

**EFFECTS OF LOW TEMPERATURES, REPETITIVE STRESSES AND  
CHEMICAL AGING ON THERMAL AND FATIGUE CRACKING IN  
ASPHALT CEMENT PAVEMENTS ON HIGHWAY 417**

by

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## Abstract

Thermal and fatigue cracking are pavement distresses that deteriorate asphalt pavements in Canada. However, the current AASHTO M320 standard specification protocol does not give satisfactory correlation between the properties measured in the laboratory to thermal and fatigue cracking performance of the asphalt in service. This thesis is aimed at validating the newly developed MTO LS-299 and LS-308 specification test methods for predicting pavement distress. A secondary objective is to determine how well laboratory-aged and field-aged binders correlate with each other in terms of their chemical and physical properties. Chemical testing using infrared (IR) spectroscopy and X-ray fluorescence (XRF), as well as physical and mechanical testing using the regular bending beam rheometer (BBR), extended BBR (eBBR), dynamic shear rheometer (DSR), and double edge notched tension (DENT) tests were performed on laboratory-aged and recovered binders from Highway 417.

Asphalt cements with significant amounts of waste engine oil residues as determined by XRF data were found to have cracked severely due to their high tendency for chemical aging. Western Canadian binders modified with styrene-butadiene-styrene polymer showed low affinity for both chemical and physical aging as determined from their carbonyl indices. Asphalt binders with smaller paraffinic structures exhibited insignificant pavement deterioration while the opposite occurred to those with low aromatic indices according to their IR data. The DSR data show that chemical aging occurs much faster in the laboratory-aged binders than the field-aged binders. The DENT test is able to separate superior performing binders from inferior ones with 86% accuracy according to their CTOD data. The regular BBR gave poor correlation between the laboratory test methods and the performance of the pavements. Good correlation exists between the laboratory test methods and the performance of the pavements in service according to the

eBBR data. Pavements without any cracks showed lower grade losses, while pavements with severe thermal cracking recorded higher grade losses after three days of conditioning prior to testing. The study has shown that the eBBR and DENT tests are better tools for predicting pavement performance and provide good specification tests for the control of thermal and fatigue cracking in modern pavements.

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## Abbreviations, Acronyms and Symbols

### Abbreviations and Acronyms

AASHTO	American Association of State and Highway Transportation Officials
AC	Asphalt Cement
AD	anno Domini
ASTM	American Society for Testing and Materials
BBR	Bending Beam Rheometer
BC	Before Christ
CTOD	Crack Tip Opening Displacement, m
DENT	Double-Edge-Notched Tension
DSR	Dynamic Shear Rheometer
eBBR	Extended Bending Beam Rheometer
EWf	Essential Work of Fracture
EVA	Ethylene Vinyl Acetate
FT-IR	Fourier Transform-Infrared Spectroscopy
HMA	Hot Mix Asphalt
LS	Laboratory Standard Test Method
LTPPBind®	Long Term Pavement Performance Binder Selection Software
m(t)	Slope of the Creep Stiffness Master Curve (m-value)
S(t)	Time-dependent flexural creep stiffness, MPa
KBr	Potassium bromide
MPa	Mega Pascal ( $10^6$ Pa)
MTO	Ministry of Transportation of Ontario
NCHRP	National Cooperative Highway Research Program
NSERC	Natural Sciences and Engineering Research Council of Canada
PCC	Portland Cement Concrete

PG (PGAC)	Performance Grade (Performance Graded Asphalt Cement)
PMA	Polymer Modified Asphalt
PPA	Polyphosphoric acid
SBR	Styrene-Butadiene-Rubber
SBS	Styrene-Butadiene-Styrene
SHRP	Strategic Highway Research Program
SIS	Styrene-Isoprene-Styrene
TFOT	Thin Film Oven Test
TTS	Time Temperature Superposition
TSRST	Thermal Stress Restrained Specimen Test
PAV	Pressure Aging Vessel
PI	Penetration Index
RTFO	Rolling Thin Film Oven
SUPERPAVE®	Superior PERforming Asphalt PAVement
WEO	Waste Engine Oil
XRF	X-Ray Fluorescence Spectroscopy
(ZDBC)	Zinc Dibutyldithiocarbamate
(ZDDTP)	Zinc Dialkyldiothiophosphate
3D	Three-Dimensional

### **Symbols**

A	Temperature susceptibility parameter (Penetration test)
a	Length of a sharp crack, m
b	Beam width, 12.5mm
B	Specimen Thickness, m
$G^*$	Complex shear modulus,
$G'$	Storage modulus

$G''$	Loss modulus
$h$	Beam thickness, 6.25mm
$K$	Stress intensity factor, $N.m^{-\frac{3}{2}}$
keV	kilo electron volt
$L$	Ligament length
$L$	Distance between beam supports, 102 mm (BBR)
$N$	Number of load repetitions
$P$	Penetration (Penetration test)
$P$	Load applied, N (BBR)
$t$	Loading time, s
$T$	Temperature, K
$W_e$	Essential fracture energy, J
$w_e$	Specific essential work of fracture, $J.m^{-2}$
$W_p$	Plastic or non essential work of fracture, J
$w_p$	Specific plastic work of fracture, $J.m^{-2}$
$W_t$	Total energy, J
$w_t$	Specific total work of fracture, $Jm^{-2}$
$\beta$	Plastic zone shape factor
$\gamma$	Phase angle
$\tau$	Shear stress
$\gamma$	Shear strain
$\delta_t$	CTOD
$\sigma_n$	Net section stress or yield stress ( $N/m^2$ )



# CHAPTER ONE

## INTRODUCTION

### 1.1 Overview

Low temperature cracking is one of the most significant modes of asphalt pavement distress among all the four types (thermal and fatigue cracking, rutting and stripping) that occur in asphalt mixtures placed in northern climates. This kind of cracking is very common in colder areas of Canada and the northern United States. Once a pavement is cracked due to excessive thermal stress, the intrusion of water and frost into the pavement layers can lead to a depression of the crack. The pumping action coming from traffic loading can result in a loss of supporting materials from the granular lower layers of the pavement. During cold temperatures in winter and spring, localised thawing of the base of the pavement and subsequent failure at the crack occurs due to de-icing solutions penetrating the cracks. Water penetrating the cracks could freeze, form ice lenses, and produce so called upward lipping at the crack edge [1]. The effect of this action results in poor ride quality and a reduction in pavement life, which requires regular and continuous maintenance and rehabilitation, which in turn drain the available economic resources from our nation.

A healthy nation requires a healthy economic stability which in turn requires a good transportation system, of which roadways are of paramount importance. Asphalt cements have been used to surface over 2.3 million miles of roads in North America [2]. In countries like Canada and northern parts of the United States, billions of dollars are spent on asphalt and road construction annually, of this huge sum, a small fraction is spent on the construction and expansion of road networks while the biggest portion is spent on rehabilitation, maintenance and

reconstruction of existing roadways. In Ontario, the annual expenditure on asphalt cement for provincial highway construction, resurfacing, rehabilitation and repairs is close to \$300 million [3]. Therefore, the construction and maintenance of asphalt-surfaced routes has a long term impact on the economic stability of every country or nation.

Harsh winter conditions experienced in Canada and the northern United States exert a heavy toll on highways and roads, which results in thermal stresses that trigger cracking in the asphalt. A number of recent reports have discussed that a large fraction of roads in Canada are in an undesirable condition. The reason for the poor condition of these pavements can be attributed to increases in overall traffic volume, poor asphalt binder quality obtained from high-tech refining processes, and a change in climatic conditions (extended freeze-thaw periods during spring due to global warming). It is therefore necessary to put good measurements in place to face these challenges.

Understanding the fundamental behaviour and properties of the roads prior to attempting to initiate or develop advance construction processes is very important. It is therefore necessary to choose the right kind of asphalt material which provides the desired or ultimate road performance with a long lifespan and reduce the maintenance rates of the roads in response to the unstable climatic conditions.

## **1.2 Asphalts**

Asphalt has different names, definitions, and descriptions, depending on its applications and the origin or location where it is found. In Europe, the term bitumen has been used to describe the dark-brown to black sticky material that binds aggregates together. In North America and in

other parts of the world, the term asphalt or asphalt binder or asphalt cement or simply binder has been used to describe the same material known as bitumen in Europe.

In a broader context, asphalt is defined by the American Society for Testing and Materials (ASTM) as a “dark brown to black cementitious material in which the predominant constituents are bitumen that occur in petroleum process” [4]. It is also defined by the American Association of State Highway Transportation Officials (AASHTO) as an “asphalt based cement that is produced from petroleum residues either with or without the addition of non-particulate organic modifiers” [5].

Asphalt has formerly been used in the construction of water tanks, which served as a sealant and glue for stone blocks, around 3000 B.C. at Mohenjo Daro in the Indus Valley [5]. It has also been used as a waterproofing material, as far back as 1595, where Sir Walter Raleigh noticed that the asphalt lake of Trinidad served as a perfect waterproofing material. One of the natural sources of asphalt has been known as the asphalt lake of Trinidad. The natural asphalt obtained from this source has limited applications in its raw or original state since it is hard in nature. However, upon refining and mixing with petroleum asphalt, it can readily be used in pavement construction and for waterproofing purposes [6]. Other sources of known natural asphalts can be obtained from rocks and from an asphalt material known as Gilsonite. The widespread use of naturally occurring asphalts dates back to around 400 B.C. Caulking for boats, cement for jewellery, sealing of bathing pool/water tanks, embalming of mummies, colouring material in paintings, lamp illumination, and device for war were some of the early applications of naturally occurring asphalts. Asphalt was first used as a road paving material in Babylon between 625–650 B.C. [6]. Modern application started in Scotland where builders began using hot coal tar to bond stones together on roadways reducing dust and maintenance in the 1860’s. In North America, the

first recognised paving project, which was completed in 1876, was constructed on Pennsylvania Avenue in Washington D.C. [7].

The synthetic production of asphalt from petroleum (crude oil) product started in the United States in 1902, which with time and an increase demand as a pavement material for road construction, turned into a large scale paving industry. Recently, the main source for the majority of asphalt used in paving is obtained from the bottom of the barrel, i.e. “the final product of the petroleum refinery process once all the other fractions such as gasoline, jet fuel, kerosene and other commodities are extracted from crude” [7]. In spite of the long term existence of asphalt in crude for over a million of years, it has been known that it does not last for very long when applied to roads. A number of factors account for the performance of asphalt, these include the source of the crude oil from which it is obtained and its composition cannot be exempted in addition to other factors such as climatic changes and the applied traffic volume on the asphalt pavement. Other factors that affect the performance of asphalt include the amount and type of aggregate that is used, the method of road construction and the moisture content [7].

Asphalt has a lot of applications which include pavement construction, waterproofing and many others. Because of its adhesive nature, asphalt was used in ancient times to glue stone axe heads to wooden handles [8]. However, about 85 percent of the asphalt obtained from petroleum refinery, which serves as the primary source of almost all asphalt today, is destined to serve as a paving material, commonly known as asphalt cement [9].

The characterisation of asphalt is not a trivial task because of its highly complex chemical and physical nature. It is made up of saturated and unsaturated aliphatic and aromatic hydrocarbon compounds with up to 150 carbon atoms. The composition of asphalt is highly dependent on the

source of the crude oil from which the asphalt is obtained. Many of the compounds contain oxygen, nitrogen, sulphur, and other heteroatoms. Asphalt typically consists of about 80% by weight of carbon, around 10% by weight of hydrogen, and trace amount of metals such as iron, nickel and vanadium, which serve as a fingerprint for the asphalt helping to identify the composition and the origin of the asphalt sample [6]. Asphalt molecular weights can range from several hundreds to many thousands. Depending on the solubility of asphalt in n-hexane or n-heptane, fractions may be classified as asphaltenes or maltenes. Asphaltenes are insoluble in n-hexane due to their high molecular weights. On the other hand, maltenes are soluble in n-hexane and have lower molecular weights. Asphalts usually contain 5 to 25% by weight of asphaltenes and may be considered as colloids of asphaltene micelles dispersed in maltenes [6].

Asphalts for road pavements are expected to be viscoelastic in nature in all weather conditions. But the opposite is usually envisaged in summer and in other high temperature regions, where the asphalt softens and suffers from permanent deformation (rutting). The natural molecules in asphalt are able to structurally arrange themselves in an organised manner during low temperatures. This is why the material hardens, becomes brittle, and cracks under the stress of heavy traffic loads. This is known as thermal or cold temperature cracking and fatigue cracking. Upon the evaporation of the volatile, lower molecular weight compounds from the asphalt, the material loses plasticity and hence it hardens, cracks and crumbles. This process is referred to as aging. Moisture from rain can also invade and damage asphalt, especially when aged or oxidized.

The performance of asphalt can be improved by using various modification techniques. Some of the techniques employ air blowing, which removes volatile compounds from the asphalt to make it more viscoelastic. Addition of polymers, adhesives, acids, bases, and filler are other ways in which the performance of asphalt can be improved to resist pavement distress in low temperature

conditions. However, while air blowing might improve the stiffness and rutting resistance, there are serious concerns that at the same time it deteriorates the fatigue and low temperature cracking resistance.

### **1.3 Asphalt Pavements**

Asphalt pavement concrete, also known as hot mix asphalt (HMA) pavement, refers to the bound layers of a flexible pavement structure. It is made of a mixture of coarse and fine aggregate and asphalt binders. It is called hot mix asphalt because it is mixed, placed and compacted at high temperatures. The typical thickness of an asphalt concrete pavement is about 4-8 inch (10-20 cm) but can reach as high as 20 inches in exceptional cases where high traffic is encountered. Road pavements can be categorised into two groups, namely, flexible pavements and rigid pavements. In rigid pavements, the surface of the pavement is covered with Portland Cement Concrete (PCC). Rigid pavements are stiffer than flexible pavements because of PCC's high stiffness, which is why such structures are referred to as "rigid" [2].

Flexible pavements achieved their name because the total pavement structure deflects, or flexes under loading. A flexible pavement is made up of three main layers or courses of materials, as shown in Figure 1.1, with each one performing a specific function to help the pavement to prevent or resist pavement distress. Loads on one layer are spread and passed on to the next available layer immediately below it. Material layers are often arranged in a descending load holding ability with the highest load bearing ability material at the uppermost part and the lowest load holding ability material at the bottom. The layers include surface course, base course and subbase course [10].

The surface course is the layer that is directly in contact with the applied load. It is made up of about 5 wt % of asphalt binder material (or about 20 vol % asphalt cement). It is useful in providing important properties such as smoothness, friction, noise control, rut and shoving resistance, and drainage. It also has the ability to prevent the entrance of excessive amount of surface water into the underlying base, subbase and subgrade. This course can be divided into two layers which are the wearing course and the intermediate course.

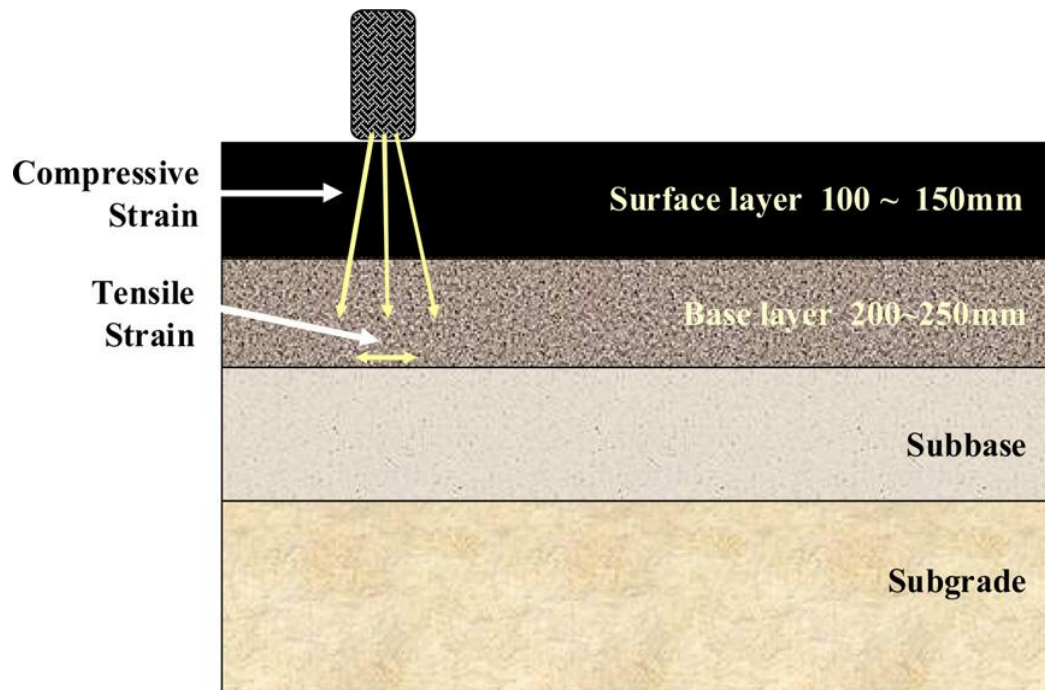


Figure 1.1: Structural layers in an asphalt concrete pavement [11].

The layer that is immediately under the surface layer is the base course. The main benefit of this course is to provide frost and drainage resistance and contributing extra load distribution. The base course is made up of aggregate and HMA. The aggregates can either be stabilized or

unstabilized and are usually obtained from durable aggregates that have the strength to resist moisture and ice damage.

The subbase course is the layer directly beneath the base course and on top of the subgrade. The primary purpose of the subbase is to serve as structural support. However, it performs other secondary functions such as improving drainage, providing a working platform for the construction, and minimizing the intrusion of fines from the subgrade into the pavement structure [10].

#### **1.4 Performance Grading of Asphalt Binders**

Asphalt binders are classified according to their performance and physical properties particularly for pavement purposes. The system used to classify the performance of asphalt binders could be simple or complicated. Among the oldest conventional specification tests used are penetration and viscosity tests. The penetration test measures the consistency or hardness of an asphalt binder at specified test conditions. The viscosity test is performed in order to determine the fundamental viscous (flow) properties of the asphalt binder. These test methods can be used to determine the binder's performance according to the test circumstances but, they cannot predict low temperature properties of the binder. The conventional test methods are considered empirical since the basic parameters and properties that correlate to the basic performance are difficult to be predicted by these test methods.

As a result of the inability of the conventional grading tests to provide reasonable agreement between binder properties and pavement performance, the evolution of a new specification grading system referred to as the Superpave<sup>TM</sup> (SUPERior PERforming Asphalt PAVEMENTS) has



been developed over the past 30 years [12,13]. This newer specification testing approach was developed under the U.S. Strategic Highway Research Program between the late 1980s and the early 1990s at a cost of nearly \$150 million. The purpose of this performance grading system and specification was to correlate binder properties to pavement performance, especially with regards to low temperature (thermal) cracking, rutting (permanent deformation), and fatigue cracking, which are the major causes of pavement deterioration. In this grading system, important factors like aging behaviour, stress (applied load), and climatic conditions, determine the performance of asphalt binders. That is, it appears to be a better tool over the conventional grading tests, because it is seen that binder properties and performance are being compared at the conditions under which it is being used.

Currently, new specification tests are developed which outperform the Superpave system. A number of research studies have revealed that binders of the same performance grade according to the Superpave specification, show tremendous differences regarding their performance at low temperatures. As a result of this failure, new specification tests have been developed which take into account performance grade such as fracture properties of the binder to distinguish fracture performance of asphalt binders using fundamental sound engineering principles and knowledge. This specification test aims at improving performance grade by resisting or reducing pavement distress at various climatic conditions especially at low temperatures [14, 15].

### **1.5 Pavement Deterioration**

The most desirable property for asphalt cement making it a good engineering material is its durability and resilience. Asphalt cement is recommended for many pavement applications because of its strength, and it is mostly preferred material for most states/provincial and federal

road projects. In spite of its high durability, it can be susceptible to deterioration due to the laws of nature. The longevity of asphalt pavements can be reduced due to poor surface preparation and construction techniques or by exposure to other environmental conditions for a long time.

Deterioration of constructed asphalt pavement is natural because the materials that constitute the asphalt pavement break down and become affected by elements such as rain, sunlight, oxygen and chemicals that come into contact with the pavement over a period of time. The binding, adhesion and waterproofing characteristics of the asphalt binder begin to loosen up in the pavement, allowing the penetration of water and frost to the base and subbase courses. As soon as this happens, the surface of the pavement becomes susceptible to a number of factors which further cause the deterioration of the pavement. Premature decline in quality of asphalt pavements can also be due to shortcomings in constructions or human error, and examples of these errors include [16]:

- insufficient or improperly compacted base below the asphalt;
- over or under compaction of asphalt;
- improper temperature of asphalt when applied; and
- poor drainage.

A properly maintained asphalt pavement wears out slowly and can last for about 25 years. Some of the key factors that cause pavement deterioration include [16]:

- Water: Water can penetrate the asphalt and wash out the base underneath it, and cause it to break down, crack and collapse over some period of time if the asphalt is not rightfully maintained.

- Sunlight: Continuous exposure of asphalt pavements to sunlight leads to ravelling and shrinking cracks which allows water to penetrate beneath the surface. This occurs because as the pavement is continuously exposed to sunlight, oxidation of the asphalt in the pavement occurs, which intend breaks down and dries out the flexible liquid asphalt that holds the aggregate together.
- Chemicals/Petroleum: Chemicals such as gas and oil which are introduced into the asphalt pavement or binder soften the asphalt and cause it to break down more rapidly.
- Cracking: There are many types of cracking that can lead to the decline in the quality of the asphalt pavements. Examples of these include alligatoring, edge cracks, slippage (caused by improper compaction), reflection (older cracks occurring in a new overlay), edge joint, shrinkage, widening, rutting, thermal cracking and fatigue cracking. Details of rutting, fatigue cracking, and thermal cracking are given in the next chapter.
- Distortion: Improper pavement construction leads to pavement distortion. This causes the deterioration of the underlying base or existing asphalt. Example of pavement distortion include channels or ruts, corrugations and shoving, grade depressions, upheaval and utility cut depressions.
- Disintegration: Examples of asphalt disintegration are potholes, ravelling, gas and oil spillage.

## 1.6 Scope and Objectives

Huge sums of money are spent annually in Canada and the United States for the reconstruction and rehabilitation of distressed asphalt-based road networks that have experienced low temperature and/or fatigue cracking. In order to account for this distress, the U.S. government funded the Strategic Highway Research Program (SHRP), which developed the Superpave specification grading system which is now embodied in the AASHTO M320 (2002) grading protocol [17]. The AASHTO M320 grading criteria accepted and used in North America does not give enough protection to asphalt pavements undergoing low temperature cracking in spite of the numerous studies that have been conducted on this form of distress. According to Hesp et al. [18] and references by these authors, pavements that are supposedly designed to withstand a certain climate at a 98% confidence level, usually experience premature and sometimes excessive cracking distress in Canada. Recent investigations have reported that the actual performance fails to meet the design expectation that, virtually no thermal cracking distress occurs for 50 years, when the current AASHTO M320 grading protocol is used. This contradiction calls for the need of a re-visitation of the AASHTO M320 protocol and making appropriate changes and corrections in order to improve the low temperature performance of the asphalt pavements.

In order to account for the shortcomings of SHRP, the Ministry of Transportation of Ontario (MTO) has embarked on an effort to develop better fatigue and low temperature specification tests and simultaneous specification tests of the asphalt cements [21], which are embodied in references [19 and 20]. One of the full-scale pavement trials that was constructed in support of the laboratory research was on Highway 417 in 2006 near Casselman, east of Ottawa [21]. The short term objective of this thesis is to validate the proposed extended bending beam rheometer

(eBBR) and double-edge-notched tension (DENT) tests and compare the laboratory and the field performance results to find the correlation between the two results and also to determine the effectiveness of the proposed specifications approach. If these two new tests are able to predict a good and effective correlation with field performance, it will cut down the high amount of money that is spent on the reconstruction and rehabilitation of the asphalt cement pavements annually that fail through thermal and fatigue cracking. This could be achieved by avoiding the use of poor quality binders in paving contracts and to design new pavements closer to the 98% confidence level as proposed by the SHRP researchers. This thesis is also aimed at evaluating the effects of low temperatures and repetitive stresses on the performance of asphalt cement pavements. The long term goal of this thesis is that these new tests methods are accepted by user and supplier agencies of modified and specialty asphalt binders in other Canadian provinces/territories and nationally.

## CHAPTER TWO

### BACKGROUND AND LITERATURE REVIEW

#### 2.1 General Knowledge of Asphalt Cement

##### 2.1.1 Chemistry of Asphalt

The chemical composition of asphalt determines its physical characteristics. Hence, fundamental knowledge of asphalt chemistry can be used to predict behaviour. Asphalt's molecular structure is extremely complex and varies in size and type of chemical bonding with each blend or source. Chemically, there are three classes of molecules that exist in asphalt structure and these are known as aliphatics or paraffinics, aromatics, and cyclics. There is interaction between these molecules in diverse ways which affect the chemical and physical characteristics of the asphalt. These molecules are chemically held together by relatively weak non-covalent bonds and are easily broken by heat or shear stress, which helps to explain the viscous nature of asphalt. When heat or constant stress or strain is applied to the asphalt, the weak intermolecular forces or bonding is destroyed, and the asphalt flows freely (viscous character), but when it cools or when the load is released, the weak covalent bonds reform and the chemical structure reverses though not necessarily to its initial shape or position (elastic character) [6].

A standard asphalt is made up of about 82-85 percent by weight of carbon, 8-11 percent by weight of hydrogen, 2-6 percent by weight of sulphur, 0-1.5 percent by weight of oxygen, and 0-1 percent by weight of nitrogen, as well as trace amounts of metals, such as iron, nickel, zinc and vanadium, which serve as fingerprints to determine the source of the asphalt. Asphalt can be divided into two chemical groups depending on its solubility and precipitation in non-polar solvents such as n-hexane. These groups are asphaltenes and maltenes [6].

Maltenes are non-polar in nature and are soluble in hexane, with low molecular weight. It is further divided into saturates, aromatics, and resins. Resins are dark brown in colour with strong adhesive properties which help asphalt to adhere to aggregates in a pavement mixture. The primary function of the resin is to disperse the asphaltenes in the oils (saturates and aromatics). It consists of carbon, hydrogen and small quantities of heteroatoms such as nitrogen, oxygen and sulphur, and the ratio of the carbon to hydrogen is 1:1. Aromatics constitute about 30 to 50 percent of the total weight of asphalt, and they are dark brown viscous liquid. They are made up of unsaturated aromatic ring systems, and they serve as a solvent for the resins and the asphaltenes. Saturates are made up of branched and straight chain aliphatic hydrocarbons together with some alkyl-aromatics. This contributes to about 5-20 percent of the asphalt's total weight. These are white in colour and appear as non-polar viscous oil.

Asphaltenes are black to dark-brown amorphous solids, insoluble in n-hexane due to their large molecular weights. They are made up of carbon, hydrogen and heteroatoms such as sulphur, oxygen and nitrogen, which help to explain their polar nature. The asphaltene content has a significant effect on the rheological properties of the asphalt: that is, decreasing the asphaltene content generates a softer, less softening point and consequently lower viscosity, of the asphalt and the opposite is obtained when the asphaltene content is increased. Their molar weights range from 1,000 to as high as 100,000 g/mol depending on the method used to determine it [25]. The ratio of carbon to hydrogen is approximately 1:1, and the size of a typical asphaltene molecule is usually between 5 and 30 nm [6].

Asphalt can be described as a colloidal dispersion of asphaltenes peptized by non-polar materials (resins) [5, 22]. In asphalt with highly aromatic maltene fractions, asphaltenes are well peptized (dispersed) and do not form extensive association. Such asphalts are referred to as sol-type

asphalts as shown in Figure 2.1(a). They exhibit high ductility, high temperature susceptibilities, low values of complex flow, low rates of age hardening, and little thixotropy [24]. On the other hand, in asphalts with insufficient aromatic maltene fractions, asphaltenes are not well dispersed and can agglomerate into connected structures, which form a continuous network throughout the asphalt in extreme cases. These kinds of asphalts are called gel-type asphalts, as shown in Fig. 2.1(b). They have low temperature susceptibilities, low ductility, significant thixotropic properties and elasticities, and high rates of age hardening [23, 24].

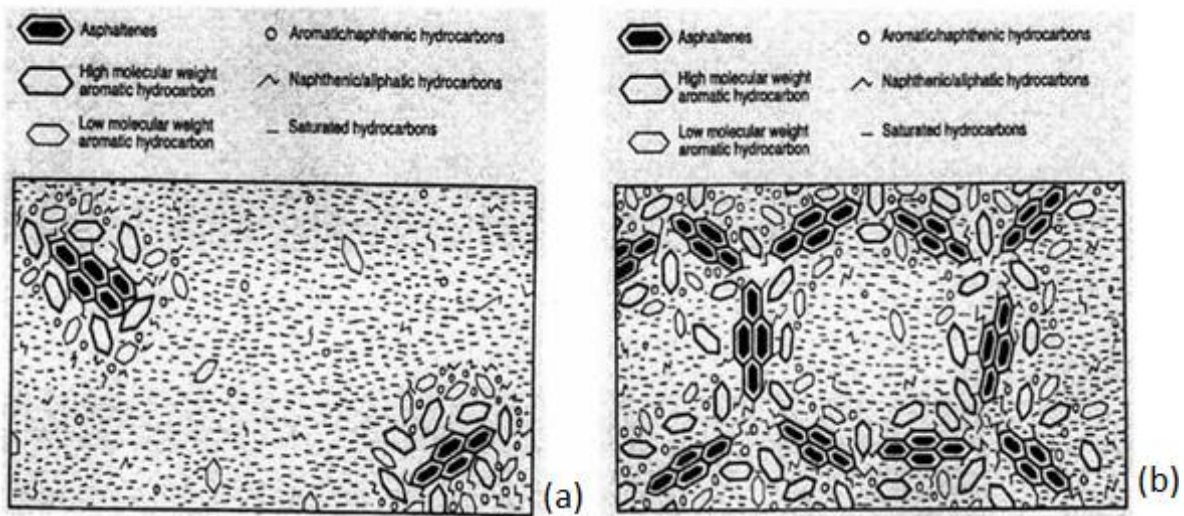


Figure 2.1: Schematic structures of (a) sol-type and (b) gel-type asphalts [6].

All the molecules contained into asphalt fall in one of two functional categories, namely polar and non-polar groups. The polar molecules form a network and give the asphalt its elastic properties, while the non-polar molecules contribute to the viscous properties of the asphalt by forming a body of materials around the network. Both the polar and the non polar molecules are uniformly distributed together in an asphalt mixture. It is expected that a good balance exists between both the polar and the non-polar molecules in order for the asphalt to show good



performance in service. At low temperatures, asphalt with large amounts of high molecular weights (non-polar) molecule become brittle and show bad performance in service [9].

Physical characteristics of asphalts and their test methods are presently used to predict adequately their performance, because there is not enough chemical knowledge to account for their properties since the fundamental asphalt binder failure cannot be chemically elucidated.

### **2.1.2 Sources of Asphalts**

There are two main sources from which asphalt can be obtained. These are natural sources and synthetic or petroleum sources.

#### *2.1.2.1 Natural sources*

Natural sources of asphalt are found within lime stones or limestone mixtures. It is estimated that about  $2.7 \times 10^{12}$  barrels of natural source of asphalt exist, which include tar sands [8]. About 95% of this naturally occurring asphalt is found as deposits of Athabasca tar sands in northern Alberta in Canada. Other major sources of naturally occurring asphalt include Pit Lake in Trinidad; Gard, Auvergne, Ain and Savoie in France; Latakia in Syria; Maestu in Spain; Butin Island in Indonesia; the Dead Sea in Israel and Jordan; as well as various locations in Central Iraq [27].

#### *2.1.2.2 Synthetic or Petroleum Sources of Asphalt.*

The refinery of petroleum products (crude oil) is the main medium through which petroleum asphalt is generated. Vacuum and atmospheric distillations are the basic units of operation. Blending, solvent deasphalting, emulsification, solvent modification, and air blowing are,

however, available to obtain different grades of asphalt depending on the source of the crude. The methods used to produce and process asphalt materials are shown in Table 2.1 [8]. Most asphalts used in North America today, are produced through the refinery of crude oil.

**Table 2.1: Methods and processes for the production of petroleum asphalt [8].**

<b>Production/Process</b>	<b>Base Material</b>	<b>Product</b>
Atmospheric and Vacuum Distillation	Asphalt-based Crude or Crude Mix	Asphalt Cement
Blending	Hard and Soft Asphalt Asphalt Cements and Petroleum Distillates	Asphalt Cements of Intermediate Consistency Cutback Asphalts
Air-Blowing	Asphalt Flux	Asphalt Cements, Roofing Asphalt, Pipe Coating, Special Membranes
Solvent Deasphalting	Vacuum Residuum	Hard Asphalt
Solvent Extraction	Vacuum Residuum	Asphalt Components (Asphaltenes, Resin, Oils)
Emulsification	Asphalt, Emulsifying Agent, and Water	Emulsified Asphalts
Modification	Asphalt and Modifiers (Polymers, Chemicals, etc.)	Modified Asphalts

### 2.1.3 Viscoelastic Nature of Asphalts

With the effect of climatic conditions, such as temperature and loading time, asphalt binders behave as viscous and/or elastic in nature. At very low temperatures asphalt binders behave like purely elastic materials; however, at intermediate temperatures, the behaviour tends to be more viscous in nature. Asphalt binders have high tendency to flow as freely as water at very high temperatures, indicating their viscous nature.

The response of a material to a load is defined by its stress-strain behaviour. When stress or strain is applied on a material, rearrangement occurs in the material as a response to that stimulus. Purely elastic (Hookean) solids or materials deform immediately to a constant strain upon application of a constant stress, and recover instantly and fully to their original shape and

position when the stress is released. The behaviour of a solid relating the applied stress to the resultant strain through a constant of proportionality, given as the modulus,  $G$ , is described by Hooke's law:

$$\tau = G\gamma \quad (1)$$

where  $\tau$  is the shear stress and  $\gamma$  is the shear strain. The stiffness of the material or its ability to resist deformation is dependent on its modulus. On the other hand, purely viscous materials (Newtonian fluids) deform continuously and permanently upon application of a constant stress, but fail to return to their original position when the stress is removed, making them non-recoverable. Newton's law gives the relationship between the applied stress and the shear rate through a proportionality constant,  $\eta$ :

$$\tau = \eta \frac{d\gamma}{dt} \quad (2)$$

where  $\eta$  is the coefficient of viscosity. A fluid is said to be Newtonian when the viscosity is independent of shear rate. A viscoelastic material shows both Newtonian and Hookean behaviours. In other words, they exhibit incomplete recovery properties but also depend on time. These materials show the related phenomenon of creep and stress relaxation as a result of their dual nature [27]. Asphalt binder has the tendency to behave as both a viscous fluid and an elastic solid at high and low temperatures, respectively, making it possess viscoelastic properties. The stress-strain behaviour of this material is dependent on time, as shown in Figure. 2.2.

The rheological properties of asphalt binders and mixtures, especially their viscoelastic behaviours have been characterised using the stress-strain behaviour of these materials in

response to load, and these properties depend on both time and temperature. The viscoelastic behaviour of asphalt is also seen under creep load, which leads to an increase in deformation.

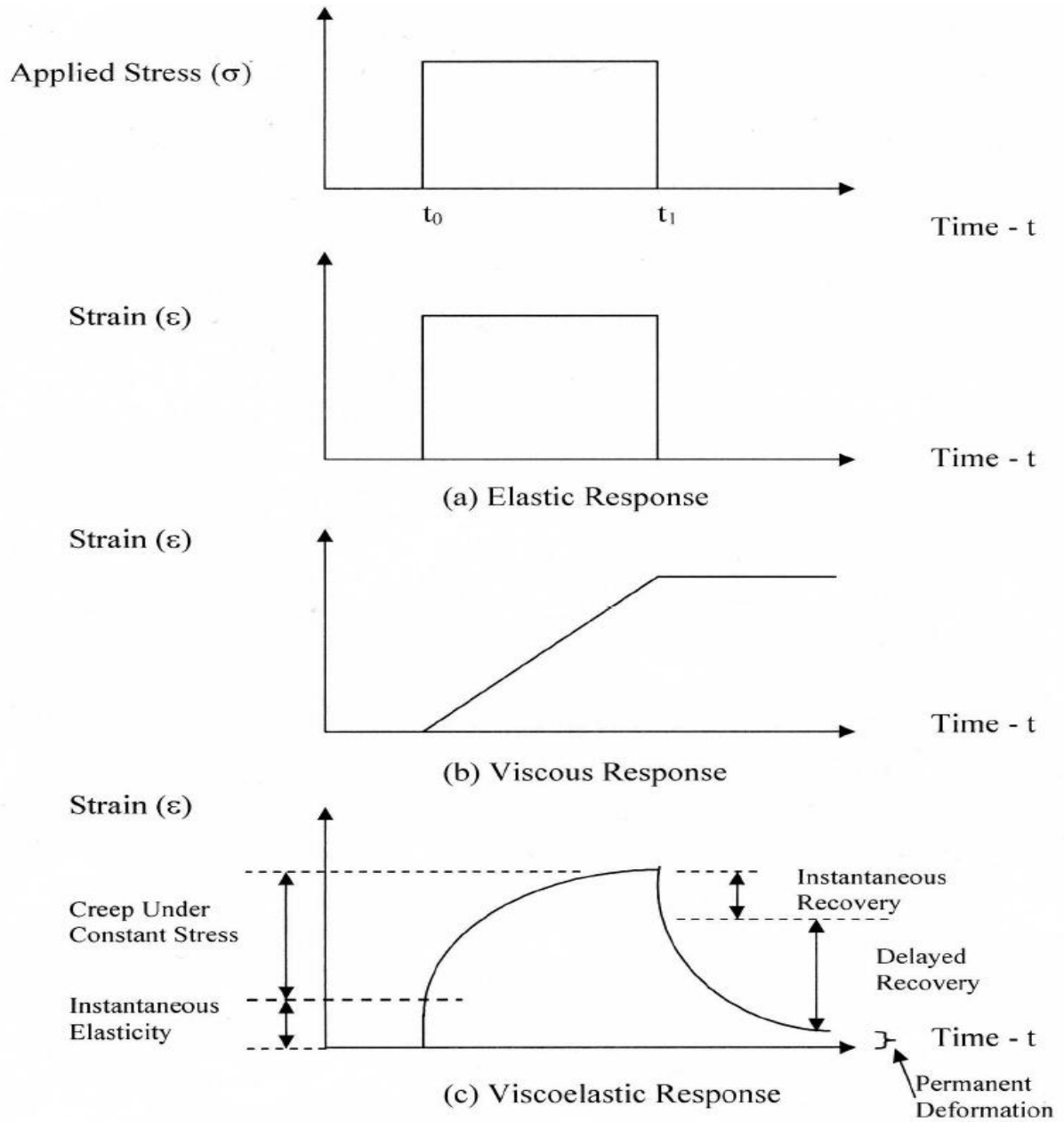


Figure 2.2: Idealized responses of elastic, viscous, and viscoelastic materials under constant stress loading [27].

#### **2.1.4 Applications of Asphalts**

Asphalt has a number of applications due to its adhesive, viscoelastic and viscous nature. It flows freely like ordinary water at high temperatures ( $> 60^{\circ}\text{C}$ ) and hardens at lower temperatures. The first use of asphalt as a road building material was reported in the 18<sup>th</sup> century in Newark, N.J. [28]. With the development of the automobile industry and the increased usage of asphalt during the 19<sup>th</sup> and 20<sup>th</sup> centuries, the use of asphalt as a road building material has progressed with time.

However, prior to the 18<sup>th</sup> century, asphalt had a remarkable history in its applications other than for road pavement. It was used to make small decorated utensils such as bowls, for coating statues, for making ornaments, and many others [29, 30]. It was used to glue various utensils together. In the Gilgal Site, a stone axe head was found which was covered with asphalt to glue it to the wooden handle [8]. With the increase in the development of synthetic glues, this use of asphalt has faded out drastically. Asphalt was used as a water proofing material around 3500 B.C., when it was used in ship building by the Sumerians and also used to waterproof temples and bands and water tanks by the ancient Phoenicians, Egyptians, and Romans. Asphalt was also used as a binding material (mortar) to join bricks and stones together. It also had some medicinal values in the ancient Middle East [31]. During the 2<sup>nd</sup> century A.D., famous doctors of medicine in the Roman and Medieval world used the Dead Sea asphalt for medicinal purposes. Recently, a valuable material called “bitupal,” which is effective for curing of certain skin diseases, was discovered by Professor A. Doctrovsky from Hasassah Medical School in Jerusalem [32]. This material has gained recent recognition and application and has been produced by the Teva Pharmaceutical Company in Israel [33]. Asphalt was also used to coat the roots of fruit trees and thus protect them against “worms and things that creep below the surface,” as described by the

Persian traveller, Nasir-I-Khusran, when he visited an area near Egypt in 1047 A.D. [34]. This beneficial effect could be made possible by the aromatic compounds and the sulphur content in the asphalt. Apart from the aforementioned applications in the ancient world, asphalt has also been used for photography [35], mummification, waterproofing, and in oil exploration [33].

### **2.1.5 Aging of Asphalt**

The aging of asphalt has received a pragmatic consideration both in the past and presently as a result of its effect on the performance of asphalt binders, especially at low temperatures. Time and temperature are the main factors that determine the properties of asphalt cement as far as aging is concerned, as exhibited by the time temperature superposition (TTS) principle [36, 37]. That is, the aging process at lower temperatures within a longer time frame happens to have the same or equivalent effect as that which occurs at higher temperatures within a short time interval. Two main kinds of asphalt aging are known, and these are physical and chemical aging.

Chemical aging occurs when there is an oxidation in asphalt cement during hot mixing, construction and in-service periods. The heteroatoms present in the asphalt, such as oxygen and sulphur, which are also found in the atmosphere, as well as changes in the temperature conditions, in addition to the presence of ultraviolet radiation on the surface of the asphalt pavement, play essential roles in the chemical aging process. Chemical aging is due to the oxidation by air (oxygen) in the asphalt binder. It leads to an alteration in the chemical and physical properties of the binder. It is known to cause increased stiffness of the asphalt binder; this makes it hard and very brittle which leads to premature cracking upon application of a constant stress/strain on it.

Physical aging, also known as reversible aging [38], occurs as a result of the aggregation/densification of the thermally unstable and low molecular weight structures within the asphalt binder. This occurs at low temperatures where the asphalt binder undergoes free-volume collapse, asphaltene aggregation, and wax crystallisation [39]. Many investigations have been conducted to evaluate the mechanisms by which physical aging takes place [39-42], but none of these mechanisms has successfully been able to predict the occurrence of the process. According to Struik [43], the reversible aging process can be accounted for by using free-volume collapse theory at low temperatures. Using Petersen's perspective [44], aging can be caused by (i) volatilization or adsorption of low molecular weight structures (saturates and aromatics), which give rise to decrease in maltene content in the asphalt binder; (ii) wax crystallization and rearrangement of asphaltene molecules; and (iii) oxidation of heteroatoms in asphalt cement. In a study carried out by Hesp et al. [39] to investigate reversible aging in asphalt at low temperatures, it was observed that "the loss in grade temperature peaks at some intermediate condition temperature, suggesting that the free-volume collapse – together with the reduction in mobility – is responsible for a large part of the aging." In other words, physical aging occurs as a result of free-volume collapse and reduction in the mobility of the asphalt binder. Asphalt binder's composition is very essential in oxidative hardening [15] and influences the interaction level between binders and the aggregates. This is because, when heated, volatilization occurs which leads to hardening properties of the binder. It is also noted that air blown asphalt binders are more susceptible to physical aging because of the oxidation which converts the resin contents to asphaltenes with significant changes in morphology, which leads to a more brittle structure and consequently premature cracking. Another cause of aging is the presence of air voids, which occurs as a result of improper compaction of the asphalt pavement. The presence of air void

allows the penetration of a high quantity of air and frost to the pavement, causing early failure of the pavement.

Aging process that occurs in the asphalt during the preparation of the hot mix asphalt, HMA (mixture of aggregate, sand, fillers, modifier and binder at high temperatures) is known as short term aging. This is due to rapid volatilization and oxidation of thermally unstable and volatile compounds in the asphalt binder. This kind of aging is simulated in the laboratory using the Rolling Thin Film Oven (RTFO) test method. On the other hand, the aging process that occurs when the HMA is exposed to different climatic conditions, especially at low temperatures and heavy traffic volumes between 5-10 years in-service, is referred to as long-term aging, and it is simulated in the laboratory using the Pressure Aging Vessel (PAV).

### **2.1.6 Modification of Asphalt Binders**

Asphalt properties can be improved by choosing the right or proper starting crude or by altering the refinery processes from which the asphalt is produced. However, there is not much crude that produces good asphalt, and “only a limited number of actions can be taken to control the refining process to make improved asphalt” [45]. As a result of these difficulties, many producers of asphalt binder have introduced additives to modify the properties. Some of these modifications include air blowing which makes the asphalt binder hard, fluxing agents or diluents oils which soften the asphalt, polyphosphoric acids (PPAs) which help to increase the temperature range in which satisfactory performances can be achieved, and other additives such as mineral acids, bases, and fillers. The addition of synthetic and natural polymers is the most frequently used method by which the quality of asphalt can be improved. Polymer addition helps to improve the asphalt by reducing viscosity at laying temperatures, increasing the stability at service



temperatures, the compatibility and the strength of the mixtures, improving fatigue resistance of the blends, improving the abrasion resistance of blends, improving oxidation and aging resistance, obtaining softer blends at low service temperatures and reducing cracking, reaching stiffer blends at high temperatures and reducing rutting, reducing structural thickness of pavements and reducing the life cycle costs of pavements [46, 47].

A number of natural and synthetic polymers are employed by various industries; however, it is important to consider certain factors when choosing polymer modifiers for a particular engineering purpose. Some of these factors are chemical compatibility with the base asphalt, mixing times and temperatures, rheological benefits, and workability of the final mix [48]. In places like airports, intersections of busy streets, vehicle weighing stations and parking lots with high stress, polymer modified asphalt (PMA) has been used with great success [49].

The use of PMA was patented as far back as 1843 [53], with much usage in Europe, compared to North America in the 1950s [52]. In places like North America, Europe and Australia, the use of PMA to optimize the performance of asphalt pavements has received pragmatic attention over the last three decades [49]. Kim et al. [51], at the Department of Transportation in Ohio, did a comparative study on three PG 70-22 binders to check their abilities to resist permanent deformation and fatigue cracking in 2001. These binders were 1) unmodified (virgin asphalt binder), ii) styrene-butadiene-rubber (SBR) modified, and iii) styrene-butadiene-styrene (SBS) modified. It was concluded that the virgin binder had less resistance to fatigue cracking and rutting when compared with the modified binders. In a report by the states department of transportation in the U.S. in 1997, it was observed that 94% out of the 50 states were using PMA and 70% showed great interest in increasing the usage of PMA in the near future [50].

Polymers used to modify asphalt binders can be divided into two general categories which are “passive” and “active” polymers [45]. Passive polymers are those in which the polymer is preformed, added to the asphalt and then mixed. This can be further divided into elastomers and plastomers. In elastomers, the molecules undergo physical cross-linking into a three-dimensional (3D) network. This helps them to achieve their strength and elasticity. This is derived by the agglomeration of the polystyrene end blocks into separate domains providing the physical cross-links for a 3D polybutadiene or polyisoprene rubbery matrix. SBS and styrene-isoprene-styrene (SIS) are typical examples of this group [6, 45]. On the other hand, the main effect of plastomers is to increase the viscosity of the binder. Examples of this group include polybutadiene, natural rubber, SBR, polyisoprene and many others. Active polymers undergo a specific chemical reaction with specific functionalities in asphalt to impart long-term phase stability. Unlike passive polymers, they do not form crosslinks or network structures with the asphalt structure itself, but form polymer networks throughout the asphalt matrix. Their function is to alter the penetration, viscosity, and softening point of the asphalt binder, but do not have any effect on its elasticity. Examples of this class include ethylene vinyl acetate (EVA), polyvinyl chloride, polystyrene, and polyethylene [45]. It is expected that the polymer content should be kept at 5% or less in order to obtain good properties of the asphalt binder at an affordable cost. In order to improve the stability, compatibility and the strength of the PMA blends, the addition of sulphur to the blend has become common in many industries. “Polymer-asphalt blends prepared with the sulphur coagglomerated polymers show improved storage stability. Also, when using the sulphur slurry, polymer use is optimized because the polymer matrix development can be completed before cross-linking occurs” [45].

## **2.2 Mechanisms of Asphalt Pavement Distresses**

In spite of the incredible values obtained from asphalt pavements, such as being quick and economical to construct, excellent ride quality, and long lasting characteristics, they can easily undergo distresses, especially when poor materials are used or poor construction techniques are adopted. It is appropriate to understand and know the type and severity of the distress in the pavement and also be able to identify the cause of it, in order to apply appropriate strategic repair methods to the distressed pavement [53]. Pavement distress begins to accumulate as pavements experience traffic repetitions and undergo aging processes. The common types of pavement distresses are rutting (also known as permanent deformation), moisture damage, fatigue cracking and low-temperature cracking.

### **2.2.1 Rutting**

Rutting is a type of pavement distress process that takes place at elevated service temperatures. It occurs as a result of the accumulation of small amounts of unrecoverable strains coming from repeated loads to the pavement. It generally occurs at the upper surface (top 100 mm) of the pavement, but it can extend to the lower layers of the pavement if desirable materials are not used as shown in Figure 2.3(a). By increasing the numbers of the load application, rutting in pavement can develop gradually, appearing as longitudinal depressions in the wheel paths sometimes accompanied by small upheavals to the sides. It is caused by densification (a decrease in volume and hence an increase in density) and shear deformation [54]. At high temperatures, the binder's viscosity is decreased and flow can easily occur under traffic loading, which leads to the formation of tracks (ruts) in the surface layer(s) of the pavement. Rutting is evaluated in the

laboratory by using the dynamic shear rheometer (DSR), where the complex shear modulus,  $G^*$ , and the phase angle,  $\delta$ , are used to predict the rutting resistance factor given as  $G^*/\sin\delta$ .

### **2.2.2 Moisture Damage**

Premature degradation in pavements may occur as a result of the stripping of the asphalt from the aggregate particles when environmental conditions such as temperature and moisture are coupled with poor construction materials and traffic loads. Stripping occurs when the aggregate tends to have a high affinity for absorbing water, which leads to the “stripping” away of the asphalt binder from the aggregate. It can bring about a loss in the quality of the mixture and, consequently, leads to a failure of the pavement as a result of ravelling, rutting, or cracking. According to Brown et al. [54], the integrity of a HMA asphalt matrix is reduced by moisture through the following mechanisms:

1. decrease or loss of the strength of the asphalt binder as a result of other mechanisms;
2. lack of bonding (adhesion) between the aggregates and the asphalt binder; and
3. freezing of the individual aggregate particles leading to failure of the pavement.

Stripping problems can be addressed by the use of appropriate anti-stripping agents such as hydrated lime. Figure 2.3(b) is a photograph of a pavement that has undergone extensive moisture damage.



Figure 2.3: Photographs of pavements that have undergone (a) permanent deformation (rutting) and (b) moisture damage [8].

### 2.2.3 Fatigue Cracking

Fatigue cracking is a pavement failure process that has received much attention from different research groups all over the world for many years. It has been defined as “the phenomenon of fracture under repeated or fluctuating stress, having a maximum value less than the tensile strength of the material” [27]. Because of its closely spaced crack order, it is often called alligator cracking because it resembles the pattern on an alligator’s back as shown in Figure 2.4(a). It usually occurs when loads which are too heavy for the pavement or more repetitions of a given load is applied to the pavement, which induces stress that is more than the fatigue limit life of the pavement, eventually causing the pavement to fracture. Factors such as inadequate pavement drainage worsen the fatigue process by causing the underlying layer to become saturated and lose strength. Fatigue cracking occurs prematurely when the HMA layers encounter high strain and when the underlying layers are weakened by excessive moisture.

Inadequate pavement thickness and/or repetitive passes with overweight trucks can also cause fatigue cracking due to poor quality control during construction [54]. In thinner pavements, fatigue cracking initiates from the bottom and migrates towards the surface because of the high tensile strain at the bottom of the HMA. On the other hand, in thicker pavements, fatigue cracking occurs downward, i.e., from the surface to the bottom, which is due to tensile strains in the surface of the HMA.

The mechanism of fatigue cracking in asphalt concrete is divided in three stages [27]: (1) crack initiation, which brings about the initiation and development of microcracks; (2) crack propagation, which involves the development and extension of the microcracks to form macrocracks as a results of stable crack growth; and (3) disintegration, which brings about the collapse and final failure of the material as a result of unstable crack growth. The fatigue life is usually consumed mostly by the propagation phase. Paris' law can be used to describe this phase, which relates the rate of crack growth to the fundamental properties of the material and experimental conditions and it is written as [55]:

$$\frac{dc}{dN} = A(\Delta K)^n \quad (3)$$

where,  $C$  is the crack length,  $N$  is the number of the load repetitions,  $\Delta K$  is the difference between maximum and minimum stress intensity factor  $K$ , in dynamic loading, and  $A$  and  $n$  are fracture parameters for the asphalt concrete. The stress intensity factor,  $K$ , is used to evaluate accurately the stress state or stress intensity near the crack tip caused by a remote load or residual stresses in fracture mechanics. The concept of fracture mechanics, which is importantly one of the major methods used to study and investigate asphalt fatigue cracking through the use of small specimen (beam bending or cylindrical specimen) to determine crack growth parameters, has

been used to study fatigue properties of the asphalt mixtures by many research groups [56]. The stresses in the immediate vicinity of the crack tip are described by the stress intensity factor.

Fatigue cracking is usually caused by a number of pavement factors that have to occur simultaneously, hence it is considered to be more of a structural problem than just a material problem. Some of the obvious factors responsible for fatigue cracking are repeated heavy loads and poor subgrade drainage which lead to a soft, high deflection pavement. Other factors include improperly designed and/or poorly constructed pavement layers that are prone to high deflection when loaded. One of the immediate and long term effects of fatigue cracking is the development of potholes when the individual pieces of HMA separate physically from the adjacent material and are dislodged from the pavement surface by the action of traffic loads. Fatigue cracking in asphalt concrete pavements can be evaluated in the laboratory using the dynamic shear rheometer (DSR), where the complex shear modulus,  $G^*$ , and the phase angle,  $\delta$ , are used to account for the fatigue cracking resistance factor of the pavement given as  $G^* \sin\delta$ .

#### **2.2.4 Low Temperature Cracking**

Low temperature or thermal cracking is known to be one of the major causes of asphalt pavement distress in Canada and the northern United States as well as other colder regions of the world. When temperature decreases to some critically low temperature, tensile strain induces in HMA, which leads to low temperature cracking of the pavement. Asphalt pavement cools upon exposure to cold temperature conditions and contracts. However, tensile stress builds up due to the resistance of the contraction of the layers as a result of the friction that exists in between the base and the pavement. When the tensile stress induced in the pavement and the tensile strength of the pavement are the same, development of microcracks occur at the edge or surface of the

pavement [27, 57]. At colder temperature or through repeated temperature cycling and loading, this crack may penetrate the full depth and width of the asphalt mix layer. This kind of pavement distress gives rise to sporadic transverse cracking (i.e., perpendicular to the direction of traffic) which occur at a consistent and regularly space intervals as shown in Figure 2.4 (b). They are spaced so regularly since the stress builds up due to the friction with the subgrade. Once the tensile stress surpasses the strength, the pavement will crack. Factors such as material, geochemical and environmental conditions have a great impact on the ability of a particular pavement to resist low temperature cracking. Of all these factors, the stiffness or consistency of the asphalt cement at cold temperatures and the temperature susceptibility of this pavement are those of much concern to pavement contractors. The stiffness of a less viscous asphalt increases at a lower rate as the temperature is reduced and this makes the material less susceptible to low temperature cracking. Penetration of water into the pavement occurs easily in this crack and causes upward lipping at the crack edge upon freezing. Air voids are also created when fine materials move through the cracks under the pavement, leading to a fracture in the vicinity of the crack upon loading. Properties of the aggregate such as low freeze-thaw loss, low absorption and high abrasion resistance also give generally an asphalt mixture with optimum resistance to low temperature cracking. Another factor that has significant influence on the low temperature cracking is the asphalt binder content. For instance, the coefficient of thermal contraction increases but stiffness decreases when the asphalt cement content is increased [27]. Fatigue phenomenon, due to the accumulative effects of cycles of cold weather can also lead to low temperature cracking. The occurrence and intensity of low temperature cracking is determined by the magnitude and frequency of low temperatures and the stiffness of asphalt mixture which is directly related to the properties of the asphalt binder [54]. Cold temperatures and faster



cooling rates also lead to greater susceptibility of the asphalt cement to thermal cracking. It has also been realised that physical and chemical aging processes are also contributing factors to low temperature cracking. Pavement design structure has significant influence on the pavement susceptibility to low temperature cracking, in that the pavement thickness, width, and subgrade types are all essential factors that help to achieve maximum resistance to low temperature cracking [27].

The rheological properties of the asphalt binders with respect to low temperature cracking are determined by both tensile strength and fracture properties of the binders [56, 58]. To understand the fracture mechanisms of asphalt pavement at low temperatures which is used to investigate the fracture properties of the asphalt mixtures, the concept of the fracture mechanics has been introduced in the past two decades [56]. The concept of the fracture mechanics is adopted over other models such as dissipated energy concept, because the properties of the material as well as the mode of loading are incorporated into the model. In addition, the influences of various material properties can be evaluated explicitly with performing fatigue test. Other methods that have been used to study the low temperature cracking in asphalt mixtures are indirect tension test, direct tension test, the direct creep tests, the flexural bending test, the thermal stress restrained specimen test (TSRST), the coefficient of thermal expansion and contraction test, and single and double edge notched tension tests. A number of research studies have shown that the TSRST test seems to be more favourable over the others when evaluating low temperature cracking susceptibility of asphalt mixture, because it stimulates field conditions, and the responses are applicable to mechanistic models [56]. However, low temperature performance is a combination of both rheological as well as fracture properties of the binder in service

temperatures. As a result of this, a comprehensive model of low temperature pavement performance must include rheological and fracture properties of the asphalt binder.

This thesis is aimed at addressing this problem by using the dynamic shear rheometer (DSR), the bending beam rheometer (BBR), and the double edge notched tension (DENT) test to account for the low temperature cracking performance and fatigue cracking properties of asphalt binders recovered from asphalt test sections on Highway 417 near Casselman, east of Ottawa, Ontario.

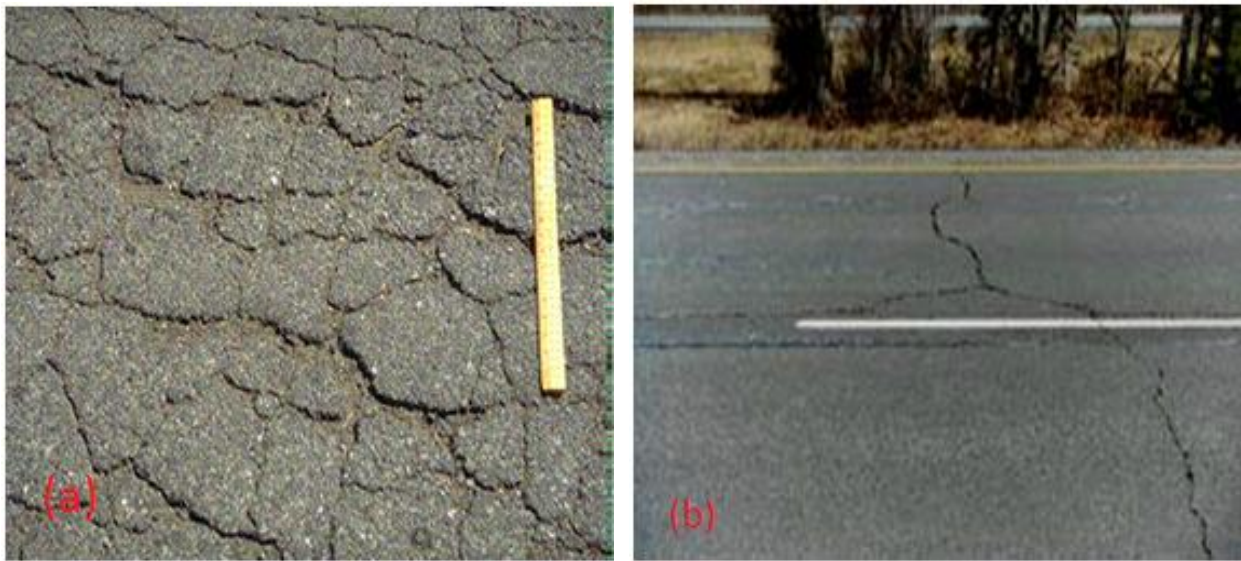


Figure 2.4: (a) Alligator-type fatigue cracks typically due to load (traffic) distress and (b) Longitudinal and transverse cracks typically due to thermal effects [56].

### 2.3 Conventional Test Methods

Conventional test methods such as penetration, softening point and viscosity were used to test asphalt binders during the early 1900s. Three sets of asphalt grading systems, which are penetration (PEN), viscosity (AC), and aged residue (AR) were used, based upon these methods.

These tests are considered to be empirical because they are not performance related. At higher or lower service temperatures, they can be deceptive to pavement performance, and also, they do not include fundamental engineering properties that can be related to the pavement performance [14]. There are four conventional grades that are used to evaluate asphalt binder properties. These are the penetration grade, the cutback grade, the oxidized grade, and the hard grade. The penetration grade is the most important one among these grades with respect to road pavement construction. These grades are determined by the combination of any two test methods or just one. The oxidized and the hard grades are determined by the combination of the viscosity and the softening point methods, whereas the penetration grade is determined by the softening point and the penetration test methods. The cutback grade is predicted by the viscosity test. The oxidized grade asphalt is used for roofing and painting, while the cutback grade asphalt is used for blending and surface coating [6].

### **2.3.1 Penetration Test**

The penetration test is the oldest test method developed in 1888 by H.C. Bowen of the Barber Asphalt Paving Company [59]. The objective of this test is to measure the consistency of an asphalt binder at a given standard conditions of temperature, time, and load with values of 25°C, 5 sec and 100 g, respectively. It is a means of classifying the asphalt rather than measuring its qualities. The unit of this test method is decimillimeters, i.e. 0.1 mm. For instance, 80 pen asphalt cement has a penetration of the needle at 25°C of 8 mm, meaning, 80 times 0.1 mm. Penetration is related to viscosity and empirical relationships have been developed for Newtonian materials. If penetration is measured over a range of temperatures, the temperature susceptibility of the asphalt binder can be established. The consistency of the binder is related to the temperature changes by the equation [60]:

$$\log P = AT + K \quad (4)$$

where P is the penetration at temperature T, A is the temperature susceptibility, and K is a constant. The value of A which is defined as the Penetration Index (PI) can be obtained from penetration measurements at two different temperatures,  $T_1$  and  $T_2$  using the equation [60]:

$$A = PI = \frac{\log(Pen @ T_1) - \log(Pen @ T_2)}{T_1 - T_2} \quad (5)$$

It has been shown that for conventional paving binder, the softening point temperature is the same as that which would give a penetration of 800 dmm. PI values can be used to evaluate stiffness of the binder at any temperature. However, PI uses the change in binder properties over a relatively small range of temperatures to characterise asphalt binders [61]. Fig. 3.5(a) is a photograph of penetration test equipment.

### **2.3.2 Ring and Ball Softening Point Test**

The ring and ball softening point is used to determine the softening point of the asphalt binder within the temperature range of 30-157 °C using the ring and ball apparatus immersed in distilled water or ethylene glycol as shown in Figure 2.5(b). This method is useful in classifying asphalt cements, as one element in establishing the uniformity of the sources of supply and also used to indicate the tendency of the material to flow at elevated temperature encountered in service. The temperature at which a steel ball in the ring pushes the soft asphalt binder to a distance is considered as the softening point of the material. In other words, the temperature at which asphalt binder can no longer support the weight of a 3.5 g steel ball is defined as the softening point of the asphalt binder. It is used to measure the consistency of the asphalt binder [61].

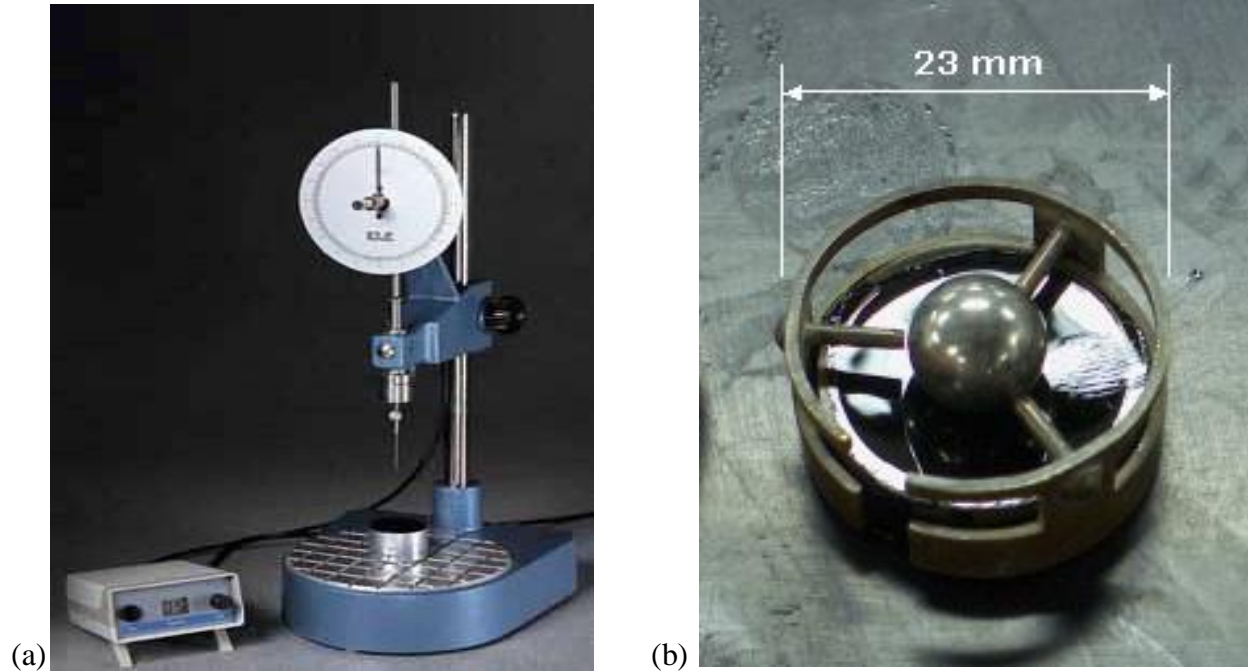


Figure 2.5: Equipment for testing (a) Penetration and (b) Softening Point of asphalt binders [60]

### 2.3.3 Viscosity Test

Viscosity is defined as the ratio between the applied shear stress and induced shear rate of a fluid. The viscosity test was developed in the early 1960s to improve upon the empirical penetration test. It has the advantage of measuring a fundamental physical property of the asphalt binder compared to the penetration test. The test can be performed on both unaged (AC grading) and RTFO aged (AR grading) asphalt binders. The AC grading test is used to characterise the properties of the binder before it undergoes HMA manufacturing processes. The AR grading test is used to stimulate the asphalt binder properties after it has undergone HMA manufacturing processes, hence it should be more representative of how an asphalt binder behaves in HMA pavements [59].

## 2.4 Superpave Test Methods

As a result of the deficiencies witnessed with the use of the conventional grading test methods due to their inability to predict a good correlation with the performance of the asphalt pavement in-service, the strategic Highway Research Program (SHRP) was initiated in the late 1980s and the early 1990s in the United States. This was developed as a coordinating effort to initiate rational specifications for asphalt binder based on performance parameters. The concept behind the development of the SHRP was to produce pavements which perform well in service. This led to the development of the Superpave (SUPERior PERforming PAVements) asphalt binder specification, which helped to group grades of asphalt binders according to their characteristic performance in different environmental conditions. The Superpave specification test aimed at limiting or reducing the potential of asphalt binder to fatigue cracking, thermal cracking and permanent deformation. This specification has an advantage of being able to measure the physical characteristics that can be related directly to field performances using sound engineering principles, and tests are carried out at temperatures that are encountered by the pavement in service. In the Superpave specification tests, the physical characteristics of the binder remains unchanged for all grades, however, the temperatures at which these properties are obtained varies depending on the climatic condition within which the asphalt binder would be used.

According to the Superpave specification test, the asphalt is assigned a grade such as Performance Grade (PG) or Performance Grade Asphalt Cement (PGAC) XX-YY, in which the XX stands for the high temperature working limit of the asphalt binder and -YY represents the low temperature limit. For example, a PG 64-34 grade is designed to be used in an environment where the average seven day maximum pavement temperature is 64°C and the minimum pavement design temperature is -34°C. It is expected that at this grade, the asphalt binder will be

able to resist permanent deformation at a maximum average-seven-day temperature of 64°C, and low temperature cracking at a lowest minimum temperature of -34°C. The next sections describe the test methods involved in the Superpave specification in some more detail.

#### 2.4.1 Rolling Thin Film Oven (RTFO) Test

The RTFO test is used to stimulate the aging that takes place in the HMA during mixing and placement of the asphalt cement concrete. It is also used to evaluate the amounts of volatile lost from the asphalt. This test was designed as an improvement over the Thin Film Oven Test (TFOT). The test is performed by heating a moving thin film of asphalt binder in an oven for 85 minutes. The moving film is created by placing the asphalt binder sample in small jars or tubes and then placing them in a circular metal carriage that rotates within the oven as shown in Figure 2.6. The alteration that occurs in the physical properties of the asphalt binder is measured before and after the oven treatment by other test procedures. The RTFO has gained recognition over the TFOT in that [62]: (i) its rolling action keeps modifiers dispersed in the asphalt; (ii) it takes a shorter time (85 minutes instead of 5 hours in the TFOT) to perform a complete test; and (iii) the test is repeatable and continually exposes fresh binder to heat and air flow.

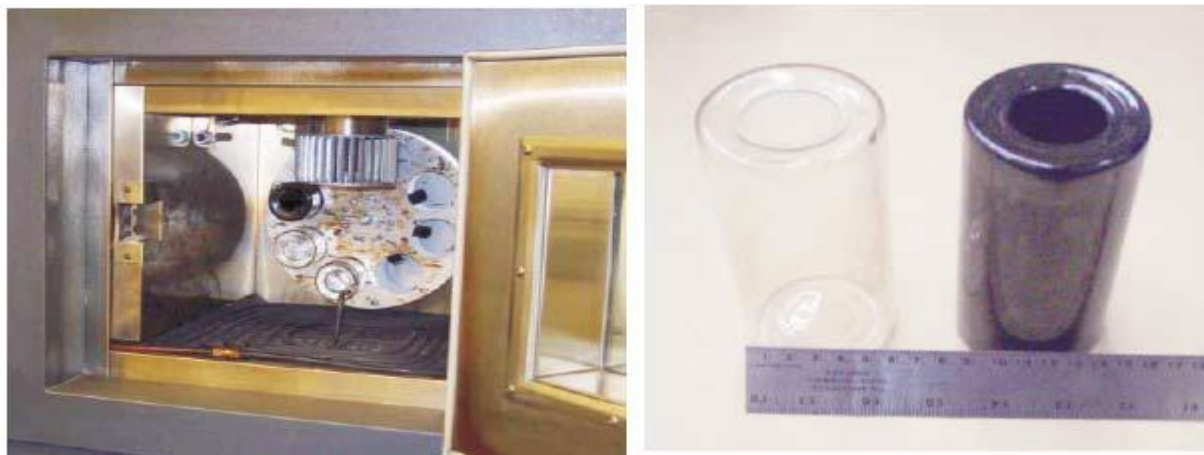


Figure 2.6: RTFO test machine for short term aging testing [56].

### 2.4.2 Pressure Aging Vessel (PAV) Test

The test is used to stimulate the long-term aging of asphalt binder that occurs after 6-10 years in service. The testing process is carried out by placing an RTOFT aged asphalt binder in the PAV vessels and aging it for 20 hours as shown in Figure 2.7. In order to stimulate the long term aging process, the device uses temperature and pressure to compress time. Depending on the temperatures changes of the environment within which the asphalt binder would be used, the test can be performed at different temperatures ranging from 95°C to 110°C [64]. PAV aged asphalt binder is used for bending beam rheometer (BBR), double edge notched tension (DENT), dynamic shear rheometer (DSR), direct tension (DT), and infrared (IR) spectroscopy tests.



Figure 2.3: Pressure aging vessel for long term aging testing [59].

### 2.4.3 Dynamic Shear Rheometer (DSR) Test

The rheological properties of an asphalt binder at intermediate to high temperatures are determined by the DSR. This is because the characteristics of the binder depend on both time and temperature and the DSR is able to predict both factors simultaneously. This is achieved by measuring the complex shear modulus,  $G^*$  (which is a measure of the total resistance of a



material to deformation when repeatedly sheared) and the phase angle  $\delta$ , (which is an indicator of the relative amount of recoverable and non recoverable deformation) [62]. It is very necessary to know the rheological properties of an asphalt binder in order to predict its end-use performance. The test is performed by placing a thin film of a disc-like shaped asphalt binder in between two parallel plates, which then undergoes a series of dynamic sinusoidal oscillations in waveform to measure the viscoelastic properties of the binder. The diameter of the asphalt binder disc depends on the temperature used for the measurement. Diameters of 8 mm and 25 mm are preferred for low and high temperature respectively, with respective gaps of 1 mm and 2 mm between the two parallel plates. The dynamic properties of the asphalt are measured when the binder sample is exposed to shear, and shear angles and torsion/torque are measured over a wide range of frequencies and temperatures.

The shear modulus is evaluated when the sample is placed in the parallel plates and subjected to a shear force in the form of torsion at a frequency and temperature of 10 rad/s and 25°C respectively. Master curves are usually generated in order to correlate with theoretical models at three different temperatures which are used for testing.

The complex shear modulus is related to the applied stress and the resulting strain by the equation [5]:

$$G^* = \frac{\tau_{\max} - \tau_{\min}}{\gamma_{\max} - \gamma_{\min}} \quad (6)$$

where  $(\tau_{\max} - \tau_{\min})$  is the total shear stress and the  $(\gamma_{\max} - \gamma_{\min})$  is the total shear strain, and it characterises the elastic component of the asphalt binder. The phase angle is used to predict the viscous component of the binder, and it is related to the time lag between the applied stress and the resulting strain. It is mathematically represented as the ratio of the viscous modulus,  $G''$ , to

the storage or elastic modulus,  $G'$ . It relates to rutting and thermal cracking at high and low temperatures respectively. Figure 2.8 is a photograph of DSR equipment.



Figure 2.8: Photograph of a dynamic shear rheometer [59].

#### 2.4.4 Bending Beam Rheometer (BBR) Test

It has been investigated in early research work that the stiffness and modulus of asphalt binder can be used as a yardstick to determine the rheological and fracture properties of both modified and unmodified asphalt binders and mixtures. Prior to the establishment of the Strategic Highway Research program (SHRP) in early 1990s [12, 63] to improve the methods used to grade asphalt binders, the concept of stiffness as a parameter for grading the performance of asphalt binder was apparently satisfactory initially, until different modification methods were employed to produce modified asphalt binders. This led to the development of the BBR, which is aimed at determining the stiffness of asphalt binders at low temperatures, which is now embodied in AASHTO M320 standard protocol (2002). This provides the specification criteria for passing/failing the BBR test [64]. The knowledge behind the AASHTO M320 specification lies on the research conducted by the Shell Laboratories in Amsterdam during the 1950s and

1960s, which led to the conclusion that a reasonable correlation exists between the binder stiffness at a constant loading time and a number of failure properties in the binder and mixture [18, 21, 56]. This hypothesis was further tested by Heukelom [72], who tested a wide range of unmodified binders and found a high correlation between binder stiffness and actual failure properties. It was later realised that Heukelom's correlation was only valid for unmodified binders whereas most of the binders used up to date have some modifiers added to improve their performances, which makes this correlation inaccurate.

The concept of standard beam properties and deflection are used to evaluate the ability of the asphalt binder to relax from the load induce stresses as well as the calculation of the stiffness upon application of load to the center of an asphalt beam. The BBR measures two important parameters. It measures the slope of the creep stiffness master curve of the binder, called the  $m$ -value and the creep stiffness  $S(t)$  at 60 seconds. Both the  $m$ -value and  $S(t)$  are used to relate to the low temperature cracking performance of the asphalt pavement. In this test, both the testing and the conditioning temperature are the same. Prior to testing at the desired temperature, the beam is first conditioned at room temperature for one hour and then conditioned at  $-10$  and  $-20^{\circ}\text{C}$  in separate cooling baths or refrigerators for another one hour before testing. The test is done by loading the specimen (the beam) in three-point bending at a temperature of  $10^{\circ}\text{C}$  above the designed pavement temperature. The increase in the temperature by  $+10^{\circ}\text{C}$  is to reduce the test time from two hours to 60 seconds, based on the assumption that the time-temperature superposition principle is valid [12, 36, 37]. In order to determine the creep stiffness master curve, the stiffness of the beam is measured at loading times of 8, 15, 30, 60, 120 and 240 seconds. The slope of the master curve is the  $m$ -value which indicates the binder's ability to relax stress due to viscous flow [64]. The sample passes the specification when the creep rate or

the slope of the creep stiffness master curve is equal to or greater than 0.3, and a maximum value of 300 MPa assigned to the creep stiffness. When the stiffness is greater than 300 MPa and/or the m-value is less than 0.3, then the material fails the specification and it can only be used in locations with warmer climatic conditions.

Recent investigations on a large number of pavement trials and regular contracts have shown that the current AASHTO M320 specification is inappropriate to predict the pavement performances in service [64-69]. It has been found out that those pavements with the same low temperature grades show different low temperature performance in service [67, 70, 71]. The inability of AASHTO M320 to predict pavement performance can be attributed to inadequate physical and chemical aging of the asphalt binder beams before testing and the lack of true failure test, since the BBR and DSR are rheological tests that only measure in the low strain regime while low temperature cracking is a high strain phenomenon [70, 71]. There is therefore the need to revisit the current AASHTO M320 specification in order to develop improved performance grading test method(s). This has led to the development of the extended BBR (LS-308) and the double edge-notched tension (DENT) (LS-299) tests at Queen's University which are now embodied in the Ministry of Transportation of Ontario (MTO) asphalt cement grading specification protocols [19, 20]. This thesis is aimed at investigating the validity of these new methods by comparing the laboratory results with the field distress results using pavement trials on Highway 417.

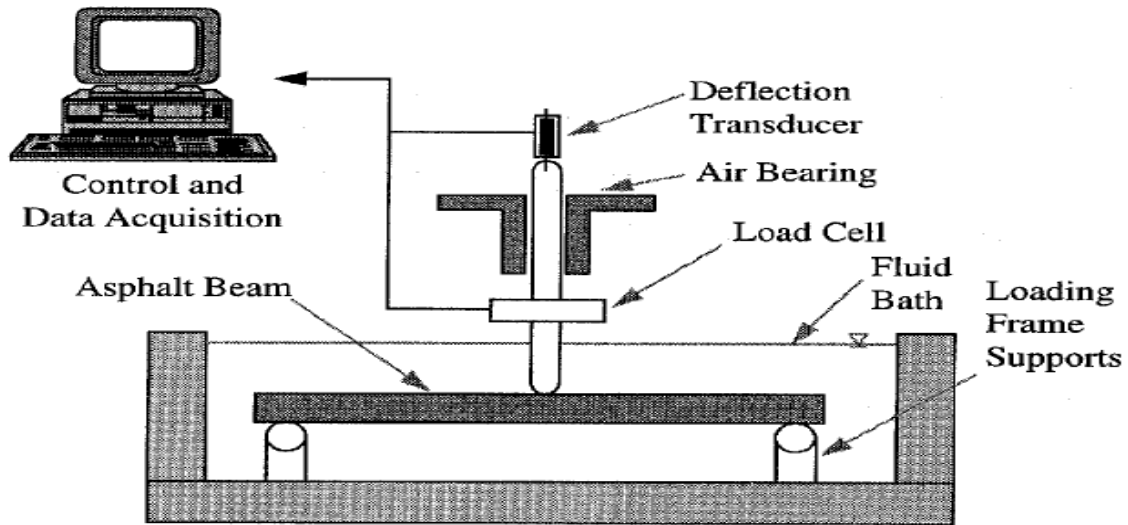


Figure 2.9: Schematic diagram of the bending beam rheometer [62].

## 2.5 Ministry of Transportation of Ontario (MTO) Newly Improved Test Methods

A number of research investigations have studied the effects of isothermal conditioning on the low temperature properties of asphalt cement since the 20<sup>th</sup> century. It is very important to take physical aging/hardening into consideration when measuring the rheological properties of asphalt cement [73-75]. According to Struik [43], it is of no use to study properties such as creep and stress relaxation without taking physical aging into consideration. To address this problem facing the AASHTO M320 specification, the Ministry of Transportation of Ontario in collaboration with Queen's University has embarked on the development of new test methods, which are: extended BBR test (LS-308) [20] and double edge notched tension (DENT) test (LS-299) [19]. It has been shown that the newly developed test methods give reproducible results that consistently give better ability to differentiate good performing binders from poor ones.

### 2.5.1 Extended BBR Test (LS-308)

Asphalt cements with lower quality are able to merge their wax/asphaltene structures, and lose their ability to relax thermal stress (decrease in m-value) to some extent when they are cooled for periods of up to several weeks and months before they are exposed to cold spells during the winter season. As a result of this, the regular BBR is not able to predict the thermal cracking performances of the pavements because they are under-designed (samples are conditioned for only one hour before testing). The rationale behind the extended BBR is that if asphalt binder beams are conditioned for periods other than one hour; they will be able to account for the low temperature cracking in asphalt pavement [74]. The extended BBR is merely an extension of the regular BBR, where asphalt beams are conditioned for 1, 24 and 72 hours at two different temperatures which are  $T_1 = T_{\text{design}} + 10^\circ\text{C}$  and  $T_2 = T_{\text{design}} + 20^\circ\text{C}$ , where  $T_{\text{design}}$  is the lowest temperature designed for the pavement, before they are tested. These two temperatures are chosen over the total range of 20 because at high temperature (above  $T_2$ ), the molecules merge and they cannot move and the thermodynamic tendency for wax crystallization and precipitation of asphaltene molecules are absent and hence physical aging tendency peaks up at very low temperatures. The objective of this method is to provide a high degree of confidence that thermal cracking is completely prevented other than to perfectly correlate with the low temperature cracking distress. It is important to protect roads that spend most of their time at a relatively warm temperatures,  $T_2$  from cold spells at  $T_1$ , even though cold spells may not happen during the initial life of the contract.

The regular test is performed to check a pass/fail temperature after each conditioning period according to AASHTO M320 criteria (where  $S(60) = 300$  MPa and m-value = 0.3). The grade temperature and the subsequent grade losses are calculated after each temperature conditioning

period. The warmest minus the coldest limiting temperature where S(60s) reaches 300 MPa and m-value(60s) reaches 0.3 is the worse grade. A maximum grade loss is set at 6°C in order to meet the LTPPBind® 98 percent confidence level after 72 hours of conditioning, which helps to ignore very poor quality asphalt cement that contain large quantities of wax and unstable asphaltene dispersions from the asphalt supply [21]. An accuracy of 95 percent has been shown by this test in separating the good from the poor performing asphalt cements [76].

### **2.5.2 Double Edge Notched Tension (DENT) Test (LS-299)**

The concept of the essential work of fracture (EWF) has been adopted to determine the fracture properties of asphalt cement at brittle-to-ductile and ductile states at Queen's University using the double edge notched tension specimen. Characterization of materials such as fibre, powder, polymers, glasses, metals and a host of other materials has been widely performed using the EWF method. The EWF is a thermodynamic method and it was originally developed by Cotterell and Reddel [77] and was further investigated by Mai et al. [78]. The method is based on the assumption that the total work of fracture at a fixed rate of loading on a DENT sample is divided into two regions: the essential fracture energy (the energy responsible for the fracture process) and the non-essential or plastic work energy (the energy responsible for the non-essential or plastic deformation outside the fracture zone) as shown in Figure 2.10. The total work of fracture is obtained from the total area under the force-displacement curve during the analysis and it is made up of the sum of the essential work of fracture energy and the non-essential or plastic work of fracture energy, i.e.

$$W_t = W_e + W_p \quad (7)$$

Cotterell and Reddel showed that the essential work of fracture component is related to the surface cross-sectional area of the plastic zone (the product of the thickness and the ligament length) and the plastic work of fracture is related to the volume of the plastic zone (the product of the square of the ligament length and the thickness), mathematically given as:

$$W_e = w_e \times LB \quad (8)$$

$$W_p = w_p \times \beta L^2 B \quad (9)$$

Therefore the total work of fracture can be written as

$$W_t = (w_e \times LB) + (w_p \times \beta L^2 B) \quad (10)$$

Where  $w_e$  is specific essential work of fracture ( $J/m^2$ ),  $w_p$  is specific plastic work of fracture ( $J/m^3$ ),  $L$  is the ligament length in the DENT specimen (m),  $B$  is the thickness of the sample (m) and  $\beta$  is the shape factor of the plastic zone, which is geometry dependent. Dividing through equation (10) by the cross-sectional area of the plastic zone ( $LB$ ), equation (11) can be obtained as:

$$W_t \backslash BL = w_t = w_e + \beta w_p L \quad (11)$$

According to Hashemi [80], a number of specific assumptions and conditions are required in order for a specimen to undergo the EWF method, of which only four of them are needed in this research and they are listed below:

- i. the volume of the plastic zone should be proportional to the product of the square of the ligament length and the sample thickness as shown in equation (9);
- ii. load-displacement diagrams should be the same for all ligament lengths;
- iii. ligaments yields fully prior to crack propagation or failure; and



- iv. fracture occurs under plane stress condition.

Plotting a graph of the specific total work of fracture,  $w_t$ , against the ligament length,  $L$  in equation (11) generates a straight line with slope and intercept on the  $w_t$  axis being equal to the specific essential work of fracture,  $w_e$ , and the product of the specific non-essential or plastic work of fracture,  $w_p$ , and the plastic zone shape factor,  $\beta$ , i.e.  $w_p\beta$  respectively [77]. It has been shown that  $w_e$  is usually able to accurately predict fatigue cracking resistance since it relates mainly to the fracture process [80]. On the other hand, in asphalt cements with the same  $w_e$ , it is believed that their  $w_p$  may be useful in explaining the differences in their performances. To be able to resist fatigue cracking and ductile fracture, it is expected that the values of both  $w_e$  and  $w_p$  should be relatively high [80].

Further studies have revealed that  $w_e$  can be used to obtain the crack tip opening displacement (CTOD) parameter using the equation:

$$\delta_t = w_e / \sigma_n \quad (12)$$

where  $\delta_t$  is the crack tip opening displacement parameter (m), and  $\sigma_n$  is the net section stress or yield stress ( $N/m^2$ ), obtained from the 5 mm ligament length DENT mould. The CTOD parameter provides a measure of strain tolerance in the ductile state under conditions of severe confinement. It has been shown that this parameter “shows promise for performance grading of both binders and mixtures for fatigue cracking at temperatures and rates of loading that cover the ductile regime” [69]. A number of research studies have been carried out both at Queen’s University and by the US Federal Highway Administration of pavement testing, to validate the EWF method [80]. It is shown that high correlations exist between the CTOD and the fatigue

properties of the asphalt cement and has an accuracy of 85 percent to predict fatigue cracking and ductile fracture properties of both binders and mixtures [76].

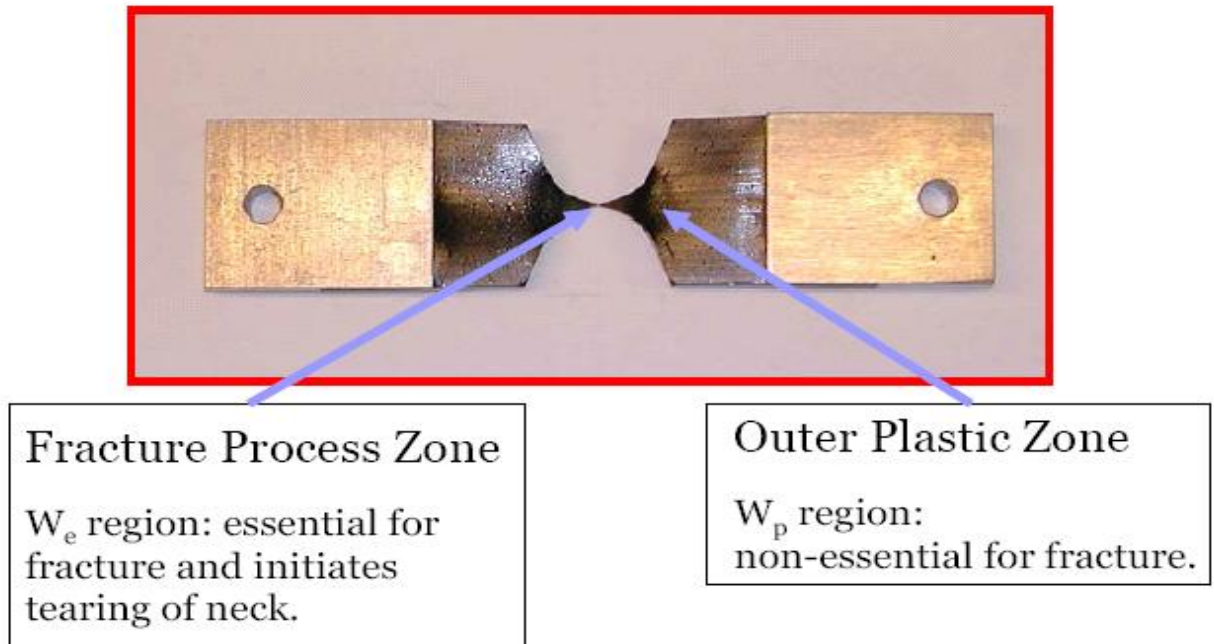


Figure 2.10: Diagram of fracture and plastic zone of asphalt binder [56].

**CHAPTER THREE**  
**MATERIALS AND EXPERIMENTAL METHODS**

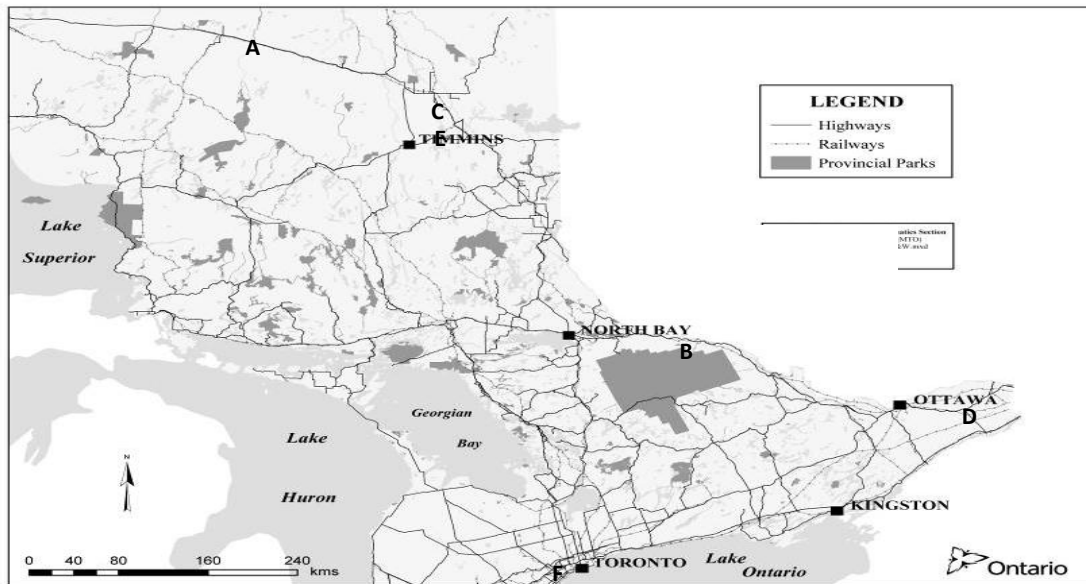
**3.1 Materials**

**3.1.1 Highway 417 Pavement Trial Materials**

Highway 417 is a major pavement trial near Casselman, east of Ottawa, as shown in Figure 3.1, constructed in the late summer of 2006. Asphalt binder samples used in these studies were obtained during the construction of the trial sections on this highway. Seven different modified asphalt binders were taken and stored in sealed cans at room temperature from their sampling during late summer in 2006 until their testing at different periods between summer 2007 [57] and winter 2011. All tested samples underwent rolling thin film oven (RTFO) aging according to standard conditions (35 g of asphalt binder aged at 160°C for 85 minutes) and pressure aging vessel (PAV) aging according to standard and modified conditions (50 g or 25 g of asphalt binder at temperature of 100°C and pressure of 2.01 MPa for 20 hours) according to the American Association of State Highway and Transportation Officials (AASHTO) procedures at the Imperial Oil laboratories in Sarnia, Ontario before they were tested at Queens University as part of this and other thesis projects.

Core samples for all sections were also obtained from the constructed highway in summer 2010 and these were stored at ambient temperature in sealed rubber bags before their recovery and testing at Queen's University in early 2011. Core samples were drilled 50 m apart from each other and five core samples were obtained from each section over a 250 m stretch. The thickness of the core samples are given in Table 3.2. A detailed list of the pertinent properties of the samples used in this investigation is given in Table 3.1. The samples are labelled 417-A to 417-

G, with the letters corresponding to the respective section in the pavement trial (417-A for 417-1, 417-B for 417-2, etc). The samples were obtained from five different commercial sources in the United States and Canada. Binders from sections 417-A to 417-C were all obtained from Alberta in Canada, but from two different suppliers. Sections 417-A and 417-B were made with base asphalt from Lloydminster while section 417-C was prepared with base asphalt from Cold Lake. However, the origins of the base asphalts from sections 417-D to 417-G are not known with any certainty. All seven sections were intended to have a low temperature Superpave grade of PG-34 according to the Long Term Pavement Performance Binder Selection Software (LTPPBind<sup>®</sup>), but sections 417-C and 417-E ended up grading as PG-40 [81].



**Figure 3.1:** Locations for Current Ontario Pavement Trials. Note: Letter D indicates the location for the Highway 417 trial between Casselman and Limoges [82].

**Table 3.1: Pertinent asphalt binder properties for Highway 417 [56]**

<b>Asphalt Binder (Sections)</b>	<b>Source of Base Asphalt</b>	<b>Modification Type</b>	<b>PG Grade</b>
417-A	Lloydminster	3 % SBS	64-34
417-B	Lloydminster	4 % SBS	70-34
417-C	Cold Lake	7 % D-1101 linear SBS	82-40
417-D	Western Canadian Blend	3.3 % Elvaloy® 1052 RET and 0.4 % PPA	70-34
417-E	Western Canadian Blend	2.1 % Elvaloy® 4170 RET and 0.4 % PPA	64-40
417-F	Unknown	SBS (unknown grade and concentration)	76-34
417-G	Unknown	Control - SB diblock copolymer and PPA (unknown grade and concentration)	64-34

Note: SBS = Styrene-Butadiene-Styrene Copolymer, PPA = Polyphosphoric Acid. SB = Styrene-Butadiene Copolymer, RET = Reactive Ethylene Terpolymer, PG = performance grade of asphalt binders.

**Table 3.2: Total and average thicknesses of Highway 417 core samples.**

<b>Sample No.</b>	<b>Core Thickness (cm)</b>	<b>Average Core Thickness (cm)</b>
417-A1	45	
417-A2	51	
417-A3	46	
417-A4	43	
417-A5	47	46.4
417-B1	50	
417-B2	43	
417-B3	42	
417-B4	48	45.6
417-B5	45	
417-C1	47	
417-C2	43	
417-C3	42	
417-C4	44	44.2
417-C5	45	
417-D1	39	
417-D2	37	
417-D3	40	
417-D4	37	38.2
417-D5	38	
417-E1	40	
417-E2	37	
417-E3	39	
417-E4	40	39.4
417-E5	41	
417-F1	39	
417-F2	40	
417-F3	40	
417-F4	39	38.4
417-F5	34	
417-G1	36	
417-G2	39	
417-G3	39	
417-G4	40	
417-G5	41	39

### 3.1.2 Recovery of Asphalt Cement from Highway 417

The asphalt cement binder was recovered from the asphalt cement-aggregate mixture through extraction with toluene. Toluene was used in preference to other solvents such as tetrahydrofuran (THF) because of its relatively low flammability and the fact that it does not contain any stabilizers such as butylated hydroxytoluene (BHT) in tetrahydrofuran.

The top layer (about 5 cm) of the asphalt cores which is in direct contact with immediate environmental conditions (traffic, climatic and others) was cut using a diamond-tipped cutting saw. The cut sample was crushed and soaked in solvent overnight. The extract was removed periodically and stored in a different container to allow fine aggregate to sediment. The solvent washing was stopped once the aggregate was relatively clean and no further asphalt cement could be removed. The extract was allowed to sediment for a minimum of 5 hours. In all, about 4-6 litres of toluene were used to extract approximately 200 g of asphalt binder from 4 kg of the asphalt mixture. The recovery of the asphalt cement from the solvent was performed using a rotary evaporator through a condensation process, where most of the solvent was condensed at a temperature of 70-80°C and aspirator pressure of 180 mbar. After sufficient solvent was removed, the temperature was increased to 150°C and the aspirator pressure was kept at 20-40 mbar for an additional one and a half hours to ensure the total removal of all the solvent from the asphalt binder and also to prevent changes in the chemical composition (oxidation) of the binder. Infrared (IR) spectroscopy analysis was performed intermittently during the recovery process to ensure that no binder oxidation occurred. IR analysis was performed on an aliquot of the:

- i. solvent and binder mixture prior to evaporation,
- ii. binder and solvent mixture after most of the solvent has been evaporated, and
- iii. asphalt binder after complete evaporation of the solvent.

This method was used as the standard procedure for extraction and recovery of all the seven asphalt cements from Highway 417.

## **3.2 Experimental Methods**

### **3.2.1 X-Ray Fluorescence (XRF) Analysis**

The presence of zinc, known to be present as a result of the introduction of waste engine oil (WEO) to improve short-term performance of the asphalt cements, was also investigated in this thesis. XRF spectra for the pure asphalt cement used for the construction of the various sections of the highway and their corresponding recovered asphalt cement samples were analyzed using a hand-held Innovative X-ray Technologies (Innov-X) model XT-440L analyzer [83]. The surface of the asphalt cement was irradiated by high-energy X-rays obtained from the instrument, and caused the removal of electrons in the inner K-shell from heavy metals present in the asphalt cements. The created holes available in the K-shell are then occupied by electrons from the outer L- and M-shells. Emission of lower energy X-ray, which is characteristic of the heavy metals present in the sample, takes place as a result of the descent of the electrons into the inner K-shell [84]. The emitted radiations are detected by the XRF analyzer, and a graph of intensity against the X-ray energy provides both qualitative and quantitative information on the presence of a range of heavy metals [83]. The presence and the quantity of a particular heavy metal are determined by the peak height in the spectrum. The presence of zinc was determined at 8.64 keV after 40 seconds scan using the intensity versus X-ray energy graph, and the quantity of zinc present in each sample was determined by comparing the amount of zinc determined in each sample to the amount of zinc found in two standard waste engine oils obtained from different suppliers, using the sample procedure.



### 3.2.2 Infrared (IR) Spectroscopic Analysis

Samples of unaged, RTFO-aged (AASHTO T240-97, 85 minutes at 160°C), PAV-aged (AASHTO PP1-98, 20 hours at 100°C and 2.09 MPa), and field aged binders were analyzed by IR spectroscopy. Potassium bromide (KBr) discs were used for this test in a Perkin-Elmer Spectrum 400 FT-IR spectrometer. The KBr disc was heated at 180°C for about 2 minutes and a small quantity of the asphalt binder was spread on the heated KBr disc with the help of a spatula to form a homogeneous thin film layer on the disc which was subsequently allowed to cool down for 5 minutes. The test was performed by placing the asphalt-coated KBr disc in the IR spectrometer and the IR software was used to run the test with 16 scans over a wavenumber range of 4000  $\text{cm}^{-1}$  to 400  $\text{cm}^{-1}$ . Prior to the test, a background scan was performed in order to calibrate the IR spectrometer.

Data analysis was done by the instrument's software. The data was analysed by integrating the total area under each peak to obtain the peak areas for the functional groups of interest.  $\text{CH}_2$  integrals were used as an internal standard because it is relatively inert to oxidative changes compared to  $\text{CH}_3$  peaks. Integration from 2850-3200  $\text{cm}^{-1}$  was assigned to the  $\text{CH}_2$  peak. The peak positions for aromatics, sulfoxides and carbonyls were assigned values of 1600  $\text{cm}^{-1}$ , 1030  $\text{cm}^{-1}$  and 1700  $\text{cm}^{-1}$  respectively [85], therefore, the integral bounds used were 1650 to 1535  $\text{cm}^{-1}$  for the aromatics, 1070 to 985  $\text{cm}^{-1}$  for the sulfoxides, and 1760 to 1655  $\text{cm}^{-1}$  for the carbonyl functional groups. The functional groups were selected because they are contained in the asphalt and they undergo oxidation upon exposure to air (oxygen) and sunlight, which has a significant influence on both physical and chemical aging processes. To understand the chemical changes in asphalt during the aging process, the indices of these functional groups were analysed as follows:

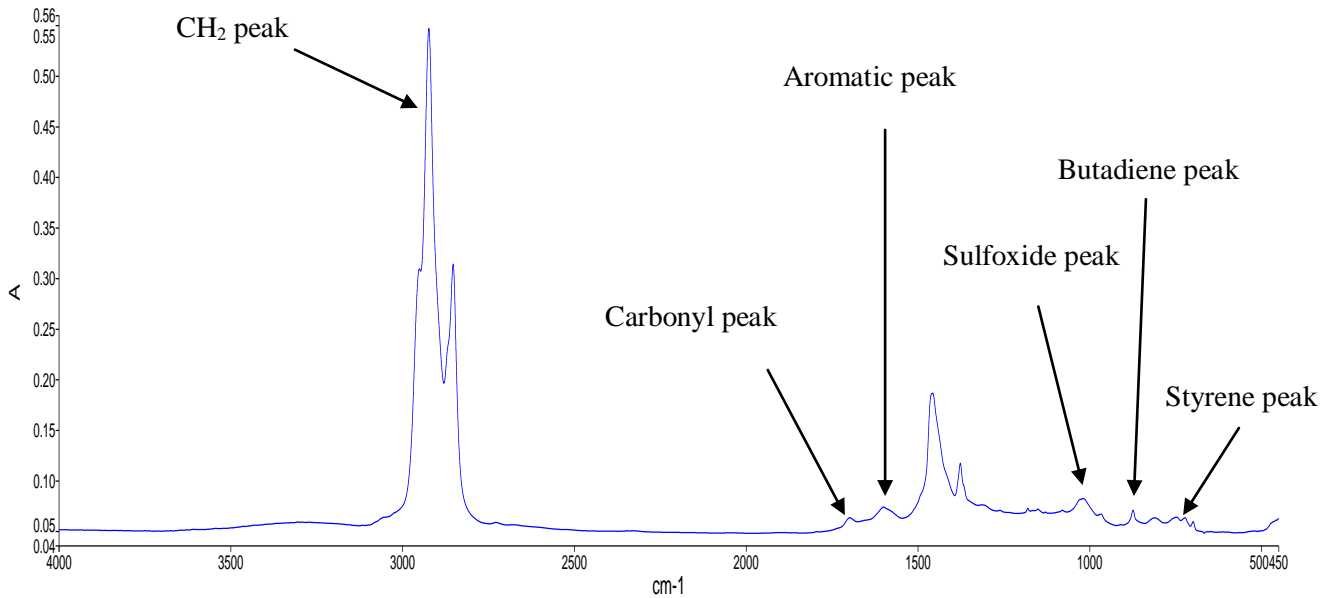


Figure 3.2: Infrared (IR) spectrum of an asphalt binder showing the peak heights of the various functional groups of interest. Note: A is the absorbance and W is the wavenumber.

1. the sulfoxide index, given as the ratio of the sulfoxide peak area to the CH<sub>2</sub> peak area,
2. the carbonyl index, given as the ratio of the carbonyl peak area to the CH<sub>2</sub> peak area, and
3. the aromatic index, given as the ratio of the aromatic peak area to the CH<sub>2</sub> peak area.

Because the asphalt cement samples tested were modified with polymers such as SBS, it was therefore necessary to study some other functional groups such as styrene at 700 cm<sup>-1</sup> and butadiene at 969 cm<sup>-1</sup>, to identify the presence of those functional groups in the asphalt binder and their effects on the performance of the asphalt binder in service. Styrene peaks were assigned limits from 710 to 690 cm<sup>-1</sup>, and the corresponding index was calculated as the ratio of the styrene peak area to the CH<sub>2</sub> peak area. Likewise, the butadiene peak area was assigned from 983 to 955 cm<sup>-1</sup>, and the corresponding index was determined as the ratio of the butadiene peak area to the CH<sub>2</sub> peak area.

### 3.2.3 Dynamic Shear Rheometer (DSR) Testing

The dynamic shear rheometer (DSR) test is used to assess the rutting resistance of the asphalt cement at high temperatures and the fatigue cracking resistance of the asphalt pavement in-service at intermediate temperatures. As a result of these parameters, the test was conducted on unaged, RTFO-aged, RTFO+PAV-aged and field aged (recovered asphalt cement) samples. The sample was first heated until it was liquid enough to be poured, and was then stirred to ensure homogeneity before pouring into the silicon moulds of specified dimensions as shown in Figure 3.3 (a). The testing temperature was selected according to the asphalt binder's specification grade or testing schedule. The upper and the lower parallel plates were heated at 46°C prior to testing of the specimen, so that the asphalt binder will be able to adhere to the surface of the plates. The DSR software was then used to move the plates together until the gap between them equals the test gap plus 0.002 inches (0.05 mm). Excess material coming out of the plates as a result of the compression was removed using a heated trimming tool as shown in Figure 3.3 (b). The test plates were then moved to the desired testing gap. This created a slight bulge in the asphalt binder specimen's parameter. The specimen was then brought to the desired test temperature. The test was started up only after the specimen had been at the desired temperature for at least 10 minutes. The DSR software was used to rotate the upper plate at a specific target torque based on the material being tested (such as unaged binder, RTFO residue, PAV residue or field aged residue). This torque was chosen in order to ensure that the measurements were within the specimen's region of linear behaviour. The DSR was used to condition the sample for 10 cycles at frequency of 10 rad/sec (1.59 Hz). The DSR test takes measurements over the next 10 cycles and the software reduces the data to produce a value for the complex shear modulus,  $G^*$ , and the phase angle,  $\delta$ . The complex shear modulus ranges from about 0.07 to 0.87 psi (500 to

6,000 Pa), while the phase angle ranges from about 50 to 90°. The complete viscous component of the asphalt binder is characterised by a phase angle of 90°. Lower values of the phase angle, usually as a result of increased elasticity, or higher values of the phase angle due to softening of the binder, are usually obtained for polymer-modified binders depending on the kind of polymer used for modification. This in turn stiffens or softens the base asphalt compared to the unmodified asphalt binders.

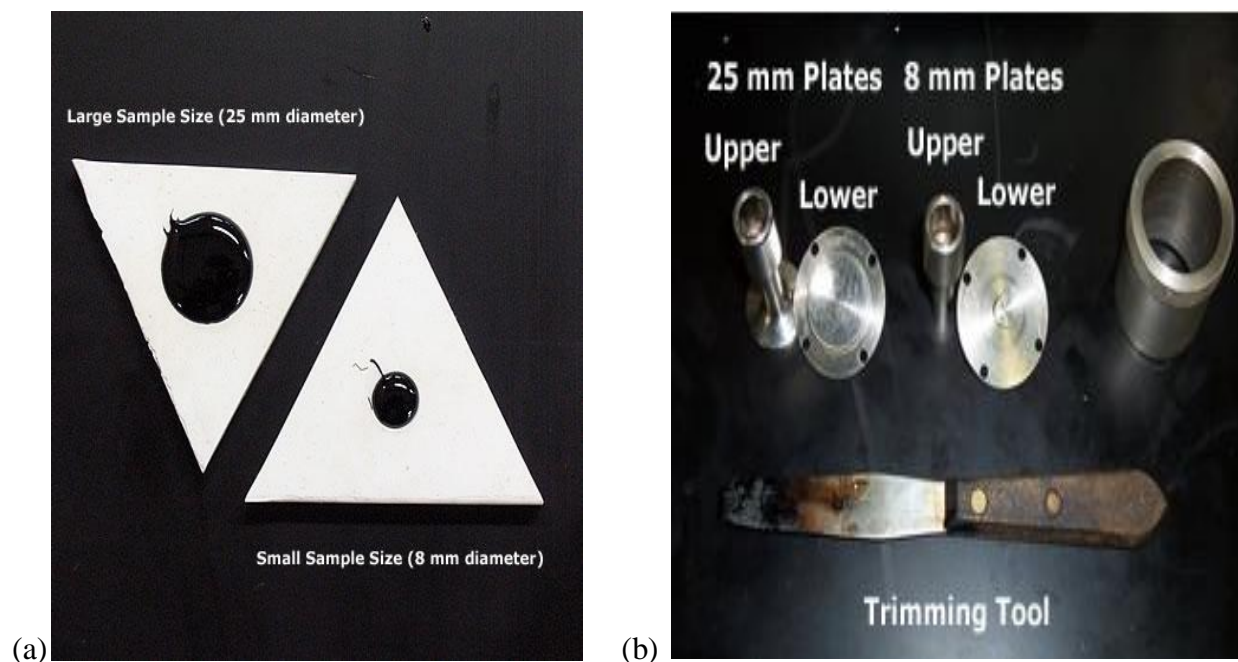


Figure 3.3: (a) Dynamic shear rheometer sample moulds showing the different sizes and (b) major dynamic shear rheometer test equipment [59].

### 3.2.4 Regular Bending Beam Rheometer (BBR) Testing

The regular BBR test was performed on RTFO-aged, RTFO+PAV-aged, and field aged asphalt cements, since the test is presumed to be used to simulate low temperature cracking and thermal fatigue cracking of the pavement in service for several years. The asphalt binder was heated at 160°C for about 45-60 minutes in an oven to ensure that it was fluid enough to be poured and

was then stirred gently afterwards to remove any air bubble present before pouring into the aluminum moulds. The aluminum moulds were prepared by greasing the surface of the aluminum pieces with Vaseline, and Mylar strips were placed against the greased surface of the aluminum pieces. The individual aluminum pieces were put together with the help of a rubber band and silicon pieces were placed at the ends in specified dimensions as shown in Figure 3.4. The asphalt binder in the aluminum moulds was conditioned at room temperature for one hour and the excess binder was trimmed with a hot spatula from the top of each mould to obtain a well-defined surface beam. The asphalt beams were then conditioned for another one hour at -10°C and -20°C in a cooling bath containing a mixture of ethanol and water. The beams were then tested at the same temperatures and sometimes at -30°C by supporting them at two points 102 mm apart. The test was performed by loading the beam at its midpoint for a period of 240 s with a load of approximately 980 mN. Figure 3.5 shows the deflection of the asphalt beam in a typical BBR test. The concept of the BBR is basically based on simple beam theory, from which the beam stiffness,  $S(t)$ , and the  $m$ -value (rate of change of the stiffness) are calculated as shown below, as the load is applied [9].

$$S(t) = \frac{PL^3}{4b\delta h^3(t)} \quad (13)$$

Where

$S(t)$  = creep stiffness at time,  $t = 60$  seconds;

$P$  = applied constant load ( $980 \pm 20$  mN) obtained using a 100 g load.

$L$  = distance between beam supports, 102 mm;

$b$  = beam width, 12.5 mm;

$h$  = beam thickness, 6.25 mm; and

$\delta(t)$  = deflection at time,  $t = 60$  seconds.

The m-value is determined as the slope of the double logarithmic graph of stiffness versus time at a specified time of 60 seconds of loading. The stiffness, also known as the creep stiffness and the m-value were calculated at a loading time of 60 seconds. The creep stiffness can have a maximum value of 300 MPa and the m-value can have a minimum value of 0.3, as specified in the AASHTO M320 standard. All the tests were performed in duplicate and excellently reproducible results were obtained. The round robin test has been used to set the approximate error for both regular and extended BBR data as  $\pm 1^{\circ}\text{C}$ , as demonstrated by the Ministry of Transportation of Ontario.

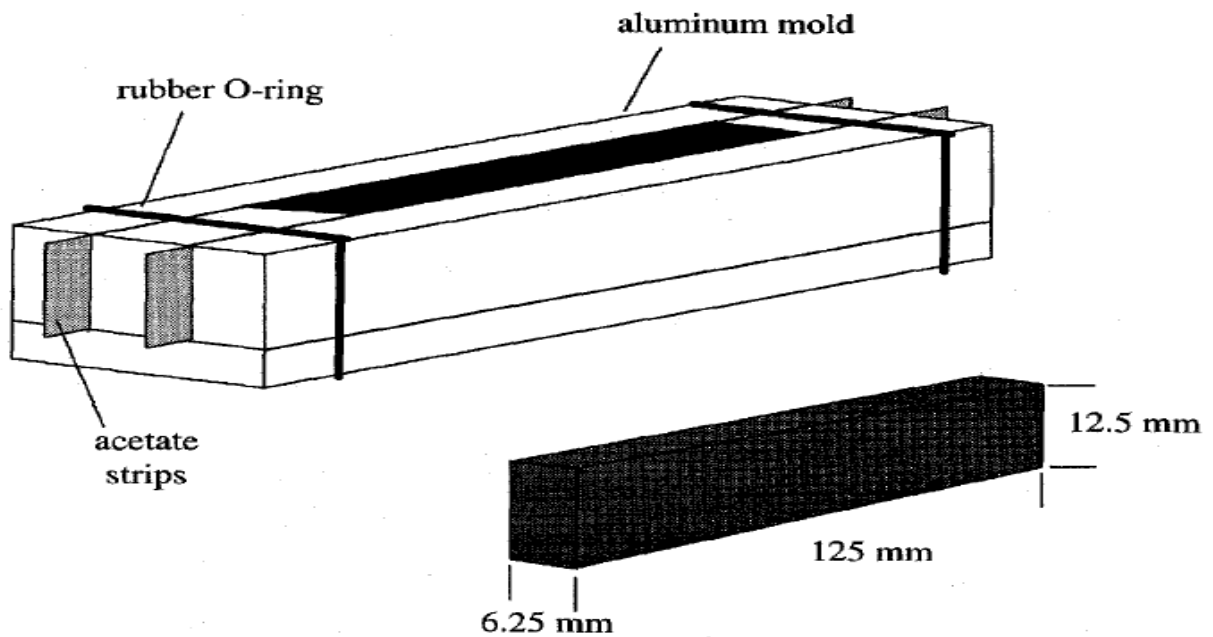


Figure 3.4: Asphalt mould assembly and dimensions of asphalt beam [62]

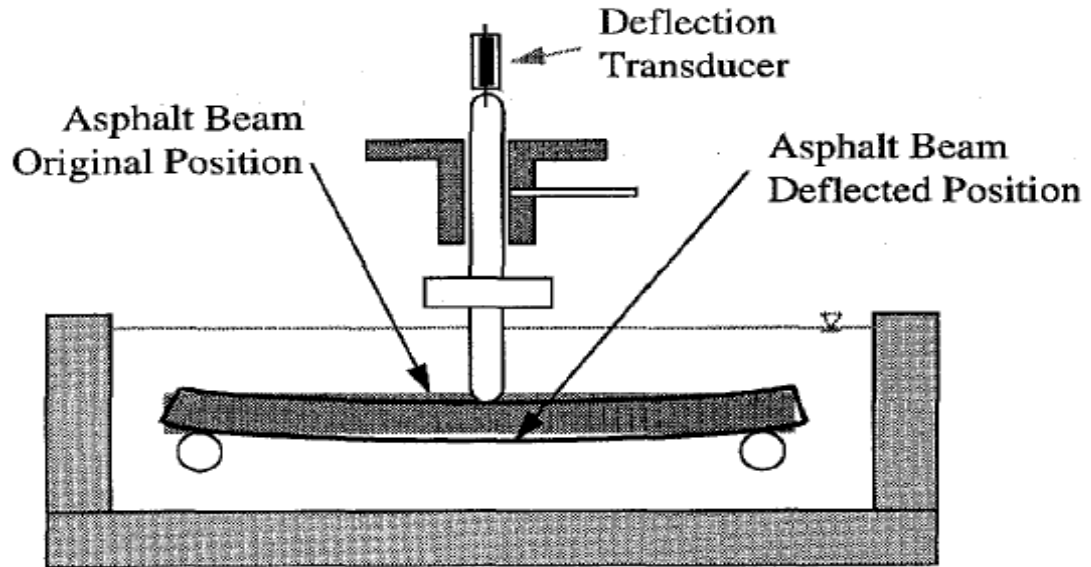


Figure 3.5: Deflected asphalt beam on bending beam test [62].

### 3.2.5 Extended Bending Beam Rheometer Testing

The extended BBR test was performed with the objective of evaluating the susceptibility of the asphalt binder to reversible aging (hardening), particularly at low temperatures. The critical cracking temperature, which is known to be the point at which the maximum stress at a given temperature is more than the strength of the material, is determined [56]. A total of 12 beams were prepared and 6 were conditioned at  $-20^{\circ}\text{C}$  and another 6 were conditioned at  $-10^{\circ}\text{C}$  in a cooling bath for 1 hour and 72 hours before testing at  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  and sometimes at  $-30^{\circ}\text{C}$ . The  $m$ -values and the creep stiffness were determined after each conditioning period and failure temperatures where  $S = 300$  MPa and  $m = 0.3$  were calculated according to the regular AASHTO M320 protocols. The temperatures of  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  were selected in order to determine a pass/fail temperature for the binder. The limiting temperature and the subsequent maximum (worst) grade loss were calculated after 72 hours of conditioning.

### 3.2.6 Double Edge Notched Tension (DENT) Testing

The DENT test method is used to account for fatigue thermal cracking as well as ductile fracture in asphalt mixes. It involves pulling of a notched asphalt binder in a water bath at isothermal condition until failure/fracture occurs. The essential work of fracture and the plastic work of fracture are determined at a constant temperature of 15°C and loading rate of 50 mm/min. The test is carried out by pouring a liquid asphalt binder into brass DENT moulds with different ligament lengths (the distance between two opposing notches), ranging from 5 mm to 15 mm in length, and conditioning them for 24 hours in an isothermal water bath before testing. The integrated area under the force-displacement curve is considered as the total work of fracture and it is made up of the sum of the essential and the plastic works of fracture. By plotting the specific total work of fracture ( $w_t = W_t/BL$ ) against the ligament length,  $L$ , a linear graph is generated with the intercept at  $L=0$  being equal to the essential work of fracture and the slope being equal to the product of the plastic zone shape factor and the essential plastic work of fracture. The crack tip opening displacement (CTOD) is used to predict the fracture characteristics of the asphalt binder and it is given as the ratio of the essential work of fracture to the net section stress of the 5 mm ligament length specimen. It measures the strain tolerance in the ductile state in the presence of significant constraint that would occur during ductile fracture in the HMA [58].

The test was performed by heating the asphalt binder for about 45 minutes to ensure homogeneity and was then followed by gently stirring to remove any air bubbles present. The liquid asphalt binder was then poured into already prepared brass DENT moulds with ligaments lengths of 5 mm, 10 mm, and 15 mm, as shown in Figure 3.6. Prior to the preparation of the DENT moulds and the heating of the sample, the water bath was set at 15°C. The asphalt binder in the aluminum moulds was conditioned at room temperature for one hour and also in the water



bath for 24 hours before testing in the same water bath at 15°C. The binder was then pulled at a constant loading rate of 50 mm/min until failure occurred as shown in Figure 3.7., and the necessary parameters were recorded. Excel spreadsheet was used to calculate the essential work of fracture, the plastic work of fracture, and the CTOD parameter. The test was repeated for each ligament lengths and excellent reproducibility was obtained for all the different ligament lengths.

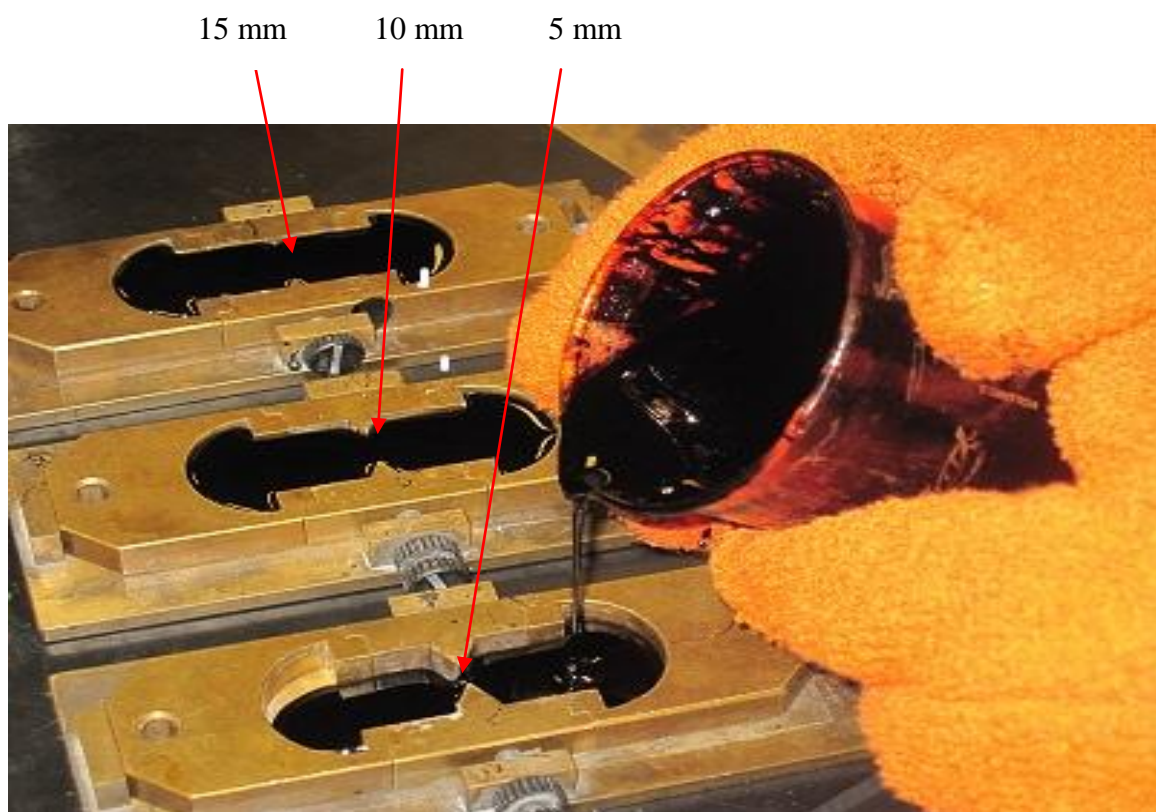


Figure 3.6: Pouring of asphalt binder into the brass moulds of different ligament lengths.

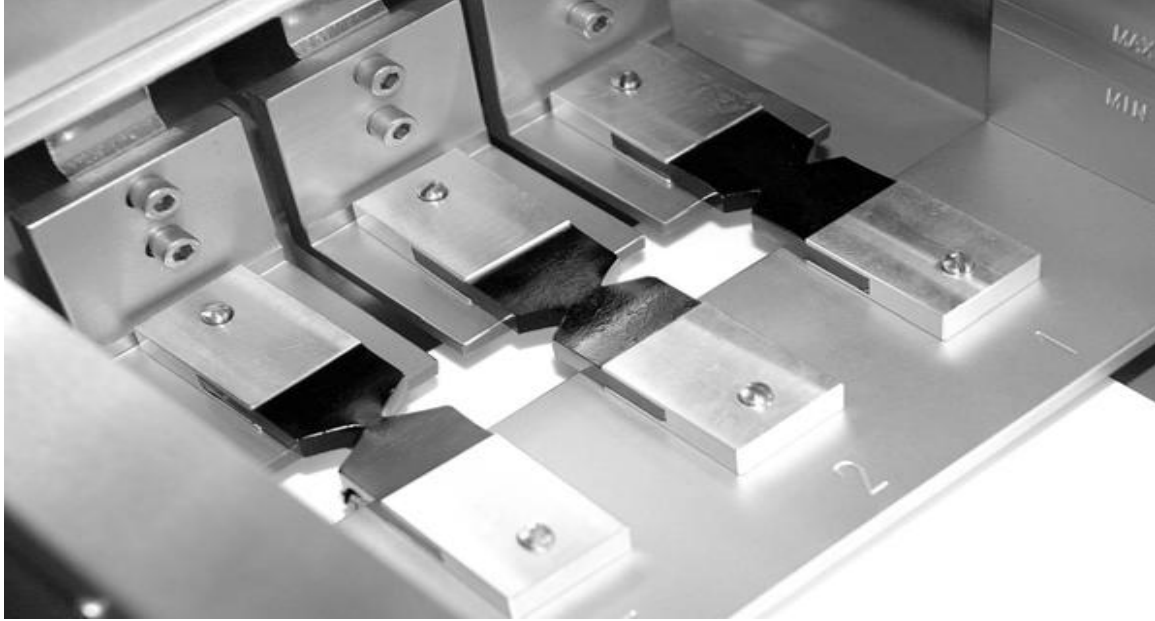


Figure 3.7: Double Edge Notched Tension (DENT) test set up utilizing aluminum moulds [8].

## CHAPTER FOUR

### RESULTS AND DISCUSSIONS

#### 4.1 Air Void Analysis

The air voids content in the hot-mix asphalt is an important parameter when it comes to asphalt pavement performance. Asphalt concrete mixes are made in such a way that they will possess sufficient air voids in the total compacted mix to account for a slight amount of additional compaction and traffic loading. A newly laid asphalt concrete is expected to have air void in the range of 4-7 %. One dimensional densification and plastic flow are the two main mechanisms by which air void reduction in a pavement can occur. Details of these two mechanisms can be found in reference [85] and the references contained in that article. Investigations on pavement performance by Von Quintus et al. [86] have shown that when the air voids are reduced to less than 3%, it is the plastic flow mechanism which dominates. The movement of asphalt binder into the voids and a reduction in the asphalt film thickness will lead ultimately to a reduction in the relative distance between the aggregate particles. As the asphalt binder flows into the voids, particle reorientation may occur. Von Quintus and coworkers [86], in the studies on pavement performance, have shown that air void between the ranges of 4-7 % in an asphalt concrete mix is satisfactory for its desirable performance in most environments. Air voids less than 3 % result in excessive plastic flow and air voids greater than 7 % leads to high air and water penetration or permeability effects. As a result, any alteration in the air voids from its optimum value during its lifetime constitutes initiation of damage and hence, in this regards, the air void reduction or increment measure can be looked upon as an asphalt concrete pavement distress parameter.

**Table 4.1: Percentage air void obtained from core samples of Highway 417**

<b>Sample number</b>	<b>Air void (%)</b>	<b>Average</b>
417-A-1	6.8	
417-A-3	6.3	6.3 ± 0.44
417-A-5	5.7	
417-B-1	5.6	
417-B-3	6.5	6.3 ± 0.61
417-B-5	6.8	
417-C-1	7.8	
417-C-3	8.2	7.3 ± 1.15
417-C-5	6.0	
417-D-1	6.2	
417-D-3	8.7	7.1 ± 1.12
417-D-5	6.5	
417-E-1	6.8	
417-E-3	6.8	6.7 ± 0.08
417-E-5	6.6	
417-F-1	11.1	
417-F-3	9.8	9.8 ± 0.62
417-F-5	8.4	
417-G-1	7.0	
417-G-3	7.3	
417-G-5	6.6	6.9 ± 0.28

Table 4.1 shows the percentage air void for all the trial sections determined on Highway 417.

The percentage air void was determined by weighing a small piece of the sample (asphalt concrete mix) under water and partially dried (wet dry), and the sample was allowed to dry completely by air for about 72 hours and then weighed again. An Excel spreadsheet was used to calculate the percentage air voids and their corresponding standard deviations, as follows;

$$\text{Bulk Specific Gravity, } \rho = \frac{\text{Weight by air dry}}{\text{Weight by wet dry} - \text{Weight under water}}$$

$$\% \text{ Air Void} = \frac{\text{Maximum Bulk Density} - \text{Bulk Specific Gravity}}{\text{Maximum Bulk Density}} \times 100\%$$

where the maximum bulk density of the asphalt cement is given as 2.55.

Three samples were used for each trial section. The heights of the core samples for the sections are given in Table 3.2. The height of all the core samples for all the sections range from 38.2 cm to 46.4 cm with Section A being the highest and Section D being the lowest. The heights for all the pavements are within the required limiting range of pavement height (> 20 cm), however, section with greater pavement heights such A, B, C and G, experienced insignificant or no cracking at all, while those with comparatively smaller heights, such as D to F, have undergone some form of cracking. Figure 4.1 shows the comparison of the cracking distress of the various trials sections with their respective percentage air voids. As can be seen from the data in Figure 4.1, Section A, Section B and Section G recorded air voids between the range of 6.3 to 6.9%, and they have so far remained free of cracking distress. Section F recorded the highest air void value of  $9.8 \pm 0.62\%$ , which exceeds the expected range of percentage air void in an asphalt concrete mix of 4-7%. Hence, it is therefore not surprising to see this section undergoing severe thermal cracking after 5 years in service. Greater values of air voids allow the penetration of water, frosts and air to the layers beneath the surface layer/course and causes the asphalt cement to undergo

oxidative aging and hardening of the asphalt binder and making it stiffer and leads to premature and excessive cracking, as witnessed in section F. Sections C and D recorded relatively the same percentage air voids of  $7.3 \pm 1.15 \%$  and  $7.1 \pm 1.12 \%$  respectively. However, Section C has experienced an insignificant cracking while D has cracked quite significantly. This difference can be accounted for by other factors such as the oxygen content in the asphalt cement, the ductile strain tolerance and ductile fracture properties of the binder, the height and thickness of the pavement, and other construction engineering principles. Section E has an air void content of  $6.7 \pm 0.08$ , which ideally was not supposed to crack, but it has experienced the most severe cracking among all the sections, and this contrast can be as result of other properties associated the with asphalt binder, the aggregate and the method used for construction. It can therefore be concluded that, an increase in the air void content of an asphalt concrete mix has a direct influence on the performance of the pavement. This causes excessive and premature cracking, as a result of the aging phenomena that occurs in the asphalt cement upon the penetration of air and water to the underlying layers of the pavement.

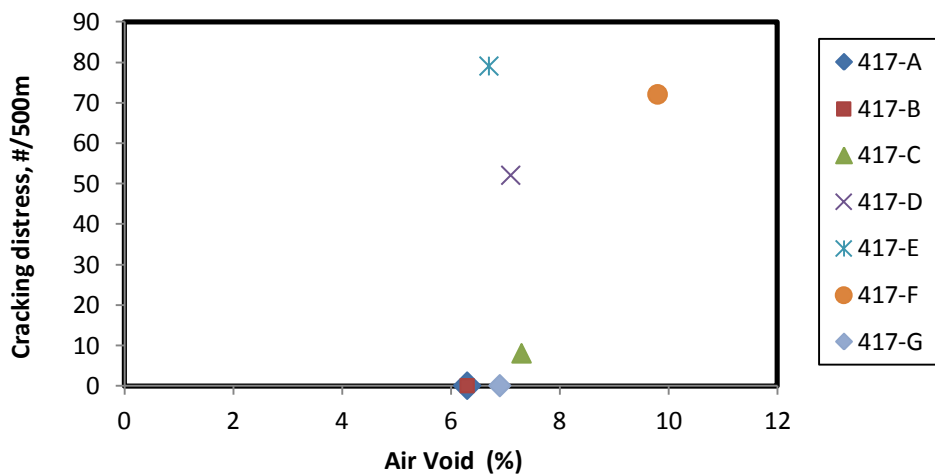


Figure 4.1: Comparison of pavement cracking distress with percentage air void of trial sections on Highway 417.

## 4.2 X-Ray Fluorescence Analysis

Aging processes in asphalt cements lead to the alteration of the chemical structure and mechanical properties of the asphalt cements over time. Oxidative aging causes the asphalt cement to harden and eventually leads to the deterioration of the asphalt pavement within a short period of time. Many techniques have been adopted to reduce the occurrence of this aging phenomenon in asphalt cements. One of these techniques involves the indirect addition of antioxidants such as zinc dialkyldithiophosphate (ZDDTP) and zinc dibutyldithiocarbamate (ZDBC) through the addition of waste engine oils to improve the resistance of the asphalt cement to carbonyl formation through oxidative processes [83]. However, waste engine oil residues have severely negative effects on the performance of the asphalt cement. The addition of waste engine oils leads to thermal cracking and permanent deformation of the asphalt pavement. Waste engine oil residue is highly paraffinic in nature and as such it causes premature hardening due to the precipitation of asphaltenes that are formed during the oxidation in service. Paraffins are also known to cause problems with a loss of adhesion to the aggregate which can lead to premature stripping (removal of the asphalt cement from the aggregate) and rutting induced by this stripping.

In order to determine the presence and quantity of the waste engine oil (WEO) residues added to the asphalt, a qualitative and quantitative analysis was conducted on all the trial sections on Highway 417 on both the neat (unaged) asphalt binder and the recovered asphalt binder. In other to compare the X-ray fluorescence to determine the percentage of waste engine oil residue added to both the neat binder and the recovered, two different waste engine oil residues obtained from

different local suppliers were scanned for 40 seconds and analysed to determine the presence and the quantity of zinc present at 8.6 keV. The spectra for the two waste engine oil residues are shown in Figure 4.2. This curve was used as the standard to determine the quantity of zinc present in each of the trial sections, and their respective percentage waste engine oil present were also determined accordingly. Table 4.2 shows the amount of zinc detected for each trial section at 8.6 keV after a 40 seconds scan. The results as obtained present a lot of interesting insights.

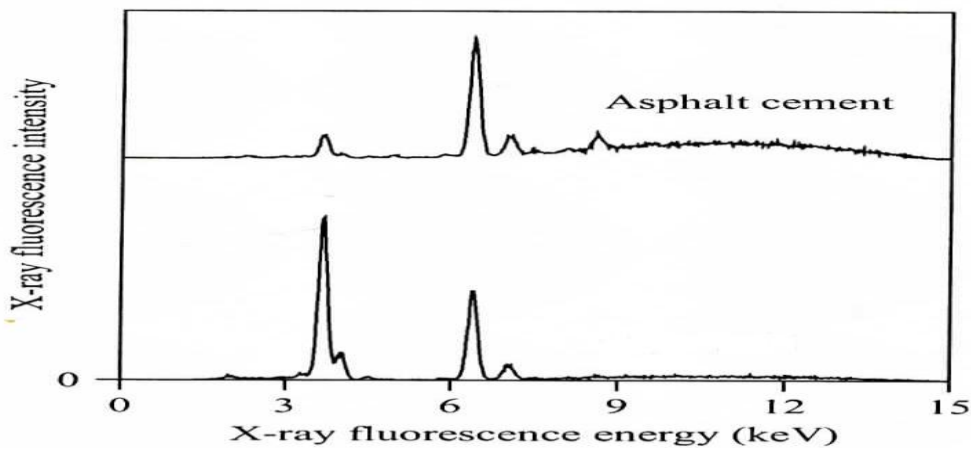


Figure 4.2: Typical X-ray fluorescence spectra for neat and recovered asphalt cement [83].

**Table 4.2: Zinc counts and percentage of waste engine oil (WEO) in trial sections of Highway 417.**

Trial Section	Amount of zinc count		Estimated percentage of WEO residue in the sample		Cracking distress, #/500 m pavement
	Neat Binder	Field-aged Binder	Neat Binder	Field-aged Binder	
417-A	92	138	0.47	0.70	0
417-B	447	115	2.26	0.58	0
417-C	413	189	2.09	0.96	8
417-D	71	174	0.36	0.88	52
417-E	1608	8.6	8.14	0.04	79
417-F	332	127	1.68	0.64	72



417-G	1833	372	9.28	1.88	0
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Note: Average count for 100% WEO from the two different suppliers is 19750.

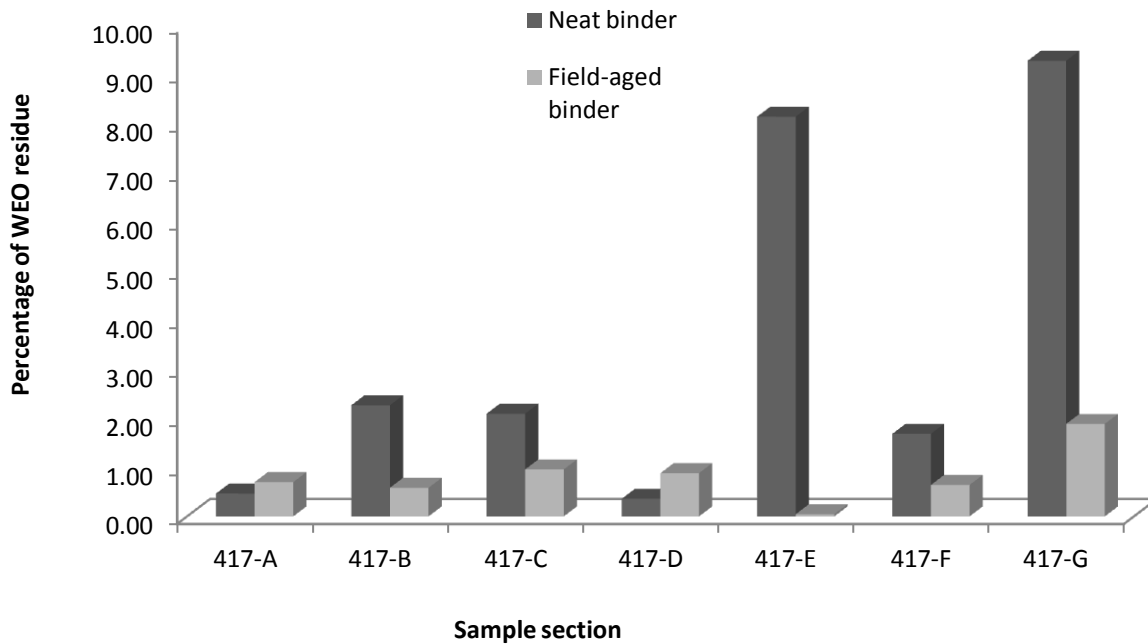


Figure 4.3: Percentage of waste engine oil (WEO) residues found in both neat and recovered asphalt cements on Highway 417 trials sections.

One important conclusion that can be deduced from Table 4.2 is that, the percentage WEO residue for the virgin binder for all the trial sections is greater than that of the recovered binders, except for sections A and D. The cause of this difference remains unknown, but it can partly be attributed to metals used to store the virgin asphalt binders, and to their media of transportation which may end up adding some quantities of other metals such as zinc, iron, manganese and many others to the asphalt cement. It can also partly be attributed to the fact that zinc present in the recovered binders, being expose to a number of environmental conditions, may undergo a series of chemical reactions and reduce its content in the recovered asphalt binder. From Figure 4.3, it is envisaged that Section A recorded insignificant amount of waste engine oil residues in

the asphalt cement. This is affirmed by the absence of any pavement cracking on this trial section during the distress survey analysis in summer 2010. The percentage of WEO for section B is respectively 2.26 % and 0.28 % for the neat and recovered asphalt binder respectively. However, the distress survey investigation shows that this trial section is free from any pavement cracking. The presence of zinc found in the neat asphalt binder may partly be attributed to the container used for the storage and transportation of the binder during construction. The percentage of WEO for Section C is 2.09 and 0.95 % for the neat and the recovered binder respectively. The distress survey shows that the pavement on this trial has 8 m/500 m pavement cracking. This minor crack can be attributed to construction variability/flaws and is unlikely related to the presence of WEO residues in the asphalt cement. Section D recorded insignificant percentage waste engine oil residues for both the recovered and the neat binders; however, the distress survey data shows that this trial section has a pavement cracking length of 52 m/500 m. This cracking can be explained by other factors such as the physical and chemical aging characteristics as well as the rheological and ductile fracture (strain tolerance) characteristics of the asphalt binder and other construction issues (pavement thickness), as discussed in the subsequent sections. Section E presents a great ambiguity, in that, the recovered asphalt binder shows no presence of waste engine oil but the virgin asphalt cement shows a significant percentage waste engine oils residues (8.6 %). However, the pavement has cracked significantly of about 72 m/500 m in length. It is therefore highly unlikely that the asphalt cement as delivered by the contractor was used for this trial section and further studies need to be done to identify the kind of asphalt used. The high amount of cracking is likely due to other factors such as aging tendency, rheological behaviour and ductile strain intolerance. Section F recorded 1.68 % of WEO in the neat asphalt cement and an insignificant amount for the recovered asphalt cement.

However, this trial section has the longest pavement crack length of 79 m/500 m. This cracking on this trial can be attributed to the significant amount of air void determined for this section as shown in Table 4.1. The presence of air voids leads to the penetration of air, water, and frost from the surface layer of the pavement to the layers beneath it, causing the oxidation and hardening of the asphalt cement and eventually, resulting in premature and excessive cracking of the pavement. Section G recorded the highest percentage waste engine oil residues of 9.28 % for the virgin asphalt binder but only 1.88 % for the recovered asphalt cement. However, the distress survey analysis shows that this pavement is free from any pavement cracking. This could be due to the fact the wrong asphalt cement was supplied to the laboratory for further testing and this asphalt binder was not the one used for the construction of this trial section. However, it should be noted that other areas of the general contract of which the trial formed just a small part cracked excessively in early life so it is likely that the asphalt cement delivered by the contractor was used in these parts.

Considering all this information it can be concluded that the presence of zinc, which indicates the addition of waste engine oil residues, is associated with the occurrence of premature and excessive low temperature distress of asphalt cement pavements. This is because waste engine oils are known to increase the rate of physical hardening and weakening of the asphalt-aggregate interface [76], hence the addition of waste engine oil to the asphalt binder either directly or indirectly can be blamed partly for the poor performance of some of the trial sections on Highway 417 and other major roads in Canada. It can be concluded that trial sections which were found to contain zinc, and hence the addition of waste engine oil residues, have all cracked severely except for Section E, whereas trial sections without any pavement cracking showed insignificant or no zinc present except for section G.

### **4.3 Infrared (IR) Analysis**

In order to draw reasonable conclusions in this research with respect to the effect of aging on the performance of the binder, a comparative study was performed to investigate the extent to which laboratory aging processes (rolling thin film oven, RTFO, and pressure aging vessel, PAV test methods) are able to account for the aging processes that take place or have taken place in the field for about 5-6 years of the asphalt cements in service. To make a validation of all the various binders, it was necessary to determine that the differences in performance of the asphalt cements observed are actually as a result of differences in performance-based properties and are not confounded by the failures or shortcomings from the laboratory aging methods. IR spectroscopy was used to account for the validation of the RTFO and the PAV methods in comparison to the field aged asphalt binders.

To be able to compare the oxidation caused by the laboratory aging processes and the oxidation that has occurred on the field for five to six years, IR spectroscopic studies were used as a tool to investigate the relative extent to which aging by rolling thin film oven test and pressure aging vessel test method are able to correlate effectively with the aging that takes place on the field. As noted in the previous section, oxidation results in the hardening of the binder and consequently leads to progressive premature and permanent cracking of the pavement. Meanwhile, it is not appropriate to predict a binder's relative changes in performance from its oxygen uptake and content since each asphalt cement reacts differently to oxidation, depending on the crude oil from which it was obtained and other environmental conditions [87]. This causes the performance of the asphalt to be dependent on its source and constituents.

It is expected that asphalt cements from the same source and with the same modifiers and modification processes, and with the same relative rate of oxidative aging, both in the laboratory and in the field, would show similar performance in service and correlate well with all the laboratory test methods. On the other hand, it is expected that such asphalt cements will show significantly different performance in service if they exhibit different oxidation rates, even though they are from the same source and of the same modifiers. IR spectroscopy analysis is basically used to determine the functional groups in organic and inorganic materials. It has also been used to monitor the oxidative changes (chemical hardening) of asphalt cements through the determination of carbonyl (C=O) functional groups at wavenumbers of 1655-1700  $\text{cm}^{-1}$  for many years. The formation of carbonyl compounds is the main oxidation product of asphalt upon exposure to air (oxygen). Other minor oxidation products of asphalt are sulfoxides, formed by the reaction of the sulphur in the asphalt with oxygen. Another functional group of primary importance to the rheological properties of asphalt is the aromatic functional group, measured from the peak around 1600  $\text{cm}^{-1}$  [84].

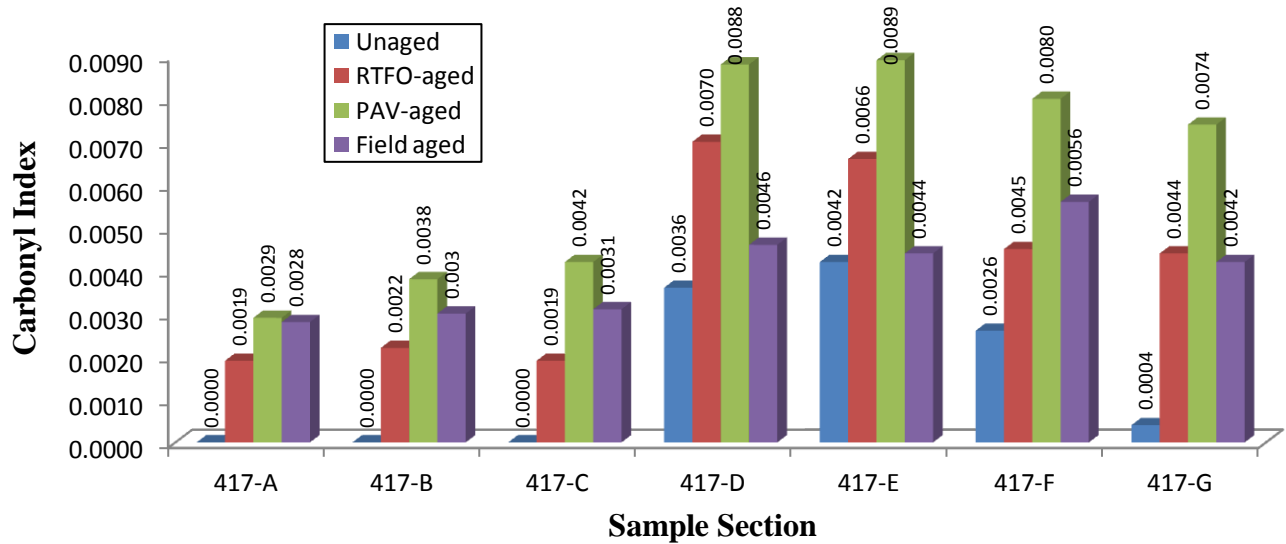


Figure 4.4: Carbonyl indices for Highway 417 trial sections.

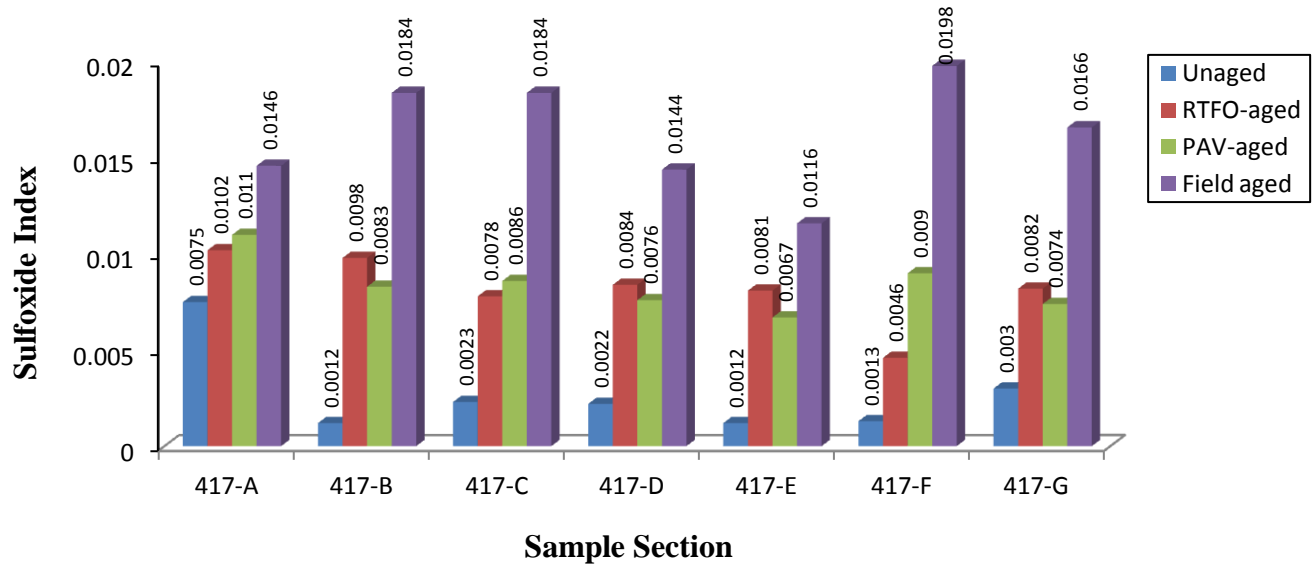


Figure 4.5: Sulfoxide indices for Highway 417 trial sections.

Figures 4.4 - 4.8 show the indices for all the functional groups of interest to this thesis. The data give several important insights to the oxidation of the asphalt cements with respect to their performance. Figure 4.4 shows the rate of oxygen uptake and oxygen content for all the aging processes for all the trial sections. The oxygen content in all the seven sections increases from unaged to RTFO-aged and then to PAV-aged residues, indicating that oxidation of asphalt depends on both time and temperature. It was expected that the oxidation rate of the field-aged asphalt cements would have been equal to or greater than the PAV-aged residues as what happened in section A, since the PAV aging process is used to simulate the oxidation of the pavement after being in service for 5-10 years. The lower values of the carbonyl indices for the field age residues can be attributed to poor extraction processes, since most of the asphalt cement formed gel structure with the solvent making it difficult if not impossible to extract from the asphalt cement-aggregate mixture of all the asphalt cements. It can also be explained by the fact that asphalt cements were recovered from the top 5 cm of the pavement while most of the aging might have occurred in the top 1 or 2 cm of the surface layer. Hence, the differences in the

carbonyl indices could be explained by the differences in aging between the surface layers and the actual sample used for analysis.

The relative high values of carbonyl indices for Sections D to G can be due to the presence of the high values of air voids obtained from these sections, and as a result leads to premature and permanent cracking of the pavement. It is therefore not surprising that Section E recorded the highest carbonyl index for the PAV residues and the second highest for the field-aged residues, and it is witnessed as the most cracked trial section among all the seven trial sections. Section F showed the highest oxygen content in the recovered asphalt cement residues. This is directly related to the higher air void content determined in this section, and it is therefore not surprising that this section has also cracked severely. The presence of higher air void than the desirable amount needed in an asphalt pavement, leads to intake of oxygen into the asphalt pavement, which causes the asphalt binder to harden and crack subsequently. Sections A to C shows relatively lower carbonyl indices compared to the other sections and Sections A and B have shown no signs of any cracking as of summer 2010 according to the distress survey analysis. Section C has only a crack length of 8m/500m, which is of less significance to pavement deterioration. Section G even though has no cracks, exhibited significant carbonyl index. This anomaly can be explained by using the rheological and ductile fracture properties of the binder, as detailed in sections 4.4 to 4.8.

The order for the oxygen content in asphalt cements according to their sulfoxide indices for Sections B, D, E and G is unaged < PAV-aged < RTFO-aged < field-aged, whereas that of Sections A, C and F is unaged < RTFO < PAV < field aged, as shown in Figure 4.5. This shows that the PAV aging process which is supposed to predict the oxygen content after 5-10 is unable to do that. This indicates that the PAV aging process needs to be reviewed to make the necessary

modifications so that it will be able to simulate correctly and accurately the aging process that occurs on the field after 5-10 years in service. The sulfoxide indices for the field-aged residues are relatively all the same, indicating that the conversion of sulphur in the asphalt cements to sulfoxides has relatively little significance on the performance of the binder in service. Sections A to C and G have relatively few or no cracks while sections D to F show significant cracks. Hence the individual sulfoxide index would not be able to account for these differences since the indices for all the seven sections for the field-aged residues are relatively the same.

The analyses made so far show that the addition of asphaltenes (carbonyl and sulfoxides) are not the only parameters to predict low temperature cracking of asphalt pavements. However, the manner in which such additional asphaltenes harden the asphalt cement is also considered important. “In highly paraffinic oils, the asphaltenes produced upon oxidation will easily form a gel structure which is unable to relax thermal stresses. In contrast, more aromatic oils will allow the asphaltenes to remain better peptized and thus able to relax thermal stresses [89].” In other words, the presence of aromatic oils are essential for the performance of the asphalt cement pavement by peptizing the asphaltenes present in the asphalt binder so that it will be able to relax thermal stresses. Figure 4.6 shows that the aromatic index also plays important role in the performance properties of the asphalt cement in service. It is observed that time and temperature, which are the basic parameters that determine the degree of aging of asphalt cements, has no influence or effect on the aromatic properties of the asphalt binder. This is because the aromatic indices obtained for all the unaged, RTFO-aged, PAV-aged and field-aged residues are relatively the same for each binder, but are different from one trial section to another. Considering the data from the field aged residues, which represents exactly the performance of the pavements in service, a number of insights can be highlighted. Sections A and G recorded relatively higher



values of the aromatic index, indicating the presence of high amount of aromatic oils in these trial sections, which is able to peptize the asphaltene content in the binder to form sol-type asphalt structure, which is able to relax thermal stress. It is therefore not surprising that these sections are free from any pavement distresses. Sections B and C even though recorded low values of aromatic indices, have not cracked significantly. This can be attributed to the reduced rate of oxidation of the binders used for construction of these trial sections as shown in Figure 4.4, and also good rheological and ductile strain tolerance characteristics of these binders as discussed in the subsequent sections. Sections E and F although recorded relatively high values of the aromatic indices, have cracked severely. This severe distress is due to the higher rates of oxidation found in these trial sections, especially for Section F due to the higher amount of air void content found in this section. This shows that the asphaltene content in the binder is higher than the aromatic oils, and hence causing the aromatic oils not being able to peptize the asphaltene completely, leading to the formation of gel-type asphalt structure, therefore leading to the binder's inability to relax thermal stress. Finally, section D recorded relatively low value of the aromatic index, hence it is therefore not surprising that this trial section has cracked quite significantly.

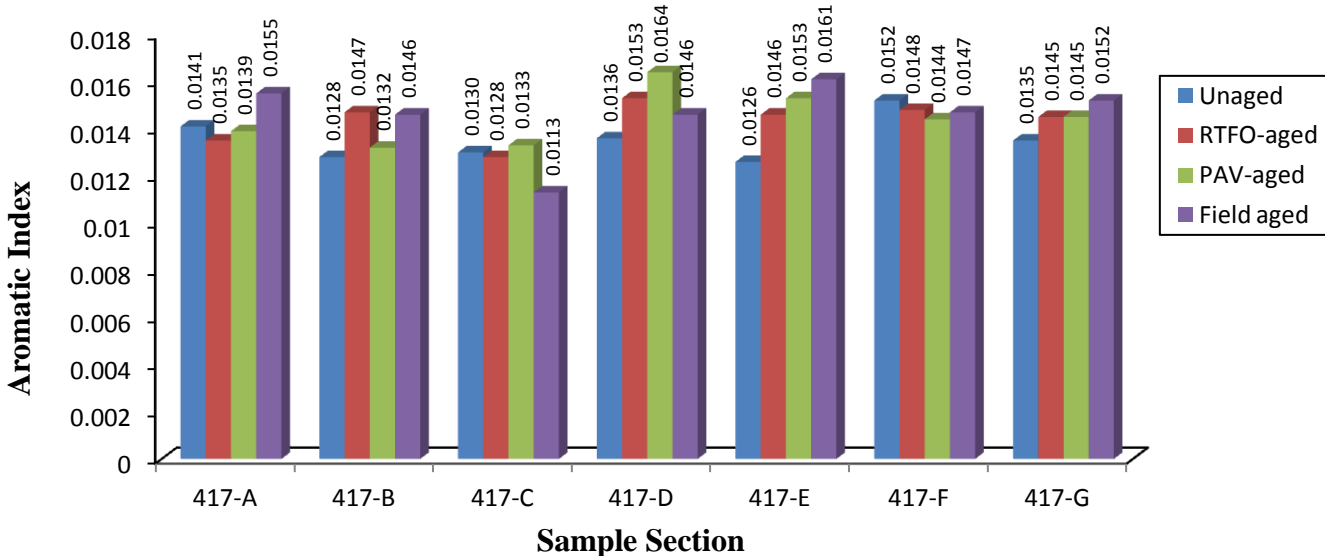


Figure 4.6: Aromatic indices for Highway 417 trial sections.

Asphalt binders modified with polymer have been used in high stress locations such as busy intersections, vehicle weighing station, parking lots and airports with much success [90]. Polymer modifiers are added to the binder to improve performance-based properties of the binder such as rutting resistance, thermal cracking resistance, and thermal fatigue cracking resistance and stripping resistance. Polymer modified asphalt cements are more elastic and durable with greater temperature stability. Styrene-butadiene-styrene (SBS) is one of the most commonly used polymer to modify asphalt cement because of its elastomeric triblock copolymer that improves elasticity of the asphalt cement to prevent permanent rutting and cohesive failure [89]. The presence of SBS was also determined to investigate the effect that is exerted on the performance of the asphalt cement in service by the addition of polymer modifiers to it. As can be inferred from the data in Figures 4.7 and 4.8, aging processes have no effect on the polymer added to the binder. Sections A, B and C were modified with 3 % SBS, about 6 % radial SBS and 7 % D-1101 linear SBS, respectively. From Figures 4.7 and 4.8, Section C had the highest

butadiene and styrene indices, which indicates higher amount of SBS contained in that pavement trial. This trial section had only about 8 m/500 m of pavement cracking as of summer 2010. The styrene and butadiene indices of Section B are almost twice the magnitude of those of Section A, indicating that the amount of SBS added to the asphalt binder in Section B is higher than that added to the binder used in Section A. Even though both trials sections have different concentrations of SBS, none of these trial sections have experienced any form of pavement distresses according to the 2010 distress survey analysis. Sections D and E were modified with respectively 3.3 % and 2.1 % of Elvaloy 1052 RET and 0.4 % PPA. It is therefore not surprising that no butadiene and styrene indices were found in these trial sections. However, SBS peak was found in an extraction of different core samples for Section D. It is therefore a further confounding issue for the interpretation of the performance of this trial section. According to the distress survey data, both Section D and E have cracked significantly. This crack may be attributed to the presence of the PPA, since studies have shown that the addition of PPA to modify the asphalt binder causes permanent deformation and low temperature cracking of the pavement. Sections F and G were modified with unknown concentrations grade of SBS. Both trial sections have almost the same styrene and butadiene indices, which explains the fact that the same amount and concentration of SBS were added to the asphalt cement used for both sections. However, section F has cracked significantly but Section G has no observable cracks. The pavement cracking observed on F may be due to presence of high percentage air void found in that trail section as discussed earlier. These results confirm the influence of the addition of polymer modifiers to improve the performance-based properties of the asphalt cements, as performed by other research studies [88-89].

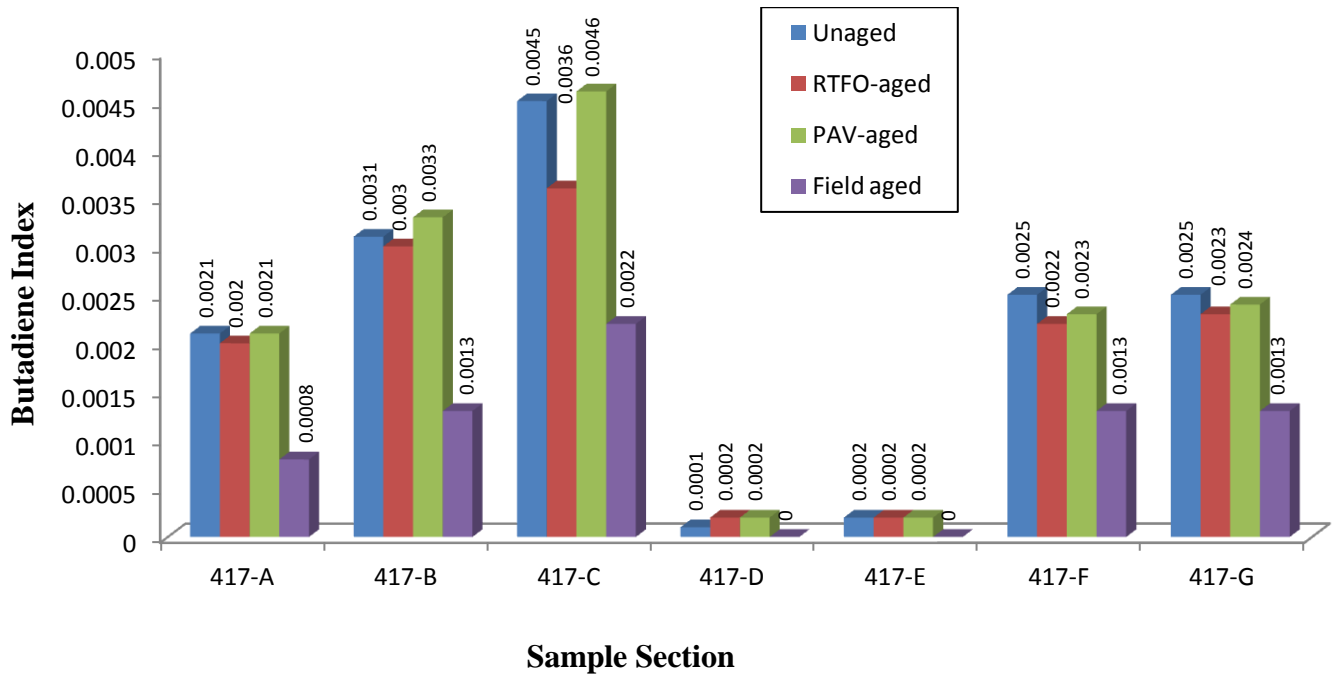


Figure 4.7: Butadiene indices for Highway 417 trial sections. (Note: SBS peak was found in an extraction of different core samples for Section D. It is therefore a further confounding issue for the interpretation of the performance of this trial section.)

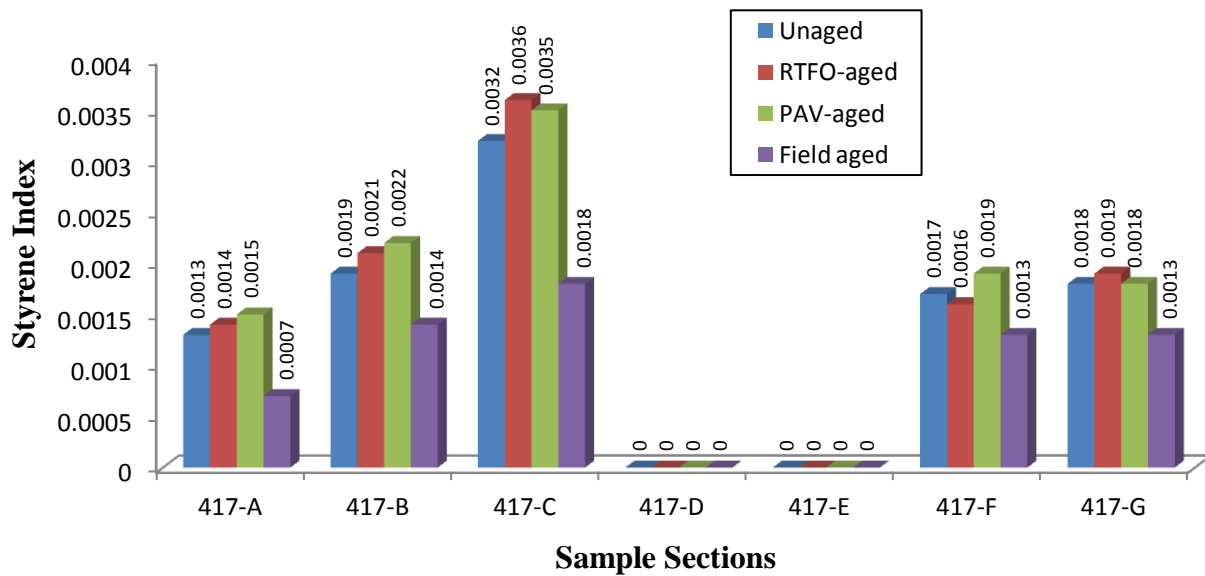
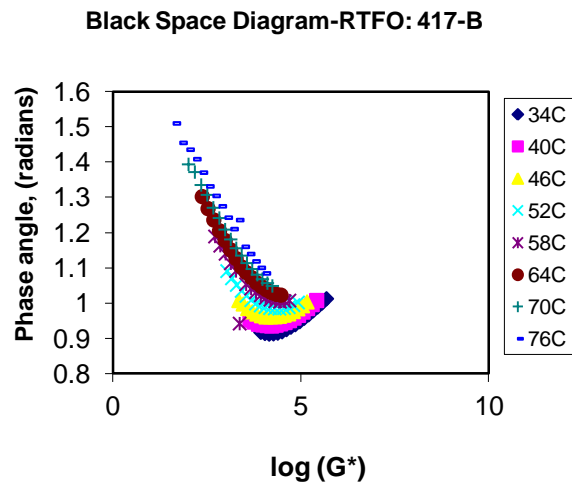
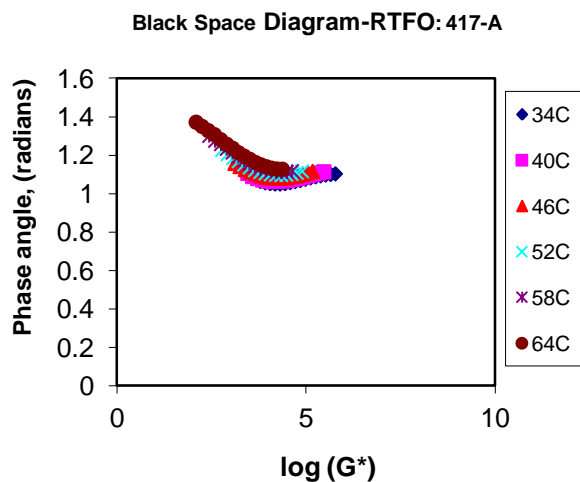


Figure 4.8: Styrene indices for Highway 417 trial sections.

## 4.4 Dynamic Shear Analysis

### 4.4.1 Black Space Diagrams

Rheology involves the studies of the deformation and flow of materials. It helps to determine whether an asphalt cement consists of homogeneous (single phase) or heterogeneous (multiple phase) system. Asphalt binders with single phase system are said to be rheologically simple, whereas those with two or more phase system are considered as rheologically complex. The Black space diagram has been used to determine whether an asphalt cement is rheologically simple or complex. Black space diagrams in which there is a smooth progression of curves at different temperatures, where the curves essentially follow the same pattern without any overlaps or otherwise anomalous behaviour, the material is said to be rheologically simple. On the other hand, if the Black space diagram is having overlaps and discontinuity at different temperatures and there is no special pattern, the material is considered to be rheologically complex. It is much easier to predict and quantify the rheologically simple material, but very difficult if not impossible to do the same for a rheologically complex asphalt binders, since their behaviour does not follow that of a conventional viscoelastic material.



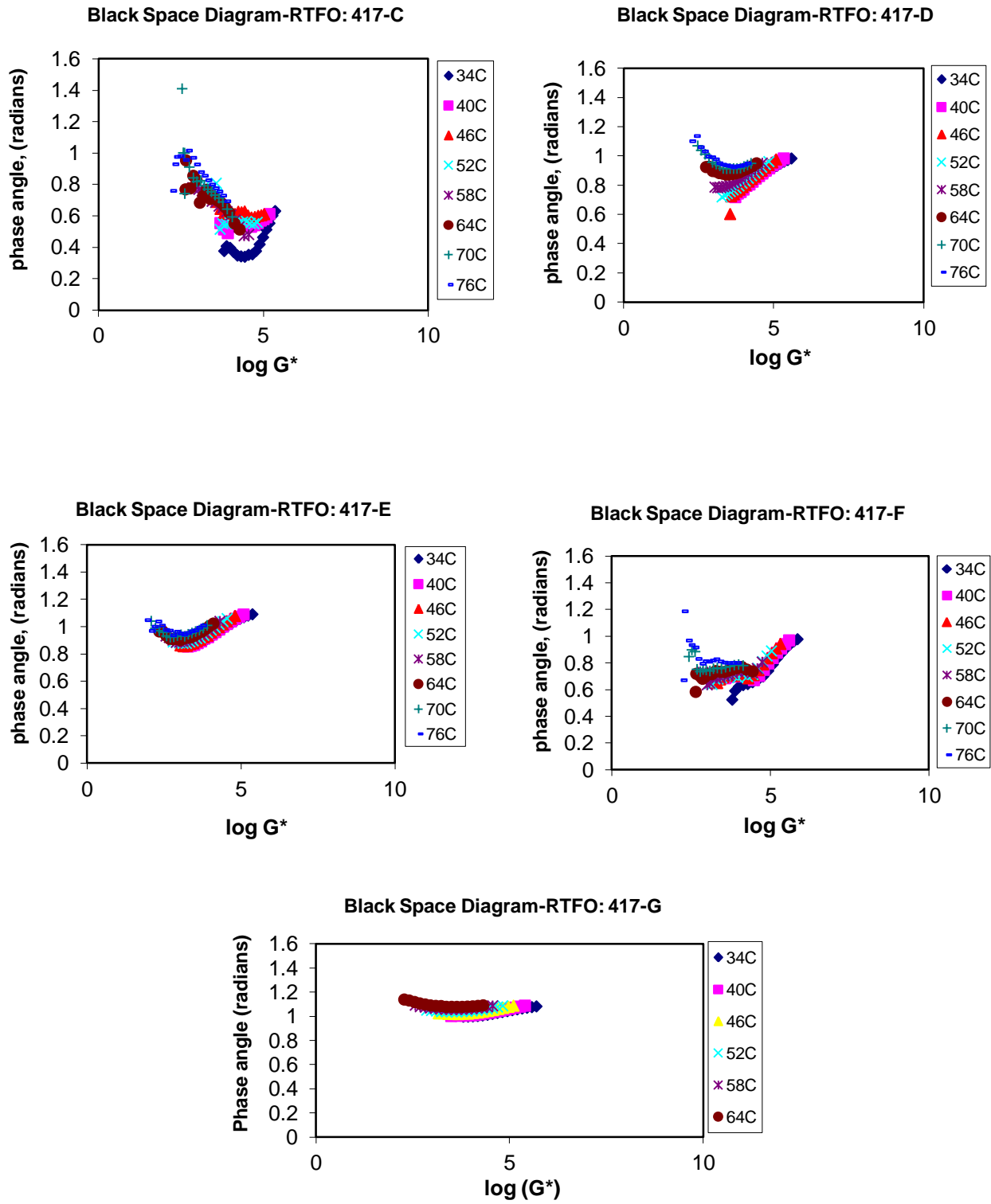
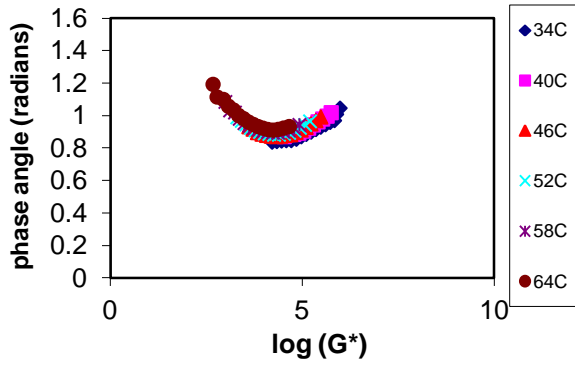
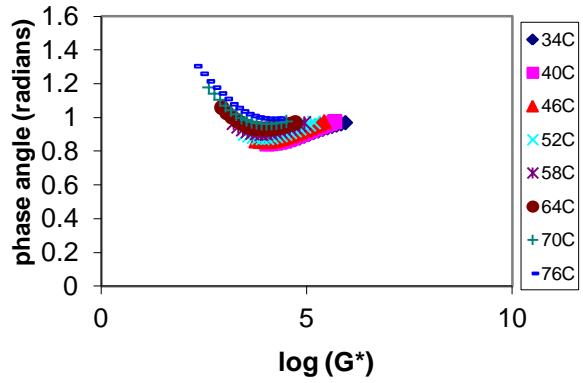


Figure 4.9: Black space diagrams for RTFO aged binders of trial sections on Highway 417 [82].

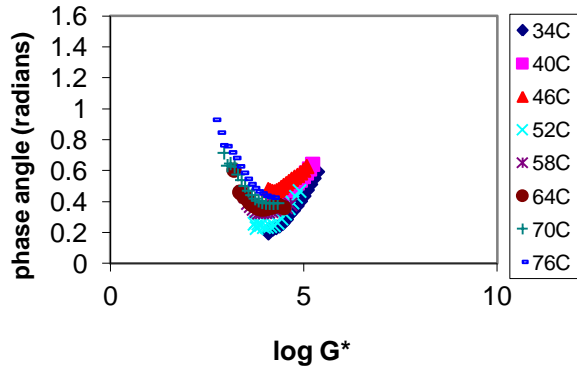
Black Space Diagram-PAV: 417-A



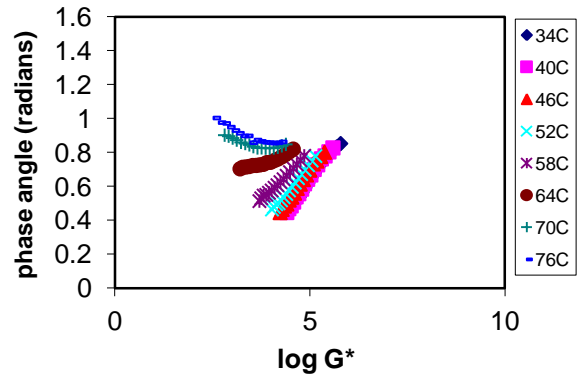
Black Space Diagram-PAV: 417-B



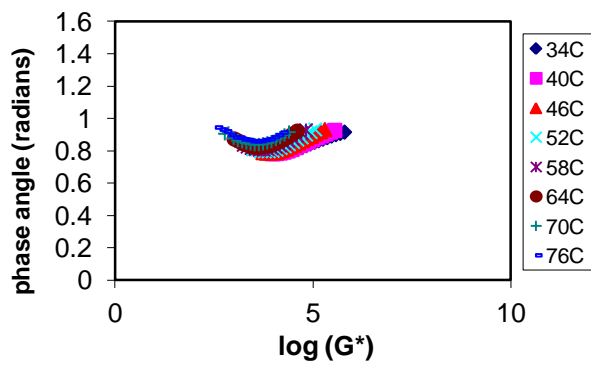
Black Space Diagram-PAV: 417-C



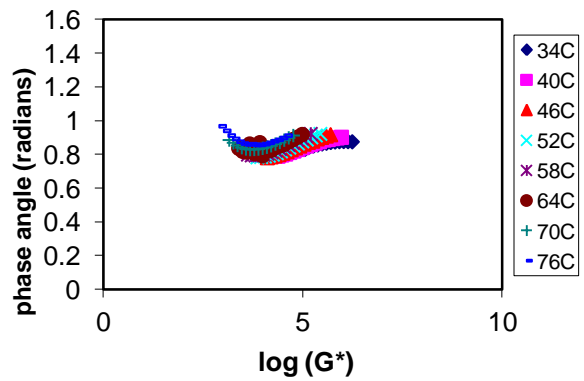
Black Space Diagram-PAV: 417-D



Black Space Diagram-PAV: 417-E



Black Space Diagram-PAV: 417-F



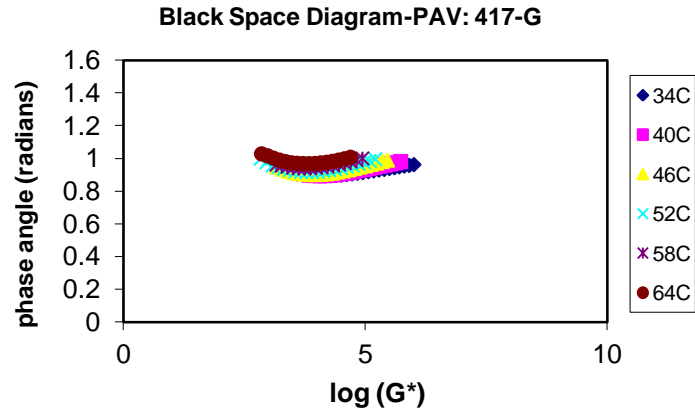
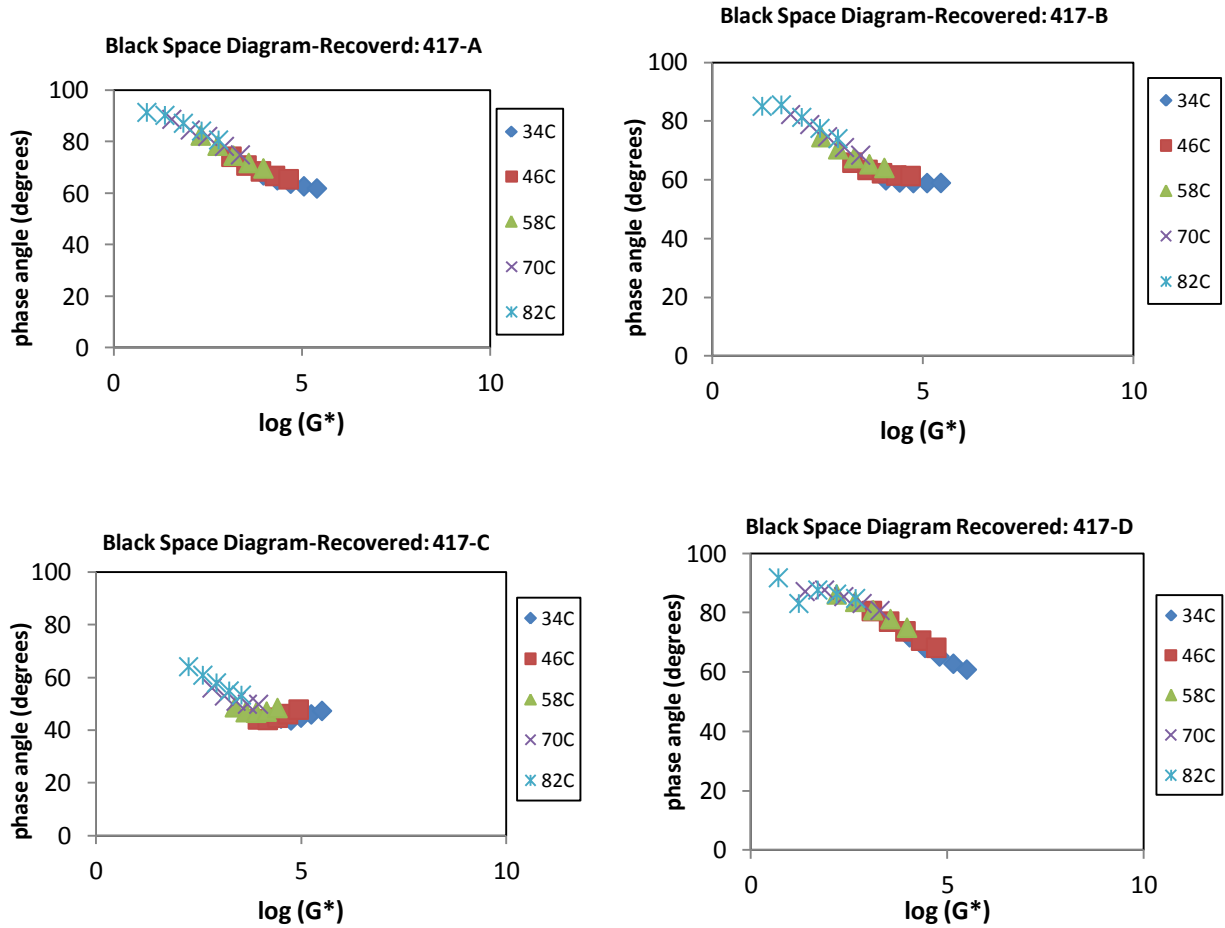


Figure 4.1.0: Black space diagrams for PAV aged binders of trial sections on Highway 417 [82].





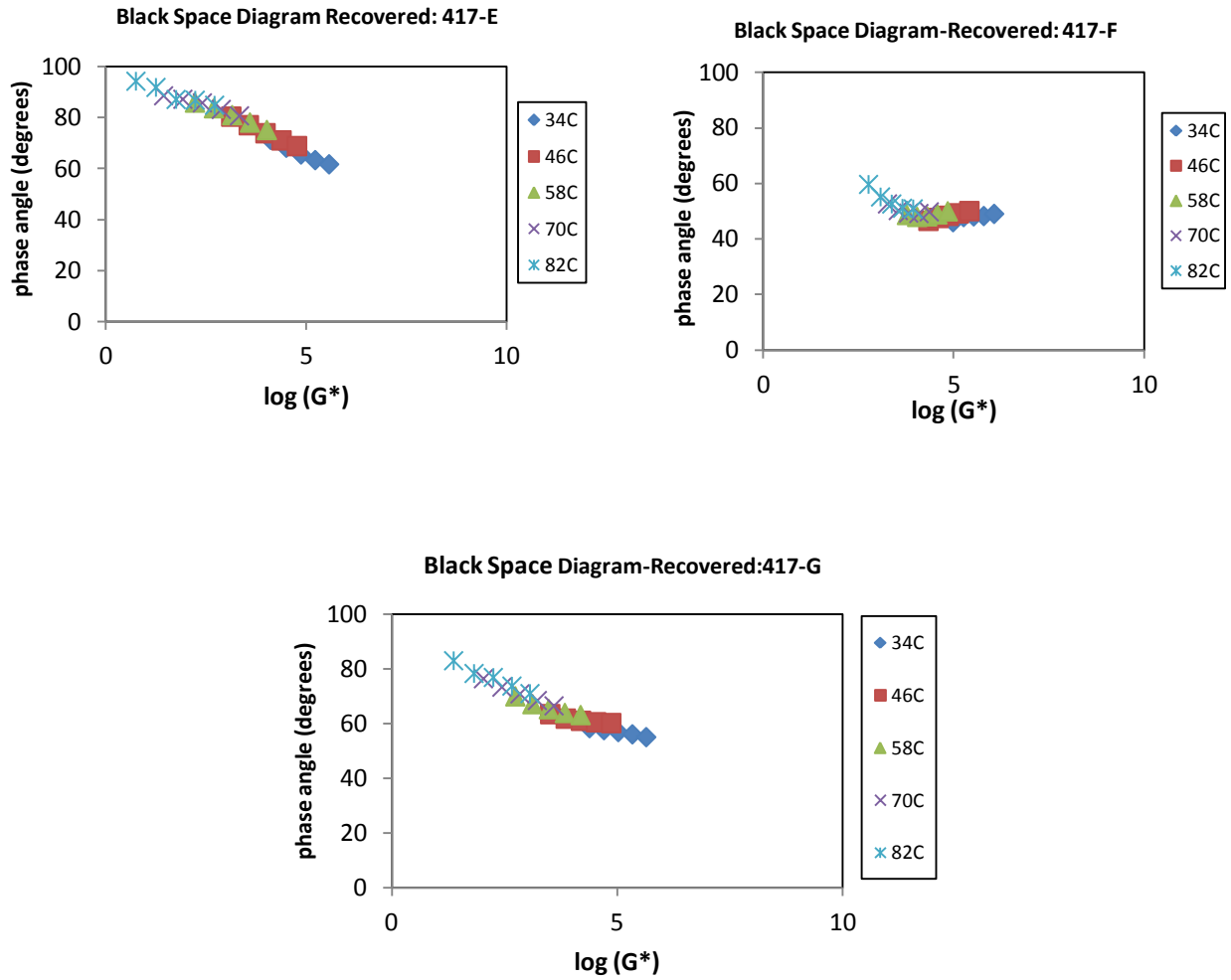


Figure 4.1.1: Black space diagrams for field aged binders of trial sections on Highway 417.

Considering the individual Black space diagrams for both laboratory and field aged samples, it can be seen that Section A was constructed with a rheologically simple asphalt cement, whereas all other sections were constructed with rheologically complex asphalt cement. The complexity of Section C, D and F may be due to the addition of polymers to those binders, which causes phase separation of the asphalt cement. In rheologically simple binders, there is a smooth transition between the curves of different temperatures without any overlaps between the curves

as shown in the Black space diagrams for Section A. There is an increase in phase angle while the complex shear modulus decreases at higher temperatures as expected, as the asphalt material tends to be more viscous fluid. However, in the Black space diagrams for Sections C, D and F, there is a discontinuous pattern between temperatures and overlaps from one temperature to another, which helps to describe how their behaviour does not follow normal viscoelastic character. The performance of asphalt cement in service depends on its physical and rheological properties, which in turn depends on the viscoelastic properties of the binder. It is expected that asphalt cement used for road pavement construction be viscoelastic at all temperatures, but this does not usually occur at extreme low and high temperatures. Based on the viscoelastic character of an asphalt binder, it is expected that asphalt that perform better in service should have lower values of phase angle at higher temperatures and higher values at lower temperatures.

By measuring the phase angle, it is very powerful to compare the behaviour of different asphalt cements. When temperature and/or strain are increased, asphalt cement encounter shear thinning-type behaviour and become a viscous fluid, hence there is an increase in the phase angle at higher temperatures and strain. It is expected that the phase angle will decrease with age when making a comparison with the effect of oxidative aging. As a result of age hardening, the asphalt stiffens over time and the storage modulus of the binder increases and it becomes more labile to elastic recovery from a given strain, which reduces the time lag and thus decreases the phase angle. At lower frequencies, the phase angle becomes larger, since the sample has much time to dissipate an amount of the applied strain as flow, delaying the response to stress. As the frequency increases, the asphalt binder does not have much time to dissipate as much of the strain as viscous flow and hence the time lag is decreased and eventually leads to reduction in the value of the phase angle. The Black space diagram is used as a graphical representation to

determine the changes in the phase angle with the complex modulus, with the phase angle being higher at higher frequencies than at lower frequencies for stiffer materials.

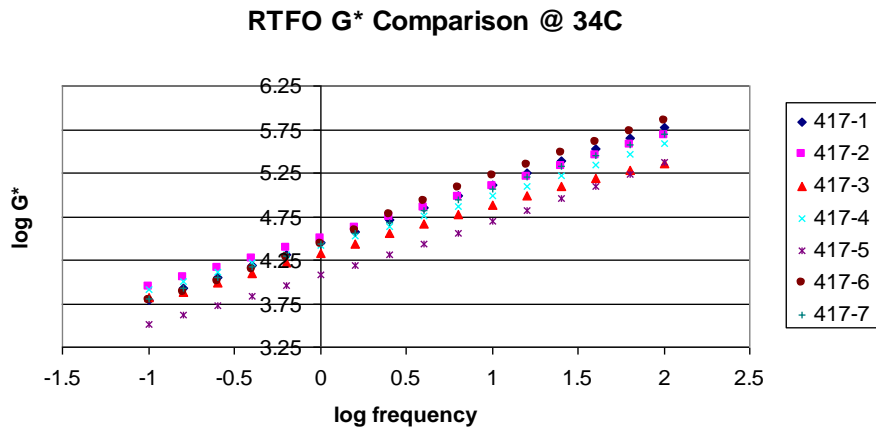
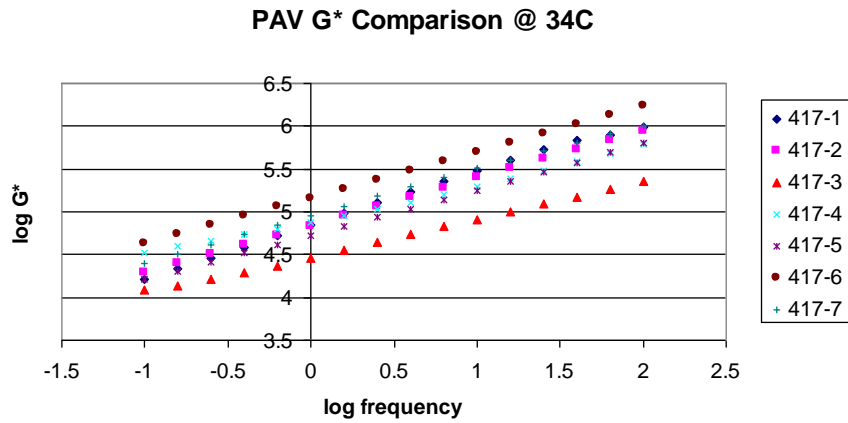
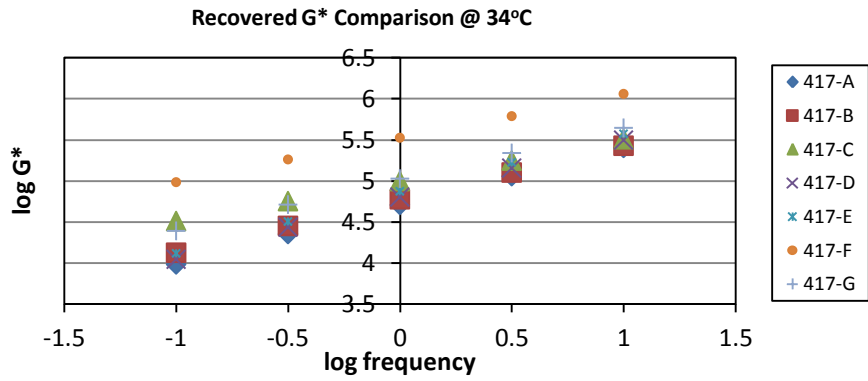
As can be seen from the Black space diagrams in Figure 4.1.0, for the PAV aged residues (representing the aging that occurs in the field after 5 to 10 years in service), Sections A, B and G show higher phase angles as expected at their maximum grading temperatures and these sections have experienced no cracking according to the 2010 distress survey data. Section C shows relatively moderate phase angle compared to the other sections and this section has experienced little cracking just as expected. Sections D to F have cracked significantly and they all showed relatively small phase angles at reduced frequencies. For a purely viscous material, it is expected that the phase angle will be approximately equal to  $90^\circ$  as the frequency increases. From the field-aged Black space diagrams, Sections A, B and G (which have not experienced any cracks and are virtually free from any pavement distress), recorded higher values of phase angles at the reduced frequencies, indicating their ability to convert the applied strain energy to viscous flow which in turn increases their loss modulus and subsequently, their phase angles, which help them to withstand low temperature cracking at lower temperatures. Sections D and E (which have cracked quite significantly), also showed relatively high values of phase angles at both lower and higher frequencies. This effect makes the binder in these sections being completely viscous and stretches without recovery at high temperatures and applied strain/stress, which leads to permanent deformation of the pavement. It is therefore not surprising that these trial sections have undergone some severe cracking. Sections C and F recorded relatively lower phase angles at lower frequencies, and these pavements have undergone some moderate cracking. By observing their individual phase angles at  $34^\circ\text{C}$  on the Black space diagrams for the field aged asphalt cements, it is expected that all the binder will exhibit viscoelastic character at

this temperature. Sections A, B and G showed phase angles within the range of 57-67°, which pronounces their viscoelastic nature at the said temperature, and hence they are able to relax stress and also recover easily to avoid low temperature cracking and rutting of the pavements. Sections D and E with the highest cracking distance showed more of viscous character, indicating their susceptibility for permanent deformation at higher temperatures. However, Section F which has also undergone severe cracking showed good viscoelastic behaviour. This contradiction is due to the high air void content determined in this section, causing the asphalt cement to undergo age hardening and subsequent cracking of this pavement.

#### **4.4.2 Master Curves**

Another important parameter that can be used to characterise the rheological characteristics of an asphalt binder is the use of the master curve. The master curve involves a graphical representation of the storage, loss or complex modulus with change in frequency. Even though master curves can be used to demonstrate the viscoelastic nature of a material, they are of no importance in characterization and comparison of materials if they are individually constructed. It is of much value to compare one master curve of one material to another in order to completely comprehend and appreciate the viscoelastic characteristics of a material, and the effects of aging on that material. In this investigation on Highway 417, a comparative study is performed on the behaviour of seven different test sections to determine the relative behaviour of each individual asphalt and the effect of different modifiers on the network structure of the asphalt binder. By comparing the modulus of the samples at the two extreme temperatures of testing, it is possible to observe the behaviour of the asphalt at these temperatures, and the changes in modulus is an indication of a shift to viscous nature as the temperature increases. At the lowest testing temperature, 34°C, it will be meaningless to monitor the behaviour of the individual asphalt

cements as the temperature is increased. Figure 4.1.2 shows master curves combining the recovered, unaged, RTFO and PAV aged samples, which is used to compare their complex shear modulus at 34°C.



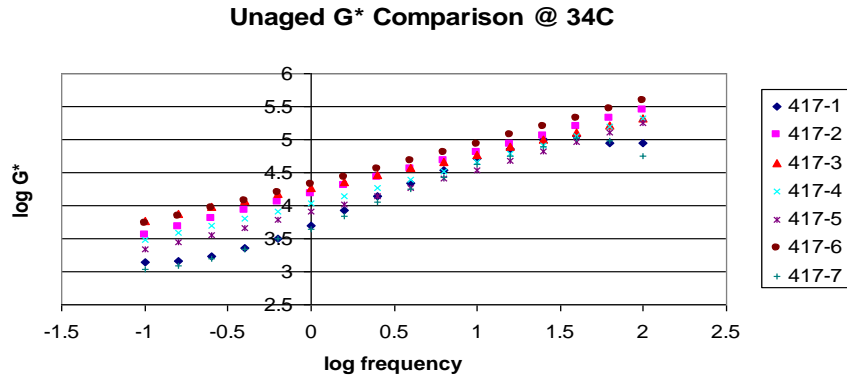
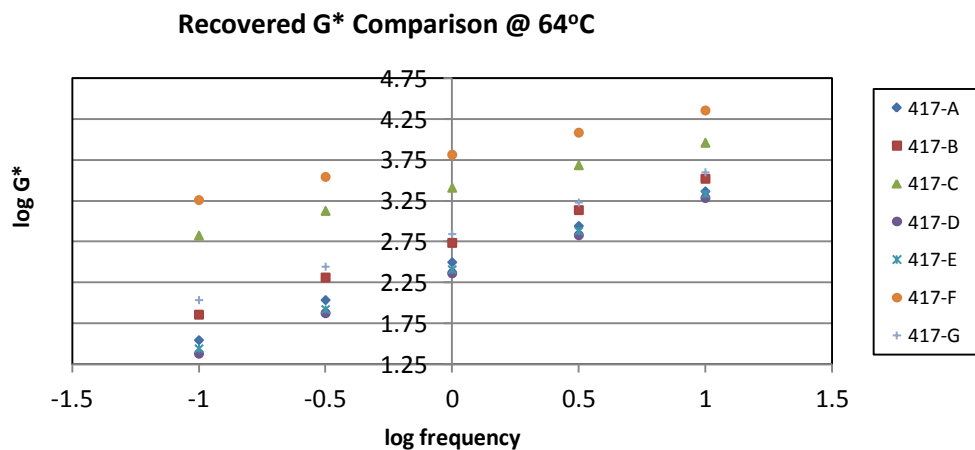


Figure 4.1.2: Complex Modulus curves of Field, PAV, RTFO, and Unaged samples at 34°C [82].

It can be observed that the modulus of each section increases with the age of the sample, with the unaged sample being the softest (low moduli) and PAV and field aged being the stiffest (high moduli). This observation is due to oxidation that takes place in the asphalt cement leading to age hardening of the binder and hence becoming stiffer. The field-aged samples of all the trial sections showed higher modulus compared to the PAV-aged samples, which indicates that the recovered samples have undergone much age hardening and hence stiffer compared to the PAV-aged samples. This shows that the PAV method needs to be looked at again, and make any necessary modifications so that it will be able to correlates perfectly to the aging that takes place in the field. The difference between the field- and the PAV-aged results and that of the unaged can be attributed to the additional intermolecular bonding that occur within the network of the asphalt molecules, resulting in a much rigid structure, capable of elastically rebounding from a strain. However, during age hardening, most of the viscous component is reduced to nothing or lost, and hence the material becomes more brittle, and as the strain increases with frequency of the strain, it becomes more susceptible to cracking, as the sample is able to distort and convert a portion of the strain energy into viscous loss energy, as a result of the strain being too infrequent.

Greater amount of the strain energy is recovered elastically at higher frequencies, since the samples are not given much time to convert the energy to viscous loss energy, thus increasing the modulus by as much as two orders of magnitude at higher frequencies. The change in viscoelastic behaviour is truly clear when the master curves for the complex modulus is monitored at higher temperatures. At higher temperatures, the weak intermolecular bonds within the asphalt binder begin to break down and the viscous nature begins to dominate, with the storage capacity reducing gradually and continually at each increase in temperature. As can be seen from the data in Figure 4.1.2, the complex modulus is greater at higher frequencies and lower at lower frequencies, just as observed at lower temperatures. However, asphalt flows much more easily at higher temperatures, and energy from the strain is converted more readily to viscous loss energy than elastic recovery, which in turn reduces the storage modulus. This decrease in the storage modulus eventually reduces the complex shear modulus in addition, as shown in the complex modulus master curves in Figure 6.1.3 at 64°C.



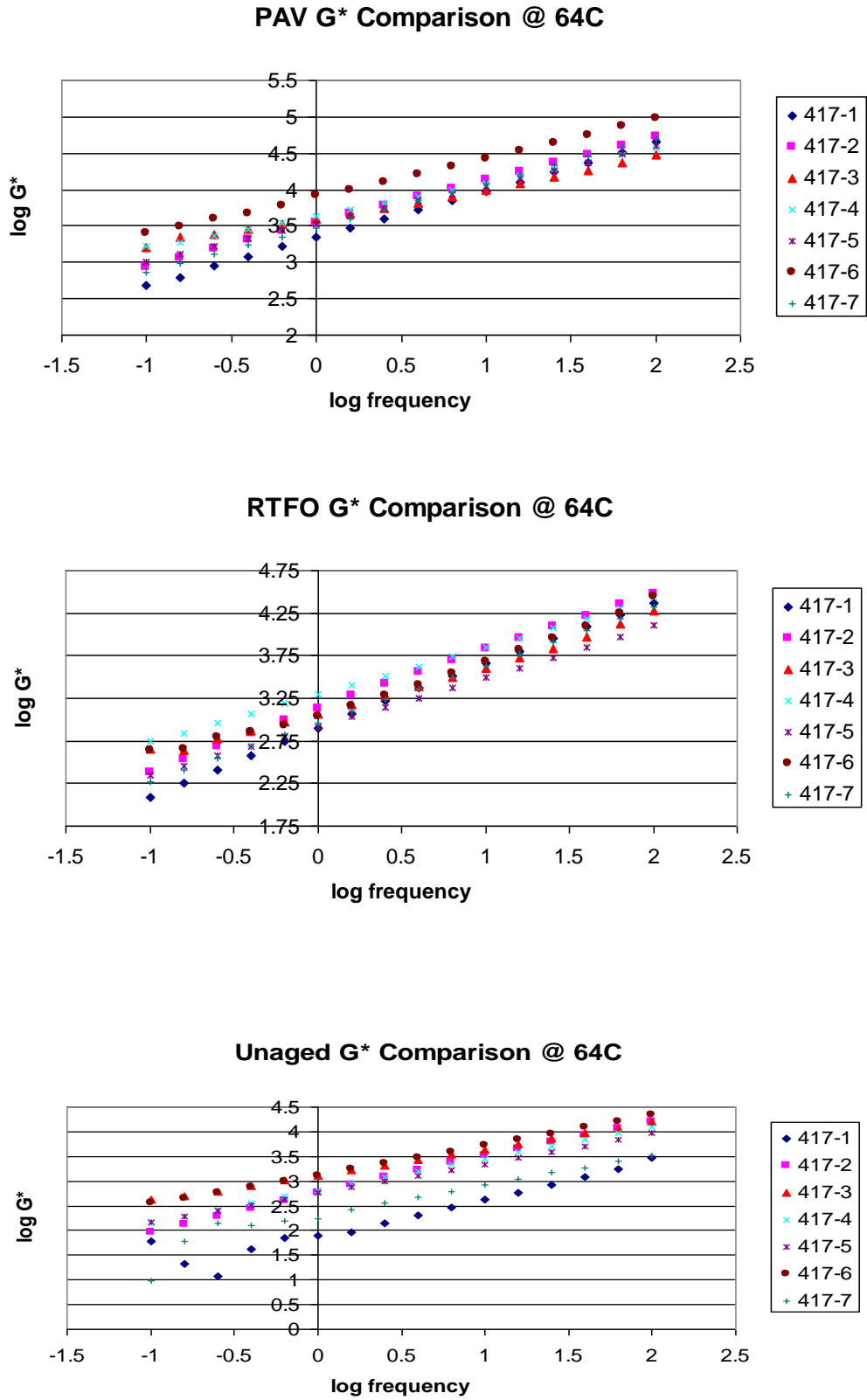


Figure 4.1.3: Complex Modulus curves of Field, PAV, RTFO, and Unaged samples at 64°C [82].

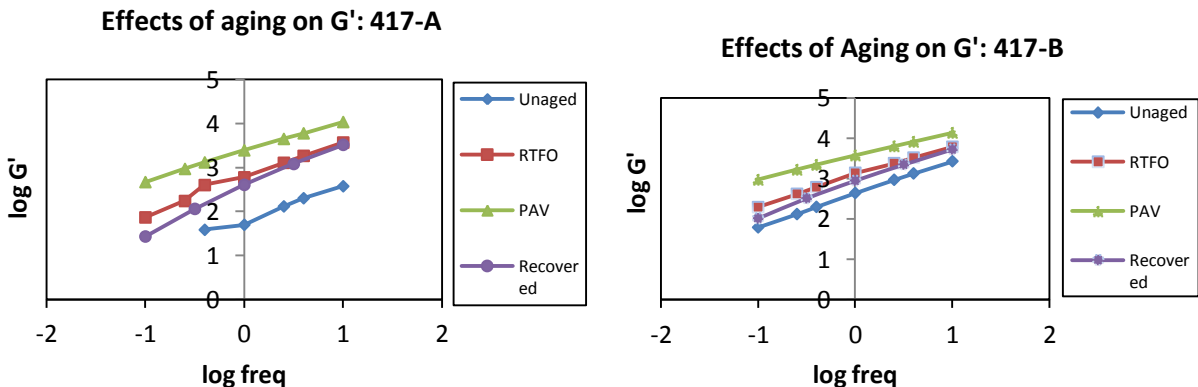


Some interesting insights can be observed about these curves. By comparing these curves to those measured at 34°C, there appears to be a seeming equality of all the binders at RTFO aged, with the range of values being not more than half of one order of magnitude, unlike the curves formed from the unaged samples at the same temperature. This could be explained by the fact that, at such elevated temperatures, the asphalt cement basically acts as viscous fluids, and hence the differences in storage capabilities between samples are equally overshadowed by the ease of flow of the material under the given strain rate. Also, considering the curves for the recovered samples at the two testing temperatures, at 34°C, the complex modulus were almost the same for all the sections except for section F, but at 64°C, the modulus is evenly spread, and one varies from the other. This occurs because, as said earlier, at such higher temperatures, the viscous component of the asphalt cement dominates and causes it to move much more easily and freely. In addition, at 64°C, the modulus for the PAV aged samples seems to be higher than that of the field aged samples. This means that age hardening is more pronounced in the laboratory aging process than the field aging process. However, recent studies have shown that the laboratory aging method is unable to account for the aging that occurs in the field after 5-10 years in service. A possible reason why the PAV aged samples have aged more than the field aged sample is that the highway was construct in 2006 with relatively good asphalt binder and good construction techniques. Another possible reason could be due to the fact that the extraction being from the top 5 cm of the road, whereas aging usually occurs at the top 2 cm of the road.

Hence, it may have not undergo much aging in the field compared to the aging in the laboratory.

### 4.4.3 Low Temperature DSR

Low temperature cracking and permanent deformation are the two major road cracking distresses experienced in Ontario and most parts of Canada as well as the northern parts of the United States. In order to compare which binder has more resistance to low temperature cracking with respect to aging, an investigation was also conducted on the trends to which the storage modulus in the asphalt binders as they undergo aging processes, since they can give early indication of the extent to which low temperature cracking correlates well with the aging of the binder. Also, another study was performed to understand and know which asphalt binder has the highest resistance to rutting at higher temperatures, since asphalt binders tend to be completely viscous during summer months and then flows much more easily without recovering to its original position or shape, leading to permanent deformation of the pavement. A comparison studies was undertaken for all the samples portraying the changes in storage modulus,  $G'$ , and loss modulus,  $G''$ , as the binder undergoes age hardening. Using this data, it is possible to predict the best quality binder, and hence the one that should theoretically and experimentally be the most durable.



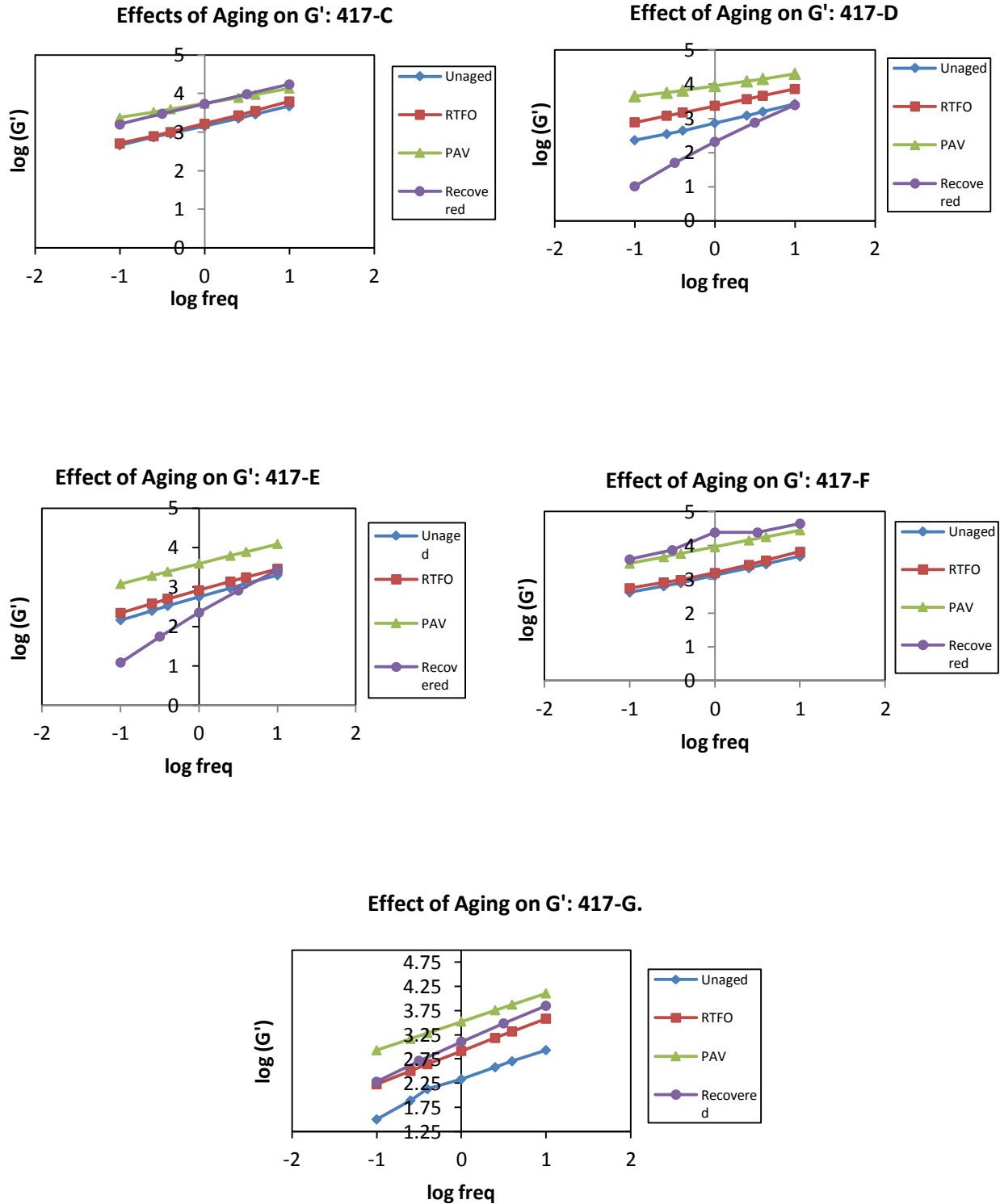
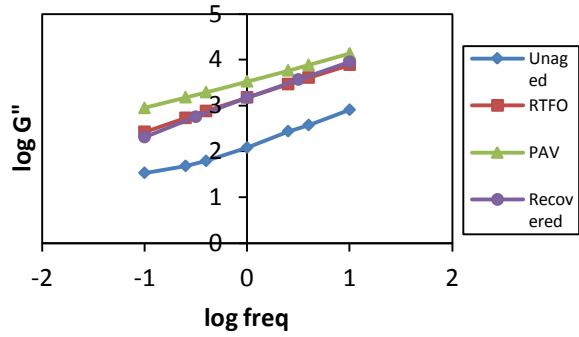
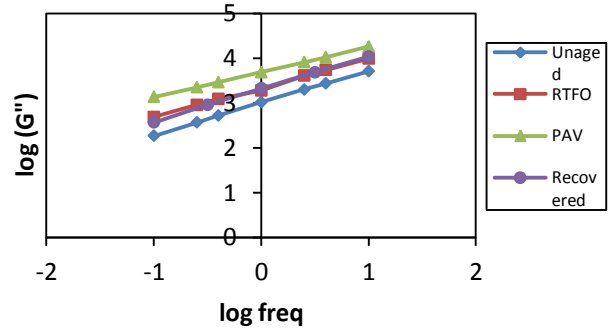


Figure 4.1.4: Comparison of loss modulus,  $G''$ , with respect to the effects of ageing

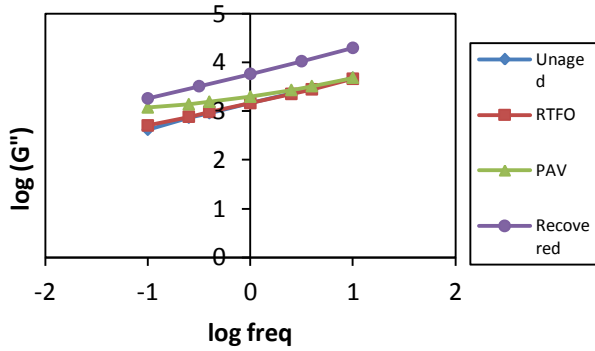
Effect of Aging on G<sup>''</sup>: 417-A



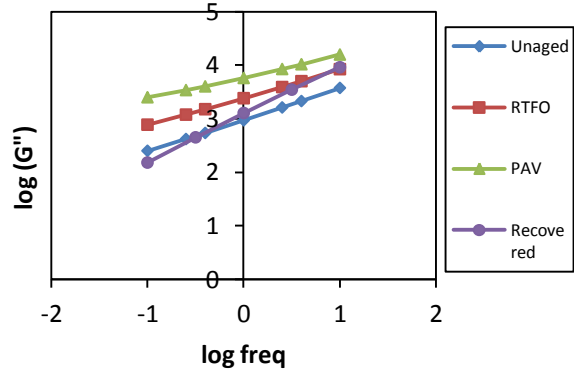
Effect of Aging on G<sup>''</sup>: 417-B



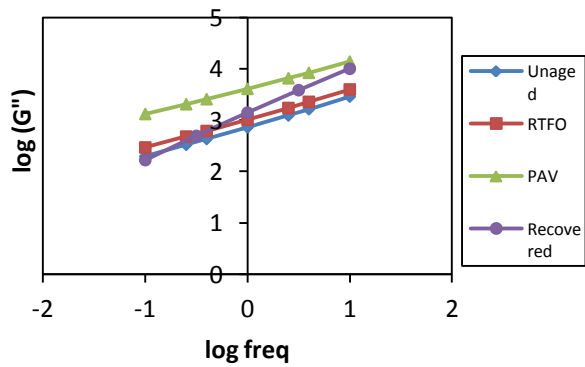
Effect of aging on G<sup>''</sup>: 417-C



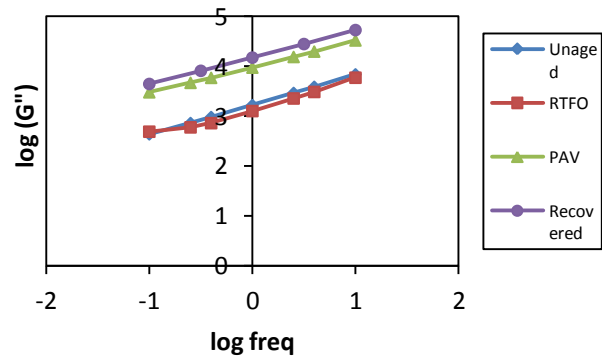
Effect of aging on G<sup>''</sup>: 417-D



Effect of aging on G<sup>''</sup>: 417-E



Effect of aging on G<sup>''</sup>: 417-F



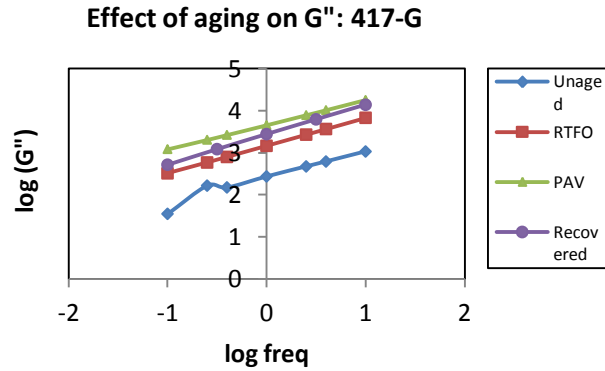


Figure 4.1.5: Comparison of loss modulus,  $G''$ , with respect to the effects of aging

Figures 4.1.4 and 4.1.5 can be collectively summarised into a table comparing the relative increase in  $G'$  and  $G''$  with age. The binder with the most similar rheological behaviour at different ages (i.e., the binders that undergo slowest or minimum aging rate) can be determined from this data. Comparison study was conducted on the RTFO-, PAV- and the field-aged binders, since they are the ones that have true meaning to aging processes. The RTFO is used to simulate aging that occurs during high temperature mixing of the binder with aggregates and other materials, while the PAV simulates the aging that occurs in the binder after 5-10 years in service. The field-aged samples reflect exactly the degree of aging in the field. The unaged samples have practically little significance since it does not represent any situation that would be encountered in the field.

The increases in  $G'$  and  $G''$  from Figures 4.1.4 and 4.1.5 are summarized in Tables 4.3 and 4.4. The changes are reported as the ratio of PAV curve to the RTFO curve in Table 4.3 and also, the recovered curve to the RTFO curve in Table 4.4, at three different frequencies, ranging from the

lowest frequency (0.1 Hz) to the highest frequency (10 Hz) common to all the three curves. The unit of the values is in percentage above the modulus value of the RTFO curve of the sample.

From Tables 4.3 and 4.4, it is evident that at lower frequencies, the aging effect is more pronounced than at higher frequencies. This is as a result of the duration of the strain, since the binder is given less time to react to the strain at higher frequencies, and hence changes in the magnitude of the modulus will be as pronounced as at lower frequencies, in which the asphalt binder has much time and the chance to behave according to its molecular character.

**Table 4.3: Relative increase in modulus with PAV age. Note: Values are in % increase**

<b>Comparison of Aging Effects PAV/RTFO</b>				
<b>Binder</b>	<b>Modulus</b>	<b>0.1 rad/s</b>	<b>1.0 rad/s</b>	<b>10 rad/s</b>
417-1	G'	30.1	18.1	11.6
	G''	17.7	9.9	5.9
417-2	G'	23.1	12.3	8.6
	G''	14.4	11.0	6.3
417-3	G'	19.8	14.1	8.1
	G''	12.0	4.0	0.6
417-4	G'	20.9	14.6	10.3
	G''	15.1	10.0	6.5
417-5	G'	23.9	18.9	15.4
	G''	20.9	16.6	13.1
417-6	G'	21.0	19.3	14.4
	G''	22.7	21.9	16.5
417-7	G'	24.0	17.3	12.7
	G''	18.64	13.48	9.98

**Table 4.4: Relative increase in modulus with field aged.** Note: Values are in % increase.

Comparison of Aging Effects Recovered/RTFO				
Binder	Modulus	0.1 rad/s	1.0 rad/s	10 rad/s
417-1	G'	23.3	6.1	1.5
	G''	4.7	0.2	1.6
417-2	G'	12.1	6.2	1.7
	G''	4.3	1.4	1.1
417-3	G'	15.4	13.9	10.4
	G''	17.0	3.4	0.2
417-4	G'	65.0	31.4	12.2
	G''	24.5	8.2	0.9
417-5	G'	53.7	19.4	1.0
	G''	10.0	4.4	10.1
417-6	G'	23.7	22.7	18.0
	G''	26.2	4.8	20.2
417-7	G'	2.2	6.2	6.9
	G''	7.8	8.1	7.5

Considering the data in the above tables, one can observe a great anomaly for the comparative loss modulus  $G''$  for 417-C. The loss modulus for both the PAV and the recovered are extremely similar to that of the RTFO at both intermediate and higher frequencies. For the PAV, the modulus reduces in magnitude to values smaller than that of the RTFO at higher frequency. This can be attributed to the heterogeneous phase system of Section C as observed from the Black space diagram, and this makes this binder behaves abnormally according to standard theoretical rheology. With respect to the rheological properties determined in Tables 2.3 and 2.4, it is possible to predict which binder performs best and which one performs worst by comparing the PAV modulus and the recovered modulus with the distress survey results simultaneously. From the data above, it appears that 417-B happens to be the most desirable binder to use in this stretch of highway according to the comparative PAV modulus. This section has the least rate of aging

with respect to  $G'$ , which means that it should be able to withstand the longest before becoming overly brittle and failing to thermal cracking. It also has relatively the least value of  $G''$  with respect to aging, indicating that it has good resistance to rutting distress. Even though 417-C shows similar and quite better characteristics with age, due to its heterogeneous phase system it will be inappropriate to predict its performance with only the result from the DSR, and hence other test methods such as BBR, eBBR and DENT would be used to account for its performance. However, based on the data in Table 4.4, it appears that Section G happens to be the most suitable binder for this stretch of highway, since it has the least or slowest aging rate according to its comparative recovered modulus. There is a high possibility that the asphalt binder for this trial section delivered to the laboratory for further analyses, is likely different from the recovered binder from this section. The order of ranking of the other sections for both the PAV and the recovered are given in Table 4.5 based on the comparative PAV and recovered modulus values. Comparing the order of the ranking from the perspectives of the PAV and that of the recovered and by comparing them individually to the distress survey results, it shows that the ranking based on the field aged correlates much better with the data obtained from the field than the ranking based on the PAV. Those binders that performed poorly in the test did so because of their higher respective rate of aging and the subsequent influence on both the storage and loss modulus, and their effect on the performance of the pavement is clearly seen in the distress survey data. On the other hand, those binders which performed better according to test and the distress survey results, did so as a result of their respective low rate of aging with respect to  $G'$  and  $G''$ . Those binder with higher values of  $G'$  and  $G''$  have undergone cracking easily due to low temperature effect, since they become brittle and not able to resist the applied stress/strain.



**Table 4.5: Ranking of test sections of binders according to changes in their modulus.**

Sample Section	PAV modulus ranking of the binders.	Field aged modulus ranking of the binders	Distress survey result, #/ 500m
417-A	4	3	0
417-B	1	2	0
417-C	2	4	8
417-D	3	7	52
417-E	7	5	79
417-F	6	6	72
417-G	5	1	0

#### **4.5 Double Edge Notched Tension (DENT) Test Analysis (Ductile Properties of the Binders)**

The concept of the essential work of fracture, EWF, was adopted not long ago in our laboratory for the determination of the strain tolerance in the ductile state regime and the failure properties of asphalt cements using the double edge notched tension (DENT) specimen test [57]. This method has been used successfully to rank asphalt binders in terms of performance-based properties and also to differentiate superior performing binders from inferior ones. In this investigative study, all asphalt binders underwent the same ductility testing by stretching them at a constant rate of loading of 50 mm/min in an isothermal water bath at 15°C until failure or fracture occurred, as according to the Ministry of Transportation of Ontario LS-299 standard testing procedure [19]. All the binders behaved according to the assumptions and conditions and/or requirements that are supposed to be met in order to be able to use the EWF model.

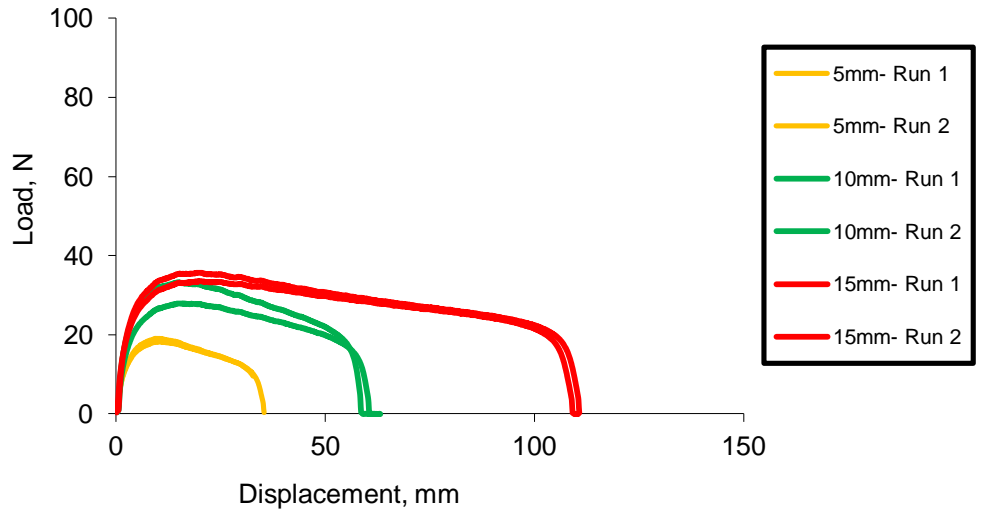


Figure 4.1.6: Representative Force-Displacement Data for the DENT Test on Binder 417-C.

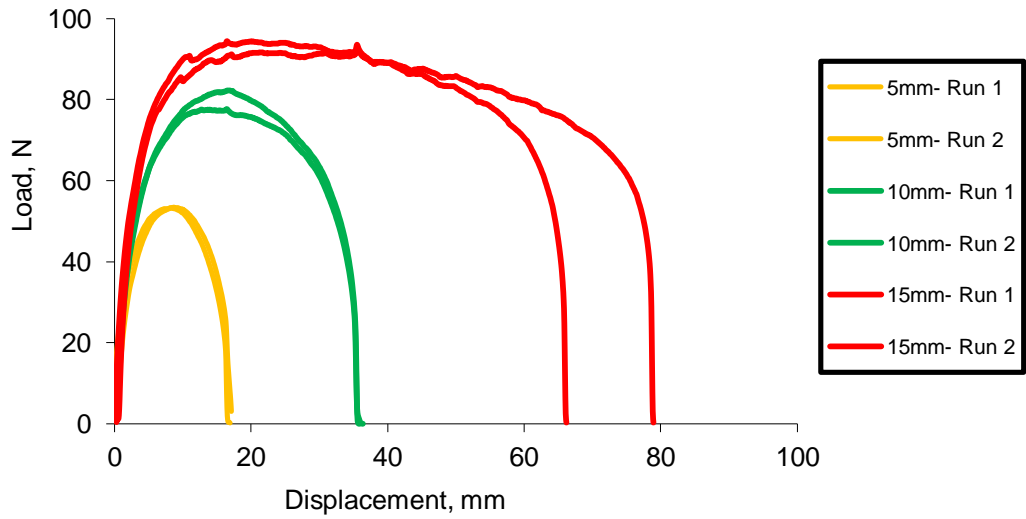


Figure 4.1.7: Representative Force-Displacement Data for the DENT Test on Binder 417-F.

This is observed by the test method being highly reproducible and that the tests for different ligament lengths were self-similar but were different for each binder, as shown in the raw data in Figure 4.1.6, which represents the duplicates force against displacement curves for a series of DENT specimen with ligament lengths ranging from 5 to 15 mm, with a difference of 5 mm from each other. Figure 4.1.6 is a representative of all the other trial sections, since they exhibited the same pattern of reproducibility except for Section F as shown in Figure 4.1.7. This gives the confirmation that all the binders went through the same process and sequence of stretching, yielding and tearing, which is a necessary requirement for the EWF test to be completed. Secondly, it is assumed in the essential work of fracture model that ligament yields fully prior to crack propagation or failure, and this is observed in the flat shapes of the force–displacement curves in Figures 4.1.6 and 4.1.7, which confirms the fact that ligament yielded fully before fracture occurred, and this is considered to occur during the sudden drop of the force at the end of each test. By plotting the specific total work of fracture,  $w_t$ , as a function against the ligament length,  $L$ , as in accordance with Equation 11, as shown in Figure 4.1.8, with the intercept being equal to the essential work of fracture,  $w_e$  and the slope being equal to the plastic work of fracture,  $\beta w_p$ , it is possible to account for the third condition which says that the volume of the plastic zone should be proportional to the square of the ligament length and the sample thickness (refer to chapter two for details). This condition is confirmed valid in Figure 4.1.8, with all the graphs of different sections of Highway 417 having a straight line with the intercept and the slope equalling to the essential works of fracture and the plastic or non essential works of fracture term. The regression coefficients of these straight lines range from 0.95 to 0.99, which indicates conclusively that this method is accurately correct.

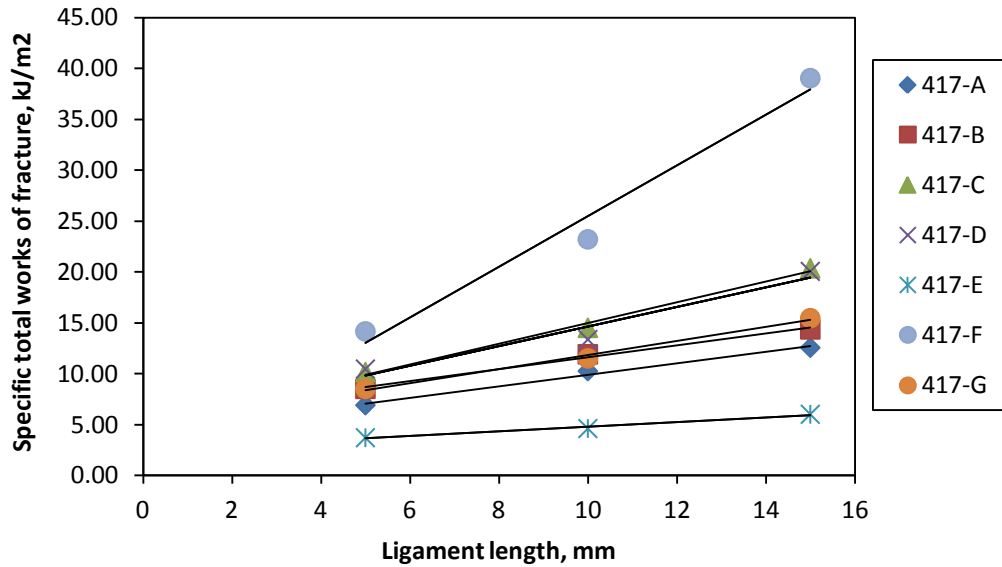


Figure 4.1.8: Essential Work of Fracture Analysis According to Equation 11 for Highway 417 Binders.

The essential work of fracture is considered as a material property which is not dependent on the geometry of the specimen (asphalt binder) with regards to performance grading of asphalt binders. However, the plastic work of fracture term is not a material property, but recent studies show that it relates to the mixture design, asphalt binder and air void. Asphalt cement mixture rich in asphalt binder content usually have high(er) plastic work of fracture, and hence are less sensitive to fatigue and low temperature cracking as a result of the higher strain tolerance deposited in them due to the higher binder content [56]. In order to use the concept of the EWF model to account for the resistance of fatigue cracking and low temperature cracking distresses in asphalt cement pavement, it is expected that the essential work of fracture value and that of the plastic work of fracture must be relatively high, since pavement cracking can occur only under flexure when the strain tolerance of the pavement is exceeded [76].

The strain tolerance and the ductile properties of RTFO-aged, PAV-aged and field-aged asphalt binders were determined using the EWF model using the DENT specimen in order to conduct a better comparative investigations on the performance of the various trial sections on Highway 417. The force-displacement curve in Figure 4.1.6 shows that the shapes of the curves are all the same for each ligament length for each binder but differ significantly from one binder to another binder. This could be due to the fact that the asphalt binder were modified with different modifiers and as a result, different modification methods leads to different morphologies which either give rise to cross-linked binders that are elastic or rubbery, stiff binders that may portray a yield stress or softer binders that are more viscous and crack easily and rapidly.

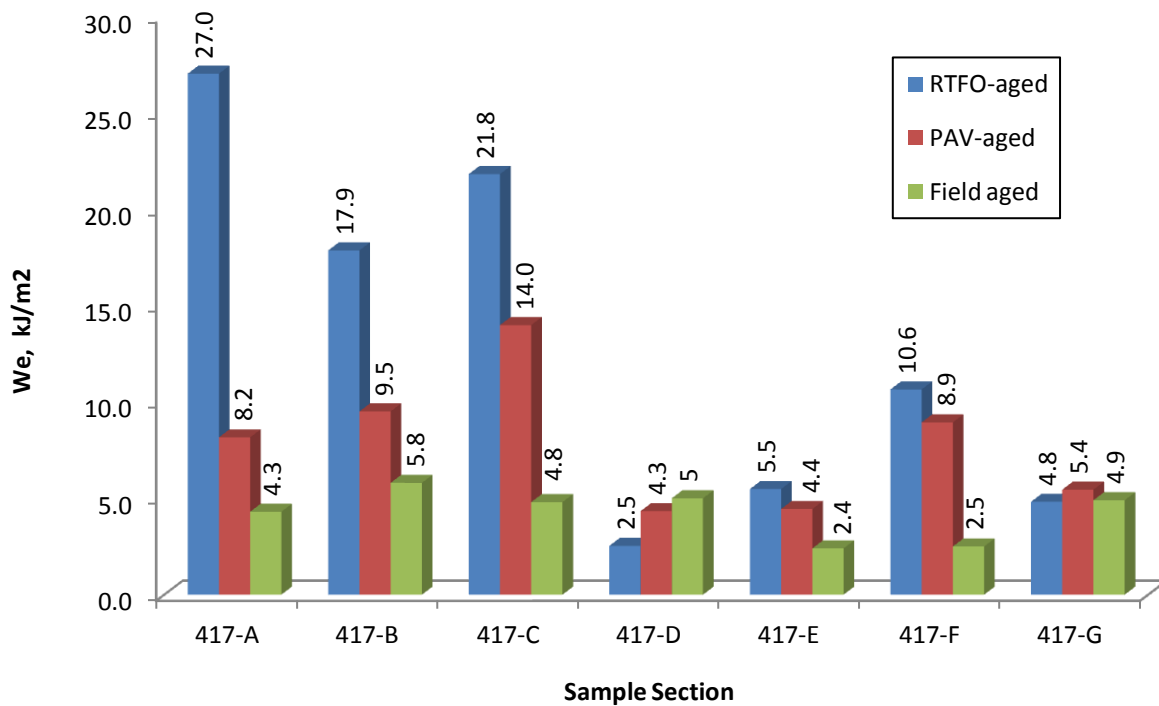


Figure 4.1.9: Specific essential works of fracture at different aging times of asphalt binders of Highway 417.

Figure 4.1.9 shows a comparison of the essential works of fracture at different aging rates both in the laboratory and in the field for all the seven section on Highway 417. It can be observed that the specific essential works of fracture values for the RTFO aged binders are higher than both the PAV aged and the recovered binder residues. This indicates that at this aging time, the binder has higher strain tolerance in the ductile state and hence it is unlikely for the binders to experience any form of pavement distress. Sections A, B and C have the highest values of the essential work of fracture with regards to the RTFO aging method, while Sections D and E have relatively lower values of the essential work of fracture. As the aging process become more pronounced as in the PAV-aged and the field-aged samples, the values of the essential works of fracture decrease. This indicates that age hardening of the binder especially at lower temperatures as a result of the oxidation of the binders, has a significant influence on the ductile strain tolerance ability of the binders and also the binders' ability to resist low temperature cracking and fatigue cracking distresses. According to the PAV-aged data, Sections A, B and C, again, have comparatively higher values of the essential work of fracture. It is therefore not surprising that Sections A and B are free from any form of pavement distress, while Section C has relatively insignificant cracks. Sections E and D show lower values of the essential work of fracture according to the PAV data. Both sections have cracked significantly according to the field distress survey results. Using the data from the recovered binders, a much better correlation is seen here compared to the PAV results. This high correlation of the field aged binder with the distress survey result can be explained by the fact that the field aged binders are exact representation of the kind of age hardening occurring in the field. Sections A to D and G show relatively higher values of the essential works of fracture,  $w_e$ , indicating that they have higher strain tolerance to resist low temperature cracking and fatigue cracking. It is therefore expected

that none of these sections should show any significant pavement distress. This is found to be true for Sections A, B and G, since they are all free from any distress. However, Section D has cracked quite significantly. Sections E and F have performed poorly in the field and their poor performance in service is attributed partly to their weak strain tolerance characteristics which is shown clearly in their lower values of the essential works of fracture.

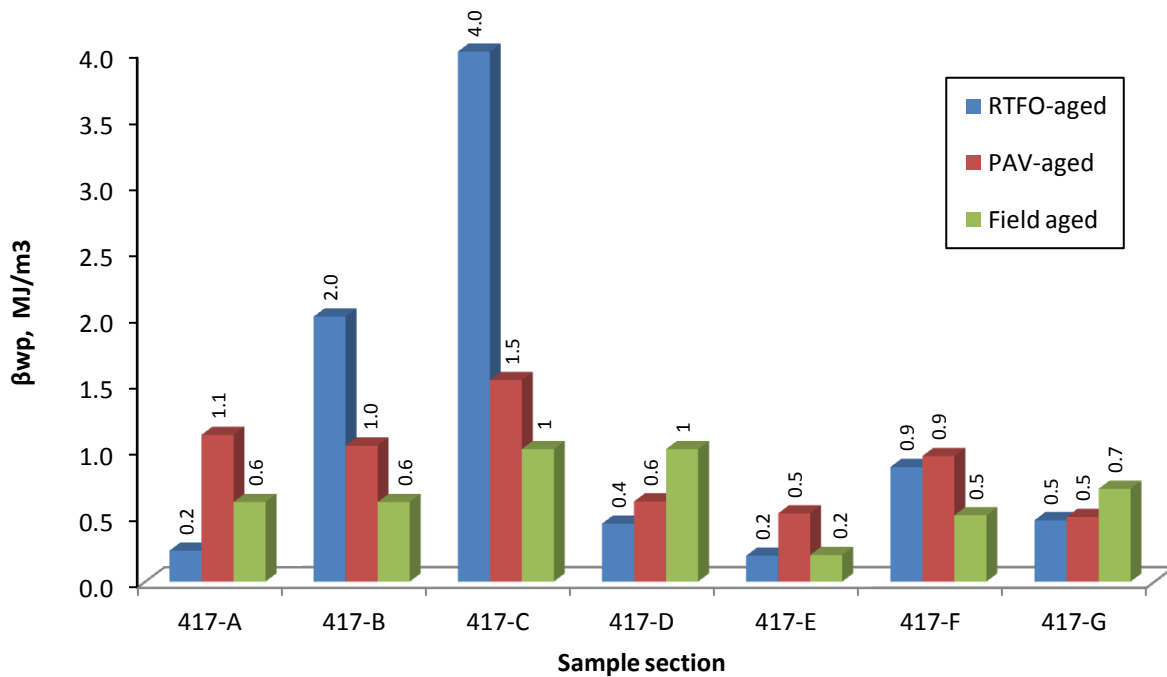


Figure 4.2.0: Specific plastic works of fracture term at different aging times of asphalt binders of Highway 417.

Figure 4.2.0 is a summary of the results of the plastic works of fracture at different aging rates for all the seven sections on Highway 417. It is observed that the values of the specific plastic or non essential work of fracture term ( $\beta_{wp}$ ) are relatively smaller compared to that of their respective essential works of fracture, and are of less importance with regards to material

properties of the binder in terms of performance in service. However, some few important conclusions can be drawn from this data. Using the data from the PAV-aged and the recovered binders, it is clear that binders with superior performance in service such as Section A to C and G, have higher values of the plastic work of fracture term, while binders with inferior performance in service such as Section D to F, have lower values of the non essential work of fracture term. This good correlation can be explained by the fact that binder with higher values of the non essential work of fracture term, have good mixture design and moderate amount (4-7 %) of air void, while those with lower values of the plastic work of fracture term are of poor mixture design properties and lower/higher air void content in the asphalt cement mixture. This is observed in Section F, having higher amount of air void in the asphalt cement mixture, leading to age hardening of the binder and subsequently resulting in premature and excessive cracking especially at lower temperature as discussed in the section 4.1.

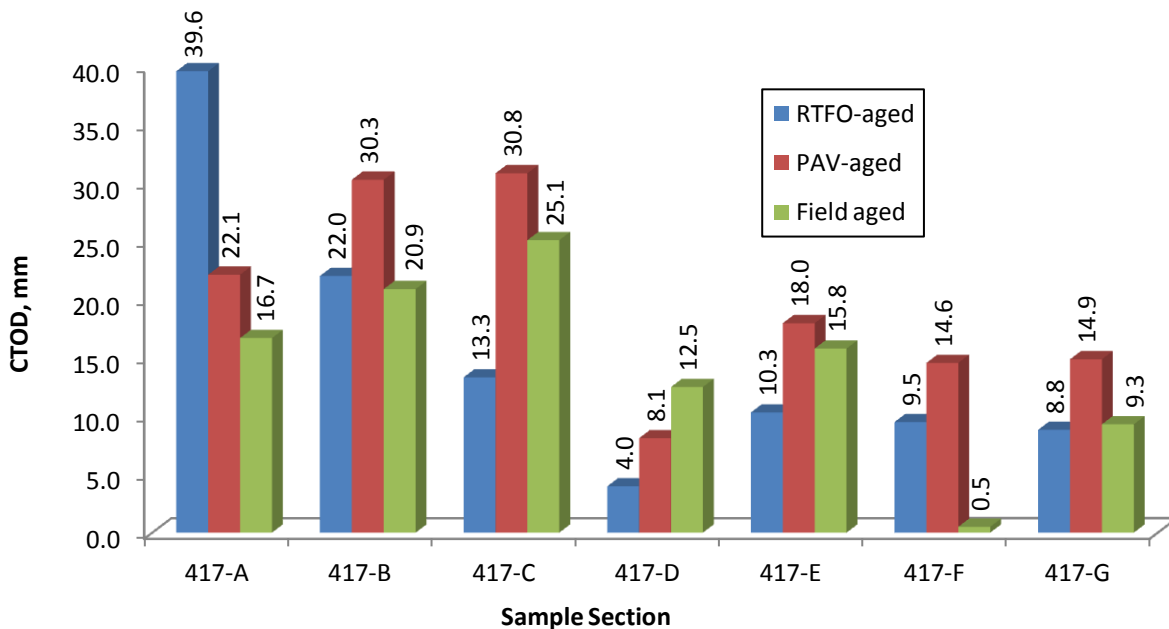


Figure 4.2.1: Crack tip opening displacement (CTOD) at different ageing rates of asphalt binders of Highway 417 at 15°C.



Another important parameter used to differentiate superior binders from inferior binders is the crack tip opening displacement (CTOD). The CTOD is given as the ratio of the essential work of fracture to the net section stress of the 5 mm ligament length DENT specimen. It provides a measure of strain tolerance in the ductile state under conditions of severe confinement. It is a good parameter for performance grading of both asphalt cement mixtures and binders for low thermal fatigue cracking and fatigue cracking at temperatures and loading rates that spread over the ductile regime [69]. Figure 4.2.1 is a summary of the data of the CTOD at different aging rates for all the seven sections on Highway 417. Only the PAV-aged and the field-aged data would be used to analyse the performance of the various binders. This is because the PAV-aged samples are used to simulate aging in the field for 5-10 years and the field aged samples represent exactly the behaviour of the binder in service. It is expected that the CTOD values of the field aged and that of the PAV aged be the same or similar, but that was not observed. This difference can be explained by the fact that the samples from the cores were extracted from the top 5 cm of the pavement while the cracking is typically starting in the top 1-2 cm layer. Further, the solvent extraction could have had some influence on the properties of the recovered asphalt cements. Sections A, B and C have the highest CTOD values for both the PAV-aged and the field-aged residues. This account for their better performance in service, by being free from any pavement distresses. Sections E and F have lower values of the CTOD in both the PAV-aged and the field-aged samples, and this indicates their poor performance in service. Both Sections E and F have cracked significantly in the field, which confirms the usefulness of this method. Section D has a moderate lower value of the CTOD, and this section has cracked quite significant but not as severe as in Sections E and F. Section G even though has not undergone any pavement distress, shows a smaller value of the CTOD. This anomaly can be explained by the fact that the

diffuse microcracking that releases the stresses but that is too small to notice at this point in time. Also, this section is not at the same location as the other six sections, it is about 2 km away from the rest, and hence the subgrade could be slightly different as what it is for the other six sections. Another reason could be that this trial section is relatively young in age (it was constructed in 2006), and will likely undergo thermal cracking rapidly in the near future. It is also possible that later, this section will likely suffer from extensive cracking and ravelling.

The CTOD has been able to differentiate between superior performing binders and inferior ones among the seven sections except for Section G. It can therefore be said that the CTOD is able to separate superior performing binder from inferior ones and also, able to rank asphalt binders with 86 % accuracy, which confirms the accuracy of this method as predicted by Hesp et al. [38, 76], in an investigation to validate the double-edge-notched tension test and extended bending beam rheometer test methods.

#### **4.6 Regular BBR Data Analysis**

The regular BBR (AASHTO M320) grades were determined for both laboratory (PAV) aged and recovered asphalt cement residues of Highway 417. The testing was performed by conditioning the asphalt samples (beams) for one hour at  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  for testing at  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$ , and the creep stiffness,  $S(60)$  and the slope of the creep stiffness master curve, m-value ( $m(60)$ ) were determined accordingly using three point bending. The limiting temperature where  $S(60) = 300$  MPa and  $m(60) = 0.3$  were determined after each test and the warmest of these two limiting temperatures was used as the minimum performance grade temperature of the material. The

laboratory-aged material data was purposed to imitate the aging that occurs in the field after 5 to 10 years in service.

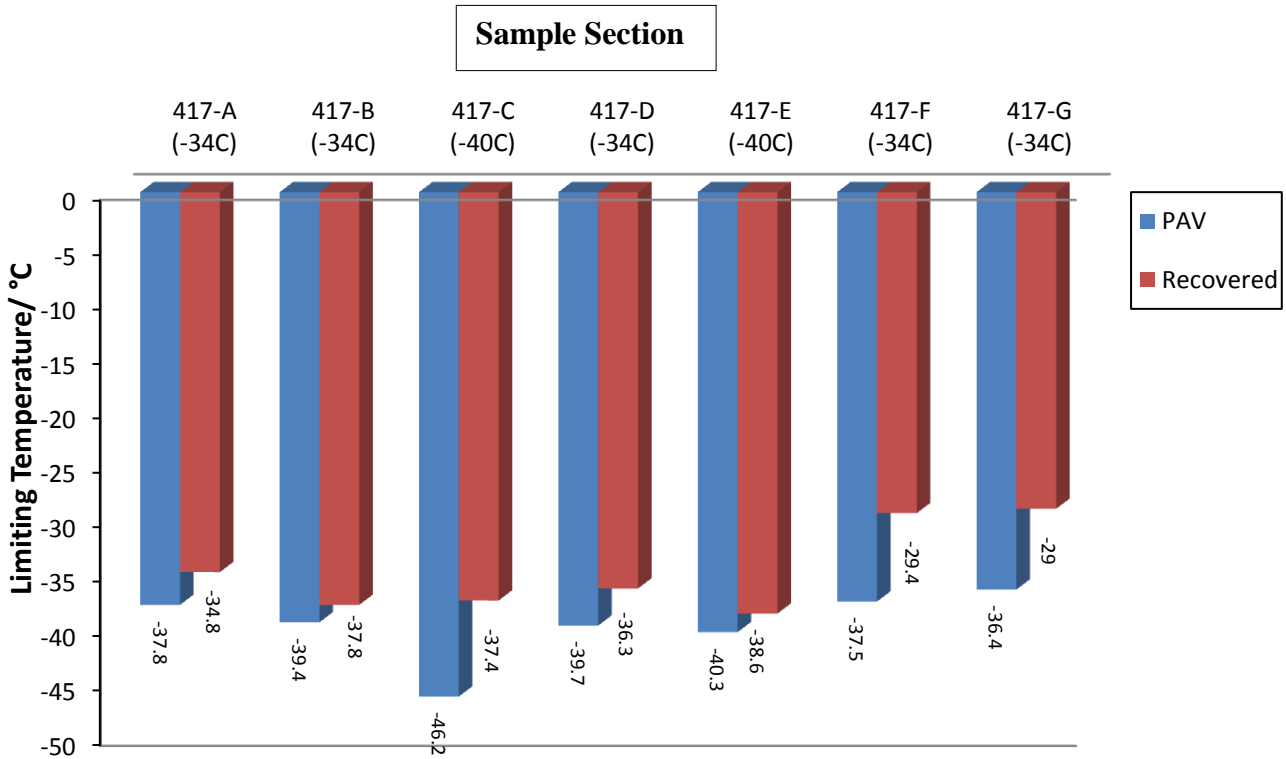


Figure 4.2.3: Limiting grade temperature according to AASHTO M320 protocol. Note: The numbers in the brackets represents the minimum performance grade temperature according to the Superpave grading method. .

Figure 4.2.3 gives a summary of the data of the limiting temperatures for all the seven sections, comparing the laboratory-aged residues with the recovered residues. From the PAV-aged data, all the trial sections grade lower than their respective minimum performance grade temperatures, which shows that all these trial section should have been free from any form of pavement distress. However, the data from the recovered residues gives limiting temperatures that are warmer than those obtained from the PAV aged residues. For instance, Section C grades  $-46^{\circ}\text{C}$

for the PAV data, which indicates that the pavement is ideally not suppose to show any transverse cracking, but the field-aged data recorded a performance grade temperature of  $-37^{\circ}\text{C}$ , a temperature that is  $\sim 9^{\circ}\text{C}$  warmer than that of the PAV-aged data and is more than a full grade ( $6^{\circ}\text{C}$ ), and it is also warmer than the minimum performance grade temperature of the binder by  $2.6^{\circ}\text{C}$ , which is almost equal to half a full grade deficit (loss). Based on the field-aged data, Sections C, E, F and G all have experienced more aging in the field than those occurring in the PAV-aged residues. The higher rates of aging in these binders can be attributed to wax crystallization and free volume collapse occurring in these binders. All these sections have their limiting temperatures warmer than their respective minimum performance grade temperatures within the range of  $1.4$  to  $8.8^{\circ}\text{C}$ . In addition, the recovered residue data is not able to predict the performance for all the trial sections. It fails to predict the performance of Section D and G. Section D has cracked quite significantly, but its limiting temperature is lower than its minimum PG grade temperature. On the other hand, Section G is free from any distress but its limiting temperature is  $7.4^{\circ}\text{C}$  warmer than its minimum PG grade temperature. This data gives the evidence that the current AASHTO M320 standard protocol has a serious problem and needs to be revisited, since it is unable to predict correctly the performance of the asphalt cement in the field due to insufficient time to allow for adequate physical aging before testing. There is therefore the need to use the Ontario's LS-308 method to correctly account for the performance of the asphalt cement in service.

A separate analysis was performed in order to determine how aging processes occurring in the asphalt cements are affected by changing the standard PAV conditions. The PAV laboratory aging conditions were altered by changing the test from 20 hours to 40 hours while maintaining the weight of the asphalt cement at 50 g, to allow more time for aging to occur in order to see if

it would be able to predict correctly the effect of physical aging on the performance of the asphalt binder. Another test was also conducted by changing the weight of the asphalt cement from 50 g to 25 g while keeping the test time at 20 hours, to determine if a reduction in the sample size (weight) can influence the rate of aging processes. The results of these tests are summarised in Figure 4.2.4. The results show that by extending the PAV aging time from 20 hours to 40 hours, and by changing the weight of the asphalt cement for the test from 50 g to 25 g, a remarkable aging phenomenon is observed in the asphalt binders. The limiting temperatures of all the PAV tests at 40 hours and also at 25 g are all warmer than their respective minimum performance grade temperatures by a factor of 6°C or more, which is equal to a full grade loss. This shows that by changing the conditions governing the standard PAV aging procedure, there is a higher possibility to account for and predict correctly the effect of aging (chemical or physical) on the performance of the asphalt binder in the pavement at low temperatures.

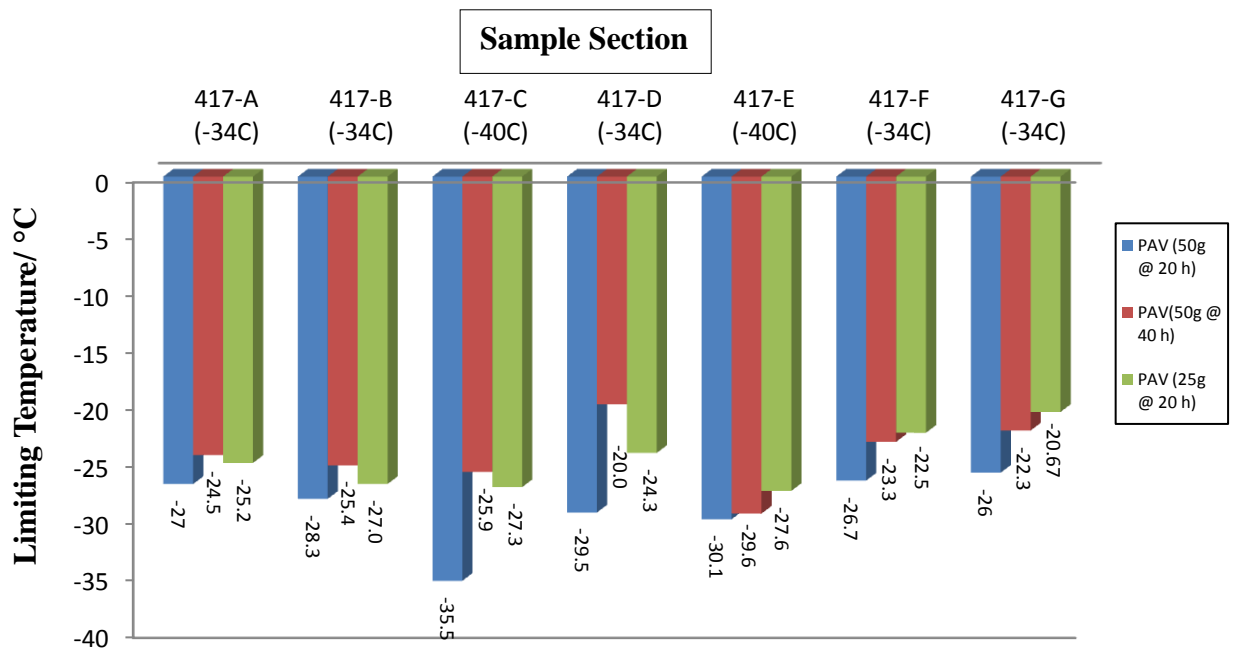


Figure 4.2.4: Limiting grade temperatures at different PAV aging conditions for asphalt binders on Highway 417.

#### 4.7 Extended BBR Data Analysis

The materials were further tested according to Ontario's extended BBR protocol proposed by the collaboration of Queen's University and the Ministry of Transportation of Ontario (MTO), which is now embodied in the MTO standard test methods LS-308 [20]. This test was performed in order to account for the greater differences in performance of the pavements at low temperatures. The materials were tested at  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  after they have been conditioned for one hour and 72 hours at isothermal conditions of  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$ . The limiting temperature where  $m(60) = 0.3$  and  $S(60) = 300$  MPa were also determined according to AASHTO M320 standard protocol and the warmer temperature among the two limiting temperatures was used as the minimum performance grade temperature of the pavement. The grade losses after 72 hours of conditioning were also determined after each test, since these grade losses are good indicators of pavement performance.

Figures 4.2.5 and 4.2.6 represent the summary of the LS-308 data for the recovered and the PAV residues at  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$ , respectively. The results show significant influence of physical hardening on the performance of the pavement at low temperatures. The minimum performance grade temperatures obtained for the PAV residues at both  $-10^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  are approximately the same for all the seven sections. However, in spite of the good reproducibility, there seems to be a problem with the laboratory aging procedure. Sections A to D and F all passed the test with their limiting temperatures lower than their respective minimum PG grade temperatures. Comparing this data to the cracking distress results, shows that Sections A, B and C are free from any pavement distress and Section D has cracked quite significantly.

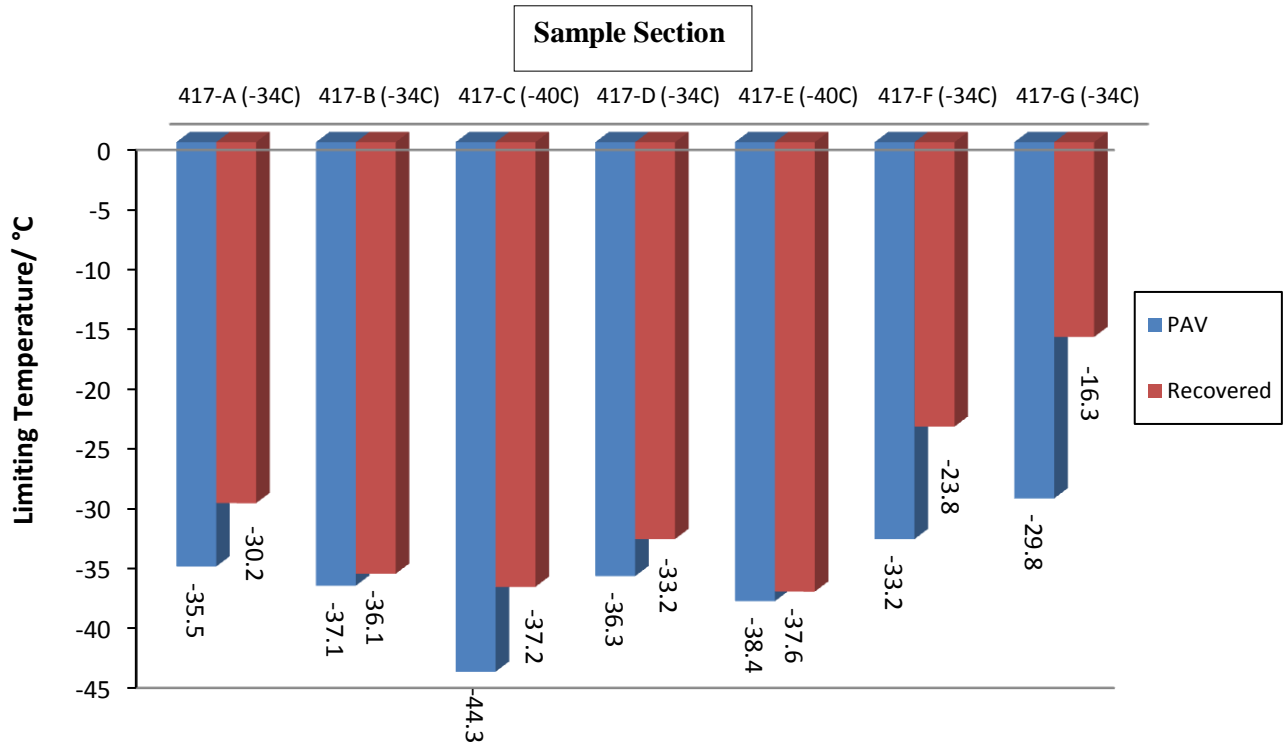


Figure 4.2.5: Limiting grade temperatures at  $-10^{\circ}\text{C}$  according to MTO LS-308 specification.  
 Note: The numbers in the brackets represents the minimum performance grade temperature according to the LTPPBind<sup>®</sup> software.

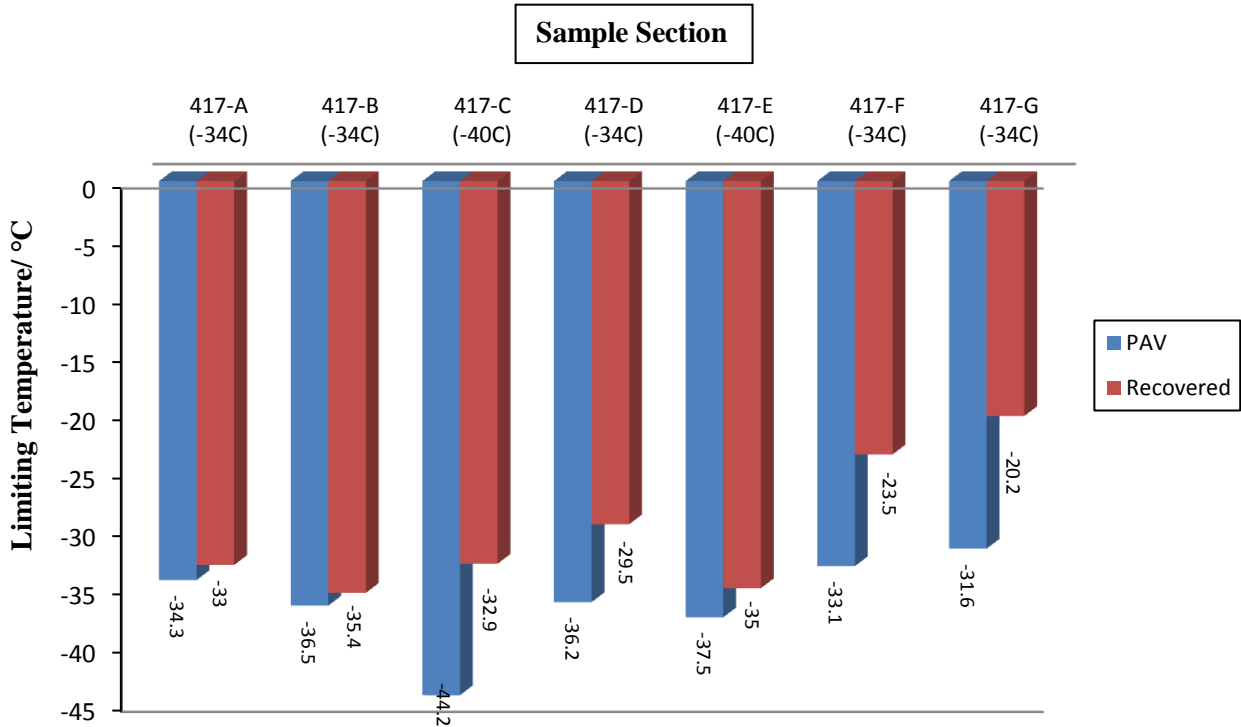


Figure 4.2.6: Figure 4.88: Limiting grade temperatures at  $-20^{\circ}\text{C}$  according to MTO LS-308 specification. Note: The numbers in the brackets represents the minimum performance grade temperature according to the LTPPBind<sup>®</sup> software.

However, Section D has lower limiting temperature compared to its minimum PG grade temperature. This gives the indication that the PAV aging protocol is not able to do a better job in terms of predicting the effect of aging on the performance of the asphalt binders.

Based on the data from the recovered asphalt cement residues, the effect of physical aging at low temperatures is clearly pronounced compared to the PAV data. Sections that have aged significantly have experienced severe cracking and those that have not undergone much aging have little or no cracking. For instance, Sections A and B aged quite insignificantly after 3 days of isothermal conditioning, and it is therefore not surprising that these sections have no observable cracks. On the other hand, Section F aged significantly with its limiting temperature



about 10°C greater (warmer) than its minimum PG grade temperature, which is close to twice of a full grade loss, which can actually reduce the confidence from the intended 98 % to about 0 % that in a given year the pavement is not exposed to damaging temperatures (LTPPBind® 1999). This greater loss in grade can be explained by the presence of greater air void content in this section obtained from the core samples, which leads to extensive physical hardening of the pavement due to penetration of water, frosts and air into the lower layers of the pavement, and subsequently resulting in premature and excessive pavement cracking. Section G has a limiting temperature that is about 13.8°C greater than its minimum PG grade temperature, which is expected technically to undergo severe transverse cracking due to the significant aging occurrence. However, the distress survey data shows that this section is free from any form of pavement distress. This could be explained by the fact that the diffuse microcracking that releases the stresses but that is too small to notice at this point in time. Also, this section is not at the same location as the other six sections, it is about 2 km away from the rest, and hence the subgrade could be different from what it is for the other six sections. Another reason could be that this trial section is relatively young in age (it was constructed in 2006), and will likely undergo thermal cracking rapidly in the near future. It is also possible that later, this section will likely suffer from extensive cracking and ravelling. From the limiting performance grade temperatures at both -10°C and -20°C, Section B ranks the best and Section F ranks the worst in terms of the effect of physical aging on the performance of the pavements at low temperatures.

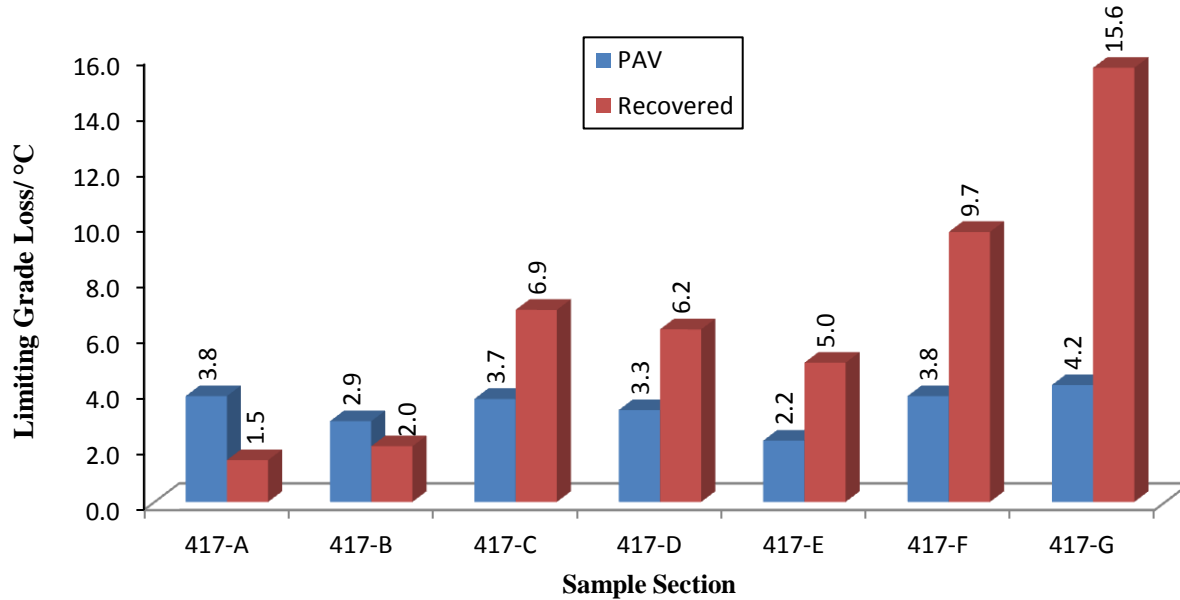


Figure 4.2.7: Three-day grade losses of asphalt cement from Highway 417.

Figure 4.2.7 shows the grade losses for both the PAV aged and the recovered asphalt cement residues after three days of conditioning. It has been shown that the grade loss is a good indicator of pavement performance, in that pavements with higher grade losses perform poorly in service while those with low grade losses perform better in service. A lower grade loss indicates a low possibility or lower tendency to physical hardening at the conditioning temperature. Those asphalt cements that exhibit low grade losses will be able to relax thermal stresses before the pavement reaches the spring thaw cycle. Therefore thermal stresses are relaxed when the pavement base thaws and serious movement occurs [87]. This shows that no stress leads to little or no cracking. The data as presented in Figure 4.2.7 shows that the grade loss is able to predict accurately the performance of the various sections on Highway 417. Sections A and B have grade losses of 1.5 and 2.0 respectively, and these losses in grades are not significant enough to cause any pavement cracking as can be witnessed in the field survey results. These sections lost

little in grade due to the effects of conditioning at low temperatures. Section C has lost significantly in grade, but this section has not cracked significantly. The good performance of this pavement is due to its good rheological properties and good ductile strain tolerance characteristics as determined by the DSR and the DENT test methods respectively. This shows that this pavement will undergo severe cracking in the near future due to its higher grade loss. Section D has a higher grade loss and this account for the reason why this pavement has cracked quite significantly. Section E, which has cracked the most also lost remarkably in grade but not as significant as in Sections D and F. Its severe cracking can be attributed to the occurrence of physical aging at low temperatures (higher grade loss) and the poor ductile strain tolerance characteristics as observed in the DENT test. Section F also has lost remarkably in grade, and this confirms why this section is the second most severely cracked among all the seven sections. This severe cracking is due to the presence of greater amount of air void determined in this section as detailed earlier. The poor performance of Section D and E may also be to the use of polyphosphoric acid (PPA) to modify these asphalt cements. This is because PPA is found to lower the ductile strain tolerance of the asphalt cement in the pavement [90]. They also performed poorly because they were made with binders that do not only lose in terms of chemical aging but also in terms of physical aging as result of the unstable colloidal structure and the presence of wax [18]. Section G presents a great deal of confusion, in that, it is the pavement with the highest loss in grade in both the PAV and the recovered data, but this trial section is free from any form of pavement distress. Even though the asphalt binder used for this section have good rheological properties as determined by the DSR method good ductile strain tolerance character (from its CTOD value), this pavement will likely undergo severe transverse cracking in the near future due to it greater grade loss as a result of physical aging at low temperatures. The

superior performance of Sections A and B and the poor performance of Sections D, E and F have been correctly predicted by this data. It also shows that Sections C and G will experience severe transverse cracking in some years to come due to their higher grade losses.

## CHAPTER FIVE

### SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

#### 5.1 Summary and Conclusions

The following conclusions and summaries could be arrived at based on the literature review, experimental procedures, results and the discussions made in the previous chapters.

- Any changes in the air void content from its optimum value during the lifetime of an asphalt pavement constitutes initiations and damage of the pavement. An increase in the air void content of an asphalt concrete mix has a direct negative influence on the performance of the pavement and causes excessive and premature cracking, as a result of the age hardening phenomena that occurs in the asphalt cement upon the penetration of air and water to the underlying layers of the pavement. Air voids less than 3% result in excessive plastic flow while air voids greater than 7% leads to permeability effects.
- All the field aged samples tested negative (i.e., no or insignificant amount) for the presence of waste engine oil residues, except for Section G. This shows that Section G, which has not experienced any visible distresses will undergo severe cracking in the near future due to the presence of the waste engine oil. Asphalt cements modified with WEO residues can pass the current AASHTO M320 standard specification protocols, however these materials are unable to provide good performance in service, hence poor returns in investment. The failure of these WEO modified pavements is due to the fact that waste engine oil residue is highly paraffinic in nature and as such it causes premature hardening due to the precipitation of asphaltenes that are formed during the oxidation in service. Paraffins are also known to cause problems with a loss of adhesion to the aggregate

which can lead to premature stripping (removal of the asphalt cement from the aggregate) and rutting induced by this stripping.

- Infrared spectroscopy is a valuable tool to determine the degree of oxygen intake for different aging processes. The degree of oxygen intake increases from unaged (neat) to RTFO-aged to PAV-aged binders. However, chemical aging (oxidative hardening) is seen to be prominent in laboratory aged binders compared to field aged binders. This difference can be explained by the fact that asphalt cements were recovered from the top 5 cm of the pavement while most of the aging might have occurred in the top 1 or 2 cm of the surface layer of the pavement, and also, these trial sections are relatively young, and will possibly undergo more chemical and physical aging in the future.
- Asphalt cements modified with styrene-butadiene-styrene (SBS) are able to perform better in service, since the addition of SBS exerts a positive influence on the performance-based properties of the asphalt cements.
- The Black space diagrams and the master curves show that Section A is a rheologically simple asphalt binder with a single phase system, whereas, the other sections are rheologically complex binders with virtually heterogeneous phase system due to the addition of polymers to these binders, which causes phase separation of the asphalt cement as seen in Sections C and F.
- By comparing the storage and the loss moduli of the PAV-aged and the field-aged residues to that of the RTFO-aged residues, there is much better correlation between the comparative moduli of the field-aged residues with the field cracking distress data, in terms of predicting and/or resisting low temperature cracking and permanent

deformation, in comparison with the comparative moduli of the PAV-aged residues with the field cracking distress data.

- The double-edge-notched tension test results (the essential works of fracture  $w_f$ , and the plastic works of fracture term,  $\beta w_p$ ) give an excellent correlation with the distress survey results of the pavement in service. Pavements with high values of both  $w_f$  and  $\beta w_p$ , indicating their high strain tolerance characteristics, provided pavements with insignificant or no cracking distress, whereas, those pavements with lower values of both  $w_f$  and  $\beta w_p$  have cracked severely.
- The CTOD has been able to distinguished superior performing binders from inferior performing binders, and it has also been able to rank the performance of the asphalt cements with 86% accuracy.
- The current AASHTO M320 standard specification procedure, which involves only one hour conditioning of the asphalt beam prior to testing, needs to be reconsidered with necessary modifications and improvements, since it is unable to account for the age hardening that occurs in the field. Asphalt cements with the same low temperature grade according to their AASHTO M320 standard specification protocols were found to exhibit very different performance in service.
- The Ministry of Transportation of Ontario LS – 308 extended BBR test method provides a better correlation with the performance of the asphalt cements in service compared to the current AASHTO M320 specification methods, hence it serves as an improvement over the regular BBR method. For instance, the grade loss for the extended BBR after 72 hours of conditioning at  $-10^{\circ}\text{C}$  ranged from  $1.5^{\circ}\text{C}$  and  $2.0^{\circ}\text{C}$  for Sections A and B respectively (which have not cracked) to  $\sim 10^{\circ}\text{C}$  for section F (which has cracked

severely). The grade losses for Sections A and B indicate that these sections have a low affinity for reversible aging at lower temperatures. The difference of grade loss of  $\sim 10^{\circ}\text{C}$  in Section F indicates the high tendency of the asphalt binder in this section to undergo reversible aging at low temperature. This high grade loss can actually reduce the confidence from the intended 98% to about 10% that in a given year the pavement is not exposed to damaging temperatures for a typical Ontario weather condition.

- By changing the conditions governing the standard laboratory aging processes (RTFO and PAV), it is possible to age the asphalt cement in the laboratory that will replicate exactly the kind of age hardening phenomena occurring in the field.

## **5.2 Recommendations and Future Work**

The following future work and recommendations can be made based on the results and discussions in this thesis.

- In order to fully correlate the performance of the binders for these trial sections to these test methods, it is recommended that new virgin (neat) binders used for construction of the various trial sections, should be supplied to the laboratory without mixing them up for further analysis.
- Extraction of the core samples should be done on the top 2 cm instead of the top 5 cm, since reversible aging occurs on the top 1-2 cm of the pavement. Different solvents can also be used for the extraction process to determine which solvent gives optimum results.
- Further X-ray fluorescence test and analysis should be carried out on Sections E and G for both the recovered and the neat binders.



- The recovered binder for Section D was found to contain SBS for the first test, a later test on different core samples from the same trial section showed absence of SBS in the binder. Further analysis should be performed on this section to determine the presence or absence of SBS in this binder.
- The cost of pavement reconstruction, resurfacing and rehabilitation in colder regions such as Canada and northern parts of the United States can be avoided by the implementation and application of the Ministry of Transportation of Ontario's LS-308 and LS-299 by asphalt cement supplier agencies and asphalt pavement contractors to enhance the long term performance of the pavements in service.

## References

1. Wang, D.; Tetteh-Wayoe, H.; K. O. Anderson; *Low Temperature Properties of Asphalt Cements and Mixtures used in the C-SHRP Lamont Test Road in Alberta*, Edmonton, Technical Report ABTR/RD/RR-92/02, Alberta, Canada, **1992**.
2. Glover I. C.; *Wet and Dry Aging of Polymer-Asphalt Blends: Chemistry and performance*: Department of Chemistry, Louisiana State University, PhD Thesis, **2007**.
3. Hesp, S. A. M. *Longer Lasting Roads*, [http://www.nserc-crsng.gc.ca/Partners-Partenaires/ImpactStory-Reussite\\_eng.asp?ID=1002](http://www.nserc-crsng.gc.ca/Partners-Partenaires/ImpactStory-Reussite_eng.asp?ID=1002), Accessed: **April, 2011**.
4. American Society for Testing and Material D8; *Standard Terminology Relating to Material for Road and Pavement*, **2002**.
5. Asphalt Institute; *Introduction to Asphalt*, Lexington, Kentucky, U.S.A., **1990**.
6. Read, J. and Whiteoak, D.; *The Shell Bitumen Handbook*, Fifth Edition; Hunter, R. N. Ed.; Thomas Telford: London, **2003**.
7. Freemantle, M. *Chemical & Engineering News*, **1999**, 77(47), 81.
8. Soleimani, A.; *Use of Dynamic Phase Angle and Complex Modulus for the Low Temperature Performance Grading of Asphalt Cements*, MSc. Thesis, Department of Chemistry, Queen's University, Kingston, Canada, **2009**.
9. Asphalt Institute; *Performance Graded Asphalt Binder Specification and Testing*, Third Edition, Superpave Series No. 1 (SP-1), **2003**.

10. Flexible Pavement Basics; [http://training.ce.washington.edu/WSDOT/Modules/02\\_pavement\\_types/02-2\\_body.htm](http://training.ce.washington.edu/WSDOT/Modules/02_pavement_types/02-2_body.htm), Accessed: **April, 2011**.
11. Lee, H. J.; Lee, J. H. Lee; Park, H. M. *Construction and Building Materials*, **2007**, *21*, 1079.
12. Anderson, D. A.; Christensen, D. W.; Bahia, H. U.; Dongré, R.; Sharma, M. G.; Antle, C. E.; Button, J. *Binder Characterization and Evaluation. Volume 3: Physical Characterization Report SHRP-A-369*, Strategic Highway Research Program, National Research Council, Washington, D.C., **1994**.
13. Anderson, D. A.; Kennedy, T. W. *Journal of the Association of Asphalt Paving Technologists*, **1993**, *62*, 481.
14. Chen, J. S; Tsai, C. J. *Journal of Materials Engineering and Performance*, **1999**, *8(4)*, 443.
15. McGennis, R. B; Shuler, S; Bahia, H. U.; *Background of Superpave Asphalt Binder Test Methods*, Publication No. FHWA-SA-94-069, Federal Highway Administration, Washington, Washington D.C., **1994**.
16. SealMaster Pavement Products and Equipment; [http://www.pavemanpro.com/article/deterioration\\_asphalt\\_causes/](http://www.pavemanpro.com/article/deterioration_asphalt_causes/), Accessed: **April, 2011**.
17. American Association of State Highway and Transportation Officials; *AASHTO M320 Standard and Specification for Performance-Graded Asphalt Binder*, **2002**.
18. Hesp, S. A. M; Kodrat, I.; Scafe, D.; Soleiman, A.; Subramani, S.; *Rheological Testing of Asphalt Cements Recovered from an Ontario Pavement Trial*, Queen's University, Kingston, Canada, **2009**.

19. Ministry of Transportation of Ontario; *LS-299 – Determination of Asphalt Cement's Resistance to Ductile Failure Using Double-Edge-Notched Tension Test (DENT)*, Revision 24 to Laboratory Testing Manual, **2007**.
20. Ministry of Transportation of Ontario; *LS-308 – Determination of Performance Grade of Physically Aged Asphalt Cement Using Extended Bending Beam Rheometer (BBR) Method*, Revision 24 to Laboratory Testing Manual, **2007**.
21. Hesp, S. A. M.; Genin, S.N.; Scafe, D.; Shurvell, H. F.; Subramani S.; *Five Year Performance Review of a Northern Ontario Pavement Trial: Validation of Ontario's Double-Edge-Notched Tension (DENT) and Extended Bending Beam Rheometer (BBR) Test Methods*, Department of Chemistry Queen's University, Kingston, Ontario, **2009**.
22. Nellensteyn, F. J. *Institute of Petroleum Technology*, **1924**, 14, 134.
23. Mack, C.J. *Journal of Physical Chemistry*, **1932**, 36, 2901.
24. Peterson, J. C.; Robertson, R .E.; Branthaver, J. F; Anderson, D. A; Christiansen, D. W.; Bahia, H. U.; *Binder Characterization and Evaluation –Volume 1-SHRP-A-369*, National Research Council, Washington, DC, **1994**.
25. Chatergoon, L.; Whiting, R.; Smith, C. *Analyst*, **1992**, 117, 1869.
26. Meyer, R. F.; De Witt, W. *US Geological Survey Bulletin*, **1991**, 1944, 14.
27. Smith, B. J.; *Low-Temperature and Dynamic Fatigue Toughing Mechanisms in Asphalt Mastics and Mixture*, MSc. Thesis, Department of Chemistry, Queen's University, Kingston, Ontario, Canada, **2000**.
28. <http://www.britannica.com/EBchecked/topic/505109/road/71907/New-paving-materials>, Accessed: **June 2011**.
29. Forbes, R .J.; *Studies in Ancient Technology*, **1964**, 4, 98, E.J. Brill, Leiden.

30. Abraham, H.; *Asphalts and Allied Substances: Methods of Testing: Industrial Raw Bituminous Materials*, D. Van Nostrand Company Inc., Princeton, New Jersey, Vol. 1, **1962**.
31. Pliny in Nature History, English translation by H. Rackham, Harvard University Press: Cambridge, Mass, **1978**.
32. Walsh, J.; *Geographical Club of Philadelphia Bulletin*, **1924**, 25, 92.
33. Nissenbaum, A. *Reviews in Chemical Engineering*, **1993**, 9(3-4), 365.
34. Nasir-I-Khusrau; *In Dairy of a Journey through Syria and Palestine*; PPTS, London: **1896**.
35. Gernsheim, H.; *In the History of Photography*; Thames, H., Ed.; London, **1969**.
36. Basu, A.; Marasteanu, M. O.; Hesp, S. A. M. *Journal of the Transportation Research Board*, **2003**, 1829, 1.
37. Marasteanu M. O.; Basu, A.; Hesp, S. A. M.; Volle, V. *International Journal of Pavement Engineering*, **2004** 5(1), 31.
38. Hesp, S. A. M.; Soleimani, A.; Subramani, S.; Marks, P.; Philips, T.; Smith, D.; Tam, K. K. *International Journal of Pavement Engineering*, **2009**, 10(3), 209.
39. Hesp, S. A. M.; Iliuta, S.; Shirokoff J. W. *Energy & Fuels*, **2007**, 21(2), 1112.
40. Anderson, D. A.; Marasteanu, M. O. *Journal of the Transportation Research Board*, **1999**, 1661, 27.
41. Bahia, H. U.; *Low Temperature Isothermal Physical Hardening of Asphalt Cements*, Ph.D. Thesis, Pennsylvania State University, PA, **1991**.

42. Lu, X., Soenen, P. and Redelius, P.; *Impact of Bitumen Wax on Asphalt Performance-Low Temperature Cracking, Proceeding of the Third Eurobitumen & Eurasphalt Congress*; Vienna, Austria, Paper 50, Section 4, **2004**.
43. Struik, L. C. E.; *Physical Aging in Amorphous Polymers and other Material*, Elsevier Scientific Publishing Co. Amsterdam, **1978**.
44. Petersen, J. C.; *Chemical Composition of Asphalt as Related to Asphalt Durability: State of the Art*. In Transportation Research Record 999, TRB, National Research Council, Washington, D.C., 13, **1984**.
45. Becker Y.; Méndez, M. P. and Rodríguez, R. Y. *Vision Tecnologica*, **2001**, 9(1), 39.
46. Lewandowski, L.H. *Rubber Chemistry and Technology*, **1994**, 67(3), 447.
47. Newman, J. K. *Journal of Elastomers and Plastics*, 1998, 30(3), 245.
48. Yildirim, Y. *Construction and Building Materials*, **2007**, 21, 66.
49. Bahia, H. U.; Perdomo, D. *Journal of the Transportation Research*, **1997**, 1586, 16.
50. Kim, S .S.; Sargand, S. M.; *Performance Evaluation of Polymer Modified and Unmodified Superpave Mixes*. In: Second International Symposium on Maintenance and Rehabilitation of Pavements and Technological Control, Auburn, AL, **2001**.
51. Terrel, R.; Walter, J. *Proceedings of the Association of Asphalt Paving Technologists*, **1986**, 55, 482.
52. Thompson, D. C.; Hoiberg, A. J.; Editor. *Bituminous Materials: Asphalt Tars and Pitches*, Robert Krieger Publishing Co., **1979**.

53. The Magazine of the Asphalt Institute; *Understanding Asphalt Pavement Distresses - Five Distresses Explained*, [http://www.asphaltmagazine.com/singlenews.asp?item\\_ID=%201812&comm=0&list\\_code\\_int=mag01-int](http://www.asphaltmagazine.com/singlenews.asp?item_ID=%201812&comm=0&list_code_int=mag01-int), Accessed: **June 2011**.
54. Brown, E. R.; Kandhal, P. S.; Zhang, J.; *Performance Testing for Hot Mix Asphalt*, National Center for Asphalt Technology, Auburn University, Auburn, **2001**.
55. Paris, P. C.; Erdogan, F.; *A Critical Analysis of Crack Propagation Laws*, In Transactions of the ASME, Journal of Basic Engineering, Series **D**, 85, No. 3, **1963**.
56. Togunde, O. P.; *Low Temperature Investigations On Asphalt Binder Performance – A Case Study on Highway 417 Trial Sections*, MSc Thesis, Department of Chemistry, Queen’s University, Kingston, Canada, **2008**.
57. Jung, D. H.; Vinson T. S.; *Low-Temperature Cracking: Test Selection*. SHRP-A-400. Strategic Highway Research Program, National Research Council, Washington, **D.C- 1994**.
58. Anderson, D. A.; Lapalu, L.; Marasteanu, M. O.; Le Hir, Y. M.; Planche, J. P.; Didier, M. *Journal of Transportation Research Record*, **2001**, 1766, 1.
59. Pavement Tools Consortium; *Pavement Interactive guide*, <http://training.ce.washington.edu/PGI/>, Accessed: **June 2011**.
60. Laboratory-Pavement Materials; *Penetration of Bituminous Materials*, School of Civil and Structural Engineering, Nanyang Technological University, **2002**.
61. American Society for Testing and Materials; *Standard Test Method for Softening Point of Bitumen (Ring And Ball Apparatus)*, D36-95, **2002**.
62. US Department of Transportation: Federal Highway Administration; *Background of Superpave Asphalt Binder Test Methods*, Publication No. FHWA-SA-94-069, **1994**.

63. Anderson, D.A; Dongre, R.; *The SHRP Direct Tension Specification Test- Its Development and Use. In: Physical Properties of Asphalt Cement Binders*, John C. Hardin (Ed), ASTM STP 1241, American Society for Testing and Material, Philadelphia, pp.51- 66, **1995**.
64. AASHTO M320; *Standard Specification for Performance-Graded Asphalt Binder* American Association of State Highway and Transportation Officials, **2002**.
65. Andriescu, A.; Iliuta, S.; Hesp S. A. M.; Youtcheff, J. S. *Proceedings of the Canadian Technical Asphalt Association*, **2004**, 49, 93.
66. Iliuta, S.; Andriescu, A.; Hesp S. A. M.; Tam, K. K. *Proceedings of the Canadian Technical Asphalt Association*, **2004**, 49, 123.
67. Zhao, M. O.; Hesp, S. A. M. *International Journal of Pavement Engineering*, **2006**, 7(3), 199.
68. Yee, P.; Aida, B.; Hesp, S. A. M.; Marks, P.; Tam, K. K. *Journal of the Transportation Research Board*, **2006**, 1962, 44.
69. Bodley, T., Andriescu, A., Hesp, S. A. M., Tam, K. K.; *Journal of the Association of Asphalt Paving Technologists*, **2007**, 76, 345.
70. Anderson, M. R.; Walker, D.; Turner, P. *Journal of the Transportation Research Board*, **1999**, 1661, 69.
71. Button, J. W.; Hastings, C. P. *Proceedings of the Canadian Technical Asphalt Association*, 1998, 43, 48.
72. Heukelom W. *Proceedings of Association of Asphalt Paving Technologists*, **1966**, 36, 359.



73. Traxler, R. N.; Schweyer, H. E. *Proceedings of the Thirty-Ninth Annual Meeting*, American Society for Testing Materials, Atlantic City, NJ, 36(II), 544, **1936**.
74. Traxler, R. N.; Coombs, C. E. *Proceedings of the Fortieth Annual Meeting*, American Society for Testing Materials, New York City, NY, 37(II), 549, **1937**.
75. Traxler, R. N.; *Asphalt – Its Composition, Properties and Uses*. Reinhold Publishing, New York, **1961**.
76. Subramani, S. K. *Validation of New Asphalt Cement Specification Test Methods using Eastern Northeastern Ontario Contracts and Trial Sections*, MSc. Thesis, Department of Chemistry, Queen's University, Kingston, Ontario, Canada, **2009**.
77. Cotterell, B.; Reddel, J. K. *International Journal of Fracture*, **1977**, 13(3), 267.
78. Mai, Y. W.; Wong, S. C.; Chen, X. H. *Application of Fracture Mechanics for Characterization of Toughness of Polymer Blends*, In: *Polymer Blends, Volume 2: Performance*. D. R. Paul and C. B. Bucknall (Eds.), Wiley and Sons, Inc., **2000**.
79. Hashemi, S. *Polymer Testing*, **2003**, 22, 589.
80. Andriescu, A.; Gibson, N. H.; Hesp, S. A. M.; Qi, X.; Youtcheff, J. S. *Journal of the Association of Asphalt Paving Technologists*, **2006**, 75, 1.
81. Togunde O. P. and Hesp S.A.M.; *Physcal Hardening in Asphalt mixtures*, Technical Paper, (in the press: *International Journal of Pavement Engineering*), Queen's University, Kingston, Canada, **2011**.
82. Faba, M. (Supervisor: Simon Hesp); *Characterization of Modified Asphalts by Parallel Plate Dynamic Shear Rheology*, CHEM 417 Independent Research Project, Department of Chemistry, Queen's University, Kingston, Canada, **2007**.

83. Hesp, S. A. M.; Shurvell, H. F. *International Journal of Pavement Engineering*, **2010**, *11*(6), 541.
84. Petersen, J. C.; *A Review of the Fundamentals of Asphalt Oxidation*, Transportation Research Circular E-C140, **2009**.
85. Krishhnan, J. M.; Rengaraju, V. R., *International Journal of Fracture*, **1999**, *97*, 337.
86. Von Quintus, H. L.; Scherocman, J. A.; Hughes, C. S.; and Kennedy, T. W.; *Asphalt Aggregate Mixture Analysis System*, NCHRP Report 338, Transportation Research Board, Washington, **1991**.
87. Hesp S.; *An Improved Low Temperature Asphalt Binder Specification Method – Final Report*, Queen’s University, Kingston, Ontario, Canada, **2004**.
88. Amit Kanabar, *Physical And Chemical Aging Behaviour of Asphalt Cements from two Northern Ontario Pavement Trials*, MSc. Thesis, Department of Chemistry, Queen’s University, Kingston, Ontario, Canada, **2010**.
89. S. Nasrazadani, D.; Mielke, T. S.; Ramasamy, N.; *Practical Applications of FTIR to Characterize Paving Materials*, Technical Report 0-5608-1, University of North Texas, Denton, Texas, **2010**.
90. Kodrat, I.; John, D.; Hesp, S. A. M. *Journal of the Transportation Research Board*, **2007**, *1998*, 47.