EVALUATION OF PHASE ANGLE AS A PRACTICAL PARAMETER FOR LOW TEMPERATURE SPECIFICATION GRADING OF ASPHALT BINDERS

by

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Abstract

The importance of road and highway systems in the development of a nation's economy cannot be overemphasized. Over the years, concerted efforts have been made by researchers to develop and design test methods with high accuracy, repeatability, and reproducibility. The goal is to reduce pavement failures and the high cost of road construction and rehabilitation. Current low temperature performance tests such as the bending beam rheometer (BBR) and extended BBR (EBBR) tests are not without limitations. They could be time-consuming and require more sample quantity even though they are rigorous and accurate. Hence, this study aims to examine the viability of limiting phase angle measurement as a credible alternative to the laborious extended BBR (EBBR) procedure.

In this study, a total of 239 samples were tested from various agencies and are grouped into tank samples, core samples and loose mix samples. Agency A and Agency B were carefully examined to ensure climatic and traffic requirements are met. High temperature performance grade (HTPG), intermediate temperature performance grade (ITPG), regular bending beam rheometer (BBR), extended bending beam rheometer (EBBR) tests were carried out on the samples. In addition to this, the data obtained from each test were correlated with limiting phase angle temperature [T(30°), T(45°)].

From the study, Agency B largely met the climatic and traffic requirements when compared with Agency A, due to a much more effective approach adopted by the agency. Furthermore, correlation studies involving ITPG, BBR, and EBBR with limiting phase angle temperature showed that core samples have the highest correlation followed by loose mix samples and tank samples. From the large data set used in this study, it is more evident that the limiting phase angle measurement is a

viable alternative to evaluate the thermal cracking performance of binders because of its higher sensitivity, easier experimental procedure, and smaller sample quantity requirement.

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Abbreviations and Acronyms

AASHTO	American Association of State and Highway Transportation Officials
AFM	Atomic Force Microscopy
ASTM	American Society for Testing and Materials
BBR	Bending Beam Rheometer
CTOD	Crack Tip Opening Displacement
DCM	Dichloromethane
DENT	Double-Edge-Notched Tension Test
DSC	Differential Scanning Calorimetry
DSR	Dynamic Shear Rheometer
DTA	Differential Thermal Analysis
EBBR	Extended Bending Beam Rheometer
EWF	Essential Work of Fracture
FTIR	Fourier-Transform Infrared Spectroscopy
GPC	Gel Permeation Chromatography
HMA	Hot Mix Asphalt
HPLC	High Pressure Liquid Chromatography
HTPG	High Temperature Performance Grade
ITPG	Intermediate Temperature Performance Grade
KPa	Kilo Pascal
LLTPG	Limiting Low Temperature Performance Grade
LS	Laboratory Standard Test Method
LTPG	Low Temperature Performance Grade

MPa	Mega Pascal
MTO	Ontario Ministry of Transportation
NMR	Nuclear Magnetic Resonance spectroscopy
PG	Performance Grade
PAV	Pressure Aging Vessel
RAP	Reclaimed Asphalt Pavement
REOB	Recycled Engine Oil Bottom
RTFO	Rolling Thin Film Oven
SARA	Saturates, Aromatics, Resins, and Asphaltene
SBS	Styrene Butadiene Styrene
SEM	Scanning Electron Microscopy
SHRP	Strategic Highway Research Program
S(t)	Time-dependent Flexural Creep Stiffness, MPa
SUPERPAVE	Superior Performing Asphalt Pavement
TGA	Thermal Gravimetric Analysis
TLC−FID	Thin-Layer Chromatography−Flame Ionization Detector
WMA	Warm Mix Asphalt
XRF	X-Ray Fluorescence

Chapter 1

Introduction

1.1 Overview

Considering the increase in goods and services, population, and infrastructural development, land transportation remains a viable option for the synchronization and coordination of our natural and human resources. The importance of road and highway systems in the development of a nation's economy cannot be overemphasized. According to records, 65 percent of the U.S.A's $15 trillion freight traffic depends on the 4-million-mile highway system in the country [1]. In other words, there is a link between the timely execution of transportation projects and the development experienced by any country [2][3].

As the major material used in pavement construction, asphalt is a residue obtained from petroleum refining. Invariably, asphalt contributes immensely to the transportation infrastructure and economic growth in developed as well as developing countries [4]. Before the 21th century, asphalt has been used for various purposes like roofing, road building and coatings. But its use in pavement construction started in the early 20th century and has accounted for up to 85 percent of asphalt application.

According to Arthur Usmani in his book titled *Asphalt Science and Technology,* "about 70 billion lb of asphalt is used annually in the U.S. alone, and asphalt usage will grow dramatically in Asia during the next 10 years" [5]. This explains the growing demands for asphalt in pavement construction in the world. Associated with the tremendous growth of the pavement industry is the increased cost of road maintenance especially in colder regions of the world. For example, in Canada and the northern part of the United States, the service life of roads is greatly affected by extreme temperatures. These extreme climate conditions also affect transportation costs. Due to

bad weather and other incidents, the United States Government annually spends over $9 billion on urban areas alone [6].

However, with the current rate of Government spending on roads and highways, pavement failures such as rutting, fatigue and, thermal cracking are still the order of the day. This explains why various governments, researchers, engineers are on the lookout for asphalts that meet the service life expectations and could withstand oxidation and harsh environmental conditions.

1.2 Mode of Pavement Failures

Those working on pavement research, design, and management found out that some roads have longer service spans while others experience a shorter spans. This can be attributed to variations in sub-grade stiffness, pavement design, climate, traffic level, and/or more elusive factors [7]. The most common types of pavement failures which will be discussed later are rutting, fatigue, moisture damage and cracking. Gavin *et al.* [7][8], explained that premature cracking is expensive, usually around $1-2 per meter for sealing; and more importantly, it could also be a sign of significantly reduced pavement life. Hence, efforts are constantly made to mitigate pavement failures.

1.2.1 Fatigue Cracking

According to Pell, fatigue is defined as: “the phenomenon of fracture under repeated or fluctuating stress having a maximum value generally less than the tensile strength of the material” [9][10]. However, a better definition proposed by Read *et al.* [9][11], was adopted. Thus, defining fatigue as “the phenomenon of cracking consisting of two main phases, crack initiation and crack propagation, and is caused by tensile strains generated in the pavement by not only traffic loading but also temperature variations and construction practices”. A study shows that fatigue cracking occurs when tensile strain approaches 30×10^{-6} to 200×10^{-6} for a standard wheel load [10]. The

dynamic shear rheometer (DSR) is widely used to evaluate fatigue failure in asphalt samples using parameters obtained at the end of the experiment. Fatigue cracking, also known as alligator cracking is shown in Figure 1.1.

Figure 1.1: Similarity between the skin of an alligator and fatigue cracking [31].

1.2.2 Thermal Cracking

Thermal cracking, as displayed in Figure 1.2, is one of the major forms of pavement failure experienced in colder climates like Canada and the northern United States. It is developed as a result of induced stress found within the asphalt layer under low temperature conditions. Various factors could be responsible for thermal cracking such as climatic condition, asphalt mix, properties of the binders among many others. It consists of three major processes as described by Yee *et al*. [12]. The first process known as single-event thermal cracking is caused by a rapid drop in temperature below a critical value at which point the stress exceeds the strength of the asphalt mixture. The second process weakens the mix as a result of repeating thermal stresses below the critical value. The third process which is also significant is a result of loading caused by regular vehicles and particularly heavy truck traffic [12].

Figure 1.2: Longitudinal and transverse cracking [32].

1.2.3 Rutting

Rutting is a leading pavement distress which affects the service life and safety of the highway. It is the permanent deformation seen on the pavement surface especially at high temperatures as shown in Figure 1.3. Due to high traffic volume, tire pressure, and axial load, many roads constructed fail to meet the structural and functional requirements within the first few years of construction [13][14]. Hence, researchers now focus on designing rut-resistant asphalt mix to reduce the cost of road maintenance and to generally improve pavement service life. The rutting susceptibility of asphalt can be evaluated from the complex modulus and phase angle obtained from a DSR experiment.

Figure 1.3: Rutting or permanent deformation [34].

1.2.4 Moisture Damage

Moisture damage is gradually gaining attention among researchers due to its ability to reduce the service life of pavement structures. It occurs when water molecules pass through the porous asphalt pavement. By implication, it means adhesion will be lost or reduced between asphalt binders and aggregates. Factors contributing to moisture damage include the presence of air voids and poor drainage systems among many others. Moisture damage could occur as a result of cracking or rutting as shown in Figure 1.4 below.

Figure 1.4: Moisture damage [33].

1.3 Scope and Objectives

Over the years, concerted efforts have been made by researchers to develop and design test methods with high accuracy, repeatability, and reproducibility. The Government of Canada spends many billions of dollars yearly on road reconstruction and rehabilitation, hence the need to reduce these cost implications. In order to reduce pavement failure and improve cost-effectiveness, a repeatable and reproducible test method that accurately predicts the performance of asphalt plays a pivotal role. Current low temperature performance tests such as the bending beam rheometer (BBR) and extended BBR (EBBR) tests are not without limitations. They could be time-consuming and require more sample quantity even though they are rigorous.

As a result of this, the primary objective of this study is to evaluate the viability of limiting phase angle measurements as a practical alternative to the somewhat laborious extended BBR (EBBR) procedure. This study uses a very large data set to examine what may be the most practical approach for determining the low temperature performance of binders.

Chapter 2

Literature Review

2.1 Literature Review

Over the years, different names have been used to describe asphalt. Based on geographical location, it is called bitumen in Europe and asphalt cement or binder in North America. The American Society for Testing and Materials (ASTM 1998) defined asphalt as a "dark brown or black cementitious material occurring in nature or obtained by crude oil refining" [15], while the Shell Bitumen Handbook by Read *et al.* [16], defines asphalt as a "road surfacing material consisting of bitumen, mineral aggregates and, fillers and may contain other additives, and includes what is described in some areas as hot mix asphalt (HMA)". In other words, asphalt contains a complex mix of fused and saturated polyaromatic structures with a small amount of nitrogen, oxygen, sulfur and trace metal. Although in principle a hypothetical structure was deduced as shown in Figure 2.1, the exact structure remains unknown [17].

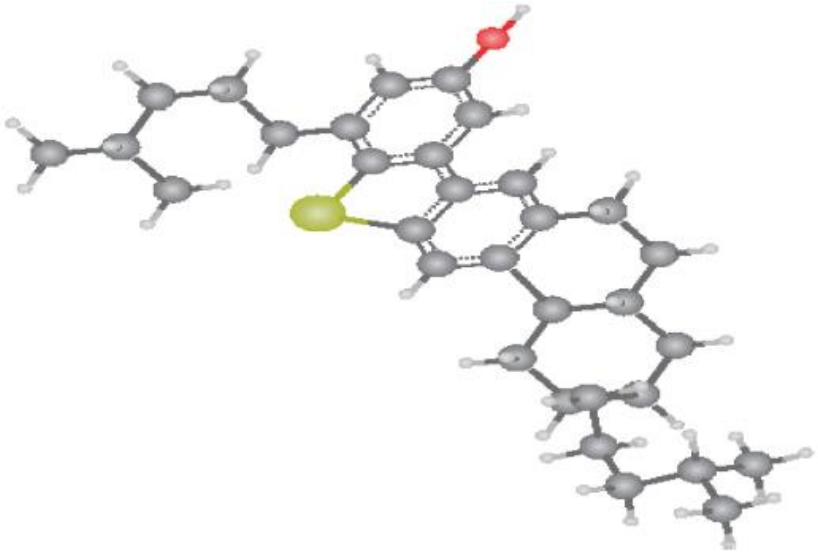

Figure 2.1: Asphalt molecule [18].

2.2 Chemistry of Asphalts

As stated earlier, the chemical structure of asphalt is highly complex which invariably makes the understanding of its chemistry very complicated. However, a practical way to evaluate the chemistry of asphalt as proposed by Redelius and Soenen [18], is to study the composition of the lighter fractions from crude oil distillates. The distillates having the lowest boiling points are well known, while the composition of the heavier distillates can be generalized. Moreover, the chemical properties of asphalt could be associated with its heterogeneity. Over the years, different techniques with varying separation principles have also evolved with the Saturates, Aromatics, Resins, and Asphaltene (SARA) technique as the most popular [18].

According to Israelachvili and Redelius and Soenen [18][19], a key factor in understanding the chemistry of asphalt is to take a critical look into its molecular interactions. The dominant molecular interaction found in asphalt is the dispersive interactions, while polar and hydrogen bond interactions—subject to the presence of electronegative element(s)—contribute to the overall interactions. Dispersive interactions could be a weak interaction between molecules but it is important in non-polar hydrocarbons and for a better understanding of the physical properties of asphalt. Two factors are vital for deriving an equation for dispersive forces. These are electronic polarizability and refractivity as shown in the equation below [18][20].

$$\alpha = \left(\frac{3}{4\pi N_A}\right)\left(\frac{M}{d}\right)\left(\frac{n^2-1}{n^2+2}\right)$$

Where d, n, N_A, and M represent density, refractive index, Avogadro's number, and molecular weight respectively. Although this equation estimates the polarizability of pure compounds, it could also be used in crude oils or asphalt. Riazi and Roomi proposed that "the same data can be used for estimating transport properties including viscosity, diffusivity, and thermal conductivity" [18][20][21].

2.3 Chemical Composition of Asphalt

One of the factors that affect the composition of asphalt is the crude oil source. In other words, asphalt from two different sources most likely have constituents with slightly different percentage composition. An analysis shown by Romberg *et al.* [22], stated that asphalts are made up of hydrocarbon molecules, with some heterocyclic compounds and functional groups such as oxygen, sulfur, and nitrogen. Asphalt was also found to contain metals in the form of metallic salt or metal porphyrins complexes [10]. Therefore, a percentage composition as reported by Read *et al.* [9], is as follows:

carbon 82–88%

hydrogen 8–11%

oxygen 0–1.5%

sulfur 0–6%

nitrogen 0–1%.

Elemental analysis of asphalt by Crump [23] also explained how the crude oil source could affect percentage composition with the result presented in Table 2.1 below.

Table 2.1: Elemental analysis of asphalt from various sources [23].

	Carbon: % w	Hydrogen: % w	Nitrogen: % w	Sulfur: % w	Oxygen: % w	Nickel: ppm	Vanadium: ppm
Range	80.2–84.3	9.8–10.8	0.2–1.2	0.9–6.6	0.4–1.0	10–139	7–1590
Average	82.8	10.2	0.7	3.8	0.7	83	254
	Iron: ppm	Manganese: ppm	Calcium: ppm	Magnesium: ppm	Sodium: ppm	Atomic ratio: H/C	
Range	5–147	0.1–3.7	1–335	1–134	6–159	1.42–1.50	
Average	67	1.1	118	26	63	1.47	

Based on solubility, asphalt composition can be classified into Saturates, Aromatics, Resins and Asphaltenes (popularly called SARA). Over the years, the most commonly used technique for SARA analysis has been (i) combination of ASTM D1319, ASTM D6560, and ASTM D2007, (ii) thin-layer chromatography−flame ionization detector (TLC−FID) method, and (iii) high-pressure liquid chromatography (HPLC) [27]. For a better understanding of the SARA acronym, a brief description is provided below [10][24].

Saturates are non-polar viscous oils containing aliphatic hydrocarbons and cycloaliphatic compounds as shown in Figure 2.1. They have an average molecular weight range and a hydrogen-to-carbon ratio of 470-880 g/mol and 2, respectively. They constitute about 5-20 % of the total asphalt composition.

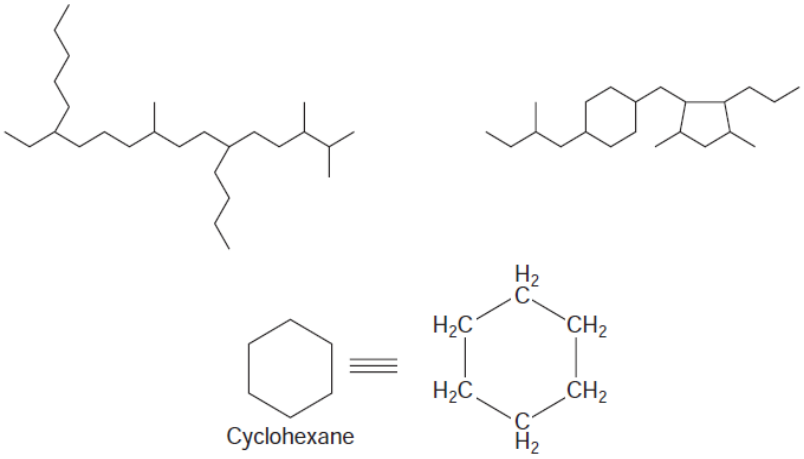

Figure 2.2: Schematic diagram of saturates [16].

Aromatics are low molecular weight naphthenic compounds constituting about 40-65 % of the total asphalt. They have an average molecular weight range and a hydrogen-to-carbon ratio of 570-980 g/mol and 1.4 to 1.6, respectively. A sample representation of aromatics is presented in Figure 2.3 below.

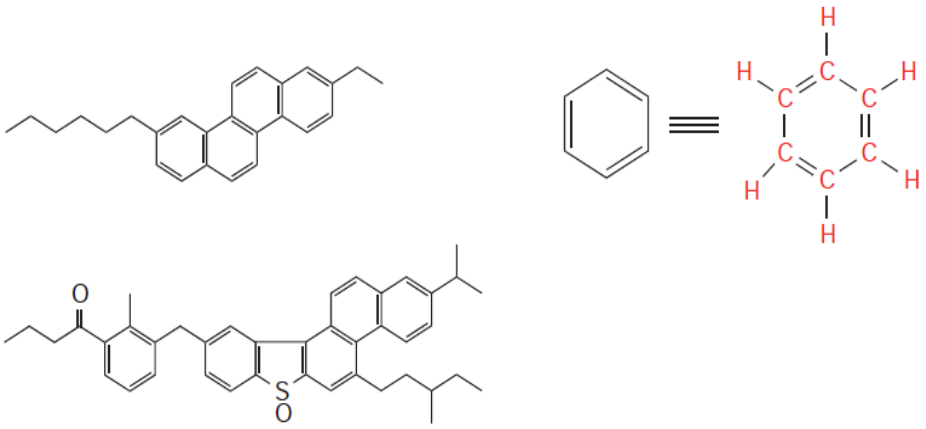

Figure 2.3: Schematic diagram of aromatics [16].

Resins are n-heptane soluble, dark brown, solid, or semi-solid constituent of asphalt. They contain carbon, hydrogen, and a small amount of sulfur, oxygen, and nitrogen. They have an average molecular weight range and a hydrogen-to-carbon ratio of 780-1,400 g/mol and 1.4-1.7, respectively.

Asphaltenes are the n-heptane insoluble portion of the asphalt. They are highly polar, having a hydrogen-to-carbon ratio of 0.98 to 1.6. Using osmometry measurements, asphaltenes are found to have an average molecular range of 800 to 3,500 g/mol. Studies have shown that the asphaltene content in asphalt could have a significant effect on its rheological properties [25]. Research by Schabron *et al.* [27], further explained how asphaltene can be separated using solvents with different or increasing solubility parameters. As shown in Figure 2.4, asphaltene also contains carbon, hydrogen, and a small amount of sulfur and nitrogen.

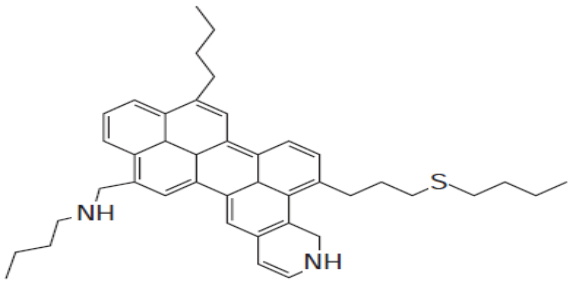

Figure 2.4: Schematic diagram of asphaltene [16].

2.4 Physical Properties of Asphalt

Durability: This is a measure of how the properties of asphalt change with aging. It describes its ability to resist weathering, traffic loading, or aging. Some pavement provides longer serviceability than the others, indicating differences in durability. According to Read *et al*. [9], durability is described as "the retention of desirable engineering and serviceability characteristics over a lifetime of an asphalt". Generally, it has been observed that as asphalt ages, it loses its viscosity and thus become stiffer [27].

Aging: Various studies have been carried out to evaluate the effect of aging on the performance of asphalt. However, aging could be short-term or long-term. The short-term aging could be a result of the production, transportation, and compaction of asphalt mixtures in the field [27][28], while long-term aging refers to the service condition of asphalt pavements and is affected by traffic loading, temperature, and several other environmental factors [27][29].

Viscoelasticity: Asphalt is a viscoelastic material that behaves like a viscous liquid at high temperatures and an elastic solid at low temperatures. The viscoelastic properties of asphalt play a major role in its performance. Thus, the viscosity and elasticity of the asphalt must be such that it suits its intended environment and applications.

Temperature Susceptibility: Asphalt is a material that is highly susceptible to temperature. In other words, asphalt is a thermoplastic material that becomes more viscous as temperature decreases [30].

2.5 Characterization of Asphalt

Analytical techniques that help in the characterization of asphalt include: include thermal gravimetric analysis (TGA), differential thermal analysis (DTA), gel permeation chromatography (GPC), scanning electron microscopy (SEM), and atomic force microscopy (AFM). These

techniques facilitate the probing of the asphalt molecules and provide insights into the properties as well.

Fourier-transform infrared spectroscopy (FTIR) is a useful analytical technique employed to study aging in the asphalt mix. It is used to characterize various functional groups present in the asphalt mix. This helps to predict the source and history of the asphalt as well as to gain insight into the additives added to modify the asphalt [17]. From Beer-Lamberts' law, the absorbance of the radiation and concentration can be deduced from the intensity of radiation. Thus, the FTIR technique provides a means to detect sulfoxide and carbonyl bonds [35].

$$A(v) = \mathrm{Log}\left(\frac{I_0}{I}\right) = \varepsilon_i(v)\ell C_\mathrm{i},$$

Where A(v) is the absorbance; I_0 is the intensity of the incident light; I is the intensity of the transmitted light; Ɛi(v) is the molar absorption coefficient; *l* is the optical path length; Ci is the studied species concentration. The two commonly used FTIR principles used for asphalt characterization are transmission and reflection mode [35].

TGA and DSC are techniques that relate the physical changes in a material to heat exchange. Examples of physical changes evaluated by TGA analysis are glass transition temperature, melting point, crystallization, heat capacity of materials [40]. Also, asphalt microstructure, entropy and enthalpy are better understood using TGA and DSC [17].

Nuclear Magnetic Resonance (NMR) spectroscopy is a versatile technique used in the characterization of asphalt. The most commonly used NMR nuclei are ^{1}H and ^{13}C NMR and they provide information on the structure of asphalt and other parameters. The environment in which the different types of hydrogens and carbons exist is also determined [37].

Gel Permeation Chromatography (GPC) uses its stationary and mobile phases to separate asphalt components based on molecular size and not molecular interactions. The use of GPC in conjunction with NMR help to elucidate possible structures for asphalt and the mechanism of aging is better understood as described by Siddiqui *et al*. [39].

Atomic Force Microscopy (AFM) typically uses a nano-sized tip attached to a cantilever to probe the surface of a material. The resulting surface-tip interactions and topography measured are recorded on the detector as shown in Figure 2.5 below. As the tip raster scans the surface, a three-dimensional image with a high resolution is formed. An observation of an asphalt surface under AFM imaging shows a bee-like structure. A study by Allen *et al*. [40], shows that asphalt microstructure seen under AFM could be affected by the asphalt's chemical properties as shown in Figure 2.6.

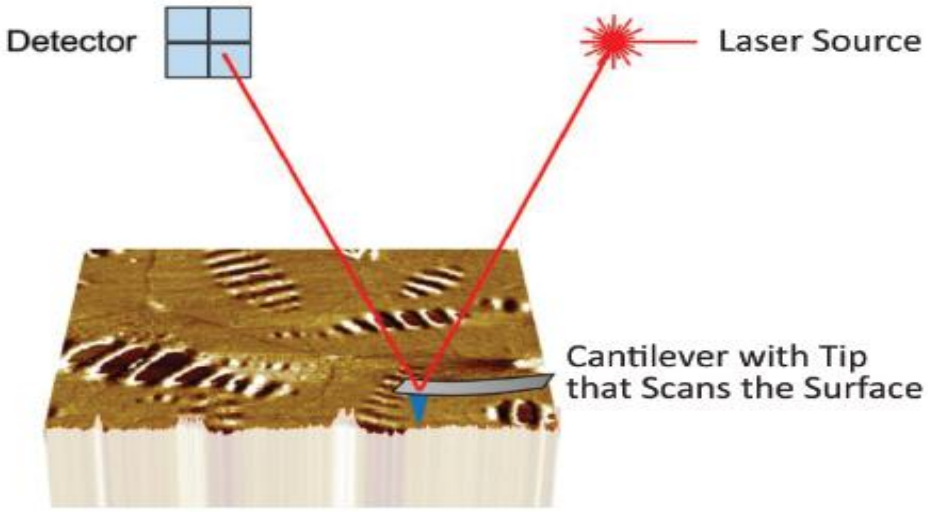

Figure 2.5: A schematic diagram of an AFM machine scanning an asphalt surface [17].

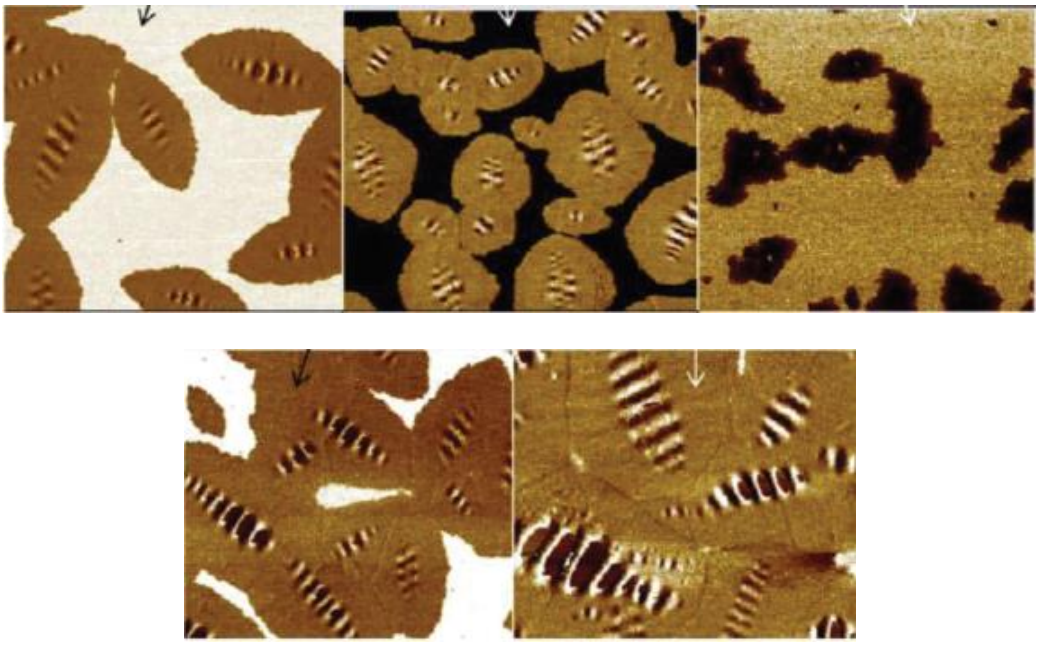

Figure 2.6: Phase images of asphalt ARC BI0002 derivatives under AFM (a) control; (b) asphaltene-doped blend; (c) naphthenic aromatic-doped blend; (d) polar (resin)-doped blend; and (e) saturate-doped blend (enlarged locations are 10 μm by 10 μm for each asphalt blend) [39].

2.6 Asphalt Modification

In response to changing global climate, environmental factors and cost, asphalt can be modified to make it suitable for its intended use. The modifiers are specifically used to alter some properties of the asphalt like increasing its resistance to deformation. The use of modified asphalt is widely accepted due to its potential for industrial applications. Over the years, researchers and chemists have experimented with the addition of asbestos, mineral fibers, plastomers, elastomers, and rubbers to asphalt for improved applications [16].

According to Roberts *et al*. [16][41], the following are some of the reasons for incorporating modifiers into an asphalt:

- Increase the stiffness of the mixture to minimize rutting
- Soften and increase the elasticity of the mixture to minimize cracking

- Improve the fatigue resistance of the mixture
- Improve asphalt-aggregate binding to reduce stripping or moisture sensitivity
- Improve abrasion resistance to reduce raveling
- Rejuvenate aged asphalt binders
- Reduce flushing or bleeding
- Improve resistance to aging or oxidation
- Reduce the structural thickness of pavement layers
- Reduce life-cycle costs of HMA pavements
- Improve overall performance of HMA pavements.

Examples of materials used as modifiers are styrene–butadiene rubber, polyisoprene, natural rubber, ethylene–vinyl acetate, polyethylene, polypropylene, polyvinylchloride, epoxy resin, phosphoric acid, crumb rubber, cellulose, etc.

2.7 Thermal Cracking: Historical Developments

For over 50 years, the phenomenon of thermal cracking has been studied by various researchers to understand the behavior of asphalt at low temperatures. Some of these early researchers are Van der Poel 1954 [60], 1955 [61], Heukelom 1966 [62], Deme & Young 1987 [65], and many others. The basis of those studies stems from the need to construct pavements that could withstand increasing traffic loads caused by economic development, industrialization, and urbanization. As a result of this, tests such as the viscosity test, the ring and ball softening point test, and the penetration test were developed to study the behavior of asphalts. Van der Poel, while working at the Shell Laboratory, developed a nomograph used for selecting hot mix asphalt. He later asserted that "as all bitumens have approximately the same breaking strength, the Frass temperature should be an equi-stiffness temperature, corresponding to the stiffness of roughly 108 N/m^2 at 11 sec"

[61][63]. Following this, Heukelom [62] studied the relationship between binder stiffness and its failure behavior at various conditions. He found that the tension measured at a specific time is directly proportional to the binder's low temperature performance. Heukelom confirmed that "stiffness can be regarded as the main parameter for fracture, and thus for the permissible deformation" [62]. The major fault with the early studies was that data obtained from high temperature behaviors of the binder were used to forecast the entire binder performance over a time-temperature domain.

Binder failure at low temperature consists of various factors that affect the lifespan of the pavement. Some studies by Deme & Young 1987 [65], Hesp 2009 [58] showed that stiffness and relaxation ability are not the only factors responsible for thermal cracking. Other factors such as moisture, asphalt mix design, traffic levels, pavement structure, paver-induced segregation, and subgrade properties could be detrimental to the pavement's lifespan [70][71][66][67]. Due to the complexities associated with the low temperature performance of binders and the shortcomings of early studies, the Superpave specifications such as (AASHTO) standard M 320 were laid out in the 1980s to predict thermal cracking [68].

2.8 Superpave Specification Tests

The strategic highway research program (SHRP) was instituted in 1987 in the United States to design specifications for asphalts based on performance parameters. The program aimed to improve the service life of pavements which are called 'Superpave' (superior performing pavements). In this specification, environmental conditions were put into consideration. Therefore asphalts used in this specification are expected to show improved resistance to thermal cracking, fatigue cracking and rutting [16]. The specification system takes into account the maximum pavement temperature YY and the minimum temperature XX in which the asphalt could retain

some or all its properties. Various publications have been written about the specification tests such as Villanueva *et al*. 2008 [44][45], Hesp *et al*. 2009 [67], Hesp and Shurvell 2010 [46][64], Rubab *et al*. 2011 and many more [66][67].

2.9 Bending Beam Rheometer Test (AASHTO M320)

The major parameters used by the AASHTO M320 specification to predict the low temperature performance of asphalt are the *creep stiffness* and *creep rate* [56]. A beam of asphalt is conditioned at the desired grading temperature. During this testing, stress is applied to a beam of asphalt through a loaded piston and the creep stiffness at 60 seconds is calculated using the equation below while the m-value is obtained from the slope of the creep curve:

$$s(t) = \frac{Pl^3}{4bh^3\delta(t)}$$

Where S(t) = creep stiffness at time t, p = applied constant load, *l* = distance between beam supports, b = beam width, h = beam height, and δ (t) = deflection at time t. The specification set its limit at 300 MPa and 0.3 for stiffness and m-value respectively. The test is designed for use in a climate where the surface temperature reaches 10 °C lower than the grading temperature once in 50 years [64].

2.10 MTO Specifications

When the Ontario Ministry of Transportation adopted AASHTO M320 specification, it was discovered that pavement failures persisted [69][70][71][72]. This gave rise to a research project to study premature cracking in 2001 [70]. The outcome of that project is the development of the double-edge notched tension(DENT) test and extended bending beam rheometer test (EBBR) [69][70][71][73][74][75]. The two specifications provided an improved prediction on binders performance on the field as described by Tabib *et al.* and Nicol *et al.* [76][77]. Studies by various

researchers such as Hesp *et al*. [72][77] and more showed that poor-performing binders lose their low temperature grade during cold storage due to lack of strain tolerance.

2.10.1 Double-Edge-Notched Tension (DENT) Test

The first ductility test was carried out by Dow in 1903 [78]. However, there is so much contention about the test due to its simplicity. Research by Kandhal opened up links to other research when he related ductility to distresses like raveling and cracking [79]. Also, efforts by Rowe *et al.* [80], to correlate ductility test to DSR test was limited especially for highly modified bitumen. As an improvement to other ductility tests, the DENT test was developed [73]. The test measures the critical tip opening displacement (CTOD) in their ductile state as described by Andriescu *et al.* [69][70]. It provides information on how much a binder can stretch before tensile damage. The DENT test was found to correlate with fatigue cracking performance and requires a large quantity of aged binders [81].

2.10.2 Extended Bending Beam Rheometer (EBBR) Test

In this MTO specification, binders are conditioned for 1, 24, and 72 hours at 10 ℃ and 20 ℃ above the low temperature grade. On completion of the test, a limiting low temperature grade and grade loss were determined [75]. Due to the extended conditioning, the EBBR gives an improved prediction of binder performance. The major setback of this specification is that it is time consuming and requires a large quantity of sample. However, there is a renewed effort to develop a test method that could predict low temperature performance at a reduced time using a smaller amount of sample.

2.11 Oxidative Aging

The term *aging* or *hardening* as described by Berkowitz *et al*. [82], is the “deterioration of viscoelastic properties in asphalt binders”. From the literature, aging has been classified into

oxidative aging and thermoreversible aging. The former is irreversible (that is, involves chemical reactions) and the latter is reversible and it involves interactions like crystallization [82].

In 2009, Petersen reviewed the fundamentals of asphalt oxidation. In his review, he found that binders go through a slow oxidation process leading to the formation of carbonyl, carboxylic acid, ketones within the binders [82][83]. Studies have shown that extent of oxidation could be a function of the binder origin. Crude oil containing high asphaltene content has a higher oxidation rate and ultimately leads to gel formation [82][83]. To take this further, a study by Bahia and Anderson [84] indicated the effect of film thickness and aging time on the rheological properties of aged binders. They also found that the effect of aging time and film thickness is a function of the crude source. Another study by Erskine *et al*. [85], demonstrated the improved properties of binders after 8 years of service when it was aged in thin films (12.5 g) or for a longer time. The data from the study was shown in Figure 2.7 below.

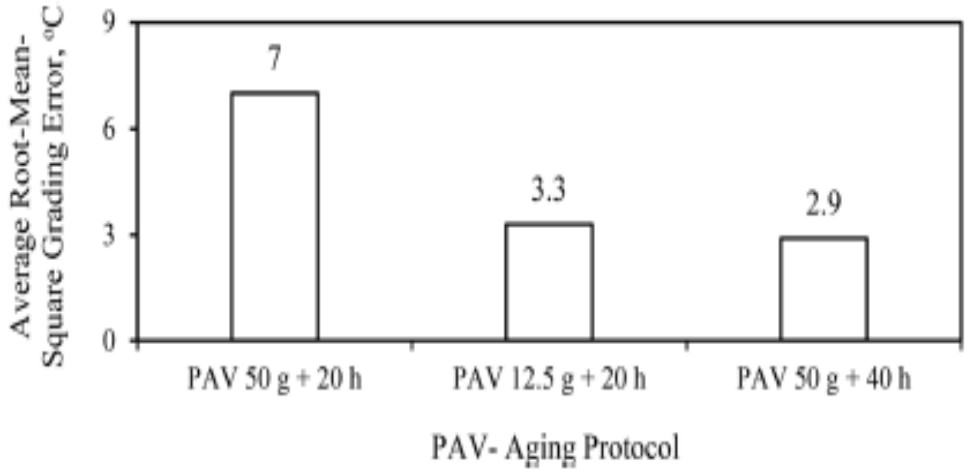

Figure 2.7: Average root-mean-square low temperature grading error for seven test section binders recovered after 8 years of service from a northern Ontario pavement trial [85].

As stated earlier, carbonyl and sulfoxide are major by-products of oxidative aging and their concentration could be measured using FTIR to determine the extent of oxidation [59][82].

Several studies such as Berkowitz *et al.* [82] and Petersen [83] have tried to relate binder physical properties such as viscosity, adhesion, and more, to the degree of oxidation. Further studies are on the way to understand the mechanism of oxidation.

2.12 Thermoreversible Aging

The study of thermoreversible aging dates back to over 100 years ago and much contribution has been made by various scientists to advance knowledge on this subject [82]. One of the early studies was carried out by Traxler *et al* [82] and Brown and Sparks [87] during which they concluded that binders of similar grades aged at different rates and that thermoreversible aging could be reversed provided the temperature is right. Hubbard and Pritchard [86] also studied the relationship between storage time and asphalt penetration. Other contributions from researchers like Blokker and Van Hoorn, Pechenyi and Zhelezko, Struik, and more have provided insight into the subject. However, a major setback on this subject is that only a few test methods have been developed to account for thermoreversible aging [82]. The SHRP program also launches investigation into this subject and some of the findings by Bahia and Anderson [88], indicate wax crystallization and shrinkage affect creep properties. Zhao and Hesp [74] also demonstrated a correlation between cracking severity and relaxation ability where m = 0.35. In another study, Hesp et al. [72], found a good correlation between grade loss after cold conditioning for three days and cracking severity in 20 eastern and northeastern pavement contracts. With the same pavement contracts, they found a good correlation between loss tangent (tan δ) and thermal cracking even after 2 hours of conditioning. Therefore, it will be worthwhile to consider the impact of limiting phase angle on cracking severity.

2.13 Effect of Engine Oil Residue on the Performance of Asphalt

Engine oil is made up of saturated, high molecular weight alkanes which gives it some characteristic properties such as low volatility, low viscosity, and high temperature oxidative

stability [42]. With the advent of the SHRP (Strategic Highway Research Program) Superpave specification in North America, there has been an influx of patents and publications on how to increase the performance grade of asphalt using various production processes and additives. The aim of this is to produce asphalt at a reduced cost. One of the additives suggested was the engine oil residue which serves as an extender for asphalt cement and a resource material during the air blowing process [43]. Further study by Villanueva *et al.* [45][42], shows that the addition of lubricating oils to air-blown asphalt makes it softer without altering the performance grade. It was later reported that the grade span for asphalt containing oil additives improved by 6 °C when compared with straight distilled asphalt.

However, other studies show that the addition of engine oil residue could be detrimental to the expected service life of pavement which is pegged at an average of at 25-40 years. One out of such studies by Hesp and Shurvell, was carried out on eastern and northern Ontario pavements. The research showed that engine oil residue was responsible for premature cracking of pavements within the region. To validate this claim, critical Crack Tip Opening Displacement (CTOD) confirms reduced strain tolerance in the samples and according to Ontario's specification tests LS-299 and LS-308, a tendency for physical hardening was discovered [42][45].

Moreover, due to the millions of gallons of recycled engine oil bottoms (REOB) generated from asphalt plants each year in the United States and Canada and the resulting environmental concerns, REOB is incorporated into asphalt in large quantity by manufacturers to lower cost. Despite the advantage that REOB increases Superpave grade spans by a reasonable amount, Paliukaite *et al.* [48], also observed that the strength and strain tolerance of the asphalt decreases with an increasing amount of REOB added. The load-displacement graph shown in Figure 2.8 explains this further.

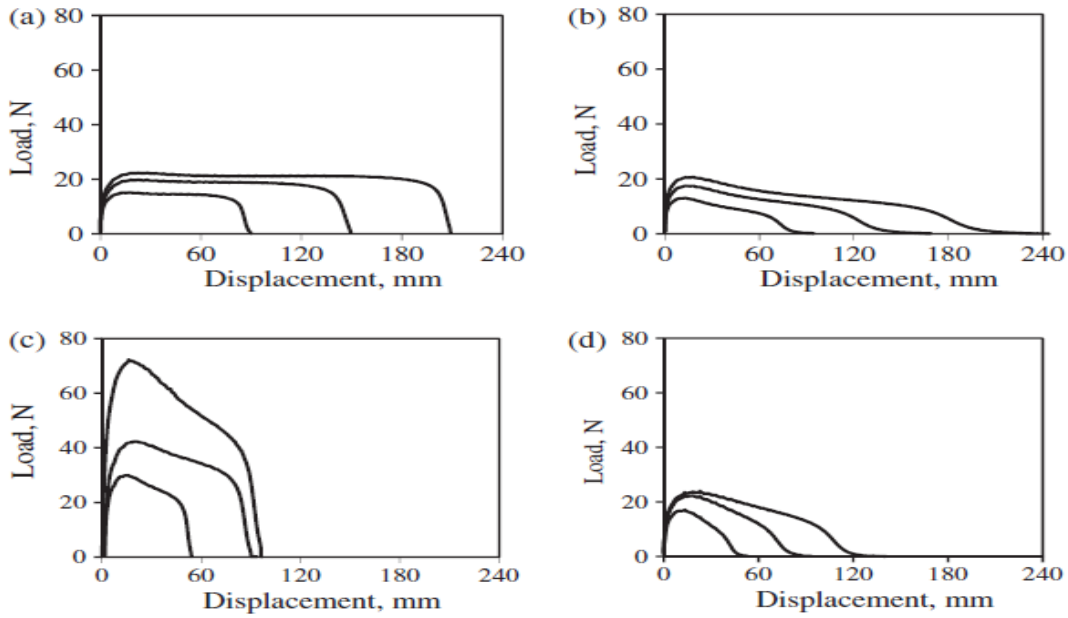

Figure 2.8: Load–displacement graphs of various modified Laguna, Venezuela binders: (a) 5 % SBS D1192, (b) 3 % SBS D1192 + 8 % REOB, (c) 4 % SBS D1192 + 20 % RAP, and (d) 2.5 % SBS D1192 + 6 % REOB + 20 % RAP [51].

2.14 Effect of Wax on the Performance of Asphalt

Wax is one of the additives added to asphalt to improve its performance and workability. Waxes are fluidifying additives commonly used in warm mix technologies to lower the viscosity of asphalt. An example of such is Sasobit. Sasobit is a synthetic hard wax comprising of long-chained aliphatic hydrocarbons produced during coal gasification using the Fischer-Tropsch process. According to Butz *et al*. [47], it is described as an "asphalt flow improver" due to its ability to improve the rutting resistance of asphalt. A DSR test carried out by Wei *et al*. [49], showed that when the wax content is increased, the complex modulus increased and the phase angle decreased for the two binders studied. A plot of complex modulus and phase angle against frequency at a reference temperature was obtained as shown in Figure 2.9 below.

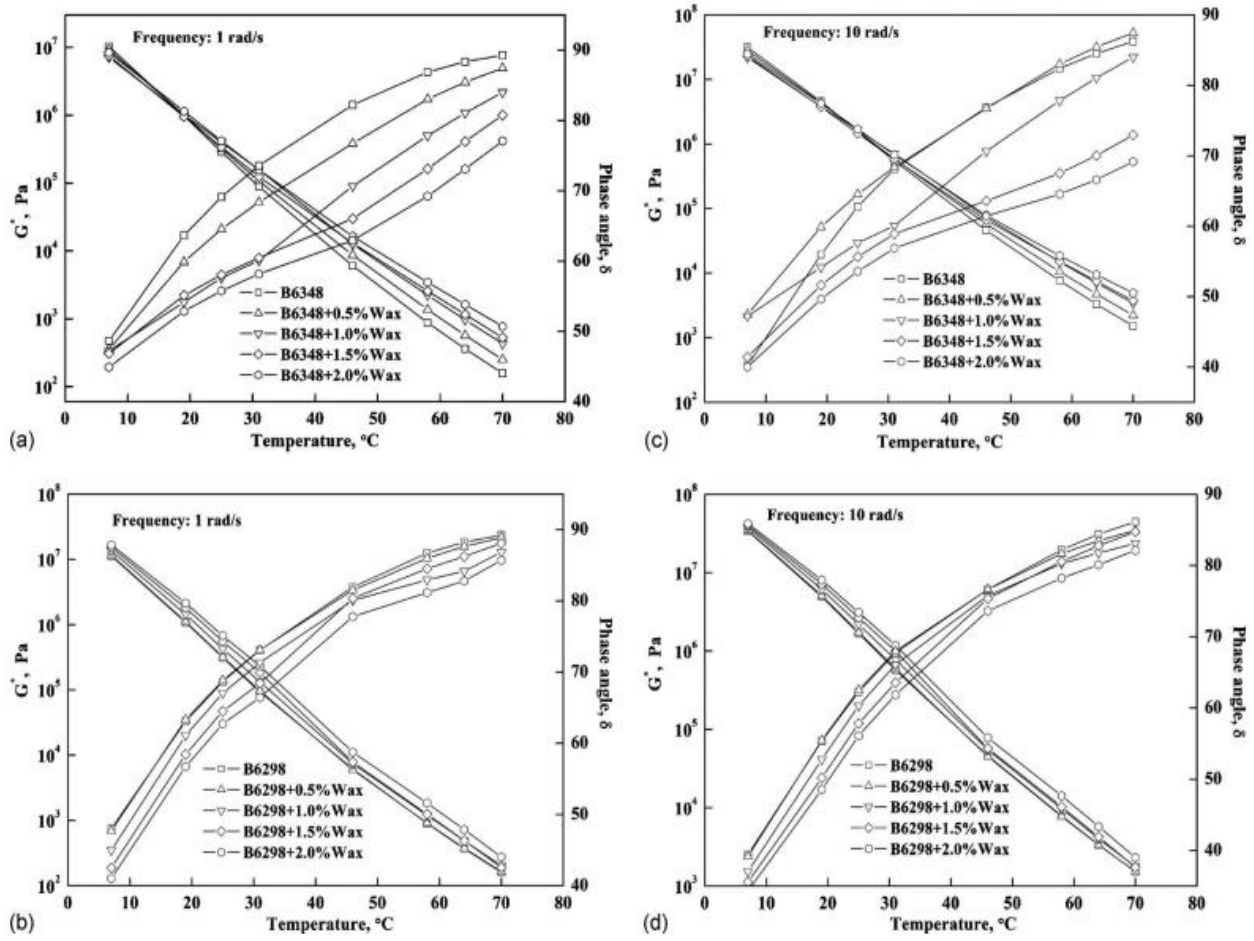

Figure 2.9: Influence of wax on the complex modulus (G^*) and phase angle of asphalt samples: [(a) and (b)] frequency 1 rad/s; [(c) and (d)] frequency 10 rad/s [49].

However, another study by Omari *et al*. [50] reveals that "wax-based, warm-mix asphalt (WMA) appears to suffer from the effects of cold conditioning with grade losses of over 6 °C in poor quality binders that were tainted with REOB or RAP". Research by Hesp *et al*. [51], also supports the aforementioned statement by Omari.

2.15 Effect of Polymers on the Performance of Asphalt

Recently, polymers are being incorporated into asphalt binder to improve their high temperature performance without altering their low temperature performance [51]. Performance of various polymer modified asphalt has been evaluated, an example of such is a study by Brule which

reiterates that the performance of polymer modified asphalt pavement is better than non-modified asphalt pavement [53][54].

However, from the literature, polymer-modified asphalts were found to have some limitations. Some of these include:

- They are more expensive
- Their stability is affected when stored at high temperatures for a long time and as a result, degradation may occur [51][52].

An example of polymers commonly used is the Styrene-Butadiene-Styrene (SBS). Despite its usefulness, the microstructure and performance of aged, SBS-modified asphalt were found to be similar to the unmodified asphalt [54].

Chapter 3

Materials and Experimental Methods

3.1 Materials

Binder samples A with varying performance grades are presented in Table 3.1 below, while binder samples B (B1-B53) were all reported to have a performance grade of PG 64-28. It is important to note that all the performance grades have a precision of +/- 1 °C. This corresponds to the interlaboratory precision requirement.

Table 3.1: Samples from Agency A and their respective performance grade.

Sample Code	Performance grade
A1	70-34
A2	76-28
A3	76-28
A4	58-28
A5	58-28
A6	58-28
A7	64-28
A8	58-28
A9	76-28
A10	64-28
A11	70-34
A12	58-28
A13	70-34

A14	64-28
A15	64-28

3.2 Extraction and Recovery of Asphalt Binder

Each asphalt mix was first heated in the oven to improve the workability of the samples. About 5 kg each of the loose mixes was soaked in dichloromethane (DCM) in a sealed clean container and allowed to stand overnight. The DCM was used as a solvent to recover the asphalt binder present in the mix. After allowing the DCM-soaked sample to stand overnight, it was decanted into a clean bottle and washed with excess DCM to recover more asphalt from the mix. The bottle was allowed to stand to ensure some sediments settle after which the extracted sample undergoes centrifugation twice into a clean labeled bottle. The essence of double centrifugation is to ensure the purity of the extract and to keep sediments to the barest minimum.

The extract was fed into the rotary evaporator as shown in Figure 3.1, to recover asphalt from the extract. It was assumed that the properties of the recovered asphalt are similar to the original asphalt binder although slight changes may occur as a result of exposure to high temperatures and other contaminations. Nevertheless, these changes are insignificant. The rotary evaporation process was carried out at a temperature between 70-80 °C and a pressure of 450 millibar to remove the solvent. Once the solvent completely evaporates, the temperature was raised stepwise to 160 °C at 20 °C intervals and the system kept at this temperature for an additional hour. The recovered binder was transferred into a clean beaker with a small portion being poured into a silicone mold for further testing.

Figure 3.1: Rotary evaporator [55].

3.3 Pressure Aging Vessel

The recovered asphalt was aged in a pressure aging vessel according to the American Association of State Highway Transportation Officials (AASHTO R28) protocol, called “Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV)”. In this test, about 50 g of the recovered samples were poured into a stainless steel pan. The pans were arranged in a rack and placed in the pressure aging vessel as shown in Figure 3.2. The samples were then aged for 20 hours in the PAV at a pressure and temperature of 2.1 MPa and 100 °C, respectively. Prior to removing the samples from the PAV (that is, after 20 hours), the pressure was first released. The samples were then transferred for further testing.

Figure 3.2: Pressure aging vessel [55].

3.4 Dynamic Shear Rheometer

DSR test was carried out on recovered binders to study their resistance to rutting and fatigue cracking at high and intermediate temperatures. The test was conducted on unaged and PAV-aged samples. Samples obtained from the rotary evaporation process are poured into silicone molds of 8 mm and 25 mm dimensions as shown in Figure 3.3 below. The 8 mm mold was used for intermediate temperature grading while the 25 mm mold was used for high temperature grading. A zero gap procedure at 46 °C between two parallel plates was performed before loading the sample and a heated trimming tool was used to remove excess samples after loading. Various parameters such as test temperatures, oscillating frequency (10 rads/sec) were set while the phase angle (δ) and the complex shear modulus (G*) were calculated by the DSR over the 10 cycles.

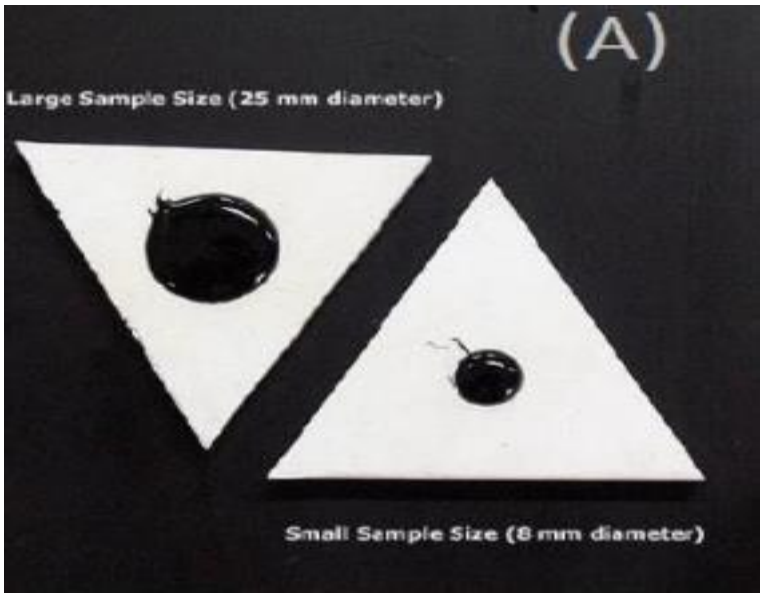

Figure 3.3: Asphalt sample in a 25 mm and 8 mm silicone mold [55].

3.5 Regular Bending Beam Rheometer (BBR)

According to the AASHTO M 320 standard test protocol, the regular BBR test is used to evaluate the performance of asphalt binders at low temperatures [56]. The BBR test measures the sample (PAV-aged and unaged) resistance to thermal cracking. Each sample was heated for about 30-45 minutes at about 150 °C and poured into a silicone mold to form a beam. The samples were allowed to cool for about an hour so they could take the shape of the mold. The beams were carefully removed from the molds and conditioned in an alcohol bath at the desired temperature of -18 °C and -8 °C. This is according to the T-design + 10 °C and T-design + 20 °C conditioning. The asphalt beam was then tested at a temperature of -18 °C and -12 °C. The test was conducted by placing a varying load of about 32 mN load at the midpoint of the beam for about 240 seconds as displayed in Figure 3.4. A graph of load and deflection versus time was then plotted automatically by the software to calculate creep stiffness as displayed in Figure 3.5 and Figure 3.6. The creep stiffness, as specified by AASHTO M320 specification, should be a minimum of 300 MPa at 60

seconds loading time and a maximum m-value of 0.3 at 60 seconds loading time to prevent thermal cracking.

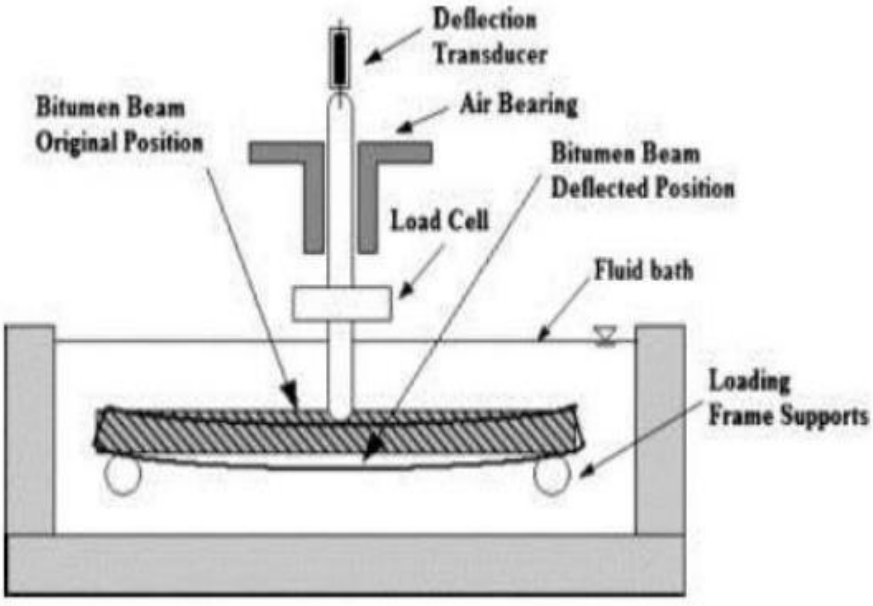

Figure 3.4: BBR test showing a deflected asphalt beam [55].

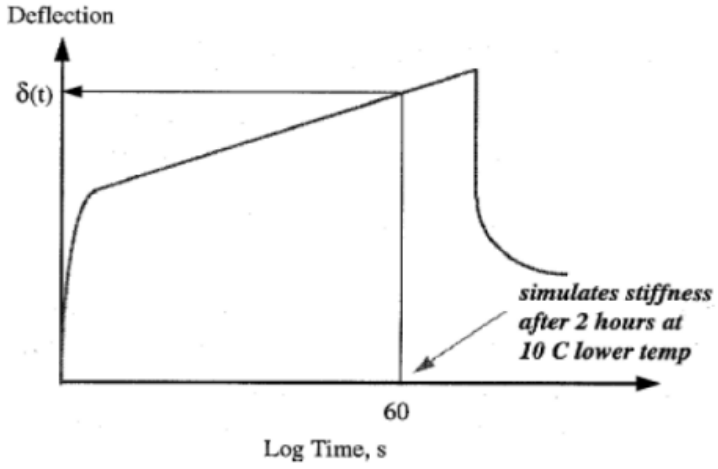

Figure 3.5: A typical graph of change in deflection with time [55].

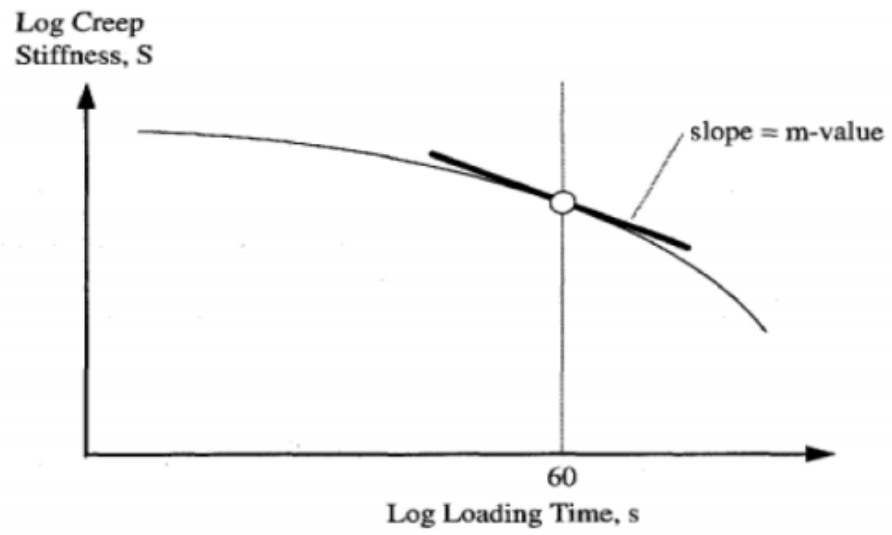

Figure 3.6: Evaluation of m-value of asphalt binders [57].

3.6 Extended Bending Beam Rheometer (EBBR)

In this method, asphalt beams were conditioned at a temperature of -8 °C and -18 °C for 1 hour, 24 hours, and 72 hours and tested at a temperature of -12 °C and -18 °C. The creep stiffness and m-values of each sample were calculated following AASHTO M320 test protocol. The limiting temperatures and worst grade loss were calculated after 72 hours. One major difference between the regular BBR and EBBR is the extended conditioning time.

3.7 Double-Edge-Notched Tension (DENT)

For this test, samples were heated in an oven at a temperature of 160 °C to facilitate free flow and homogenization. The samples were then poured into three different notched silicon molds with different ligament lengths (that is, 5mm, 10mm, and 15mm) as shown in Figure 3.7. The asphalt binders were allowed to cool off at room temperature for an hour. The essence of the aforementioned is to get it solidified and to allow easy removal from the silicone mold.

Each of the molded samples was then conditioned in a water bath at 15 °C for an hour after which the thickness of each sample was measured. The testing occurred at a speed of 50 mm/minute until the samples are torn apart. Three samples were loaded and tested at once to ensure reproducibility

in the result. The calculated parameters such as total work of fracture and average CTOD were calculated and exported to an excel spreadsheet.

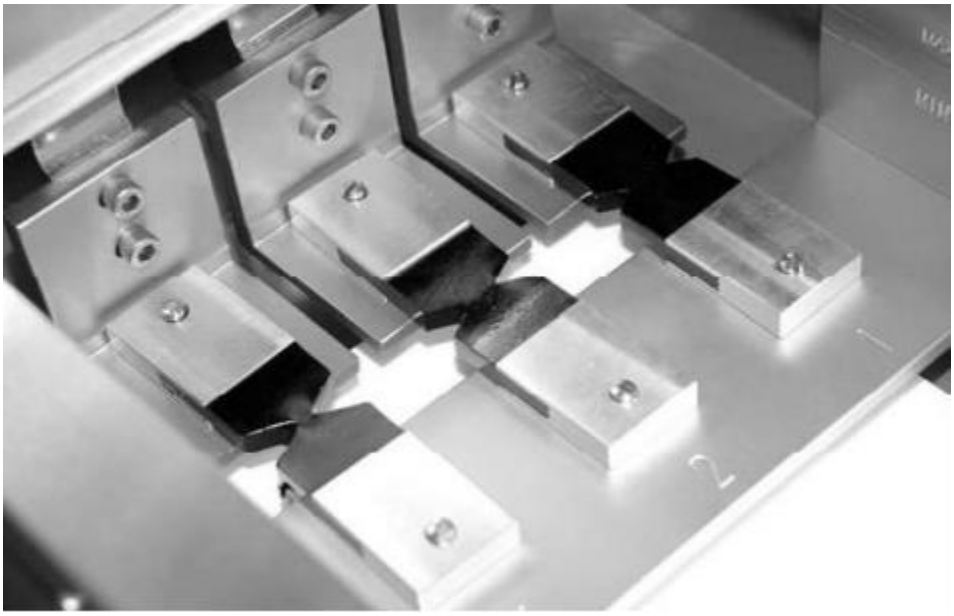

Figure 3.7: DENT test set up [55].

Chapter 4

Results and Discussion

For this thesis, a total of 14 samples were fully processed to compare the binder properties with climatic and traffic requirements. A further 338 samples from previous research were analyzed. Of these, a total of 98 samples were excluded because the limiting phase angle temperatures could not be determined with sufficient accuracy by interpolation, or by extrapolation by less than 3 °C. A single outlier was also excluded from the data set. As a result of these exclusions, the remaining 239 samples were analyzed to compare limiting phase angle temperatures with various Superpave and EBBR specification limits. It is important to note that Agency B has implemented acceptance criteria based on extracted and recovered binder properties while Agency A has yet to implement this approach. Our involvement has been at a high level to evaluate the appropriateness of the acceptance criteria (effectiveness of the specification approach), rather than to test whether specific samples passed or failed as part of the quality assurance testing according to contract requirements.

4.1 Data Selection and Validation

As stated earlier, various data sets were obtained from previous research carried out in the lab. However, to ensure data accuracy and repeatability, some data were excluded because they do not meet the interpolation or extrapolation requirements. For T(30°), the selected data were those within a 3 °C range from the lowest temperature while for T(G*(60 MPa)), a 6 °C range was chosen. This instills a higher level of confidence in the data sets.

4.2 Data Repeatability and Reproducibility

Repeatability is a means to evaluate the precision of an experiment to determine if any variation occurs when the same procedure is repeated by the same researcher. For this study, repeatability is determined as an experimental procedure carried out by a researcher, using the same machine

under the same condition, within a short period. Reproducibility, on the other hand, involves an experiment carried out by different researchers with different machines in a different lab.

4.3 Contract Requirements

The high temperature performance, intermediate temperature performance, and low temperature performance of binders was examined to check if climatic requirements are met.

4.3.1 High Temperature Performance Grade Test

The high temperature performance was determined using the Superpave grading protocol. It is the temperature at which G/sinδ equals 1.0 kPa for unaged samples or 2.2 kPa for RTFO aged samples. The HTPG data obtained for Agency A and B were compared with the contract requirements and are presented in Figures 4.1 and 4.2 respectively. From Figure 4.1 below, all the binders but A1, A2, A9, A11 and A13 met the contract requirement. While A2, A9, A11, A13 are within the acceptance margin, A1 missed the requirement by a considerable amount. The deviation could be due to extraction and testing errors. However, A4 and A5 might contain some additives which gave them a bump grade with a stiffness that is four times higher than what is required according to climate and traffic levels. This may negatively affect the long-term cracking performance of the pavement.

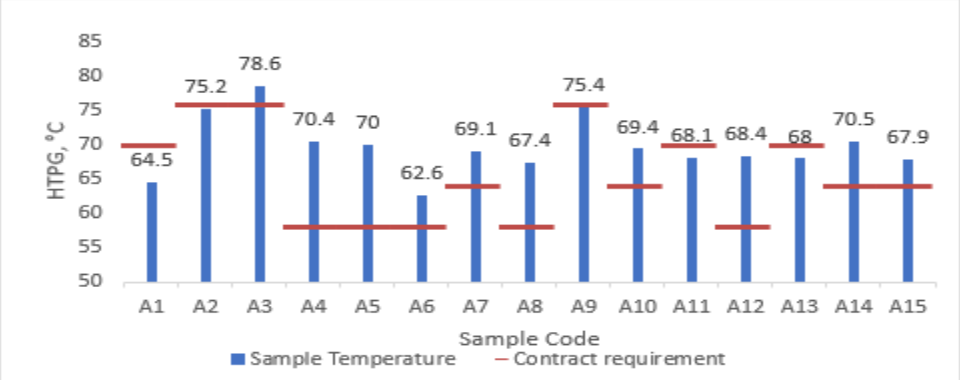

Figure 4.1: High temperature grade of sample A1-A15 in comparison with the climatic and traffic requirement.

From Figure 4.2, all but B48 met the contract requirement. B48 fell short of the requirement possibly due to DSR testing error or the presence of unrecovered polymers. Samples B9, B14, B23, B30, B44, B49, B50 and B51 all have a performance grade that is as much as four times stiffer than the required. The binders might be polymer modified. Moreover, due to the high cost of keeping multiple storage tanks, the asphalt company may have supplied different performance grades to replace the required grade. As stated earlier, the excessive stiffness could show a negative cracking performance in the long run.

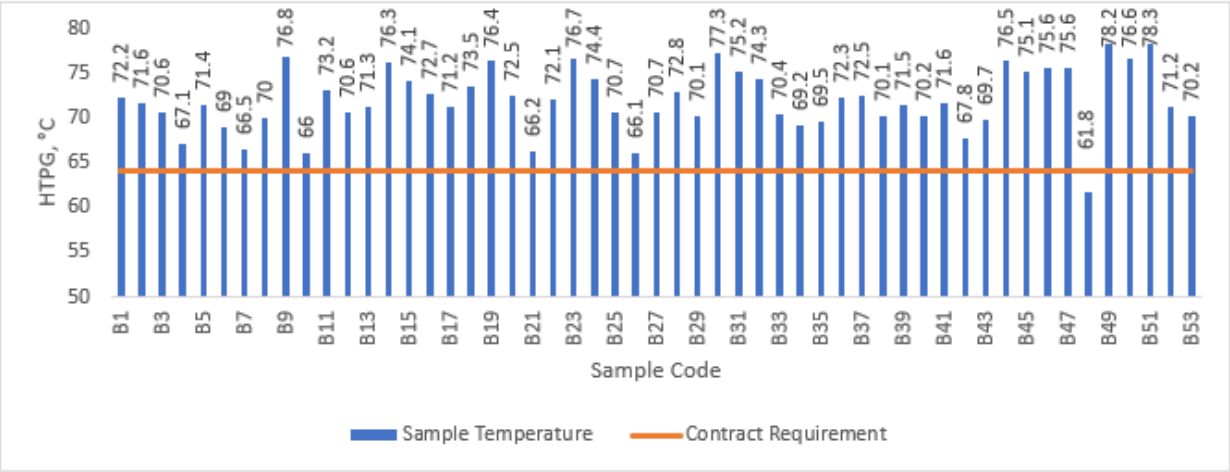

Figure 4.2: High temperature grade of sample B1-B53 in comparison with climatic and traffic requirement.

4.3.2 Intermediate Temperature Performance Grade

The temperature at which G*sinδ equals 5.0 MPa gives the intermediate temperature performance grade. Figure 4.3 shows that all but A4, A5, A8, A14, and A15 met the contract requirement. This could be because the agency failed to implement the extraction and recovery approach. Also, G*sinδ does not accurately relate to the cracking performance of the binders.

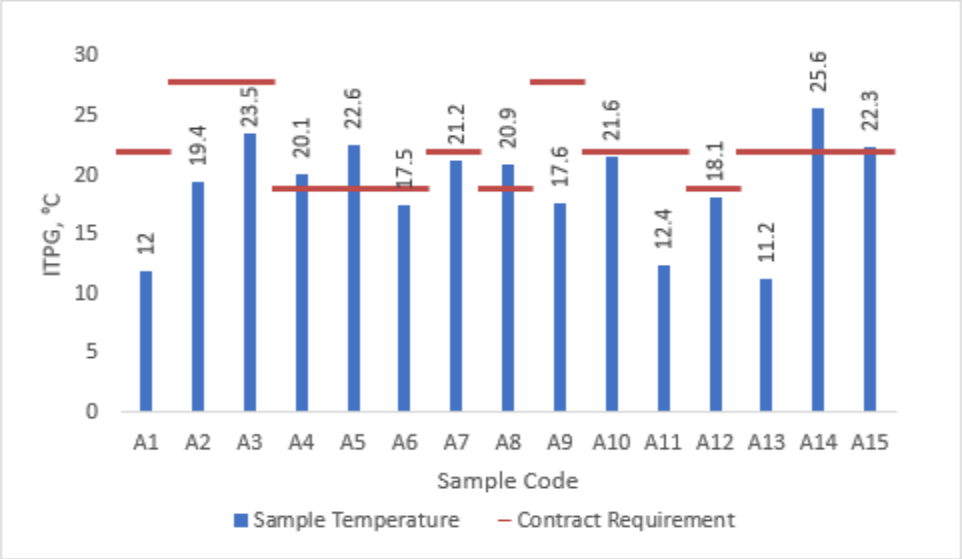

Figure 4.3: Intermediate temperature grade of sample A1-A15 in comparison with climatic and traffic requirement.

As shown in Figure 4.4, all the binders met the contract requirements. In contrast with Agency A, Agency B already implemented the extraction and recovery approach. This approach shows significant success over the conventional method.

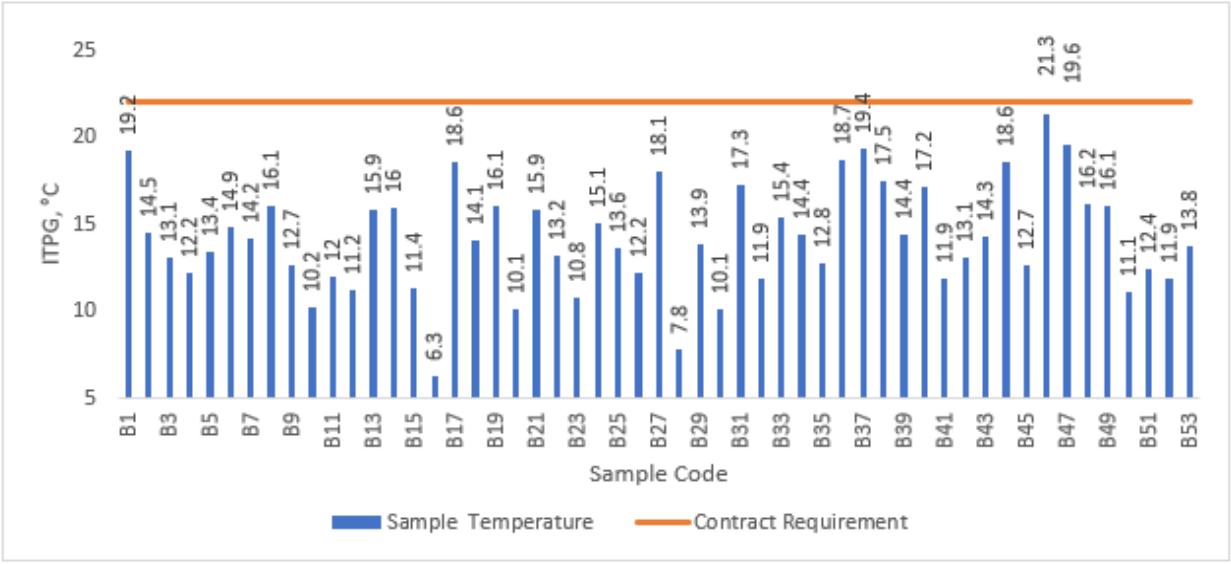

Figure 4.4: Intermediate temperature grade of sample B1-B53 in comparison with climatic and traffic requirement.

4.3.3 Low Temperature Performance Grade

The low temperature performance grade of each sample was determined using the LS-308 protocol at 10 °C and 20 °C above the pavement design temperature with a conditioning time of 1 hour and 72 hours respectively [90]. The limiting low temperature grade is the warmest temperature at which creep rate at 60 seconds, m(60), reaches 0.300 or the creep stiffness at 60 seconds of loading, S(60), reaches 300 MPa is the LLTG [91].

Figures 4.5 and 4.6 show that all the binder samples passed the LTPG test. However, attention is drawn to samples B16 and B28. They are both approximately 10 °C below the contract requirement. A possible reason is that the binders were over-designed and may age faster in service than the others.

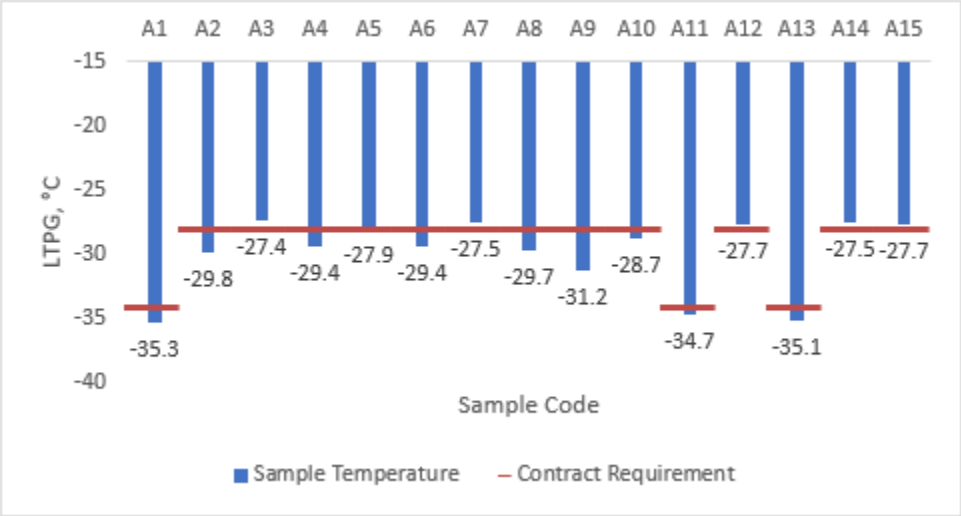

Figure 4.5: Low temperature grade of sample A1-A15 in comparison with climatic and traffic requirement.

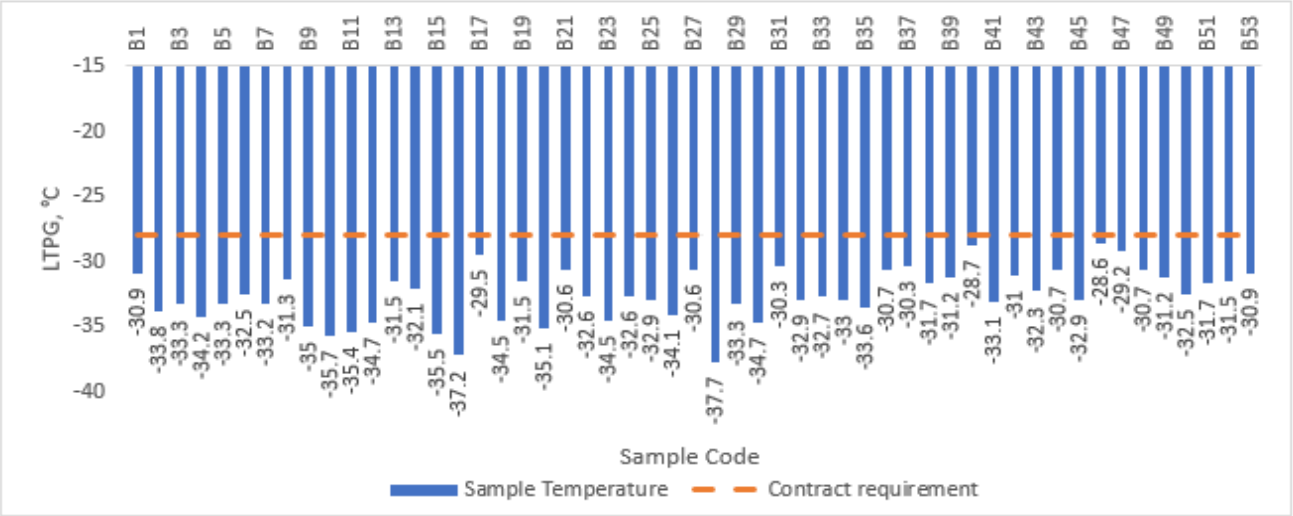

Figure 4.6: Low temperature grade of sample B1-B53 in comparison with climatic and traffic requirement.

4.3.4 Limiting Low Temperature Performance Grade

After cold conditioning for 72 hours, a considerable number of samples fail the requirement. Figure 4.7 shows that all the binders fail to meet the mark by varying amounts. A major explanation for this is that Agency A has yet to implement specifications based on extracted and recovered binders. These binders may contain RAP and have higher stiffness leading to premature cracking.

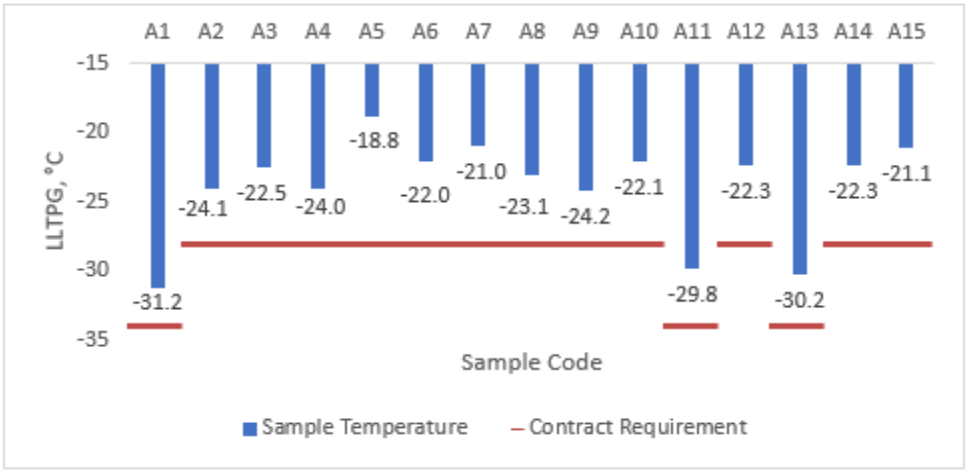

Figure 4.7: Limiting low temperature performance grade temperature for sample A1-A15 in comparison with climatic and traffic requirements.

In contrast with Agency A, Figure 4.8 shows that all but B1, B17, B19, B27, B37, B40, B44, B46, B47, and B48 meet the contract requirement. The failed samples all fall within the acceptable margin of 2 °C, except B17 which has a significant failure of 5.2 °C. The supplier might have supplied the binder with the wrong performance grade or added some RAP to it. This will negatively impact the long-term cracking performance of the binders.

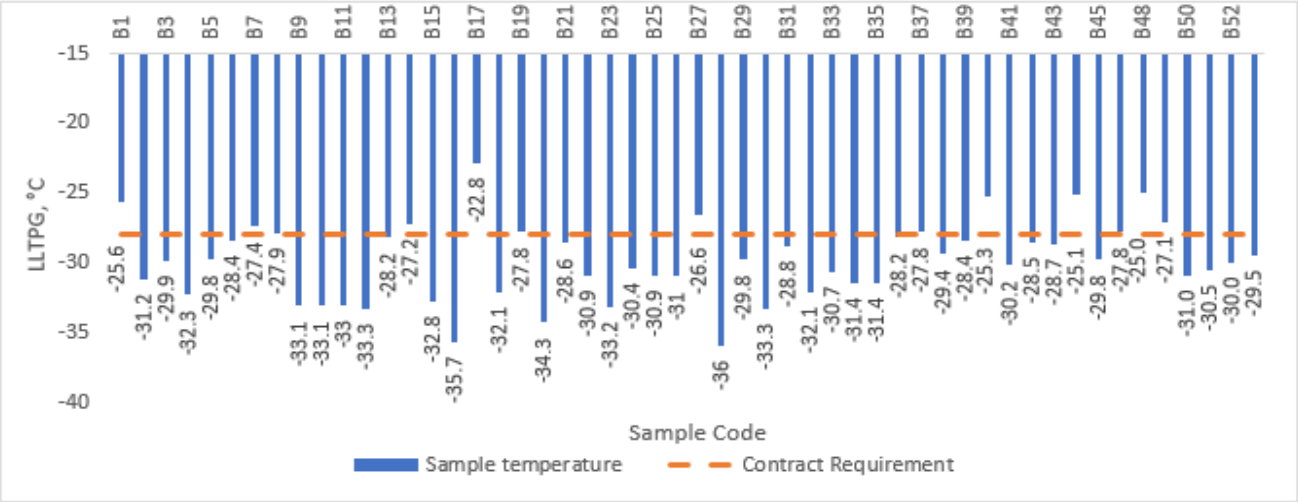

Figure 4.8: Limiting low temperature performance grade temperature for sample B1-B53 in comparison with climatic and traffic requirements.

4.3.5 Grade Loss

Grade loss is the difference between the low temperature grade obtained at 1 hour and 72 hours of cold conditioning. From Figure 4.9, about 50 % of the total samples (A5, A6, A7, A8, A9, A10, A15) fail to meet the grade loss requirement. This shows that the approach deployed by Agency A is less effective in characterizing the performance of the binders. Closer attention is drawn to A5 (9.1) and A15 (9.8) because they are significantly higher than the 6 °C limits. The failure could be attributed to the addition of RAP which increases the stiffness more than twice as well as the thermal stress. This ultimately leads to failure. Studies show that grade loss of 6 °C reduces the confidence level from 98 % to about 50 % in any year the pavement is not exposed to adverse

temperatures [92]. Other factors that could affect the grade loss are production errors such as overheating, the use of low quality binders, and more.

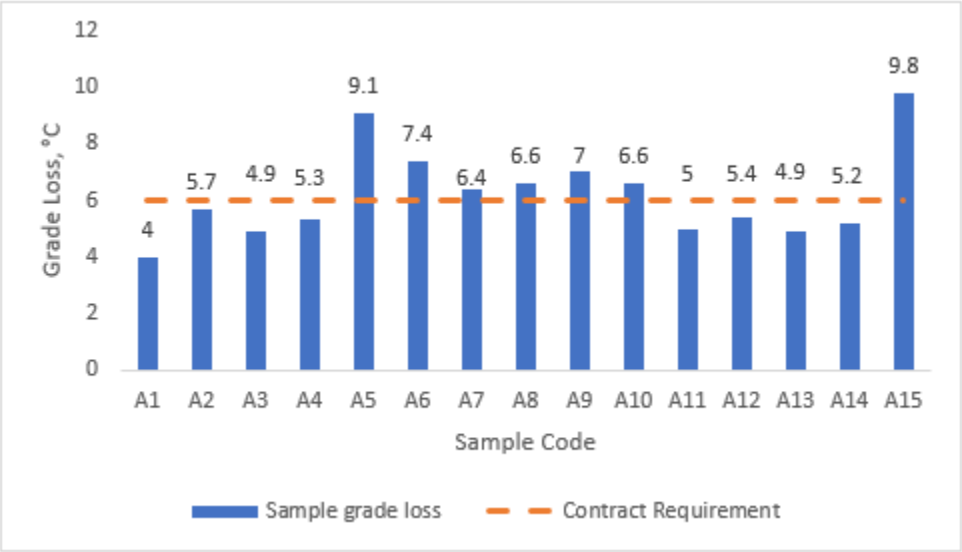

Figure 4.9: Grade loss for sample A1-A15 in comparison with climatic and traffic requirements.

Figure 4.10 shows that all but B17 pass the contract requirement. This is another evidence that the approach adopted by Agency B is more effective. The B17 sample might probably have a wrong specification or include RAP.

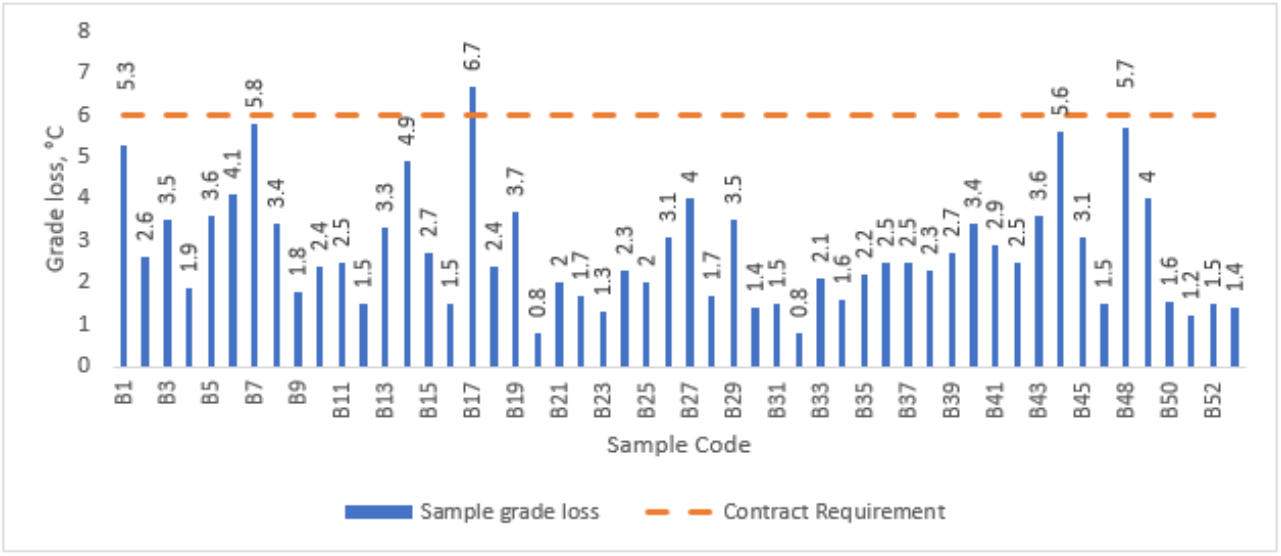

Figure 4.10: Grade loss for sample B1-B53 in comparison with climatic and traffic requirements.

4.3.6 Double-Edge-Notched Tension Test

Figure 4.11 shows that all but A9, A11, A13 fail to meet the contract requirement and are likely to fail in the climate they were intended to be used. While A1, A2, A3 are still within the acceptance margin, others are below. In a climate of -28 °C and -34 °C, a CTOD value of 14 mm and 20 mm are specified respectively. On the other hand, samples from Agency B largely meet the contract requirement except for B1, B8, B17, B27, B46. In other words, there is only 8 % failure for Agency B as compared to 80 % failure for Agency A. It is important to note that B12, B15, B22, B23, B25, B26, B30, B32, B40, B45 all have CTOD value that is far above the limit. The supplier might have supplied the wrong performance grade or added RAP to improve the ductility of the binders. The low failure rate in Agency B could be attributed to the approach adopted. This is an indication that the CTOD value is effective in characterizing binder performance.

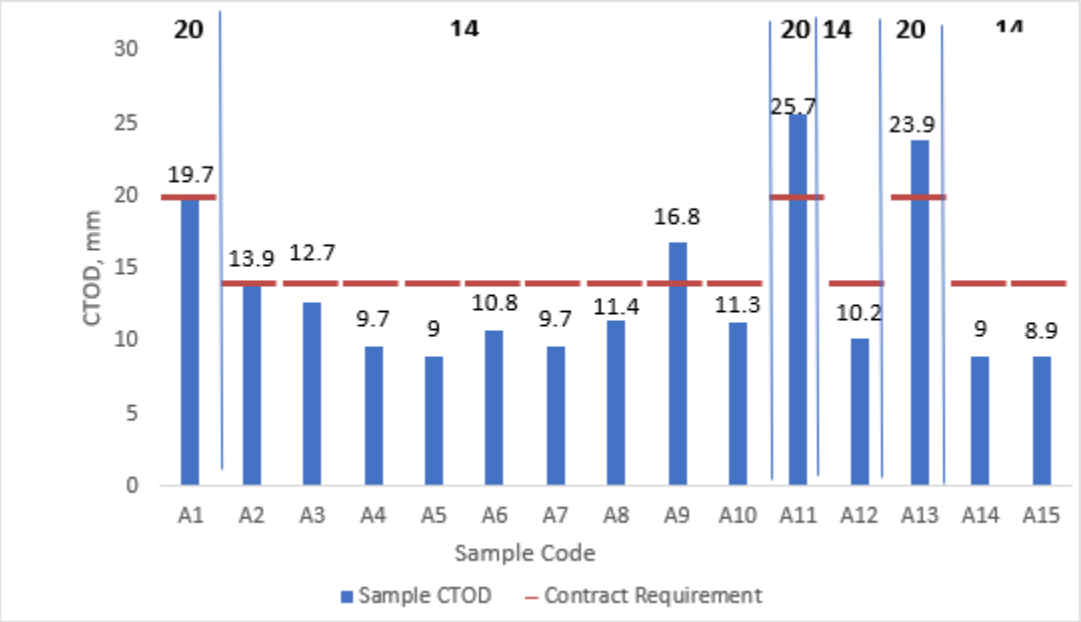

Figure 4.11: CTOD of sample A1-A15 in comparison with climatic and traffic requirements.

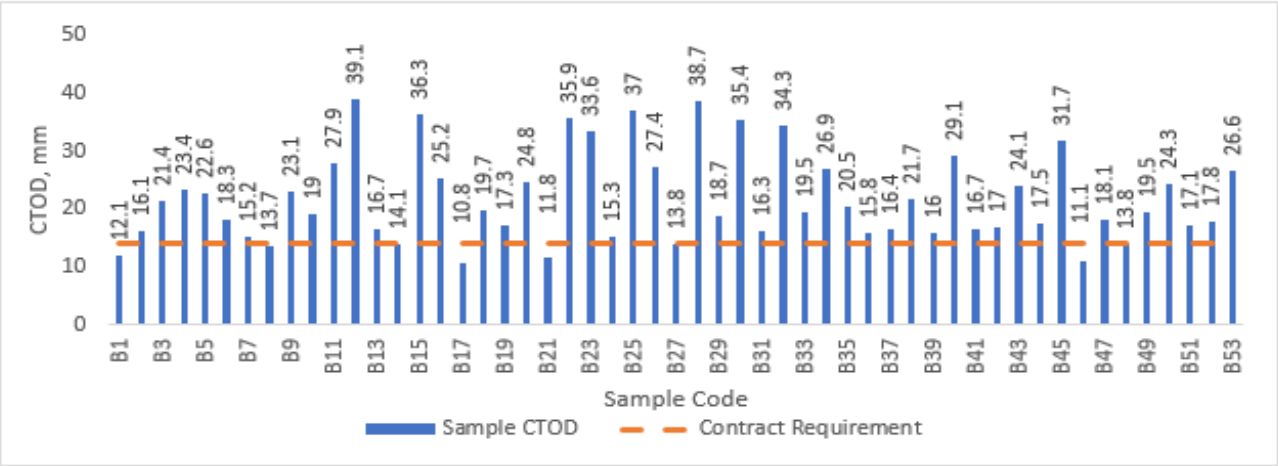

Figure 4.12: CTOD of sample B1–B53 in comparison with climatic and traffic requirements.

4.3.7 Difference in Limiting DSR Temperature (Tcd)

The difference in limiting DSR temperature (ΔT_{cd}) for Agency A and B were presented in Figure 4.13 and Figure 4.14. The ΔT_{cd} is given as $(T(30°) - T(G^*(60\ MPa)))$ and could give an insight on whether a sample is a gel or sol. Samples from Agency B has a higher range of -19.7 to +2.8 as compared to -2.6 to 4.5 in Agency A. Because ΔT_{cd} is a DSR parameter, it can be obtained at a higher temperature and faster equilibration [91]. Thus, giving it an advantage over the extended BBR (EBBR) procedures.

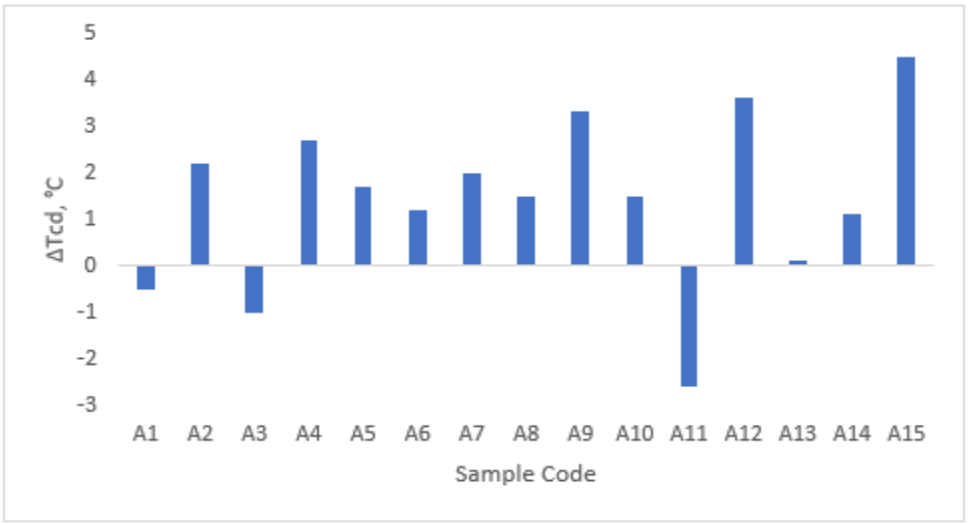

Figure 4.13: Difference in limiting DSR temperature (ΔT_{cd}) for samples from Agency A.

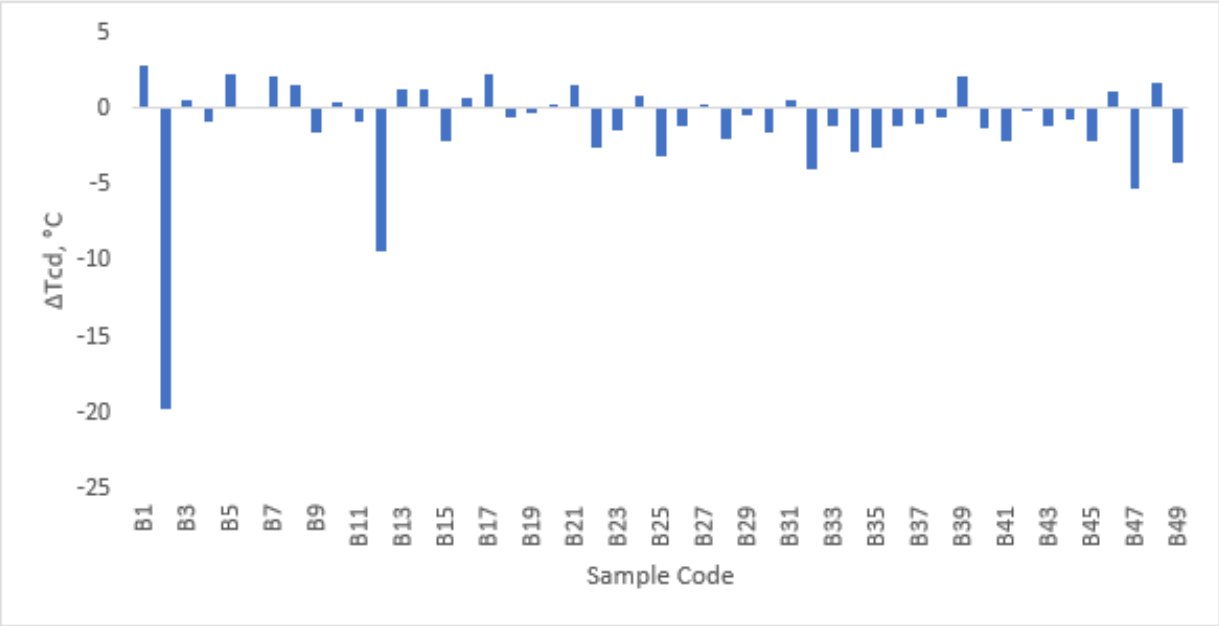

Figure 4.14: Difference in limiting DSR temperature (ΔT_{cd}) for samples from Agency B.

4.4 Correlation of HTPG with Phase Angle [T(30°), T(45°)]

Figure 4.15 depicts the relationship between high temperature performance grade and limiting phase angle temperatures. The core and loose-mix samples have the highest and lowest correlation between HTPG and phase angle, respectively. The higher correlation observed with core samples could be due to their sensitivity to the rutting factor. In other words, core samples are likely to have a higher resistance to deformation at high temperatures. Since each sample was tested at a higher temperature or low frequency, the storage modulus is higher, and thus exhibits higher viscous behavior. This explains why T(45°) has a higher correlation.

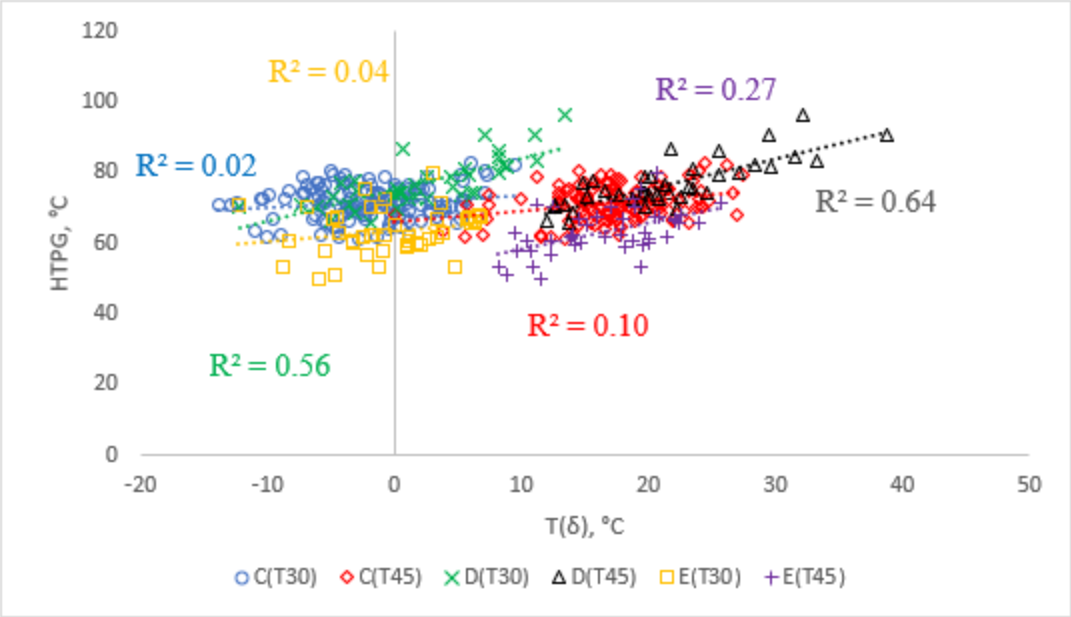

Figure 4.15: Correlation of HTPG with limiting phase angle temperature [T(30°), T(45°)].

Note: Legends C, D, E represent loose mix, core, and tank samples respectively.

4.5 Correlation of ITPG with Phase Angle [T(30°), T(45°)]

From Figure 4.16 below, ITPG shows a higher correlation with T(30°) when compared with T(45°). Using a ranking system, at T(30°), the core samples have the highest correlation followed by loose mix and tank samples respectively. A repeat of the order would have been expected at T(45°) but the reverse is the case. This shows that ITPG does not give an accurate and efficient prediction of binder performance. Worthy of note is the data range for T(30°), T(45°) and ITPG. They have a range of 27.2, 38.8, and 26.3 respectively. It can be concluded that both T(30°) and T(45°) have higher sensitivity when compared to ITPG. T(45°) was found to be 48 % more sensitive to ITPG, most likely because the elastic storage modulus is higher than the loss modulus at that phase angle and the gelation point is closer [93]. Thus, the measurement of phase angle could predict binder performance, however, further study is advised to ensure confidence, repeatability, and reproducibility.

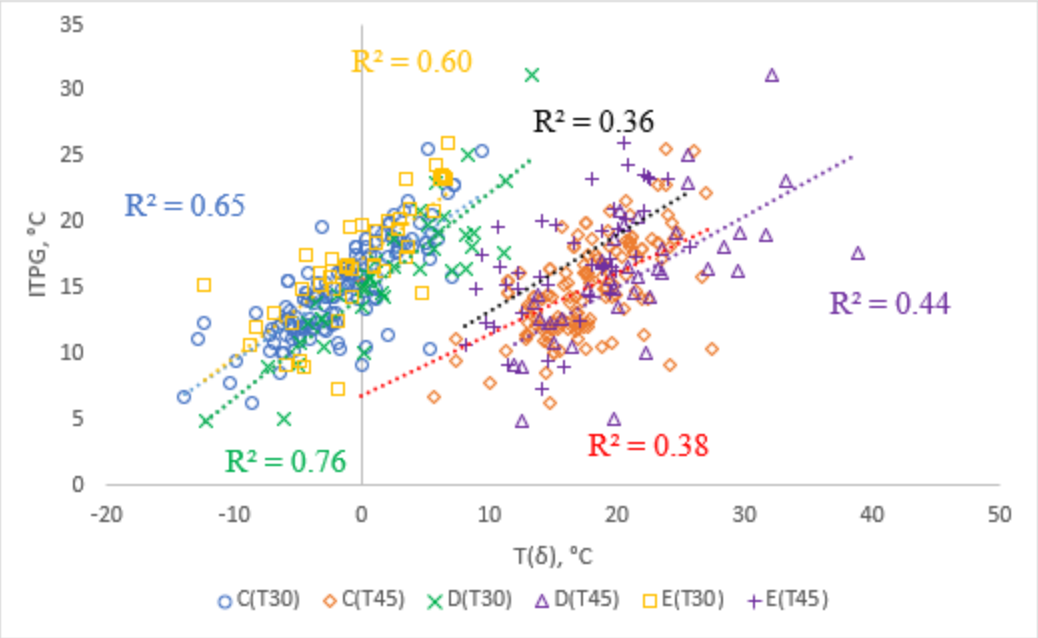

Figure 4.16: Correlation of ITPG with limiting phase angle temperature [T(30°), T(45°)].

Note: Legends C, D, E represent loose mix, core and tank samples respectively.

4.6 Correlation of LTPG with Phase Angle [T(30°), T(45°)]

As shown in Figure 4.17, core samples exhibit a higher correlation while tank samples show the least correlation. This once again showed that acceptance base on extracted and recovered binders can predict performance more efficiently when compared to acceptance based on tank samples. Phase angle was found to have a wider data range (52.6) while LTPG has a shorter data range (22.9). In other words, for this set of data the phase angle is 2.3 times more sensitive than LTPG. This is an indication that phase angle measurement could predict thermal cracking more efficiently.

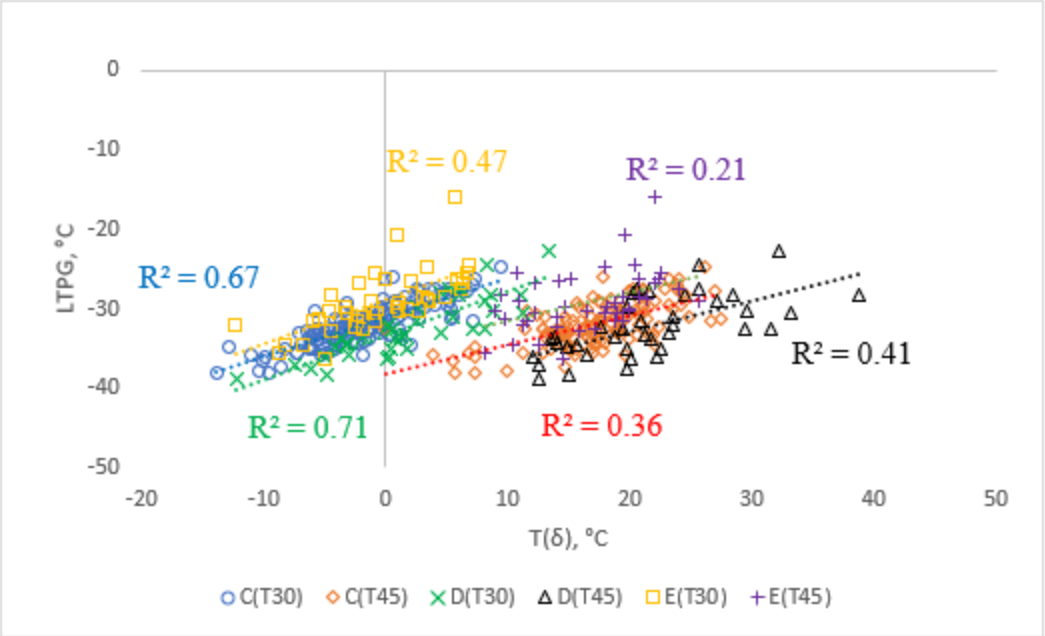

Figure 4.17: Correlation of LTPG with limiting phase angle temperature [T(30°), T(45°)].

Note: Legends C, D, E represent loose mix, core and tank samples respectively.

4.7 Correlation of LLTPG with Phase Angle [T(30°), T(45°)]

When compared with Figure 4.17 above, Figure 4.18 showed a higher correlation. This is not surprising because the extended BBR (EBBR) test accounts for the effect of aging and thus predicts thermal cracking behavior better than the regular BBR. Also, after 72 hours of cold conditioning, the grades are influenced by the m-value, which further explains the higher correlation observed [93][90]. At T(30°), the data range for LLTPG and phase angle is given as 21.7 and 27.2 respectively. This indicates that phase angle measurement is 25 % more sensitive than LLTPG. In contrast with the extended BBR (EBBR) test which requires more labor, time, and samples. Phase angle measurement could be carried out easily with a very small sample and in lesser time. While a wide range is good, caution must be exercised to reduce error and ensure relative precision over the range. Therefore, phase angle measurement could be a viable alternative to the extended BBR (EBBR) test in characterizing the low temperature performance of binders.

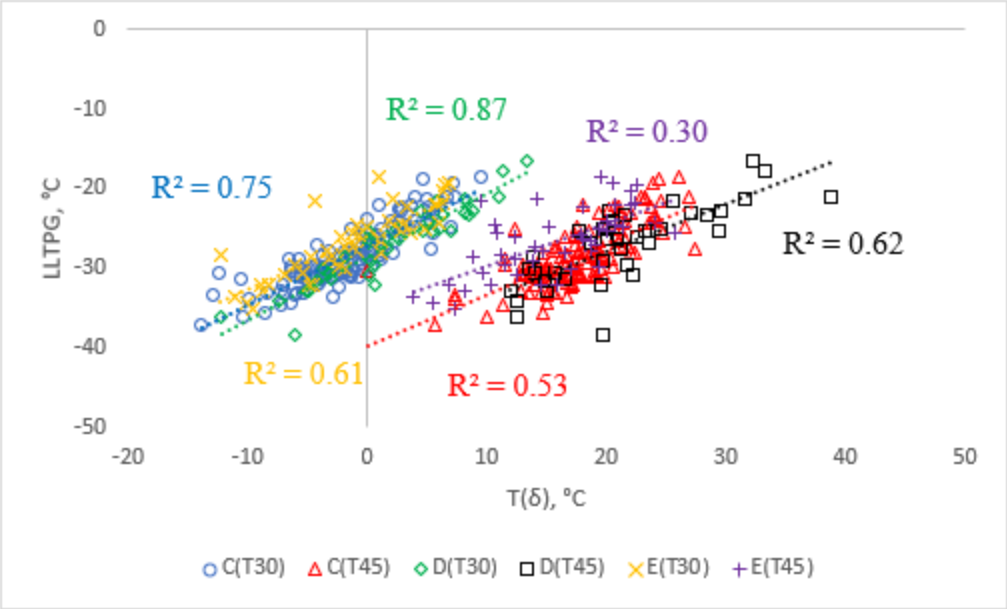

Figure 4.18: Correlation of LLTPG with limiting phase angle temperature [T(30°), T(45°)].

Note: Legends C, D, E represent loose mix, core and tank samples respectively.

4.8 Correlation of Grade Loss with Phase Angle [T(30°), T(45°)]

Grade loss can be obtained from the extended BBR (EBBR) test after 72 hours of cold conditioning. It is the difference between the grades obtained at 1 hour and 72 hours. From Figure 4:19 below, grade loss for loose mix samples has the highest correlation with phase angle. Of the three groups of samples, tank samples have the least correlation. Grade loss has a small data range and could be the reason for its lower correlation with phase angle. Unlike the extended BBR (EBBR), grade loss does not provide a piece of conclusive information at the moment unless if further study is carried.

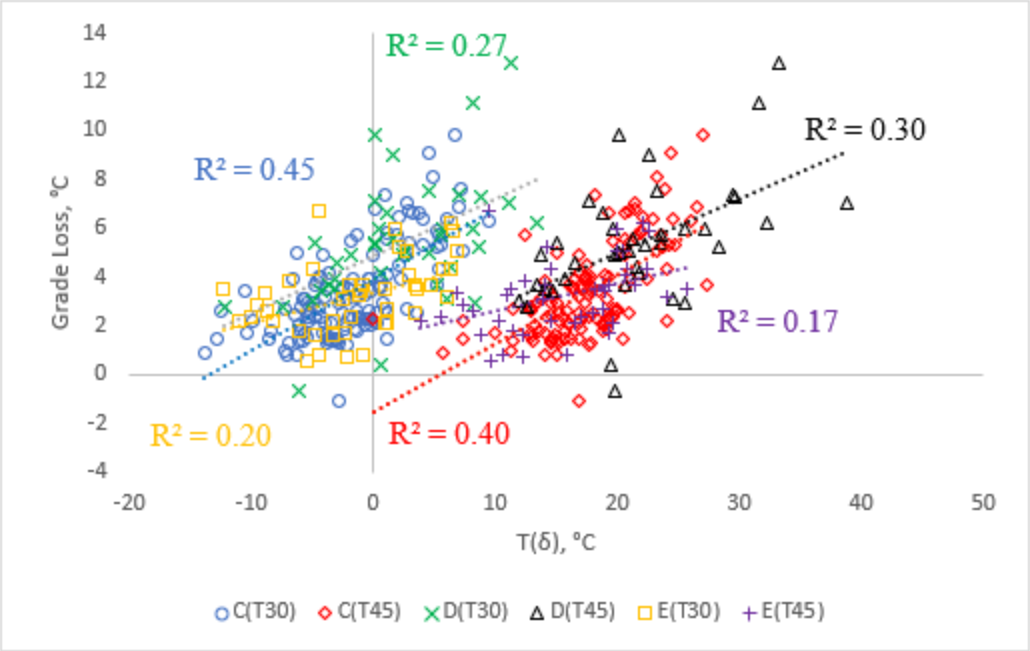

Figure 4.19: Correlation of grade loss with limiting phase angle temperature [T(30°), T(45°)].

Note: Legends C, D, E represent loose mix, core, and tank samples respectively.

Chapter 5

Summary and Conclusions

The summary and conclusions outlined below are a reflection of all that has been discussed in this study, including the literature review and experimental methods.

- From the different tests (HTPG, ITPG, DENT, BBR, EBBR) carried out on samples from Agency A and Agency B, Agency B largely met the climatic and traffic requirements. Agency A uses an approach based on tank samples while Agency B has recently adopted an approach based on extracted and recovered samples. The approach adopted by Agency B thus proves to be much more effective in controlling binder and therefore long-term pavement performance.
- The HTPG analysis carried out on both groups of samples showed that samples from Agency B performed better overall—an indication that they can better prevent rutting at high temperatures. The ITPG test passes almost all the samples from Agency A and B. This further proves that G*sin(δ) will not accurately control cracking performance in service.
- The CTOD analysis from the DENT test (LS-299) provides a better means of distinguishing fatigue cracking performance of superior performing binders from the poor ones. This is evident because most samples from Agency A failed the test while the majority of Agency B passed the test.
- From the DENT analysis, we can deduce that some of the samples from Agency B were likely modified with polymers because they are much higher in CTOD than the set limit. Although these samples have higher strain tolerance at 15 °C, it does not necessarily translate to how they will behave at extremely low temperatures.

- In contrast with the extended BBR (EBBR) protocol, the BBR passes all the extracted and recovered binders. A major setback for the BBR procedure is that it fails to account for the thermoreversible aging in the binders. Therefore, it is unable to satisfactorily separate good binders from poor binders. This is one of the reasons why asphalt binders undergo significant thermal cracking in colder climates. On the other hand, the EBBR procedure accounts for thermoreversible aging in binders, thus providing a deeper insight into the thermal cracking performance. However, the EBBR procedure involves a large sample requirement, significant time, and labor. Furthermore, samples with grade loss higher than 6 °C (especially A5 and A15) tend to lower confidence and perform badly in extreme weather conditions.
- Different correlations were done to further understand how various parameters relate to phase angle. A broader data set including core samples, loose mix samples, and tank samples was used to determine if a limiting phase angle temperature measurement is a viable alternative to predict binder performance at low temperatures. From this study, $T(\delta = 30°)$ and $T(\delta = 45°)$ showed a moderate correlation with HTPG (tank samples) because the latter test was carried out at higher temperature. The storage modulus is also higher, and thus exhibit viscous behavior particularly close to $T(\delta = 45°)$. Other correlation studies involving ITPG, BBR, and EBBR with limiting phase angle temperature showed that core samples have the highest correlation followed by loose mix samples. Tank samples were found to poorly correlate. From the large data set used in this study, it is becoming more evident that limiting phase angle temperatures correlate strongly with EBBR grades, require less time and material, and possess higher sensitivity. Hence, T(30°) and perhaps T(45°) will provide viable alternatives to evaluate thermal cracking.

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