilsands bitumens showed the highest content, ranging from

27% to 38%. The aromatic contents were found to represent approximately 28% of content for the oilsands bitumens irrespective of distillation temperature, while the aromatic contents of PG 64-22 and PG 58-31 were found to be 20% and 28%, respectively. The lower saturate content observed in the oilsands asphalts, it should be noted, may affect their brittleness (slightly reducing the brittleness) and more flexible relative to the crude oil asphalts (PG 64-22 and PG 58-31).

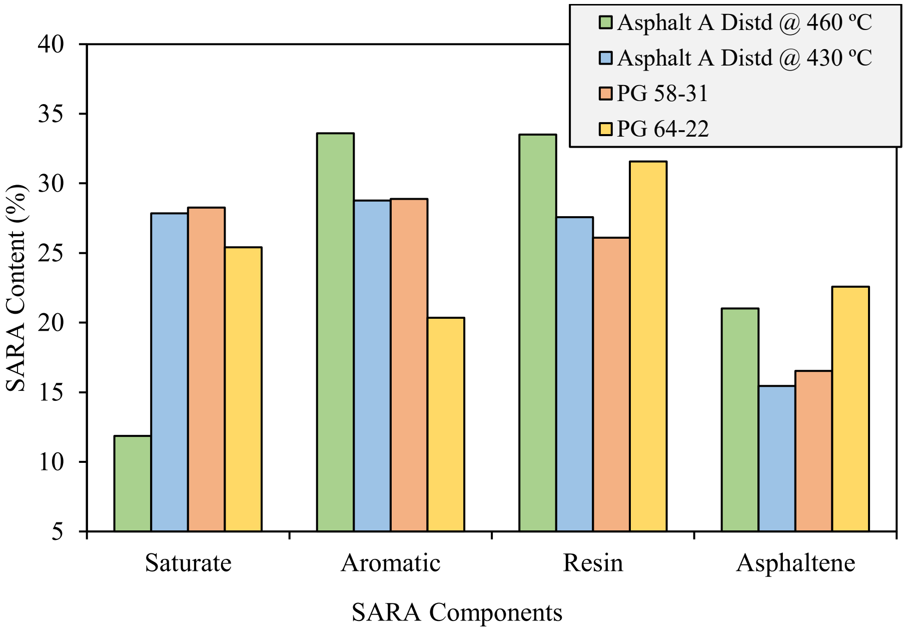


Figure 6-7. Comparison of SARA Components of Asphalt A with PG 58-31 and PG 64-22

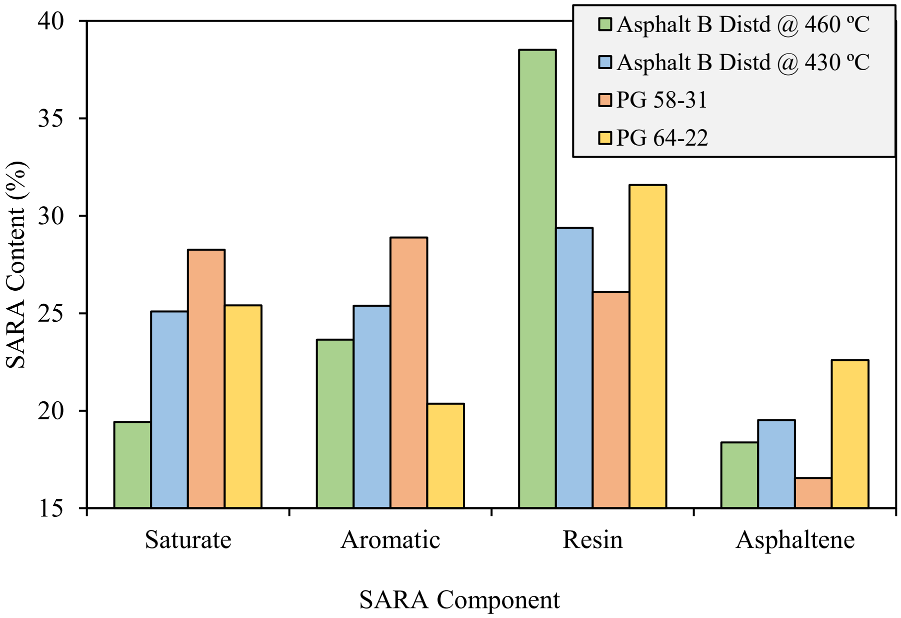


Figure 6-8. Comparison of SARA Components of Asphalt B with PG 58-31 and PG 64-22

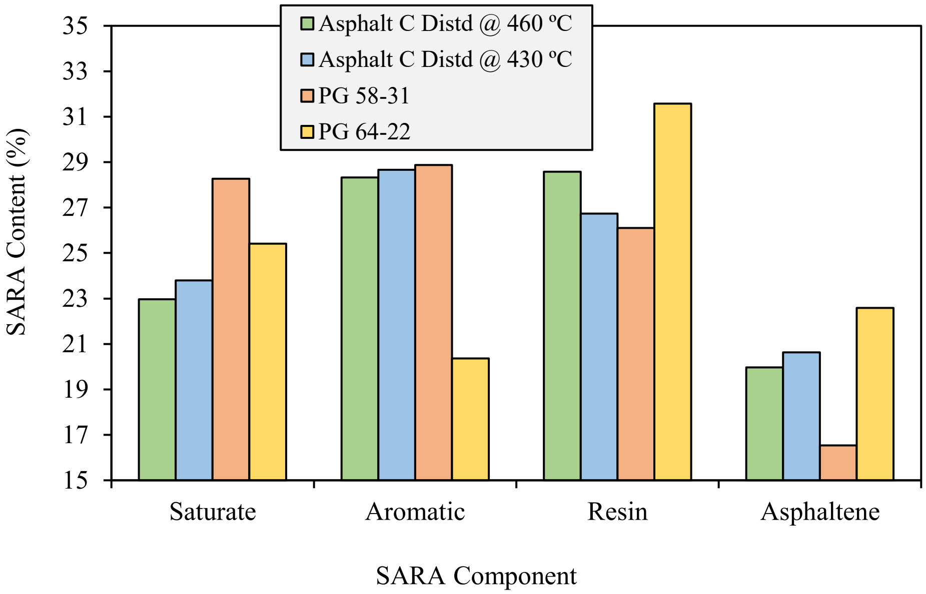


Figure 6-9. Comparison of SARA components of asphalt C with PG 58-31 and PG 64-22

6.4.8 Colloidal Instability Index (CI)

The colloidal stability of the asphalts, which is the ratio of dispersed medium (Asphaltenes+Saturate) to dispersing medium (Aromatic+Resin), were calculated as presented in Table 6-4. If asphalt has a CI value below 0.7, it should be noted, it is regarded as stable, whereas if the CI value of the asphalt is greater than 0.9, the asphalt sample is considered to be unstable. When the CI value falls between 0.7 and 0.9, the asphaltene stability is considered uncertain (176). For oilsands bitumens, it can be seen that the CI value decreases with an increase in distillation temperature. This indicates that oilsands bitumens distilled at 460°C are more stable than those distilled at 430°C, since an increase in CI value marks a decrease in asphalt stability. Comparing the CI values of PG 58-31, PG 64-22, and the oilsands bitumens, it is apparent that the oilsands asphalts distilled at 460°C show the lowest CI values. The lower value of CI indicates that asphaltene micelles were well dispersed in the dispersing medium (aromatics and resins), making the asphalt more stable than others. On the other hand, the CI value of PG 64-22 was found to be greater than 0.9, indicating that it is the most unstable of the asphalts investigated.

Table 6-4. Colloidal Instability Index Results

Distillation Temp (°C)	Asphalt	Asph+Sat	Arom+Res	Colloidal Index (C.I)	Stability
N/A	PG 58-31	44.81	54.98	0.82	Uncertain
	PG 64-22	48	51.94	0.92	Unstable
430	Asphalt A	43.31	56.35	0.77	Uncertain
	Asphalt B	44.62	54.76	0.81	Uncertain
	Asphalt C	44.42	55.39	0.80	Uncertain
460	Asphalt A	32.88	67.1	0.49	Stable
	Asphalt B	37.79	62.17	0.61	Stable
	Asphalt C	42.93	56.91	0.75	Uncertain

Asph + Sat: Asphaltene + Saturate, Arom + Res: Aromatic + Resins

6.5 Conclusions

This study investigated the impact of distillation temperature on the rheological properties and PG of oilsands bitumens binders. Based on the results obtained and the analysis conducted, the following conclusions were drawn:

- In general, oilsands bitumens binders distilled at 460°C were found to be more resistant with regard to rutting resistance compared to the oilsands bitumens distilled at 430°C and the crude asphalts PG 58-31 and PG 64-22.
- Oilsands bitumens distilled at low temperatures and crude oil asphalt PG 58-31 were found to have the lowest viscosities, meaning significantly lower mixing and compaction temperatures required during asphalt production and thus reduced energy consumption. Oilsands Asphalt B was found to have a greater viscosity value than the other oilsands bitumens (A and C) at all distillation temperatures.
- The oilsands bitumens distilled at 460°C from Source A and Source B were found to be capable of achieving high-temperature grades of PG 76-22 and PG 82-16, respectively, indicating ideal rheological characteristics at high performance temperatures.
- The BBR low-temperature analysis indicated that an increase in distillation temperature corresponds to an increase in high temperature grade and a decrease in low temperature grade for the oilsands bitumens, which reduces the suitability of the oilsand bitumens distilled at high temperatures to cold weather applications.

- The SARA fractions of the asphalts were found to considerably change based on the source of the asphalt, thus affecting the rheological behaviours of the asphalt binders, while the CI value also decreased with an increase in distillation temperature, with oilsands asphalts distilled at 460°C being the most stable among all the asphalts.
- It was found that crude oil asphalts require modification to achieve high PG for high modulus asphalt application, while oilsands bitumens was found to be capable of achieving high PG for high modulus asphalt applications in moderately cold climate regions without further modification.

Chapter 7. Summary and Conclusions

7.1 Summary

High modulus asphalt concrete (HMAC) is a premium type of asphalt mixture with main feature of high modulus or stiffness at intermediate temperatures. Key elements of HMAC mixtures, including a hard grade binder along with a strong continuous mineral skeleton provide this type of asphalt mixture high mechanical performance, including high elastic stiffness, high deformation resistance and good fatigue resistance, with workability, durability, and impermeability. The advantages of HMAC mixes are not limited to mechanical performance properties, as application of HMAC in base courses, causes the thickness of the asphalt layers to reduce considerably, which through saving materials brings economic and environmental benefits. As a performance-based mixture design, HMAC mixtures are designed to pass certain criteria when tested for performance testing, and choosing the right binder plays an important role in meeting these specifications. Modified asphalt binders are one common category of binders commonly used in HMAC applications. Sustainability concerns on one hand and high cost of polymer modifiers on the other hand, recently have increased the application of waste to modify and improve asphalt mixtures. Asphaltenes is one of the waste materials obtained through the deasphaltation process in refineries. Despite its high rate of production, asphaltenes has generally been considered a waste with minimal value and insignificant applications in industry.

The focus of this study was to investigate the potential application of asphaltenes to modify conventional asphalt binders for HMAC applications. In addition, other specific objectives of this research study are to evaluate binders derived from Alberta oilsands for HMAC applications, to compare the performance of asphalt binders from different sources, and to investigate effects of the distillation temperature on performance of oilsands asphalt binders from different sources. For this purpose, various percentages of asphaltenes were first added to different asphalt binders, from crude oil and oilsands refineries. Then the unmodified and modified binders were tested for their rheological properties and compared to investigate the effects of different factors on their performance. Also, a predictive model was used to estimate the dynamic modulus values of chosen asphalt binders, which was then verified by performing dynamic modulus test on actual asphalt mixtures.

7.2 Conclusions

This research study investigates the potential application of asphaltenes to modify asphalt binders processed from crude oil and oilsands binders. In this regards, the effects of different factors, including modifier content, asphalt binder type and source, and distillation temperatures in case of oilsands binders were investigated by conducting different tests on unmodified and modified asphalt binders and mixtures. The main conclusions drawn from the study are summarized as follows:

- Regarding the low temperature performance, with similar high PG grades, asphaltenes-modified binders fail at lower temperatures than the common hard grade binders in HMAC applications. This indicates that in regions with cold climates asphaltenes-modified binders could perform better in preventing low temperature cracking.
- According to rheological test results, addition of asphaltenes increases the stiffness and elasticity of the asphalt binder, which in turn results in a considerable improvement in resistance against permanent deformation at high temperatures. On average, and regardless of the source of binder, a 6% increase in asphaltenes content corresponds to a one-interval increase in high PG temperature grade of the asphalt binder. At low temperatures, however, every 10-20% increase in asphaltenes content corresponded to a low PG grade increase of one interval; however, this was found to be dependent on the binder type.
- The improving effects of asphaltenes on high temperature performance of asphalt binder are more pronounced as the binder ages through short-term aging.
- According to the 1999 Witczak prediction model, the desirable PG grades of asphalt binders to satisfy the requirement of dynamic modulus at a loading frequency of 10 Hz and a temperature of 15°C being higher than 14 GPa was found to be PG 82-16 and PG 88-16. Also, the minimum asphaltenes content to reach a high PG grade of 82ºC was observed to be highly dependent on the PG grading of the base binder.
- Dynamic modulus test results on one asphaltenes-modified binder with a continuous PG grading of 82-16 showed that this binder along with a well-graded aggregate gradation could meet the requirement for dynamic modulus of more than 14 GPa at a loading frequency of 10 Hz and a temperature of 15°C. This result verified the predications from the 1999 Witczak model.

- Regardless of the source of the asphalt binder, every 2% increase in asphaltenes content was found to increase the binder viscosity at 135ºC by approximately 0.1 Pa·s, and increase the mixing and compaction temperatures by approximately 2.6ºC.
- By comparing different trends in the rheological properties of the various asphalt binders with SARA test results, it was observed that an increase in polar fraction content corresponds with an increase in the stiffness, elasticity, and viscosity of the asphalt binder.
- Modifying the Alberta oilsands asphalt binders with asphaltenes and comparing the results of different tests with those of asphaltenes-modified crude oil asphalt binder it was determined that binders from the Alberta oilsands modified with asphaltenes could achieve the performance grade requirements for HMAC applications.
- Binders sourced from Alberta oilsands bitumen have lower colloidal index values, which signifies their higher stability (stability of asphaltenes phase in maltenes matrix) for the asphalt binders compared to crude oil asphalt binder PG 70-22.
- By comparing different trends in the rheological properties of the various asphalt binders with SARA test results, it was observed that an increase in polar fraction content correlates well with an increase in the stiffness, elasticity, and viscosity of the asphalt binders.
- Investigating different oilsands asphalt binders distilled at different temperatures showed that an increase in distillation temperature corresponds to an increase in high temperature grade and a decrease in low temperature grade for the oilsands bitumens reduces the suitability of the oilsand bitumens distilled at high temperatures to cold weather applications.
- It was found that depending on distillation temperature and source of asphalt binder, oilsands binders are capable of achieving premium temperature grades of PG 76-22 and PG 82-16, indicating ideal rheological characteristics at high performance temperatures, and for high modulus asphalt concrete applications.
- The SARA fractions of the asphalts were found to be considerably change based on the source of the asphalt, thus affecting the rheological behaviours of the asphalt binders, while the colloidal index value also decreased with an increase in distillation temperature, with oilsands asphalts distilled at 460°C being the most stable among all the asphalts.
- In spite of crude oil asphalts that require modification to achieve performance grade requirements for high modulus asphalt applications, using the right distillation temperature

and source, oilsands bitumens were found to be capable of achieving high PG for high modulus asphalt applications in moderately cold climate regions without further modification.

7.3 Future Research

A comprehensive investigation of asphaltenes-modified binders with asphalt binders from different sources is covered in this thesis, as well as confirming their eligibility to be used for high modulus asphalt concrete applications in regard of dynamic modulus criterion. However, more research is required to study the performance of HMAC mixes composed of asphaltenes-modifed binders with the focus on low temperature performance tests, and compare the results with the performance of hard grade binders common worldwide in HMAC applications.

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