QUALITY AND DURABILITY OF RUBBERIZED ASPHALT CEMENT AND WARM RUBBERIZED ASPHALT CEMENT

by

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A thesis submitted to the Department of Chemistry

in conformity with the requirements for

the degree of Master of Science

Queen's University

Kingston Ontario, Canada

(APRIL, 2013)

Abstract

This thesis discusses and documents findings from an investigation of performance-based testing of asphalt cement (AC), warm mixed asphalt cement, asphalt rubber (AR), and warm asphalt rubber. A number of control, warm, and asphalt rubber binders from Ontario construction contracts were investigated for their compliance with conventional Superpave® test methods such as rolling thin film (RTFO), pressure aging vessel (PAV), dynamic shear rheometer (DSR), and bending beam rheometer (BBR), as well as additional specification tests such as extended BBR and double edge notched tension test. The quality and durability of those binders were determined. Quality means the ability of asphalt binder to reach a set of specific properties whereas durability is the measure of how well asphalt retains its original characteristics when exposed to normal weathering and aging process.

One warm AC and two field-blended asphalt rubber samples showed high levels of physical hardening which can lead to premature and early cracking. The warm asphalt cement lost 8 °C when stored isothermally for three days at low temperatures according to Ontario's extended bending beam rheometer (BBR) protocol (LS-308). The two asphalt rubber samples lost 10 °C and 12 °C following the same conditioning. Many of the studied asphalt samples showed deficient strain tolerance as measured in Ontario's double-edge-notched tension (DENT) test (LS-299).

In a study of warm rubberized asphalt cement with improved properties, a number of compositions were prepared with soft Cold Lake AC and a small quantity of naphthenic oil. These binders showed little chemical and physical hardening and reasonable critical crack tip

opening displacements (CTOD). Strain tolerance was much improved by co-blending with a high vinyl type styrene-butadiene-styrene (SBS) polymer and a small amount of sulfur.

Acknowledgements

I would like to express my sincere thanks to my supervisor, Dr. Simon Hesp, for his continuous support in my whole study period. His constant encouragement has been a great help. Without his help this thesis would not have been possible. I would also like to thank my supervisory committee, Prof. Dr. Donal Macartney and Dr. Gary VanLoon for their continuous encouragement and advice.

I also wish to thank undergraduate students Hattie Xu and Andrew McIntyre for their assistance in collecting data for my thesis. Hattie helped me in collecting data for BBR and DENT test and Andrew helped me in collecting DSR data. I would also like to express thanks to my fellow lab mates Senthil Kumar Paul Samy, Henry Agbovi, Kelli-anne Johnson, Prashant Agrawal and Samuel Abiodun for their contribution in my study period. I would also like to express thanks to all faculty, staff and students of the Department of Chemistry at Queen's University for their valuable support.

I also wish to express thanks to the National Sciences and Engineering Research Council of Canada (NSERC), the Ministry of Transportation of Ontario (MTO), Imperial Oil of Canada, and E.I. du Pont Canada for their financial support.

Finally, I would like to give thanks to my parents, my wife Rita Adhikari and my son Manas Adhikari for their kind love, continuous support and encouragement.

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Abbreviations, Acronyms and Symbols

AASHTO	American Association of State and Highway Transportation Officials
AC	Asphalt Cement
ASTM	American Society for Testing and Materials
BBR	Bending Beam Rheometer
BC	Before Christ
CRM	Crumb Rubber Modifier
CTOD	Crack Tip Opening Displacement, mm
DENT	Double-Edge-Notched Tension
DSR	Dynamic Shear Rheometer
HMA	Hot Mix Asphalt
eBBR	Extended Bending Beam Rheometer
EWF	Essential Work of Failure
EVA	Ethylene Vinyl Acetate
FB	Field Blend
LS	Laboratory Standard Test Method
LTPPBind®	Long Term Pavement Performance Binder Selection Software
m(t)	Slope of the Creep Stiffness Master Curve (m-value)
MPa	Mega Pascal (Pa)
MTO	Ministry of Transportation of Ontario
NSERC	Natural Sciences and Engineering Research Council of Canada
PAV	Pressure Aging Vessel

PI	Penetration Index
PMA	Polymer Modified Asphalt
PPA	Polyphosphoric acid
RTFO	Rolling Thin Film Oven
SBS	Styrene-Butadiene-Styrene
SHRP	Strategic Highway Research Program
SIS	Styrene-Isoprene-Styrene
S(t)	Time-dependent Flexural Creep Stiffness, MPa
SUPERPAVE®	SUperior PERforming Asphalt PAVEment
TB	Terminal Blend
TFOT	Thin Film Oven Test
WEO	Waste Engine Oil

Symbols

a	Length of a Sharp Crack, m
A	Temperature Susceptibility Parameter (Penetration Test)
b	Beam Width, 12.5 mm
B	Specimen Thickness, m
G*	Complex Shear Modulus, Pa
G’	Storage Modulus, Pa
N	Number of Load Repetitions
P	Penetration (Penetration Test)

P	Load applied, N (BBR)
T	Temperature, K
T	Loading time, s
W_e	Essential Failure Energy, J
w_e	Specific Essential Work of Failure, $J.m^{-2}$
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
β	Plastic zone shape factor
δ	Phase angle
τ	Shear stress
γ	Shear strain
δ_t	CTOD
σ_n	Net section stress or yield stress (N/m2)

CHAPTER 1

INTRODUCTION

Rubberized asphalt cement is also called asphalt rubber. It is a blend of asphalt cement, tire rubber, and certain additives (such as extender oil, natural rubber and polymers) in which the rubber component is at least 15% by weight of the total blend [1]. Approximately 12 million tires are used annually in rubberized asphalt cement in Ontario [1]. It is mixed at high temperatures so that it can react with the rubber and cause swelling of the rubber particles. The use of rubberized asphalt cement as pavement material has great attraction nowadays due to its high durability and high resistance towards aging. Rubberized asphalt cement exhibits more elasticity and reduces road noise. The pavement with conventional asphalt requires some sort of maintenance each year [1]. But the pavement with asphalt rubber generally has longer service life and it reduces maintenance cost due to its resistance to cracking and aging behaviour. Asphalt rubber being stiffer improves the environment by decreasing road noise and reducing disposable waste tires. However, the use of conventional asphalt rubber (AR) requires high mixing and compaction temperatures in hot mix asphalt that could raise emissions of harmful fumes and energy costs. In addition, the increase of rubber content in asphalt cement decreases the value of resilient modulus of mixture and thereby increases the flexibility. Furthermore, the increase in rubber content can reduce the indirect tensile strength of modified mixture [1]. So, the addition of warm mix additives on conventional asphalt rubber (i.e. warm mix asphalts) involves a significant reduction of mixing and compaction temperatures of asphalt mix by lowering the viscosity of asphalt binders. It reduces emissions and odours from the plants and makes the working condition better at the plant and paving site [3]. In addition, it saves time in production as well as in surfacing roads. It reduces the time and labor cost for compacting the mix because it makes compaction easier. Lower temperature also allows more asphalt mix to be hauled for longer distances and it reduces the transportation costs [2]. Warm mix asphalt can be produced by adding additives like Aspha-min®, Sasobit®, Rediset®, etc. to the asphalt binder before mixing. This results in lower consumption of fossil fuels and releases less carbon dioxide, aerosols and vapour. Aspha-min® is synthetic zeolite (sodium–aluminum–silicate) which is hydro-thermally crystallized as a very fine powder. It contains approximately twenty one percent crystalline water by weight. On addition of it to an asphalt mix, crystallised water is released which produces a volume expansion of the binder, and as a consequence binder viscosity decreases. Sasobit® is an organic wax having long chain aliphatic hydrocarbons. It is obtained from coal gasification. It is completely soluble in asphalt binder at temperatures above 120 °C. It forms a lattice structure in the binder after crystallization that can stabilize the binder. It can lower the viscosity of binder and thus reduce the working temperature.

During relatively short term conditioning at lower temperature in winter and long term exposure to oxygen, hardening of asphalt takes place and it has high chances of cracking. The rate of hardening depends on climate, air voids and components (i.e. types and quantities) of the asphalt binder. Traffic and low temperature exposure during winter increases the stress levels within pavement beyond their critical limits and it causes pavement cracks [4]. Finally, we have to spend lots of money for early rehabilitation and reconstruction of cracked pavement. Road construction plays a vital role in the development of sustainable infrastructure for all economies in all countries. Countries without roads and highways can be considered as underdeveloped and they have to do lots of struggle to achieve the level of wealth as developed countries. Countries like the United States and Canada spend billions of dollars for the repairing and development of their highway transportation system. The Ontario government spends around $300 million annually on the purchase of asphalt concrete for the construction and maintenance of roads and highways. So, there is an increasing interest for the detailed study of quality and durability of rubberized asphalt cement and warm mixed asphalt rubber [4]. Quality means the ability of asphalt binder to reach a set of specific properties whereas durability is the measure of how well asphalt retains its original characteristics when exposed to normal weathering and aging process.

1.1 Definition and Origin of Asphalt

Asphalt has different names and forms depending on the application, origin and location where it is found. In Europe, asphalt is called *bitumen* which is preferred to describe the semi-solid glue used for road paving while in North America and other countries of the world, the same material is called *asphalt cement or asphalt binder or simply binder* [10]. According to the American Society for Testing and Materials (ASTM 1998), asphalt cement is defined as a *"dark brown or black cementitious material occurring in nature or obtained by crude oil refining where the predominate material is mainly bitumen"* [10]. It varies widely in consistency from solid to semisolid at normal temperature. When heated sufficiently, asphalt softens and becomes a liquid, which allows it to coat aggregate particles during hot-mix production.

Asphalt binder behaves an elastic solid at low temperatures, a viscous liquid at high temperatures, a semi-solid at intermediate temperatures, or a solid mixture when combined with filler, sand, and aggregates. In this latter state it is often called *hot mix asphalt (HMA) or asphalt concrete*, the material used for the construction of flexible pavements [6].

The word “asphalt” is originates from the Greek word “asphaltos” which means “secure”. Similarly, the word “bitumen” originates from the Sanskrit word “jatu-krit” that means “pitch creating” [7]. In ancient times, asphalt was used as a mortar between bricks and stones, ship caulking and as a water proofing material. It was mainly used for adhesive purposes where water-proofing properties were desired. According to the Oxford English Reference Dictionary asphalt is defined as “*A tar like mixture of hydrocarbons derived from petroleum naturally or by distillation and used for road surfacing and roofing*” [8]. According to the American Heritage Dictionary, *“A brownish-black solid or semi-solid mixture of bitumen obtained from native deposits or as petroleum by-product is termed as asphalt that is used in paving, roofing and water proofing”* [9].

People have been aware of the adhesive and waterproofing properties of asphalt from long time. Surface accumulations of petroleum, forced upward by geological forces, leave behind naturally occurring lakes of asphalt that have hardened after exposure to the elements. Some examples of these deposits include Trinidad Lake Asphalt, on the island of Trinidad off the northern coast of Venezuela, and the La Brea ‘’Tar’’ Pits near Los Angeles. Natural asphalt is also found impregnated within porous rock such as sandstone or limestone called rock asphalt that was used by the ancient Babylonians, Egyptians, Greeks and Romans as road-building and waterproofing materials. The evolution of asphalt as widespread ingredient in paving material did not occur even with the long history of asphalt usage until the development of modern petroleum-refining techniques. These techniques were developed in the early 1900’s [10].

The use of rock asphalt as a side walk surface occurred was first documented in France in 1802 and later in Philadelphia in 1838. In 1870, the first asphalt pavement was constructed in Newark, New Jersey [8]. The first sheet asphalt (fine sand mix) pavement was built in

Washingston, D.C. in 1876 with imported lake asphalt. Later, in 1902 asphalt began to be refined from petroleum, which led to the development of the asphalt paving industry in the U.S. [10].

1.2 Asphalt Concrete Pavement

Asphalt concrete pavement is commonly familiarized as hot mix asphalt (HMA) pavement because it is mixed, placed and compacted at high temperatures. It represents the bound layers of a flexible pavement structure. Asphalt concrete is placed as HMA for most applications that is a mixture of course and fine aggregates and asphalt binders. The HMA is usually applied in 4-8 inch thick layers with the lower acting to support the top layer known as the surface or friction course. The aggregates in the lower layers are selected to prevent rutting and fatigue failure, while the top layer or the aggregates in the surface course are chosen for their friction properties and durability [11]. In this case, a properly and well mixed asphalt binder and aggregates are produced by heating to a temperature of approximately 180 °C in a central mixing plant. The main purpose of heating the asphalt is to decrease its viscosity and enhance the formation of a homogeneous mixture. When the asphalt mixture is still hot through the use of a mechanical spreader and rollers, paving and compaction is done. Thus, construction of the asphalt pavement involves the use of surface and binder courses while other additives can be added to increase the strength of the pavement [10].

A pavement's structure consists of three main components: foundation, base and surface layer. All the components play an important role in the overall performance of a pavement. The foundation is made up of the subgrade or the sub-base. The foundation carries the loads produced by construction traffic. From the structural point of view, it is the final layer to which stress is transferred. The base layer is a main structural element of the design and can have several layers.

The base layer spreads the wheel load so that the foundation is not over stressed. Its stiffness and its fatigue resistance characterize its behaviour in the pavement structure. The surface layer ensures adequate skids resistance and acts as a protective layer for the underlying materials [10]. Surface layers are characterized by their stiffness, creep resistance and moisture resistance. Other characteristics of surface layers include their resistance to low temperature cracking, fatigue resistance and skid resistance. Road pavements are divided into two groups namely flexible pavements and rigid pavements. Flexible pavements are surfaced with asphalt materials called hot mix asphalt. The total pavement structure bends or deflects due to traffic loads so these types of pavements are called flexible. A flexible pavement structure is generally made up of three layers or courses as shown in Figure 1.1.

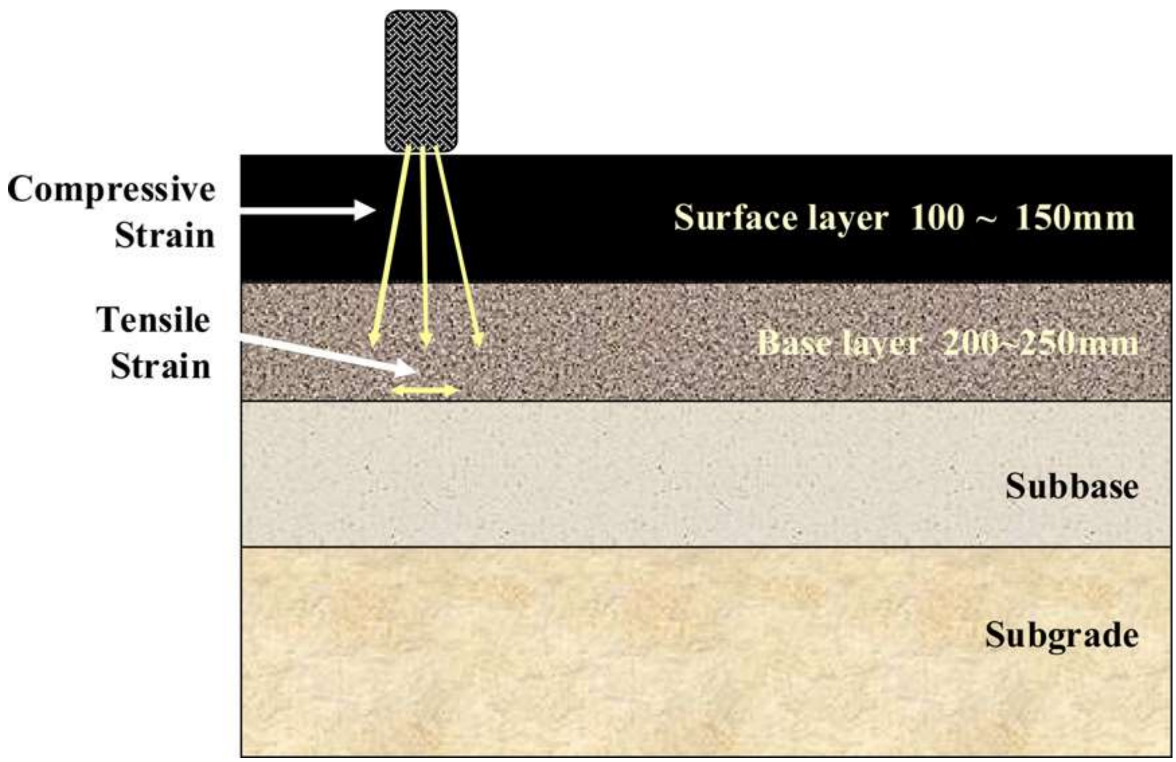


Figure1.1: Structural layers in an asphalt concrete pavement [12].

Rigid pavements are surfaced with Portland cement concrete (PCC). Since they are substantially stiffer than flexible pavements due to PCC's high stiffness, they are called rigid pavement.

Superpave® was developed in the early 1990's with funding from the U.S. Federal Highway Administration. According to Superpave® specification parameters, the design of a HMA pavement requires the knowledge of the traffic load, the drainage system and the sub-grade soil support. This becomes necessary since pavement life can significantly be altered by different traffic loads. Poor drainage systems and the load bearing ability of the underlying soil also can have significant effects on the general performance of the asphalt pavement. So, the following factors are considered for a good design of an asphalt pavement [13]:

(i) foundation thickness;

(ii) performance grading of asphalt binders;

(iii) air voids and

(iv) aggregate sizes

1.3 Source and Nature of Asphalt

There are two main sources of asphalt. These are natural asphalt and petroleum asphalt:-

<u>Natural Asphalt</u>: Natural asphalt is obtained from nature and it is found in "asphalt lakes'' around the world. Natural sources of asphalt are found in limestones or limestone mixtures [12]. The total amount of asphalt including tar sands has been estimated at 2.7×10^{12} barrels. About 95 % of natural asphalt cement referred to as tar sand is found in Canada. In reality, there is a

large deposit of Athabasca tar sands in Northern Alberta. The major sources of natural asphalt are in Pit Lake, Trinidad; Gard, Auvergne, Ain and Haute Savoie in France; Central Iraq; Latakia, Syria; Maestu, Spain; Butin Island, Indonesia; and the Dead Sea area in Israel and Jordan [15].

Table 1.1: Methods Used to Produce and Process Asphalt [12].

Production/Process	Base Material	Product
Atmospheric and Vacuum Distillation	Asphalt-based Crude or Crude Mix	Asphalt Cements
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

Petroleum Asphalt: Petroleum asphalt is obtained during the refinery process of heavy crude oils. Asphalt used for road construction is mainly produced from the refinery process. Different type sand grades of asphalt can be produced by using different processes. Vacuum and atmospheric distillations are the main processes that used in oil refineries to produce asphalt and other useful products [12]. Nowadays, most of asphalts used in North America are produced through the refinery of crude oil. The methods used to produce and process asphalt materials are shown in Table 1.1.

1.4 Composition of Asphalt

Some asphalt is manufactured from crude petroleum, a product formed naturally from organic matter over millions of years under different conditions of temperature and pressure. It is a highly complex material that needs to be characterized properly. It mainly consists of saturated and unsaturated aromatic as well as aliphatic compounds with estimated 100-200 carbon atoms. In fact, the composition of the asphalt depends on the type of crude oil from which it is derived. Basically, the molecular compounds within asphalt contain oxygen, nitrogen, sulphur and other heteroatoms. However, asphalt mainly consists of approximately 80 percent by weight of carbon, 10 percent by weight of hydrogen, 6 percent by weight of sulphur and remaining percent of oxygen, nitrogen and trace amount of metals like iron, nickel and vanadium [14]. The constituent compounds in the asphalt have molecular weights range from several hundred to many thousands. The hetero atoms present in asphalt contribute many of asphalt‘s unique chemical and physical properties. The type and amount of heteroatoms that exist in asphalt are function of both the crude source and its exposure to aging. Heteroatoms especially sulphur reacts more easily with oxygen and get oxidized [16]. These metals act as fingerprints enabling us to identify the source of the asphalt cement. Sometimes asphalt can be fractionated and evaluated by two methods, namely Corbett chromatography which uses the solubility in asphalt-suitable solvents and the Rostler method which uses precipitation techniques [16]. The molecular structure of asphalt is extremely complex and varies in size and type of chemical bonding with each blend or source. Basically, there are three basic types of molecules: aliphatic, cyclic and aromatics. The aliphatic molecules are also called paraffinic. They are linear, three-dimensional chain-like molecules and oily or waxy in nature. The cyclic molecules are also called naphthenic and they are three-dimensional saturated rings of carbon atoms with various atoms attached with it. The chemical bonds that hold the molecules together are relatively weak and they are easily broken by heat or shear stress. This explains the viscoelastic nature of asphalt. As the asphalt is heated, the intermolecular bonds are destroyed and it flows freely. As the asphalt becomes cool, the weak intermolecular bonds reforms and the chemical structure returns but not necessarily to the same as the original structure.

Most asphalt behaves pseudo-plastic or semi-solid at room temperature but it behaves like a Newtonian liquid at temperature above 60 °C. Asphalt is a visco-elastic material so its behavior can be explained by spring and dashpot models. The viscous behavior of asphalt is due to the presence of non-polar high molecular weight components of asphalt while the elastic behaviour of asphalt is due to the presence of polar components of asphalt. High molecular weight content of asphalt may result in brittle failure at low temperature conditions where as high polar content of asphalt gives more flexibility to asphalt [15]. Polymer-modified asphalt also shows the better performance at low temperature conditions following a similar trend with polar components. At high temperature conditions like in a desert climate, asphalt acts like a viscous liquid while at very low temperatures, it undergoes failure like an elastic solid. Both the polar and non-polar components should be at the right balance for better performance of roads [15]. Due to its complex composition, asphalt is often separated in two different chemical classes of constituents called asphaltenes and maltenes. Maltenes can further be classified into three groups: saturates, aromatics, and resins [17]. The compositions of saturated compounds are straight and branched-chain aliphatic hydrocarbons along with some alkyl-aromatics and alkyl naphthenic. Asphalt cement contains around 5 to 20 % saturates. Approximately 30 to 50 % of the total amount of asphalt consists of aromatics which are dark brown viscous liquids. Aromatics act as a solvent for other higher molecular weight hydrocarbons such as resins and asphaltenes and are made up of unsaturated ring systems. The resins are made up of hydrogen and carbon along with small amounts of oxygen, sulphur and nitrogen. They appear dark brown in color and are strongly adhesive to the aggregate in a pavement mixture. The main function of resins is the dispersion of the asphaltenes in the oils [8].

1.5 Properties of Asphalt

1.5.1 Chemical Properties of Asphalt

Basically, asphalt is composed of various hydrocarbons and traces of sulphur, oxygen, nitrogen and other elements. When asphalt is dissolved in a solvent such as heptane, it can be separated in two major parts: asphaltenes and maltenes. Asphaltenes do not dissolve in a linear hydrocarbon such as n-heptane because they are polar compounds with a high molecular weight. Their molecular weight ranges from 1,000 g/mole to as high as 100,000 g/mole on the basis of the method used to determine them [38]. Asphaltenes are usually black or dark brown in color as they are separated from maltenes and look something like course graphite powder. Asphaltenes give asphalt its color and hardness. Maltenes dissolve in heptane. They are viscous liquids composed of resins and oils. The resins are usually amber or dark-brown heavy liquids and the oils are lighter colored. The resign provides the adhesive qualities like stickiness in asphalt while the oil act as a medium in which the asphaltenes and resins are carried. The proportions of asphaltenes and maltenes in asphalts can change due to a number of factors such as high temperatures, exposure to oxygen and light, type of aggregate used in the pavement mixture and the thickness of the asphalt film on the aggregate particles. The changes that occur include evaporation of more volatile components, oxidation, polymerization, and other chemical reactions that can significantly influence the properties of asphalt. During these reactions, the resins gradually change in to asphaltenes and the oils change in to resins resulting in an overall increase in asphalt viscosity [39].

1.5.2 Physical Properties

Asphalts are mainly characterized by their physical properties because we do not have enough chemical knowledge of asphalt to predict its performance and its failure mechanisms that can be described chemically. The main physical properties of asphalt include durability, adhesion, temperature susceptibility and hardening and aging [39, 69].

1.5.2.1 Durability

Durability is the measure of how well asphalt retains its original characteristics during construction and service life. This is a property tested primarily through pavement performance and therefore difficult to define in terms of asphalt alone. The property is tested by Thin Film Oven Test (TFOT) and Rolling Thin Film Oven Test [39].

1.5.2.2 Adhesion and Cohesion

Adhesion is the ability of asphalt to stick to the aggregate in the paving mixture. Cohesion is the ability of asphalt to hold the aggregate particles firmly in place in the finished pavement. It is measured by ductility test.

1.5.2.3 Temperature Susceptibility

Temperature susceptibility is also an important physical property of asphalt. That characteristic of asphalts due to which they become harder as their temperature decreases and becomes softer as their temperature increases is called temperature susceptibility. All the asphalt show thermoplastics behaviour due to temperature susceptibility. Temperature susceptibility varies between asphalts from different petroleum sources even if the asphalts are of identical grade.

1.5.2.4 Hardening and Aging

Asphalt tends to harden or aged in the paving mixture during mixing and construction. The hardening of asphalt is primarily caused by oxidation. Oxidation is a process that takes place most readily at higher temperature and thin asphalt films. Asphalt coats the aggregate particles both at higher temperature and in thin films during mixing. This makes mixing most severe oxidation and hardening.

1.6 Asphalt Cement Performance Grade

Asphalt cement performance grade is also called Performance Graded (PG) binders is selected on the basis of climate and traffic at the location where it will be used. An example of a PG binder designation is PG 64–34. The first number 64 is the high temperature in degrees Celsius that the pavement is expected to reach. The second number –34 is the lowest service temperature expected. PG 64–32 asphalt is “soft” enough to resist low temperature thermal cracking down to a temperature of –34 °C, but “stiff” enough to prevent a pavement from rutting due to traffic during a very hot week when pavement temperatures reach 64 °C. Some PG binder specifications require the modification of asphalts with polymers or other chemicals to increase its properties and meet performance requirements [14]. These modifiers can reduce temperature susceptibility and age hardening, resist low temperature cracking and improve stiffness of the asphalt at high temperatures. However, the cost of modified asphalts is nearly twice as much as unmodified asphalts and so they are unlikely to be used on most local roads. There are not many problems with the high temperature grading test because it is reasonably able to predict rutting performance. However, the time given for the samples to condition in the low temperature test is insufficient. The low temperature at which asphalt cement is graded relates to the one-in-fifty-year lowest pavement surface temperature such that 98 % confidence needs to be obtained that in a given winter the pavement is not exposed to damage [14]. A 98 % confidence limit is supposed to be used because thermal cracks are very hard to repair once present. An error of only a few degrees can easily reduce the confidence level to less than 50 % [14].The current specification sets a limit on the creep stiffness and the slope of the creep stiffness master curve to limit damage at the pavement design temperature [14]. The high temperature grade is based on limiting rheological properties at the average highest seven-day surface temperature for an average summer in the contract location. The rheological properties relate better to rutting than those of the BBR that relates to cracking. In addition, the high temperature performance is affected to a large degree by the properties of the aggregate [18].

The Superpave® specification, a new grading method, is based on a measurement of the asphalt’s fundamental physical properties that are directly related to field performance through sound engineering principles. However, recent experiences have shown that the Superpave® specification that would show improvement over historical penetration and viscosity methods still needs significant improvement because asphalt binders graded by this method can show significant performance variations [19].

1.7 Deterioration of Asphalt Pavement

Deterioration of asphalt pavement refers to any break-down in the surface of pavement allowing more serious problems such as ravelling (loss of pavement material from the surface downward). This may be due to the various reasons such as ultraviolet exposure, traffic frequency, weather conditions, asphalt mix design, and compaction of the asphalt during construction. As the asphalt binder in the pavement wears, it turns the pavement surface gray from the black color of fresh asphalt and aggregate particles begin to break away. Consequently, it allows exposing the coarse aggregate. Deterioration of constructed asphalt pavement is natural because constituents of asphalt begin to break down over time and become affected by various factors such as rain, sunlight and chemicals that come into contact with the pavement surface. The liquid asphalt binder named “glue” of the pavement begins to lose its natural resistance to water allowing it to penetrate into the pavement. Then, the surface can quickly fall gradually to a number of different types of deterioration [24]. Furthermore, the presence of air voids is also one of the key factors responsible for the pavement failures or distress. This is due to the inhomogeneity between aggregate and asphalt binders which lead to adverse effects on hot-mix asphalt (HMA) due to significant stress and strain concentration at the interface between the two phases. The premature deterioration of asphalt pavement occurs usually due to failures in construction or human error.

1.7.1 Types of Deterioration

1.7.1.1 Cracking: All the pavement failure begins with crack. The cracking of asphalt is mainly due to the effects of sun and moisture and ground movement. When the crack is open, moisture reaches the pavement surface softens and expands it. The pavement begins to deteriorate around the crack. There are many different types of cracking that can occur in the asphalt pavement. They include alligator crack, edge cracks and slippage that is caused by improper compaction and reflection that is caused by older cracks occurring in a new overlay, etc. [24].

1.7.1.2 Distortion: This is also one of the important types of deterioration. This is caused by improper pavement construction, deterioration of the underlying base or existing asphalt and high load factors.

1.7.1.3 Disintegration: This type of asphalt deterioration includes potholes, ravelling, and gas and oil spillage. This kind of deterioration can be prevented by sealing the pavement with quality asphalt [24].

1.7.2 Causes of Deterioration

Deterioration of asphalt pavements can be due to different factors. When asphalt pavement is constructed and maintained properly, it deteriorates slowly and can sustain traffic for up to 25 years or more. It can be protected from the external factors by proper maintenance. Factors that cause deterioration in pavement are mentioned below [24].

1.7.2.1 Water: After long period of service life and especially without proper maintenance, water penetrates the asphalt and washes out the base underneath it causing it to crack. Finally it breaks and collapse.

1.7.2.2 Sunlight: Asphalt undergoes aging or oxidation over time in the presence of light. Oxidation breaks down and dries out the once flexible liquid asphalt that holds the aggregate together. This causes ravelling and shrinking cracks which allow water to penetrate beneath the surface.

1.7.2.3 Chemicals: The introduction of chemicals to asphalt including gas and oil can soften the asphalt and eventually cause it to break down more rapidly.

1.8 Scope and Objectives

The automobile industries in the world have developed very rapidly in recent years resulting in high amounts of scrap tire every year. The scrap tire may cause serious environmental effects if they are not properly disposed [71]. The pavement with conventional asphalt requires some sort of maintenance each year. The pavement with asphalt rubber generally has longer service life and it reduces maintenance cost due to its resistance to cracking and aging behaviour. The attraction of Crumb Rubber Modifier (CRM) in asphalt mixtures is that it increases the durability of the pavement by reducing the cracking and rutting potential of the pavement. The crumb rubber modifier binders can perform the same performance with styrene – butadiene - styrene (SBS) modified binders. However, the use of crumb rubber is preferred because it can provide a significant cost savings due to the high price of SBS and on the other hand it can also prevent the accumulation of waste [72]. The binder viscosity is affected by the aromatic oil and light fractions contents of the asphalt binder. As the crumb rubber is added into asphalt at high temperatures, the rubber particles absorb the aromatic oil and light fractions in the asphalt and swell in size resulting in a higher viscosity. Due to the process of swelling the free spaces between rubber particles decrease and they have less freedom to move into the binder matrix or move around and causes higher binder's viscosity than before interaction. The binder's viscosity at high temperature can be considered an important property as it represents the capacity of binder to pump through an asphalt plant, to coat aggregate thoroughly in asphalt concrete mix and to form a new pavement surface. So, the binder with crumb rubber requires a high mixing and compaction temperatures compared to the conventional hot mix asphalt [72]. Warm-mix asphalt (WMA) technology has been popular in recent years due to its capability to reduce the mixing and compaction temperatures of asphalt mixtures. Besides this, it makes the working conditions better and minimizes the effect on the environment. Warm mix asphalt lowers mixing temperatures at least 15 % as compared to conventional hot mix asphalt [22]. The mechanism by which it reduces the mixing and compaction temperature than conventional hot mix asphalt is that it reduces the binder's viscosity. The reduction of binder's viscosity permits the aggregate to be well coated at temperatures below than those used for hot mix asphalt. The use of warm mix reduces fuel consumption and reduced emissions from the plant. This can reduce the fuel costs around 25 % [22]. Besides these, there are several other advantages of using warm mix asphalt such as longer paving seasons, longer hauling distances, reduced aging of binders, reduced oxidative hardening of binders and thus reduced cracking in the pavements. A detailed study of rubberized asphalt cement with additives is necessary especially for the long term performance of asphalt pavement [22].

The objective of this project is to determine the quality and durability of warm mix rubberized asphalt cement. For this we determine high temperature and low temperature performance grade of various commercial samples from warm mix and rubber mix asphalt contracts as well as a number of laboratory samples by Superpave tests: Rolling Thin Film Oven (RTFO), regular and modified Pressure Aging Vessel (PAV), Bending Beam Rheometer (BBR), Dynamic Shear Rheometer (DSR) tests. Furthermore, it is aimed to determine maximum worst grade loss of the various asphalt binders by extended BBR test. Finally, we have an objective to obtain superior performing warm rubberized asphalt cements that could be used in future Ontario pavement trial to allow for an evaluation of pavement performance and reduced emissions for northern climates [4].

CHAPTER 2

BACKGROUND AND LITERATURE REVIEW

2.1 Reversible or physical hardening of asphalt

Physical hardening is a reversible process that takes place below room temperatures. As the asphalt binder is cooled and stored at a constant low temperature, the stiffness of binder increases and physical hardening takes place. On the other hand, when the asphalt binder is heated to room temperature or above, the effect of physical hardening is completely removed. Physical hardening in polymers was first reported by Struik [60] and it was later introduced in asphalt binders during the Strategic Highway Research Program (SHRP), i.e. a recent investigation of the rheological properties of paving grade of asphalt cement. Physical hardening is similar to physical aging for many amorphous solids such as polymers [27, 60]. Physical hardening for amorphous materials occurs below glass transition temperature whereas the physical hardening for asphalt binder is observed both above and below the glass transition [27]. When the asphalt cement is cooled down from high temperature, its volume shrinks due to a reduction in free volume and finally it attains equilibrium. Once the glass transition region is reached, the molecular transport mobility is reduced and achieves in non-equilibrium volumes. In this case, the asphalt is in a metastable state causing the material to continuously shrink isothermally. More closely packed molecular arrangement and reduced molecular mobility is obtained due to the decrease in free volume. The viscoelastic properties change with time and the material hardens. The physical hardening is time dependent and it depends on the temperature. The lower the temperature, the higher the hardening phenomenon and hardening rate. The hardening rate also depends on the chemical composition of the asphalt binder such as length of molecular chains, wax content, etc. [27, 60]. The hardening phenomenon may still occur at temperatures well above Tg due to partial crystallization of some components of the asphalt binder [69]. Physical hardening is important cause of asphalt cracking in the pavement.

2.2 Aging

Aging is broadly defined as the change of properties in asphalt pavement taking place in time and it is accompanied by significant hardening of the asphalt cement. The aging behaviour of asphalt binders are simulated in the laboratory to evaluate the effect of such aging on short- and long-term service conditions of pavement. Aging may be short-term or long-term. Aging that takes place in the asphalt during the preparation of the hot mix asphalt is called short- term aging and it occurs due to the rapid volatilization and oxidation of thermally unstable and volatile compound. This kind of aging is tested in the laboratory by the Rolling Thin Film Oven (RTFO) test. In contrast, the aging that takes place when the HMA is exposed to different climatic conditions especially at low temperatures and heavy traffic volumes between 5-10 years in-service is called long-term aging. This kind of aging is simulated in the laboratory by using the Pressure Aging Vessel (PAV) test. The oxidation occurs in the preparation and laydown of hot mix pavements as well as in service life of pavement due to environmental aging. The aging behavior of asphalt binders depends on temperature and time. For example, a high temperature within a short time appears to be equivalent to a lower temperature and longer time [25]. Oxidation of asphalt is generally considered to be a major factor contributing to the hardening and embrittlement of asphalt pavement. The hardenings of asphalt pavements happen due to the following changes:

i. changes in the chemical composition of asphalt;

ii. molecular structuring that produces thixotropic effects or steric hardening and; and

iii. loss of the oily components of asphalt by volatility or absorption by porous aggregates.

The asphalt binder becomes more brittle in nature as the oxidation takes place in presence of the organic molecules present in asphalt which causes an increase in viscosity resulting the pavement distress of the asphalt mixture.

There are two types of aging namely physical aging and chemical aging. Physical aging occurs as a result of the aggregation or densification of the thermally unstable and low molecular weight structures within the asphalt cement. It is also called reversible aging. It occurs at low temperatures where the asphalt cement undergoes free volume collapse, asphaltene aggregation and wax crystallization. Many studies have been done to evaluate the mechanisms by which physical aging takes place but, none of these mechanisms has successfully been able to predict the occurrence of the process. This is due to the complex composition of asphalt that changes widely with different sources. According to Petersen [26], three main causes for physical aging involve: (1) amount of composition present in asphalt changes with oxidation reactions; (2) decrease in maltenes content due to volatilization or adsorption and (3) slow crystallization of waxes and rearrangement of asphaltene and resin molecules. According to Struik [27], the reversible aging can be explained by using free-volume collapse theory at low temperatures. In a study done by Hesp et al. [28] in reversible aging of asphalt at low temperatures, they found 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. So, it is understood that physical aging occurs as a result of free-volume collapse and reduction in the mobility of the asphalt binder. The composition of an asphalt binder is very essential in oxidative hardening and affects the interaction between binders and the aggregates

[29]. The reason behind it is as the asphalt binder is heated, volatilization occurs which leads to hardening properties of the binder. It is also noted that air blowing of asphalt binders also increases the aging and hardening. Chemical aging occurs in asphalt cement due to oxidation during hot mixing, construction and in service periods. It is due to the oxidation of asphalt cement in air. Asphalt is a complex mixture of organic molecules so the presence of oxygen in the atmosphere, ultraviolet radiation on its surface, and changes in temperature influence the chemical aging process. It results in the increases in stiffness of the asphalt binder and makes it hard and very brittle which leads to premature cracking [12].

2.3 Failure Modes in Asphalt Pavement

The failure modes are also known as distresses in asphalt. The failure modes in asphalt arise because of two factors: environmental due to weathering and aging and structural caused by repeated traffic loadings. The rate at which pavement distress appear depends on its environment, traffic loading conditions, original construction quality and interim maintenance procedures. Poor quality materials or poor construction procedures can significantly reduce the life of a pavement. Consequently, two pavements constructed at the same time may have significantly different life times or certain portions of a pavement may deteriorate more rapidly than others. Therefore, timely and effective maintenance can extend a pavement’s life. Crack sealing and seal coating can reduce the effect of moisture in aging of asphalt pavement. The four major distress conditions affecting the performance of asphalt pavements are permanent deformation or rutting, fatigue or load associated cracking, low temperature or thermal cracking and moisture damage [31].

2.3.1 Permanent Deformation (Rutting)

Permanent deformation or pavement rutting is the accumulation of permanent deformation in all or a portion of the layers in a pavement structure that forms a distorted pavement surface. It happens at high temperatures during summer because the asphalt becomes softer at higher temperature [30]. Heavy loads at low frequencies or high temperatures are the major causes of this failure. Rutting occurs primarily under hot climatic conditions where the binder viscosity is reduced and flow can occur under traffic loading. Eventually, it results in the formation of ruts or tracks in the asphalt surface layers. Strain accumulates in the pavement and permanent deformation takes place. Deformation results as a formation of ruts or tracks on the surface of asphalt pavement as shown in Figure 2.1 [30]. Surface depression occurs in the wheel path. Pavement uplift may take place along the sides of the rut. Ruts are particularly visible in a rain when they are filled with water. There are two basic types of rutting: mix rutting and subgrade rutting. Mix rutting occurs when the subgrade does not rut yet the pavement surface exhibits wheel path depressions as a result of compaction or mix design problems. Subgrade rutting takes place as the subgrade exhibits wheel path depressions due to loading. In this situation, the pavement settles into the subgrade ruts causing surface depressions in the wheel path. As it pulls vehicles towards the rut path only, it may be hazardous. Repair or maintenance is done by levelling up the deeper ruts or by overlaying a new lift of asphalt concrete. However, ruts which form in lower layers of the pavement will return very fast after a single lift overlay [31].

Figure 2.1 Ruts on wheel path [30].

2.3.2 Fatigue Cracking

This mode of failure happens in asphalt pavement due to cycling loading of vehicles at moderate and low temperature conditions. The environment and climatic condition along with the pavement structure, mixture composition and construction play important roles for the fatigue cracking [31]. This is also called alligator cracking because it looks like the skin of an alligator. Cracks start at the bottom of the asphalt surface where tensile stress and strain are highest under a wheel load. Initially, the cracks propagate to the surface as a series of parallel longitudinal cracks. After repeated traffic loading, the cracks connect forming many sided and sharp-angled pieces that develop a pattern same as the skin of an alligator. The pieces are generally less than 0.5 m or 1.5 ft on the longest side. Alligator cracking takes place only in areas where repeated traffic loading such as wheel paths occurs. Early repair is one of the best ways to reduce this kind of crack. This can be characterized by loss modulus obtained from dynamic shear rheometer (DSR) test.

Figure 2.2: Alligator-type fatigue cracks caused by load or traffic distress [32].

2.3.3 Thermal Cracking

Thermal cracks are transverse cracks of pavement perpendicular to the road centerline and are generally equally spaced along the road as shown in Figure 2.3. Thermal cracking occurs predominantly in a cold region. This kind of cracking is mainly observed on asphalt pavements in North America and the northern US. This is because of unusually large drops in temperature that can occur at regular intervals during cold winter months. It may occur in southern climates where harder asphalt that will crack at higher temperatures is typically used to combat hot weather pavement problems such as rutting [31]. At low temperature, shrinkage of the asphalt surface takes place and cold temperature significantly produces thermal stresses in asphalt pavement. Asphalt pavement is a heterogeneous material so the asphalt binder in pavement is shrinking more than the mix aggregates particles when temperature drops. This results the asphalt film to get thinner around aggregates. Thermal cracking is initiated in the asphalt pavement as temperature drops below the point where asphalt binder becomes brittle. In other words when the pavement contracts with falling temperatures, the asphalt binder cannot relieve the stresses and it cracks. Thermal cracking happens as a result of following three different phenomena [33]:

- A sudden drop in temperature below the limit that causes stress build-up above the strength of the mix. This is called single-event cracking.
- The loss of strength of asphalt mix as a result of repeated thermal stresses below the temperature limit. This is referred as thermal fatigue cracking.
- Freeze and spring thawing cycle with addition to heavy traffic and repetitive loading. This is called mixed load or thermal distress cracking.

The thermal cracks in the asphalt pavement usually appear during the first years after road construction. These types of cracks can be repaired by sealing of the cracks or by overlaying the surface with one or more lifts of asphalt concrete. However, these cracks sometimes rapidly reappear in the surface through so called reflection cracking [33].

Figure 2.3: Longitudinal and transverse cracks due to thermal effects [33].

2.3.4 Moisture Damage

Moisture damage is defined as the progressive deterioration of asphalt pavement due to loss of adhesion between asphalt binder and aggregate surface or loss of cohesion within the binder due to water [31]. Air voids and lack of proper compaction are major reasons for moisture damage in asphalt. Stripping takes place between the asphalt cement and the aggregate particles in the presence of water. This causes a lack of cohesion between the asphalt layers and allows water to seep in because the aggregates are attracted towards water while the asphalt cement is hydrophobic in nature. Thus the water wets the aggregates forming a layer between the asphalt cement and aggregates which results in stripping. This may happen after fatigue cracking and rutting. This failure damages the base structure due to this poor chemistry between aggregates and surface as shown in Figure 2.4 [34]. Moisture damage can be prevented by proper drainage, the use of anti-stripping agents and proper compaction.

Figure: 2.4 Moisture damage [12].

2.4 Viscoelastic Nature of Asphalt

The deformation of asphalt binders takes place when subjected to loads. The properties of asphalt also change with varying temperatures. The deformation is a combination of elastic response and viscous flow [35]. They exhibit both elastic and viscous behavior and are thus called visco-elastic materials because they show viscous as well as elastic properties depending on climatic conditions such as temperature and loading time. They show a purely elastic behavior at very low temperatures whereas at intermediate temperatures they behave viscous materials and at high temperature they can flow completely same as other liquids. The stress-strain behavior defines the response of materials to a load. A typical elastic, viscous, and viscoelastic response to an applied stress is shown in Figure 2.5. An elastic material experiences recoverable deformation when subjected to a constant creep load as shown in Figure 2.5a. An elastic material will immediately deform and maintain a constant strain when loaded as shown in Figure 2.5b. The material will immediately return to its initial shape when the creep load is removed. A viscous Newtonian material by applying a constant load will deform at a constant rate until the load is removed as shown in Figure 2.5c. However, the deformation of the viscous material will remain after the load is removed. Thus, viscous materials show non-recoverable deformation. A viscoelastic material on applying a creep load experiences an immediate deformation followed by a continued time-dependent deformation as shown in Figure 2.5d.

The immediate deformation regarding to the material's elastic response and the time-dependent deformation corresponds to the material's viscous response. As the stress or strain is applied on a material, rearrangement take place inside the material [36]. These types of materials are called elastic because they are able to return to their original shape after the removal of stress. It means that when a constant load is applied to an elastic material, the strain of the material is proportional to the applied stress (Hooke's law, $\tau = G\gamma$, where τ is the shear stress and γ is the

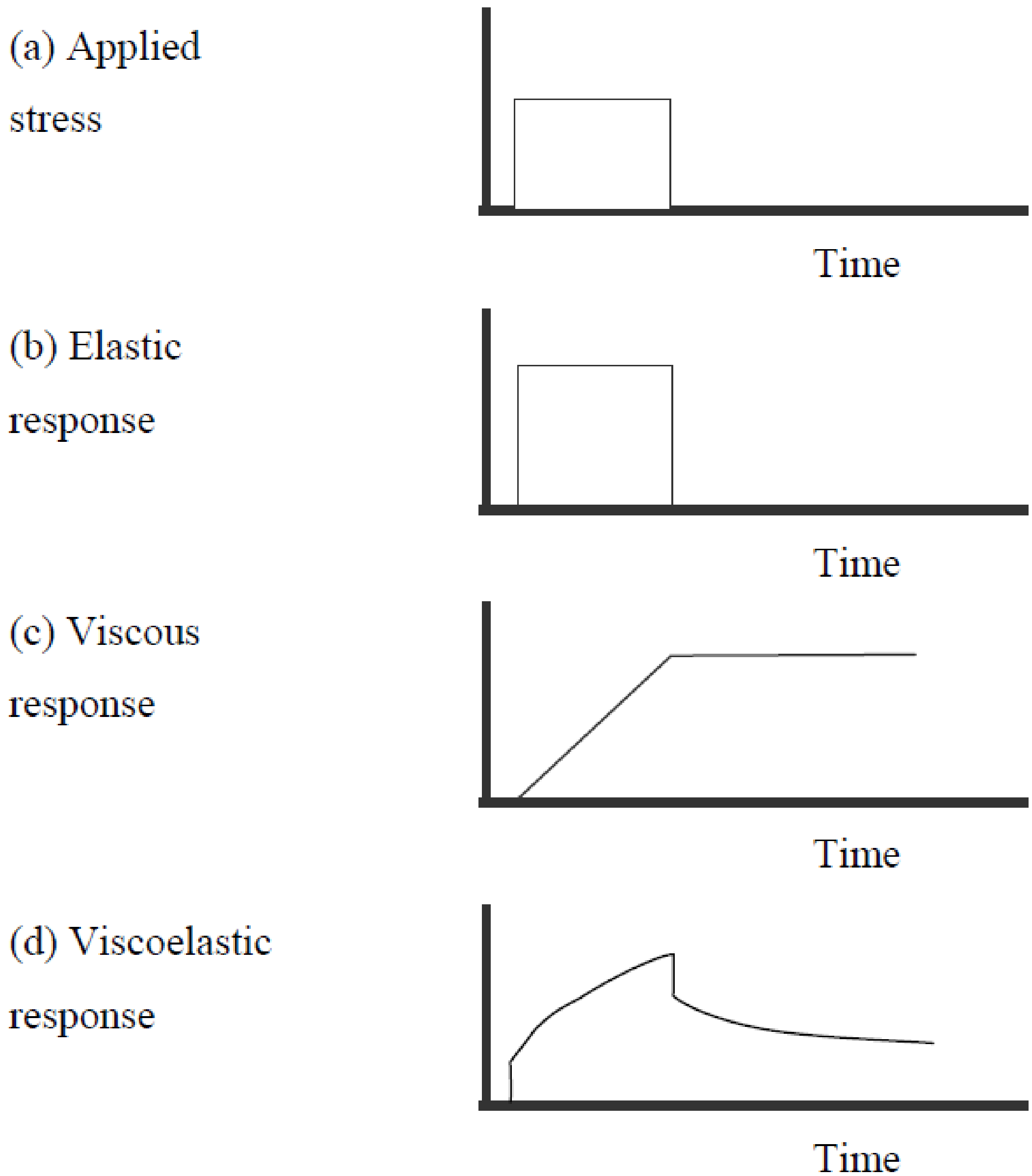


Figure 2. 5 Idealized responses of elastic, viscous and viscoelastic material under constant stress loading [35].

shear strain) and it gains complete recovery to the original position when the applied stress is removed from the material. The stress-strain behavior of these materials is time-independent and can be characterized by an elastic modulus. On the other hand, purely viscous materials deform continuously and permanently by applying a constant stress and they cannot return to their original position when the stress is removed. So, they are called non-recoverable materials and are characterized by viscous modulus. Some materials dissipate the input energy which leads to permanent deformation. The stress-strain behaviors of these materials are time-dependent and they are characterized by their elastic and viscous moduli. They exhibit both elastic and viscous behavior and are thus called visco-elastic materials. Hence, a viscoelastic material experiences only a partial recovery of the deformation resulting from creep loading. The viscoelastic behavior of asphalt can be characterized by its deformation resistance and the relative distribution of that resistance between the elastic component and the viscous component within the linear range. The relative distribution of the resistance between the elastic component and the viscous component is dependent on the asphalt cement characteristics, temperature and loading rate. The linear loading response is characterized by the deformation being directly proportional to the applied load at any time and temperature. Nonlinear loading responses are difficult to model for viscoelastic materials like asphalt [37, 38].

2.5 Modification of Asphalt

By selecting the proper starting crude materials or by changing the refinery processes, the properties of asphalt binders can be improved. Many asphalt industries have introduced additives to modify the properties of asphalt binder. To obtain improved performance, different varieties of polymeric materials are used to modify asphalt binders. Some asphalt binders may be required modifiers such as polymers to meet low and high temperature requirements. Some of these modifications include polyphosphoric acids which help to increase the temperature range in which satisfactory performances can be achieved, air blowing which makes the asphalt binder hard, fluxing agents or diluents oils which soften the asphalt 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 [12]. Although modifiers may affect many properties, the most of the modifiers attempt to decrease the temperature dependency and oxidation hardening of asphalt binder mixtures [43]. Modification of asphalt binders with different polymers, fillers, and fibers may reduce the susceptibility of asphalt pavement to low temperature cracking [40]. There are mainly two groups of polymers used as asphalt modifiers: plastomers and elastomers. Plastomers are those materials which when stretched will largely remain in the stretched position after releasing load .For example, ethylene vinyl acetate (EVA), and ethylene-acrylate (EA), polyethylene (mostly low density polyethylene, LDPE), etc. They are used in asphalt pavements to decrease permanent deformation or rutting. On the other hand, elastomers are those materials which will largely return to their original shape after removing the load. They are 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. For example, styrene-butadiene rubber latex (SBR), diblock styrene-butadiene (SB) and triblock styrene-butadiene-styrene (SBS), etc. They also reduce rutting, fatigue cracking and thermal cracking. The asphalt modified with styrene-butadiene copolymers is aimed at improving the rheological properties of the original asphalt. This gives mechanical resistance under particular load characteristics and types of climate in the case of pavements. In addition, it improves adhesion and mechanical properties and flexibility in the case of waterproofing agents. Furthermore, it improves mechanical properties under variable deformation in the case of sealants. Besides these groups, other groups are anti-stripping additives and acid modifiers. Anti-stripping additives are those which are used to reduce moisture damage and increase adhesion in asphalt binders and aggregates. For example, polyamines and fatty amino-amines, etc. [43]. Acid modification such as polyphosphoric acid (PPA) is nowadays used to modify asphalt. It is used to increase the softening point of a binder without lowering the penetration value. The use of such modifier helps to increase the grading range at high temperature but have some negative effects on long term i.e. fatigue performance according to the source of asphalt [41, 42]. Polymer modification of asphalt results in improvements in performance and durability, reduction of pavement distress as compared to unmodified asphalt. Such modifications have showed demonstrable reductions in rutting, thermal cracking, and increased resistance to many forms of traffic-induced stress. The major benefits of asphalt modification include the following [43]:

- Improve binder-aggregate adhesion
- Improve rutting resistance
- Improve consistency
- Improve stiffness and cohesion
- Reduce temperature susceptibility
- Improve flexibility, resilience and toughness
- Improved resistance to in-service ageing

The modification of asphalt binders had started as far back as 1843 with much usage in Europe as compared to North America in 1950. The application of various modifiers in asphalt has great attraction in the places like Europe, North America and U.S. to improve the performance of asphalt pavements over the last three decades [44].

2.6 Applications of Asphalt

Since asphalts binders are viscoelastic materials, they become liquid and flow like water at very high temperature and very hard at extremely low temperature. Therefore, they are used in different purposes. In ancient time, asphalts were used as adhesives, waterproofing material, mummification, etc. In the Gilgal Site, a stone axe head was found which was covered with asphalt to glue it to the wooden handle [45]. Asphalts were used for making small decorated utensils such as bowls, for coating statues in making ornaments and so on. Asphalt had used as a water proofing material around 3500 BC in ship building by the Sumerians and also used to waterproof temples and bands and water tanks by the ancient Phoenicians, Egyptians and Romans. They were also used asphalt as a binding material for bricks. The most interesting used of asphalt was as medicine in the ancient Middle East, 2nd century AD [13]. The most famous doctor of medicine in the Roman and Medieval world used the Dead Sea asphalt for medical purposes. Professor A. Doctrovsky from Hasassah Medical School in Jerusalem discovered a valuable material recently called "bitupal," which is effective in treatment of certain skin diseases [46]. This material still is in use and has been manufactured by the Teva Pharmaceutical Company in Israel [47]. Besides these uses, they were also used in photography and in oil exploration in ancient time. The main application of asphalt as road building materials had reported in the late 18th Century. The use of asphalts as a road-building material has increased exponentially during 19th and 20th century along with the growth of the automobile industries. Asphalts play a vital role in maintaining and rehabilitating pavements. For every type of asphalt pavement failure or distress, there is at least one asphalt type that can treat the damage. There are four major types of pavement failures: cracking, distortion, disintegration, and slippery surface. Crack sealing is a very effective maintenance treatment of pavement failure if it is done early in the service life of the pavement. Hot mix asphalts (HMA) are commonly used in highways, airports, parking lots and other areas which carry heavy or high volume traffic. It is also used in paint and marker inks to increase the weather resistance and permanence of the paint and ink. In addition, it also makes the color much darker [48].

2.7 Test Methods

2.7.1 Conventional Test Methods

2.7.1.1 Penetration Test

The penetration test is the oldest asphalt cement grading test and it was introduced by Baber Asphalt Paving Company in 1888 [48].

Figure 2.6: Penetration test equipment [34].

This test has been used according to an ASTM standard since 1959. This test involves the measuring of the depth of a sewing needle pushed into a sample of asphalt under a specific condition of loading time and temperature. It classifies the asphalt rather than measuring its qualities. In this test, a needle of specified dimensions is allowed to penetrate a sample of asphalt cement, under a known load of 100 g, at a fixed temperature of 25 °C and for a known time of 5 s as shown in Figure 2.6 [49].

The distance travelled by the needle in to the asphalt cement is known as penetration. It is measured in units of decimillmeter, i.e. 0.1 mm. The lower the value of penetration, the harder the asphalt cement. For example, 80 Pen asphalt cement will have a needle penetration at 25º C of 8 mm means 80 times 0.1 mm. The main advantage of this method is that it is cheap and it gives a better result than the high temperature viscosity method in terms of low temperature performance. Penetration is related to viscosity and empirical relationships have been developed for Newtonian materials. As penetration is measured over a range of temperatures then the temperature susceptibility of the asphalt binder can be established. The consistency of the binder is related to the temperature changes by the equation log P = AT + K, where

P = Penetration at temperature T

A = Temperature susceptibility and K = Constant. The value of A which indicates the Penetration Index (PI) can be obtained from penetration measurements at two different temperatures T_1 and T_2 using the equation A = log (pen at T_1)-log (pen at T_2)/ (T_1-T_2). The PI values can be used to determine the stiffness or modulus of asphalt cement at any temperature and loading time [50].

2.7.1.2 Viscosity Test

Viscosity is the internal resistance offered by the liquid to flow and is a fundamental characteristic of asphalt cement. It shows how the material will behave at a given temperature and over range of temperature. The viscosity test was developed in the early 1960. The viscosity test measures the viscosity of asphalt. Both the viscosity test and the penetration test measure the consistency of asphalt at some specified temperatures and are used to determine the grades of asphalts. The advantage of using the viscosity test as compared with the penetration test is that the viscosity test measures a fundamental physical property rather than an empirical value. Viscosity is defined as the ratio between the applied shear stress and induced shear rate of a fluid**.** Viscosity tests measure the time required for the asphalt cement to flow through a calibrated glass tube. Viscosity values are obtained from this test at 60 ºC and 135 ºC.

2.7.1.3 Ring and Ball Softening Test

In this test method, the softening point of asphalt cement is determined in the range from 30 to 157 °C using the ring-and-ball apparatus immersed in distilled water at 30 to 80 °C or ethylene glycol at 30 to 110 °C as shown in Figure 2.7. It is also an indirect measure of viscosity [5]. In this test method, the hard asphalt sample is converted into a soft substance and temperature is measured at that point. The temperature at which the steel ball in the ring pushes the soft bitumen samples to a standard distance is measure of the softening point. It is used to measure the consistency of the asphalt binder. For an asphalt binder of a given penetration determined at 25 °C, the higher the softening point means the lower the temperature sensitivity. The softening point is important tool to classify bitumen, in establishing the uniformity of sources of supply, and indicates the tendency of the material to flow at elevated temperatures encountered in service [52].

Figure 2.7: Softening Point Sample [34].

2.7.2 Superpave Specification Tests

Due to the failure of conventional grading tests that provided binder properties with little or no correlation of pavement performance in service, a Strategic Highway Research Program (SHRP) was developed in the late 1980s and the early 1990s in the United States. The Strategic Highway Research Program (SHRP) spent nearly $150 million and ran for a period of five years for the development of a range of asphalt cement and mixture tests. The purpose of the test was to get better design pavements so that it lasts for the desired time. These new test methods have led to a new specification known as Superpave (Superior Performing Pavement) asphalt binder. This new specification was successful to group the grades of modified and unmodified asphalt binders according to their characteristic performance in different climatic conditions. The unique feature of Superpave is that the specified criteria remain constant but the temperature at which the criteria must be achieved varies for the various grades of the binders. The Superpave test measures physical properties that can be directly related to field performance by pavement designer's principles and are also carried out at temperatures that are encountered in –service. The intention of Superpave specification is to improve the performance by limiting the potential for the asphalt cement to contribute toward permanent deformation, low temperature cracking and fatigue cracking in the asphalt pavement. 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 58-28 grade is designed to be used in an environment where the average seven day maximum pavement temperature is 58 ^{o}C and the minimum pavement design temperature is -28 ^{o}C. At this grade, the asphalt binder will be able to resist permanent deformation at a maximum average seven day temperature of 58 ^{o}C and low temperature cracking at a lowest minimum temperature of -28 ^{o}C. There are not many problems with the high temperature grading test as compared to low temperature in that it is reasonably able to predict rutting performance. The time given for the samples to condition in the low temperature test is very much inadequate so, many pavements in northern climates are suffered from thermal cracking. The next sections will explain the Superpave specification tests in some more detail.

2.7.2.1 Rolling Thin Film Oven (RTFO) Test

The Rolling Thin Film Oven (RTFO) test was proposed by the California Department of Transportation in 1959 and approved by ASTM in 1970 and accepted by AASHTO in 1973. This test was developed as an improvement over Thin Film Oven test (TFOT) for short term aging of the asphalt binder. This is because aging time in RTFO (85 minutes) is less as compared to TFOT (5 hrs), rolling action of carousel can disperse modifier the effectively in RTFO and continuously exposes the fresh binder in heat and air flow in RTFO. This test is done to simulate aging that occurs during the hot mixing process or the early stages of the pavement life. It is expected from this test that the aged samples would reflect the rheological changes in the properties of the asphalt binders. It also provides a quantitative measure of the volatiles lost during the aging process. This test is performed by heating a moving thin film of asphalt binder in a RTFO oven at 163 °C for 85 minutes by placing the asphalt binder sample in small jars in a circular metal carriage that rotates within the oven as shown in Figure 2.8a [53].

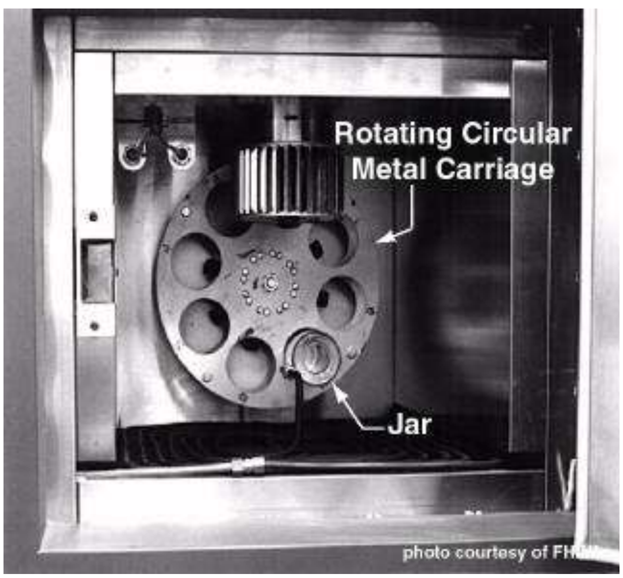


Figure 2.8a Rolling thin-film oven test [53].

Figure 2.8b RTFO bottles [53].

This aging process continuously exposes a film of asphalt binder to heat and oxidation similar to conditions experienced in the production of hot mix asphalt concrete. The asphalt binder's viscosity is increased due to the loss of volatiles from the asphalt binder which mainly occurs during the processes of manufacturing and placement. The RTFO has problems with highly viscous binders (e.g. some polymer modified asphalt binders) because they do not flow properly in the bottles as they are rotated.

2.7.2.2 Pressure Aging Vessel (PAV)

2.7.2.2.1 Test Origins

Two general approaches were considered in developing the procedure to simulate long term aging by oxidation. They are Oven Test and Pressure Test.

Oven Tests: These are relatively simple and quick and older than pressure tests. They depend on high temperatures and thin asphalt films to accelerate the oxidation process. However, asphalt binder samples lost a significant amount of volatiles during this aging process as well. In-service, the constituent asphalt binders did not lose significant amounts of volatiles was observed by field tests.

Pressure Tests: This approach has been around for over 40 years, although it was originally less popular than oven tests. These tests work by using high pressure to increase the diffusion rate of oxygen into an asphalt binder sample. In general during aging of asphalt binder, this approach limits the loss of volatiles.

SHRP researchers adopted the Pressure Aging Vessel (PAV) test to simulate the effects of long-term asphalt binder aging that occurs as a result of 8 to 10 years pavement service. Pressure Aging Vessel (PAV) test is the currently used method of choice for simulating the long-term aging of asphalt materials for physical property testing. The PAV is a combination of an oven and a pressure vessel method which is done using RTFO aged samples that are exposed to high air pressure of 2.1 MPa and temperature 90-110 °C for 20 hours as shown in Figure 2.9. PAV aged asphalt binder is used for later testing such as bending beam rheometer (BBR), double edge notched tension (DENT), dynamic shear rheometer (DSR), differential scanning calorimeter (DSC), direct tension (DT), and infrared (IR) spectroscopy tests, etc.

Figure 2.9: Pressure Aging Vessels (PAV) [53].

2.6.2.2.2 Reasons for PAV Time and Temperature [53]

This process is generally conducted for 20 hours at 90, 100 or 110 °C which were more based on practical approach instead of theoretical. Originally, these experiments were performed for 6 days at 60 °C and 2.1 MPa. The test period was deemed too long and the test results showed up insufficient aging. Hence the test temperature was raised in order to increase the aging rate by producing shorter test times. Initially, the temperature for the test was chosen as 100 °C but during field validation, it was found to be too mild for hot climates and overly harsh for cold climates. Thus, three elevated temperatures are used which simulate a different general environmental condition.

Table 2.1: Three elevated temperatures to simulate different environment condition

Temperature	Simulation
90 °C cold	cold climate
100 °C	moderate climate
110 °C	hot climate

2.7.2.3 Dynamic Shear Rheometer Test [53]

Dynamic shear rheometer (DSR) is used to evaluate rheological properties (complex shear modulus and phase angle) of asphalt binders from low to high temperatures and it is used to mainly to characterize the rutting resistance and fatigue cracking in asphalt binder. The complex shear modulus (G*) represents the sample's total resistance to deformation when repeatedly sheared and it is related to the applied stress and the resulting strain by the equation $G^* = \tau_{max} - \tau_{min} / \gamma_{max} - \gamma_{min}$, where $(\tau_{max} - \tau_{min})$ is the total shear stress and the $(\gamma_{max} - \gamma_{min})$ is the total shear strain. The complex shear modulus characterises the elastic component of the asphalt binder. The phase angle (δ) represents the lag between the applied shear stress and the resulting shear strain. It is used to predict the viscous component of the binder. The larger the phase angle (δ), the more viscous the material. For purely elastic material: $\delta = 0°$ and for purely viscous material: $\delta = 90°$. Mathematically, it is the ratio of the viscous modulus (G") to the storage or elastic modulus

(G’).i.e. δ= G”/ G’. The elastic modulus (G’) represents the elastic behaviour of the asphalt binder and loss modulus (G”) represents the viscous behaviour of asphalt binder.

The principle of this test is: a thin asphalt binder sample is sandwiched between two circular plates as shown in Figure 2.10. The lower plate is fixed while the upper plate oscillates back and forth across the sample at 25 ºC and 10 rad/sec to create a shearing action. The dimensions of the circular plates used are 8 mm for higher stiffness conditions or 25 mm for lower stiffness conditions and the gap between them is 2 mm. Three different temperatures are taken for the testing and a master creep curve is created to correlate with theoretical models. DSR tests are carried out on unaged, RTFO aged and PAV aged asphalt binder samples. The test is largely software controlled.

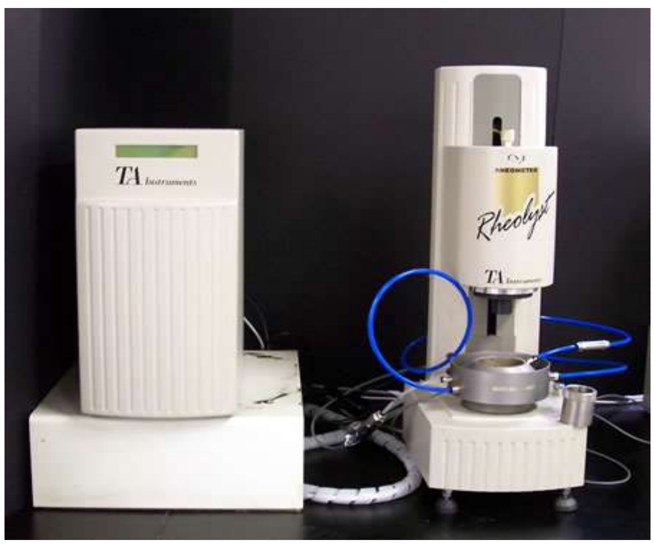


Figure 2.10: Dynamic Shear Rheometer (DSR).

According to DSR specification, the value of $G^*/\sin\delta$ should be minimum 1.0 kPa for unaged and minimum 2.0 kPa for the RTFO binder to prevent from rutting resistance and $G^*\sin\delta$ should be maximum 5000 kPa for the PAV residue to prevent from fatigue cracking.

2.7.2.4 Bending Beam Rheometer (BBR)

The bending beam rheomter (BBR) as shown in Figure 2.11 was developed under the Strategic Highway Research Program and is performed to simulate low temperature cracking of asphalt pavement in the service life. The specification criteria for passing or failing the BBR test is provided by the AASHTO standard M320 protocol [53]. The BBR specification test determines the low temperature cracking of asphalt as a major distress form for asphalt pavements. It measures of low temperature stiffness and relaxation properties of asphalt binders. These variables indicate the asphalt binder's ability to resist low temperature cracking.

Figure: 2.11: Bending Beam Rheometer (BBR).

In this test method, both the testing temperature and conditioning temperature are the same. The binder samples are conditioned for one hour at room temperature and then conditioned at 10 ºC and 20 ºC above the pavement designed low temperature grade in refrigerators for one hour before testing and low temperature grade is determined. A small asphalt beam that is simply supported and immersed in a cold liquid bath in a basic BBR test as shown in Figure 2.12. The deflection against time is measured when a load is applied to the center of the beam. A measure of how the asphalt binder relaxes the load induced stresses is measured along with the stiffness

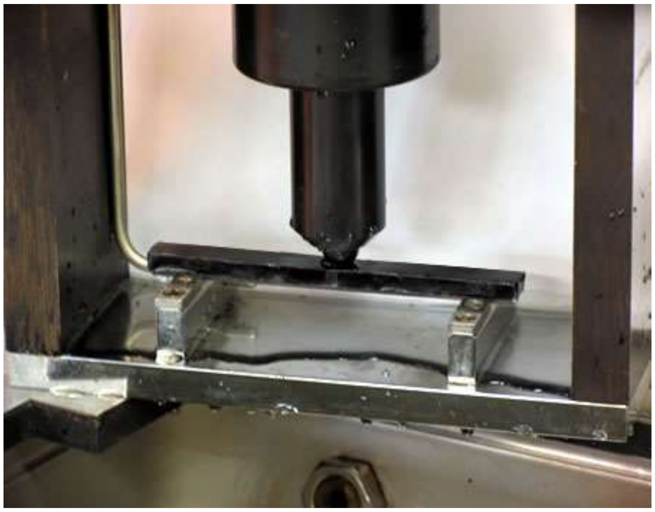


Figure 2.12 BBR beam on support [54].

that is calculated on the basis of measured deflection and standard beam properties. Creep stiffness is a measure of the thermal stresses in the asphalt binder resulting from thermal contraction. The higher the thermal stresses, the higher will be the chances of cracking. Higher thermal stresses are determined by higher creep stiffness. The BBR calculates beam stiffness (S (t)) on the basis of beam theory as follows:

$$S(t) = PL^3/4bh^3\delta(t)$$

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

P = applied constant load, 980 mN

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, $t = 60$ sec

The condition for passing the test is: stiffness (S-value) is below 300 MPa and the creep rate or slope of the creep stiffness master curve (m-value) is greater than or equal to 0.300. If either the stiffness is above 300 MPa or the m-value is below 0.3 then the asphalt cement fails the specification and it cannot be used in a very cold climate location.

2.7.3 Ontario Improved Low Temperature Specification Tests

A significant number of researchers have studied the effects of isothermal conditioning on the low temperature rheological properties of asphalt cement in the 20th century. Traxler became cautious that physical hardening mechanisms need to be considered when measuring rheological properties at low temperatures in asphalt cement [55, 56]. According to Struik, it is useless to study properties such as creep and stress relaxation without taking physical aging into consideration [57]. The Ministry of Transportation of Ontario (MTO) in collaboration with Queen’s University has embarked on the development of new test methods namely extended bending beam rheometer, eBBR test (LS-308) and double edge notched tension (DENT) test (LS-299) to deal this problem facing the AASHTO M320 specification [58]. The newly developed test methods give a reproducible result which consistently gives the better ability to predict either good or poor performance of asphalt cement.

2.7.3.1 Extended BBR Testing According to MTO Method LS-308

Extended bending beam rheometer (eBBR) is extension or modified form of BBR test. In this test, asphalt beams are conditioned for 24 hours and 72 hours instead of only one hour and then limiting temperature and maximum grade loss is determined after 72 hours. Asphalt binders with low quality contain large quantities of wax and unstable asphaltene dispersions. They lose their ability to relax thermal stress, i.e. decrease in m-value to some extent when cooled for periods of up to several weeks and months during the winter season. Therefore, the regular BBR cannot predict the thermal cracking performances of the pavements because they are under-designed, i.e. samples are conditioned for only one hour before testing [60]. The regular tests are performed to determine a pass and fail temperature after one hour of conditioning. The test temperatures are selected so as to determine a pass and fail temperature according to regular AASHTO M320 criteria (S = 300 MPa or m = 0.3). The m-value and creep stiffness is determined as per the extended BBR protocol where they are stored for one hour, 24 hours, and 72 hours conditioning at 10 ºC and 20 ºC above the lowest temperature designed for the pavement before they are tested [59] . The two temperatures are selected over the total range of 20 because the molecules merge and cannot move at higher temperature, i.e. above 20 and then there is absence of thermodynamic tendency for wax crystallization and precipitation of asphaltene molecules. As a result, physical aging tendency enhances at very low temperatures. The limiting temperatures are recorded and from which the maximum worst grade loss is calculated. The worst grade loss is the warmest minus the coldest limiting temperature where S reaches 300 MPa or m reaches 0.3. The worst grade is also observed along with worst grade loss after three days of conditioning. The worst grade temperature is determined at -10 ºC from the warmest limiting temperature where either S = 300 MPa or m = 0.3. According to LS-308 specification, the maximum grade loss should be less than 6 ^{o}C than the regular BBR after three days of conditioning which helps to detect very poor quality asphalt cement. This test provides 95 % accuracy and is also easy to repeat and reproduce. This method guarantees minimum levels of quality and durability because it provides a vast improvement over the AASHTO M320 specification. The objective of this method is to provide a reasonable degree of confidence that thermal cracking is completely prevented.

2.7.3.2 Double Edge Notched Tension (DENT) Test (MTO LS-299)

The double-edge-notched tension test (DENT) was recently developed at Queen's University [12] and it is performed to determine the ductile fracture or failure of asphalt binder at low temperature. It involves pulling of a notched asphalt binder in a water bath at isothermal condition i.e. at 15 °C and loading rate of 50 mm/min until fracture takes place. The essential work of fracture and the plastic work of fracture are determined. The integrated area under the force-displacement curve gives the total work of fracture which is 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 formed with the intercept on w_t axis gives the essential work of fracture and the slope gives the product of the plastic zone shape factor and the essential plastic work of fracture.

$$W_t = W_e + W_p = LBw_e + \beta L^2 Bw_p \quad (1)$$

$$\text{and } w_t = W_t/LB = w_e + \beta w_p L, \quad (2)$$

where w_t is the specific total work of fracture (J/m^2), W_e is the total essential work of fracture (J), W_p is the total plastic work of fracture (J), L is the ligament length (m), B is the sample thickness (m), β is a geometry factor to account for the shape of the plastic zone around the fracture zone, w_e is the specific essential work of failure (J/m^2), and w_p is the specific plastic work of failure (J/m^3).

It is found that w_e relates mainly to the fracture characteristics can predict fatigue cracking resistance accurately. On the other hand, for the asphalt cements with the same w_e, w_p may be useful in explaining the differences in their performances. It is expected that the values of both w_e and w_p should be relatively high to be able to resist fatigue cracking and ductile fracture [66].

The ratio of the essential work of fracture to the net section stress of the smallest ligament length specimen 5 mm is called the crack tip opening displacement (CTOD) which is used to predict the fracture characteristics of the asphalt binder.

$$\text{CTOD} \sim w_e / \sigma_{\text{net, 5 mm}}$$

It measures the strain tolerance in the ductile state that would occur during ductile fracture in the hot mix asphalt.

CHAPTER 3

MATERIALS AND EXPERIMENTAL PROCEDURES

3.1 Materials

The materials tested were obtained during the construction of Ontario contracts and trials as well as from various commercial sources. A list of all asphalt binders, warm asphalt binders, and AR samples evaluated in this project is provided in Table 3.1. The Highway 7 contract and the Highway 115 FB trial were constructed during the summer of 2011. The Highway 11 TB trial was constructed during the summer of 2010. The Highway 35 FB trial was constructed during the summer of 2011. The QEW and Highway 402 warm AC was produced using a wax-type additive. The Highways 7 and 62 warm AC was produced using a surfactant-type additive.

The superior warm AR blends were prepared with a soft Cold Lake base obtained from the Nanticoke, Ontario, refinery of Imperial Oil of Canada. This material is normally sold as a roofing asphalt flux (RAF) and it grades as a PG 50-34 (continuous grade). Naphthenic type oil sold under the Raffex name and obtained from Tricor Refining in Bakersfield, California, was added at 6 % by weight of the warm AR binders. Crumb rubber modifier (CRM) passing the 30 mesh screen was obtained from Liberty Tire Recycling in Brantford, Ontario, and was added at 18 % by weight of the AR binders. Warm asphalt additives evaluated included Evotherm®, SonneWarmix™ and Rediset® WMX 8017. One of the warm AR binder was prepared with 2 % of a high vinyl grade styrene-butadiene-styrene copolymer (D1192) obtained from Kraton Polymers and reacted with 0.1 % of sulfur in the presence of the CRM. Warm AR were prepared by mixing the base asphalt, oils, warm additives, and CRM at temperatures between 180 °C and 190 °C for a minimum of 1 hour under moderate shear.

Table 3.1: Pertinent Asphalt Binders

Sample	Highway	Binder Type	PG Grade	Comments
A B C	7 7 7	TB FB AC	58-34 58-34 58-34	The Highway 7 contract was constructed during the summer of 2011
D E	115 115	FB AC	64-34 64-34	The Highway 115 FB trial was constructed during the summer of 2011.
F G	11 11	TB AC	58-28 58-28	The Highway 11 TB trial was constructed during the summer of 2010.
H I	35 35	FB AC	58-34 58-34	The Highway 35 FB trial was constructed during the summer of 2011.
J K	QEW 402	Warm AC Warm AC	70-28 64-28	The QEW and Highway 402 warm AC was produced using a wax-type additive.
L M	7 62	Warm AC Warm AC	58-34 58-34	The Highways 7 and 62 warm AC was produced using a surfactant-type additive.
N	-	RAF	Unknown	The RAF was a straight Cold Lake roofing flux from the Nanticoke, Ontario refinery of Imperial Oil.
O	-	AR	Unknown	The regular AR was produced with 18% CRM in the RAF.
P	-	Warm AR	Unknown	The warm AR was produced with 1.5% of the same surfactant-type additive as used for the Highway 7 and 62 contracts, 6% naphthenic oils, and 18% CRM.
Q	-	Warm AR	Unknown	The warm AR produced with 1.5% of the same wax-type additive as used for the QEW and Highway 402 contracts, 6% naphthenic oils, and 18% CRM.
R	-	Warm AR	Unknown	The warm AR was produced with 1.5% of a cationic surfactant/rheology modifier warm mix additive, 6% naphthenic oils, 2% high vinyl SBS, 0.1% sulfur, and 18% CRM.

Note: AC = control asphalt cement; AR = asphalt rubber; FB = field blend AR; TB = terminal blend AR; QEW = Queen Elizabeth Way; and RAF = roofing asphalt flux (soft AC from Cold Lake).

3.2 Experimental Procedure

3.2.1 Binder Aging

Strategic Highway Research Project (SHRP) developed in the early 1990s proposed an aging procedure to simulate short and long term field oxidative aging of asphalt binders in order to understand the failure mechanism of asphalt pavement. This proposal developed the Rolling Thin Film Oven (RTFO) and the Pressure Aging Vessel (PAV) for simulation of short and long term aging, respectively. RTFO samples of asphalt binder is used to simulate short term aging such as hot mixing of asphalt and aggregate and PAV asphalt sample is used to simulate in service aging of binders as a result of the effect of time, temperature, traffic load and environment. In other words, these procedures are meant to mimic field aging of binders in order to understand changes in rheological properties of the binders [3].

3.2.1.1 Rolling Thin film Oven (RTFO)

First of all, the temperature of the RTFO machine was set to 163 ^{o}C and the air flow adjusted to 4 L/min prior to testing. A sample of asphalt binder was heated in RTFO oven until it was changed to liquid enough to pour. The sample was stirred with glass rod to ensure homogeneity and to remove any air bubbles in the sample. About 35-40 g of unaged asphalt binder samples was poured in cylindrical glass bottles or jars and samples of asphalt binders in the tubes were placed in a rotating carriage at the rate 15 rpm of RTFO machine as shown in Figure 3.1. The sample was allowed to rotate continuously for 85 minutes in order to induce oxidative hardening or age hardening. This is kind of aging that simulates the aging that occurs during the hot mixing process or the early stages of the pavement life and it is expected that the aged samples would reflect the rheological changes in the properties of the asphalt binders [62].

Figure 3.1: Rolling thin film oven (RTFO) used for short-time aging of asphalt binders [61].

The bottles were removed from the carousel and RTFO residue were transferred to the another container which was used for future testing like PAV aging and DSR test. The aging time of 85 min was arbitrarily selected to classify all binders to the same kinds of aging. It is expected that this aging represents a majority of conditions where the asphalt is considerably aged from the first exposure to the plant burner and contact with hot aggregates throughout hauling and paving sites. This test method is designed to determine the effect of heat and air on the moving film of asphalt cement. This test shows the approximate change in properties during conventional hot mixing.

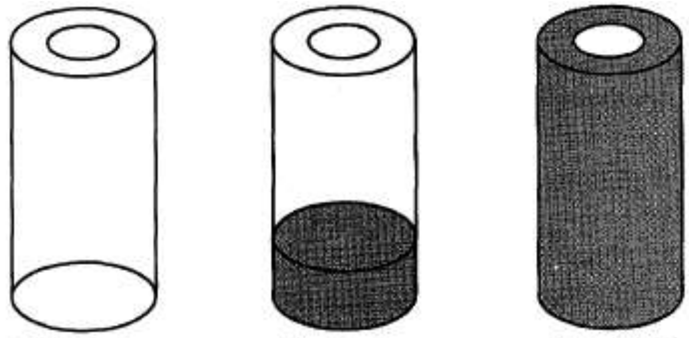


Figure 3.2: RTFO bottle, sample before and after aging.

3.2.1.2 Pressure Aging Vessel (PAV)

The pressure aging vessel (PAV) was applied by Superpave to simulate the effects of long-term asphalt binder aging that occurs as a result of 7 to 10 years hot mix asphalt pavement service. The PAV-oxidised asphalt binder is expected to estimate the rheological properties of binders after significant aging on the road.

First of all the samples of asphalt binder were aged by the RTFO method as explained above and 50 g of hot RTFO sample was poured into thin stainless steel pan. The sample was spread around the pan. The pans were placed in pan holder and placed inside the pre heated PAV as shown in Figure 3.3. The RTFO samples thus aged for 20 hours in a PAV pressured with air to 2.1 MPa at a temperature of 100 ^{o}C. Since the pressure aging vessel system is considered to simulate the aging caused by oxidation during 7-10 years of pavement service, it is expected that PAV-oxidised asphalt binders could be used to estimate the rheological properties of binders after significant aging on the road. A schematic diagram of the PAV system is shown in Figure 3.3.

Figure 3.3: Pressure aging components used for long term aging [63].

At the end of aging period, the pressure was gradually released and the pans were removed from PAV. For modified PAV, 50 g of the RTFO sample was aged for double time than regular PAV i.e. 40 hours and 12.5 g of RTFO sample was aged in PAV for 20 hours. The pans were placed in RTFO oven at 163 °C for 15 minutes and the asphalt samples were scraped in another container and kept for future tests.

3.2.2 Dynamic Shear Rheometer (DSR) Testing

The dynamic shear rheometer (DSR) test is used to evaluate the rheological properties of asphalt cement at intermediate to at high temperature. The purpose of this test is to characterize the rutting resistance of the asphalt cement at high temperatures and the fatigue cracking resistance at intermediate temperatures. The test was conducted on unaged, RTFO-aged, PAV-aged samples. At first the RTFO oven was preheated to 325 °F and RTFO or PAV samples heated in the oven untill it changed in to a fluid enough to pour. A small amount of the melt was poured into the mold of silicon rubber for parallel plate samples as shown in Figure 3.4(a).

Figure 3.4: (a) DSR sample moulds showing the different sizes and (b) major DSR test tools [64].

There are two parallel plate dimensions of diameters of 8 mm and 25 mm used for testing. The smaller plate (8 mm) was used for lower temperatures while the larger plate (25 mm) was used for higher temperatures. Then, the testing temperature was selected according to the asphalt binder grade or testing schedule and the DSR was set to a particular test temperature. This preheated the upper and lower plates which allowed the specimen to adhere to them. The asphalt binder sample was placed between the test plates. The lower plate was oscillated while the upper plate was kept fixed. After that, using software, the test plates were moved together until the gap between them equals the test gap plus 0.002 inches (0.05 mm). The excess material around the edge of the test plates was then trimmed using a heated trimming tool. The test plates moved together to the desired testing gap. This creates a slight bulge in the asphalt binder specimen's perimeter. The specimen was then brought to the test temperature. The test was started up only after the specimen had been at the desired temperature for at least 10 minutes. Using the DSR software, a target torque at which to rotate the upper plate was determined based on the material being tested (e.g., unaged binder, RTFO residue or PAV residue). This torque was chosen in order to ensure that the measurements were within the specimen's region of linear behavior. The specimen for 10 cycles at a frequency of 10 rad/sec (1.59 Hz) was conditioned using the DSR. The software reduces the data to produce a value for the complex modulus (G*) and the phase angle (δ) once the DSR takes test measurements over the next 10 cycles. The range of the phase angle (δ) of asphalt binder obtained from DSR test is about 50 to 90°, and while that of the complex modulus (G*) from about 0.07 to 0.87 psi (500 to 6000 Pa).

3.2.3 Regular Bending Beam Rheometer Test (AASHTO M320)

The PAV residue was heated at 160 °C in the oven approximately 30-45 minutes to change to fluid enough to easily pour. Sometime modified asphalt takes more time compared to unmodified asphalt. After heating, the sample was stirred with glass rod to make sure the sample is homogenous and to remove any air bubble present before pouring into the aluminum molds. Then, the sample was poured into aluminum molds as shown in Figure 3.5. 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 3.5.

Figure 3.5: Bending beam rheometer molds.

The asphalt binder in the aluminum moulds was conditioned at room temperature for 60 minutes and after 60 minutes the excess binder was trimmed from the top of each mold with a hot spatula. The asphalt beams were then conditioned for another one hour at -14 ^{o}C and -24 ^{o}C in a cooling bath. The cooling bath usually contains mixture of ethyl alcohol and water. The beam specimen was supported at two points 102 mm apart in a controlled temperature fluid bath. Then, the beam was loaded at the midpoint with a load of approximately 1000 mN for a period of 240 s. The deflection of the asphalt beam in a typical BBR test is shown in Figure 3.6. Creep stiffness (S (t)) and the slope of creep stiffness master curve (m (t)) were calculated at a loading time of 60 s. The creep stiffness should be a minimum of 300 MPa at 60 second loading and m-value should maximum 0.3 at 60 second loading time to prevent from low temperature cracking according to the AASHTO M320 specification. All samples were tested in duplicate and the reproducibility was found to be excellent. The measurements of creep stiffness and m-value are shown in Figures 3.7 and 3.8, respectively.

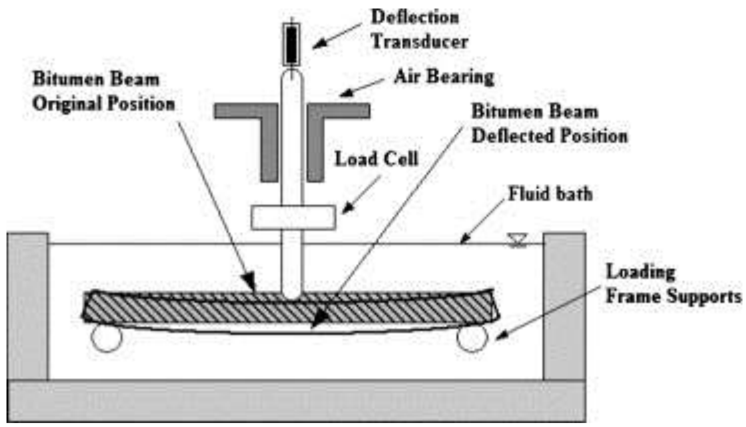


Figure 3.6: Deflected Asphalt Beam on Bending Beam Test [65].

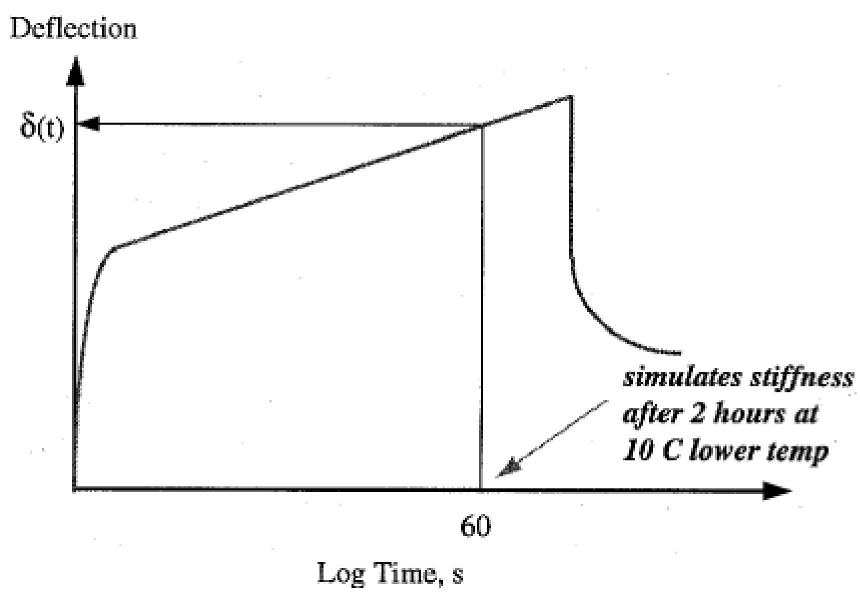


Figure 3.7: Creep stiffness of asphalt binders based on change in deflection with time.

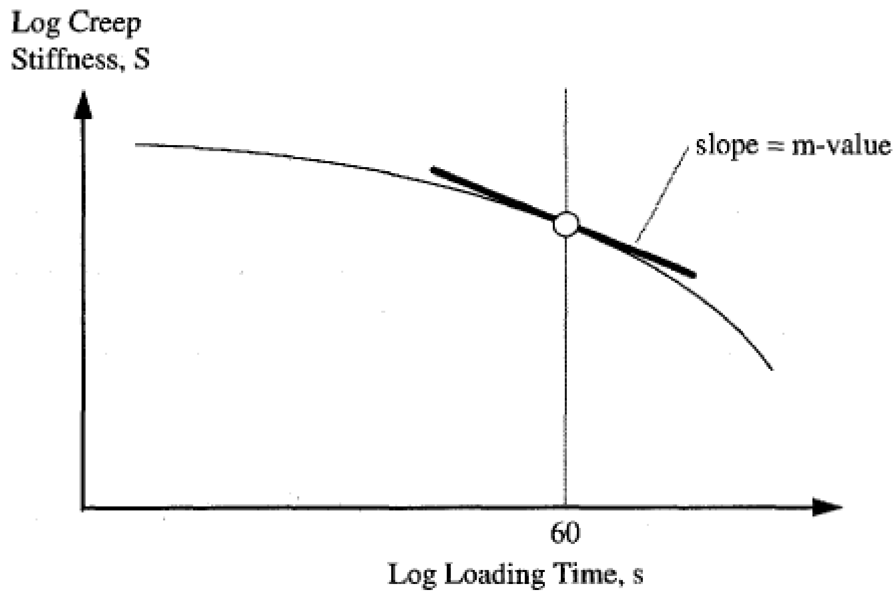


Figure 3.8: Evaluation of m-values of asphalt binders.

3.2.4 Extended BBR Testing (MTO LS-308)

The objective of extended BBR test is to evaluate the susceptibility of the asphalt binder to reversible aging or hardening particularly at low temperatures. The critical cracking temperature at which the maximum stress at a given temperature is more than the strength of the material is determined. A total of 12 BBR beams were prepared according AASHTO M320 specification. Asphalt beams were subsequently conditioned at both 10 ºC and 20 ºC above the pavement design temperature for one, 24 and 72 hours. Six beams were used at each conditioning temperature after each conditioning period; the beams were tested at two different temperatures. The m-values and the creep stiffness(S) 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. After 72 hours of conditioning, the limiting temperature and the subsequent maximum worst grade loss were calculated.

3.2.4 Ductile Failure Testing or Double Edge Notched Tension Test Method (LS-299)

To determine the ductile fracture or failure and fatigue cracking of asphalt binder at low temperature, the double-edge-notched tension (DENT) test was developed. It involves pulling of a notched asphalt binder in a water bath at isothermal condition until 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. In this method, the temperature of water bath was set at 15 ^{o}C before heating of the PAV sample and the preparation of the DENT moulds.

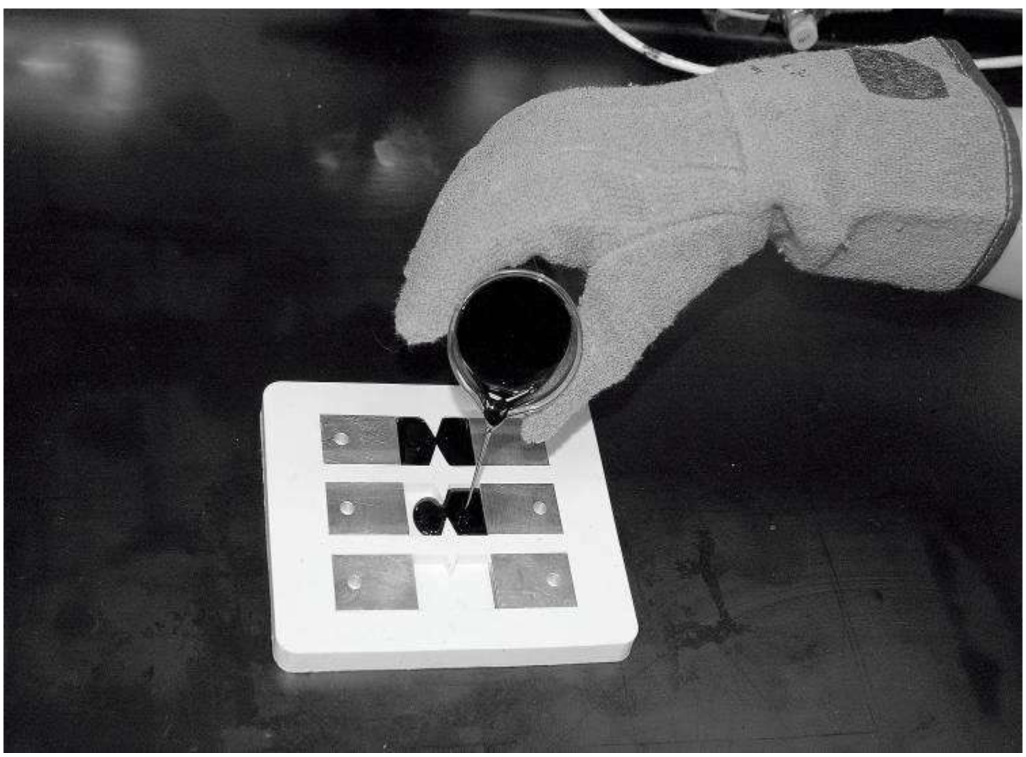


Figure 3.9: Sample Preparations in DENT Test [30].

The PAV residue of asphalt binder was heated for about 45 minutes in RTFO oven at 163 ^{o}C to change in to fluid enough to pour and it was then stirred with glass rod to make the binder homogenous and to remove any air bubbles present in the sample. The hot sample was poured in between two aluminum inserted with three different notch depths (ligaments between notch tips include 5, 10 and 15 mm) as shown in Figure 3.9.

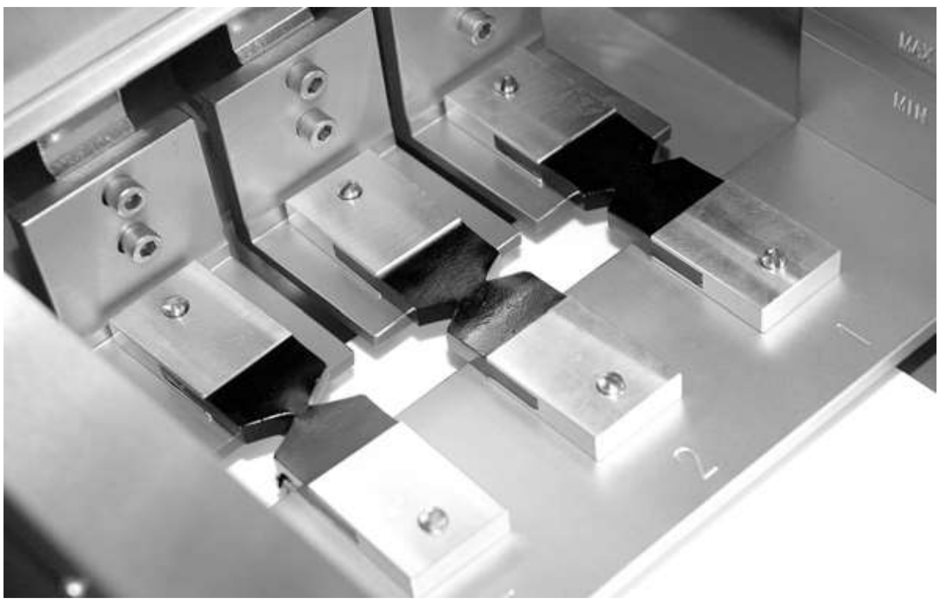


Figure 3.10: Double-edge-notched tension test set-up [30].

Samples were slightly over-filled in order to accommodate some minor shrinkage during cooling. The excess of asphalt binder was trimmed with hot spatula. The samples were conditioned at room temperature for 1 hour before they were loaded into the tensile machine. The samples were kept for 24 hours conditioning at 15 °C prior to testing in the DENT bath. Samples were tested in DENT bath by pulling at a constant speed of 50 mm/min until facture of binder took place. Figure 3.10 shows a photograph of DENT test set-up. The essential work of fracture, the plastic work of fracture, and the CTOD parameter were calculated in Excel spreadsheet. The test was repeated for each ligament lengths and excellent reproducible results were obtained in all cases.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Dynamic Shear Rheometer Analysis

4.1.1 High Temperature Superpave Grading

The high temperature Superpave performance grades were determined for all regular asphalt cement, warm asphalt cement, asphalt rubber, and warm asphalt rubber according to standard procedures in a dynamic shear rheometer (DSR) test. The high temperature Superpave grades measures the rutting resistance of asphalt pavement at higher temperature. The limiting temperatures of regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber are summarized in Figure 4.1-4.4.

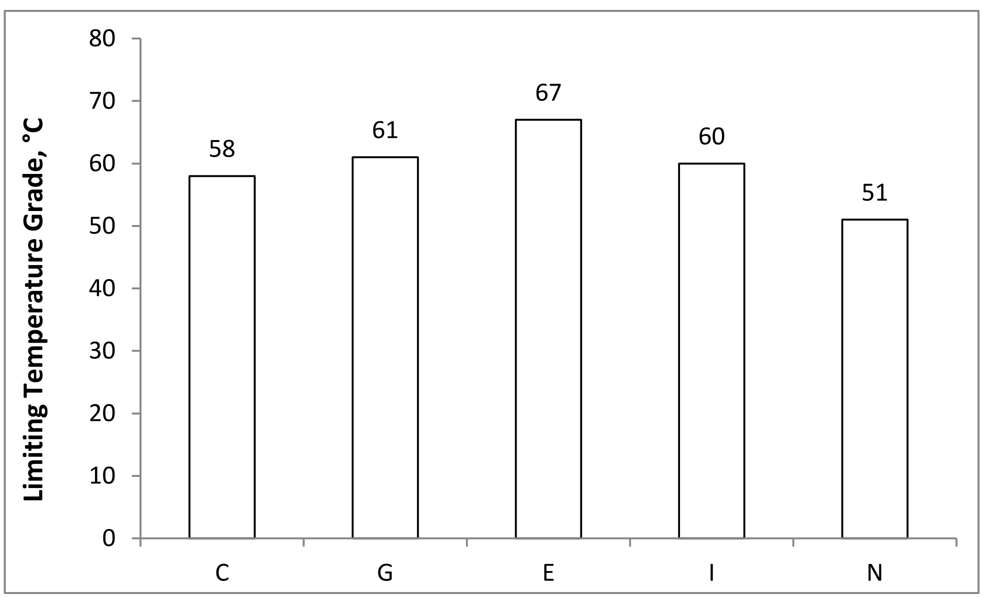


Figure 4.1: Limiting temperature of high temperature grade of regular asphalt.

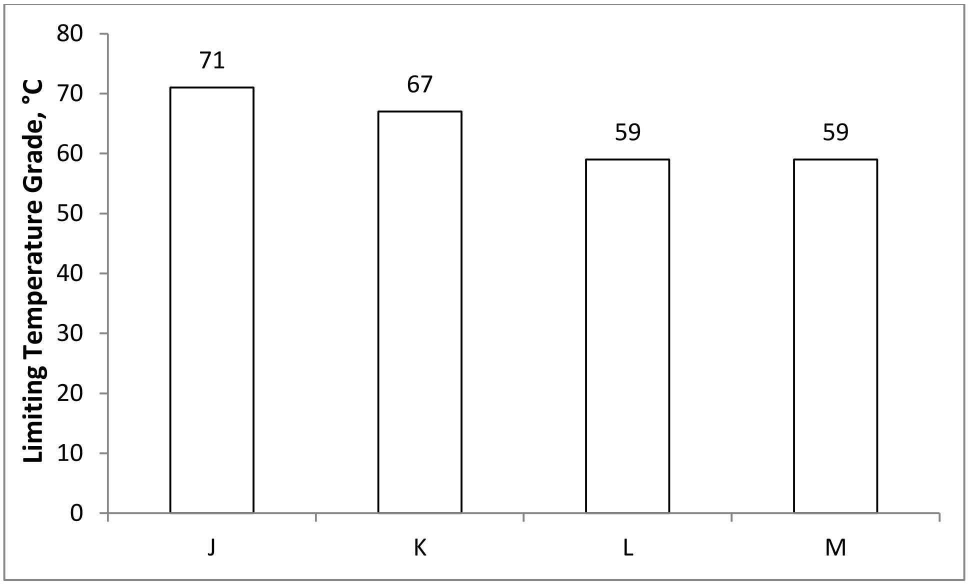


Figure 4.2: Limiting temperatures of high temperature grade of warm asphalt cement.

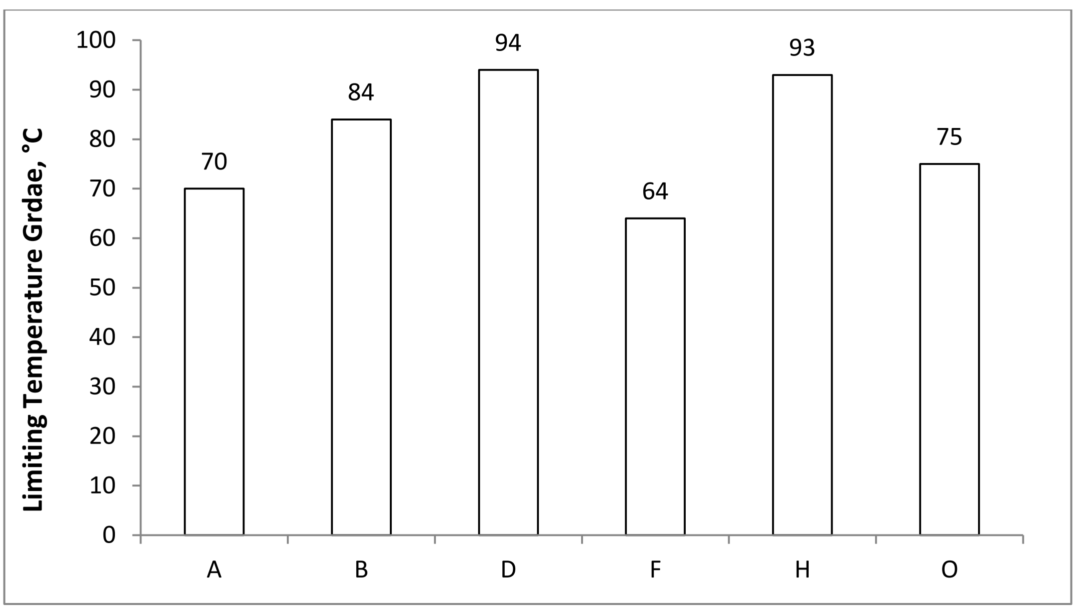


Figure 4.3: Limiting temperature of high temperature grade of asphalt rubber.

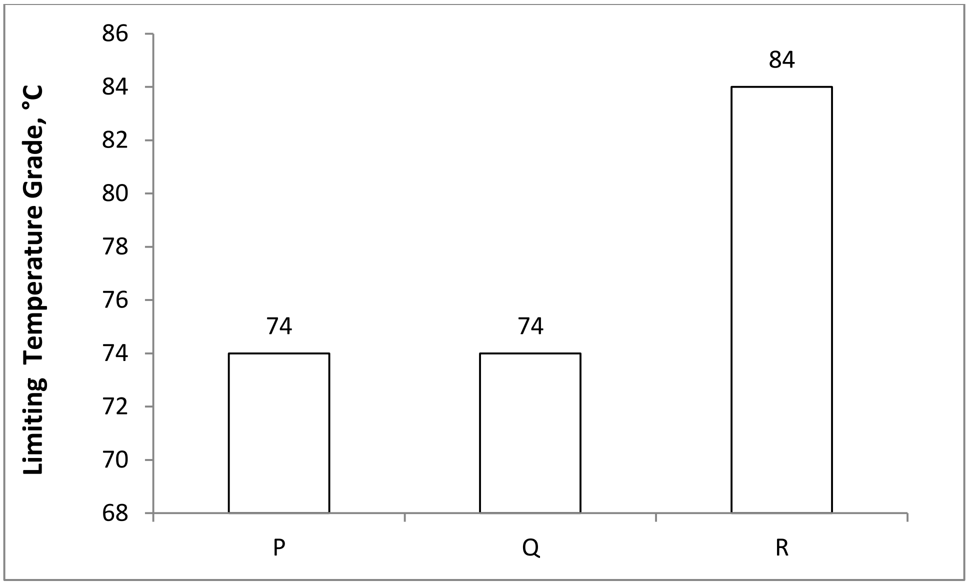


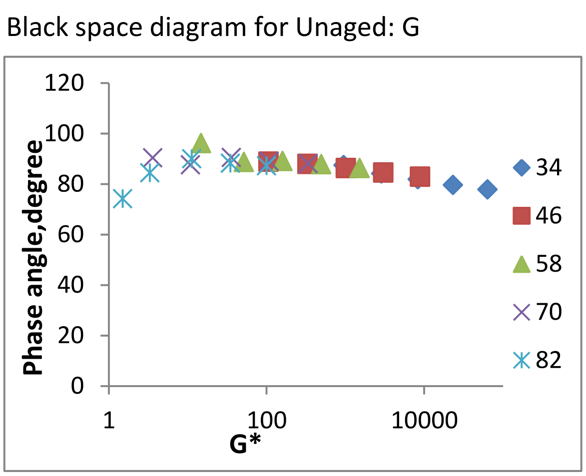
Figure 4.4: Limiting temperature of high temperature grade of warm asphalt rubber.

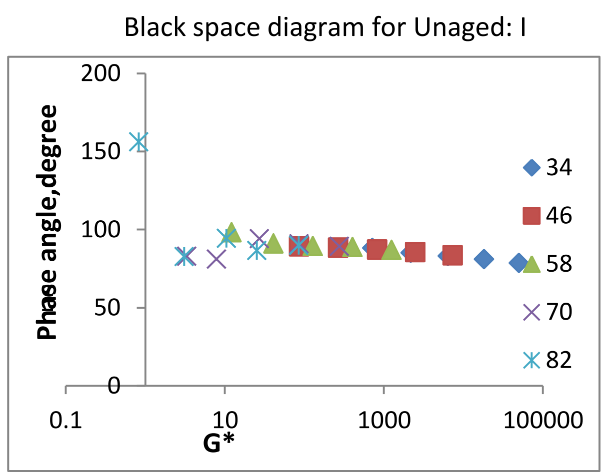
It can be observed from Figure 4.1-4.4 that the asphalt rubbers and warm asphalt rubbers have comparatively higher limiting temperature at which unaged or RTFO aged samples pass the DSR specification. For the unaged sample, $G^*/\sin\partial$ should be a minimum of 1.0 kPa and for the RTFO aged sample $G^*/\sin\partial$ should be a minimum of 2.0 kPa. Regular asphalts and warm asphalts have comparatively lower limiting temperature performance grade. The high value of limiting temperature of asphalt rubber indicates that they have high resistance to rutting at higher temperature and these types of binders would show good performance at high temperature climate or during summer in more moderate climates such as those in most of southern Ontario. This could be due to the presence of high content of elastic component and low content of viscous component of asphalt rubber. These types of binders would show low stiffness and low viscous behaviour at higher temperature and would show high durability at high temperature region. For all binders, Sample H of asphalt rubber showed the highest limiting temperature and the Sample N of regular asphalt cement showed the lowest limiting temperature. In fact , Sample

N of regular asphalt would have high content of viscous component and it would change in to liquid at higher temperature. It could result in permanent deformation at higher temperature showing low durability at higher temperature. Also, it can be noted that warm asphalt cements have higher limiting temperature performance grade as compared to regular asphalt. This could be due to the presence of additives. The additives improve the rutting properties of binder and improve the performance of binders at higher temperature. Based on the result, it can be concluded that the rubberized asphalt binders can perform very well at higher temperature and have higher rutting resistance than regular asphalts and warm asphalts. This shows that the pavement with asphalt rubber shows higher elastic nature at higher temperature and low chances of permanent deformation at higher temperature.

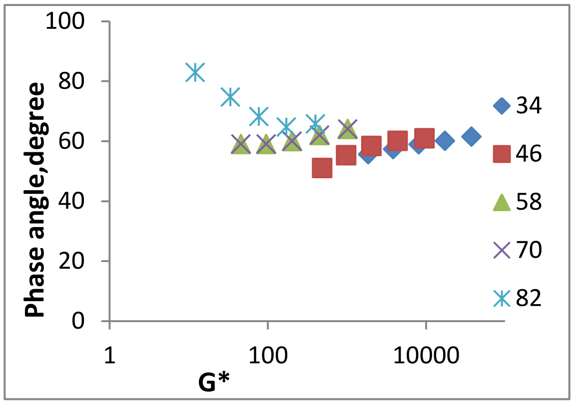
4.2 Black Space Diagrams

One of the important parameters that can be used to characterise the rheological characteristics of an asphalt binder is the Black space diagram. DSR test is performed for the study of the rheological properties of asphalt binder. Rheology is the branch of polymer science where the deformation and flow of materials is studied. With the help of this it is determined whether an asphalt binder consists of a homogeneous composition having a single phase or a heterogeneous composition having multiple phases. Asphalt binders which have single phase system are said to behave rheologically simple whereas those binders which have two or more phase system are known as rheologically complex. Black space diagrams are used to determine whether asphalt cement is rheologically simple or complex. A Black space diagram in which there is a smooth progression of curves at different temperatures and there is no any anomalous behaviour then binder is called rheologically simple. There is special pattern in the diagram in this type of material. On the other hand, for a Black space diagram in which there is no smooth progression of curves at different temperatures, or a diagram which shows discontinuity at different temperatures, the binder is said to be rheologically complex. There is no special pattern in this type of material, i.e. phase separation takes place. A Black space diagram is simply a graphical representation to determine the change in phase angle with the complex modulus at different temperatures. We can predict and quantify the rheological simple material very easily but very difficult for rheologically complex asphalt binders because their behaviour does not resemble conventional viscoelastic materials. Generally, an asphalt cement can be considered as good quality if it has a high phase angle at high stiffness (reducing thermal cracking due to stress relaxation) and a low phase angle at low stiffness (preventing rutting). The black space diagrams for unaged, RTFO-aged and PAV-aged of regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt are shown in Figures 4.5, 4.6 and 4.7 respectively.

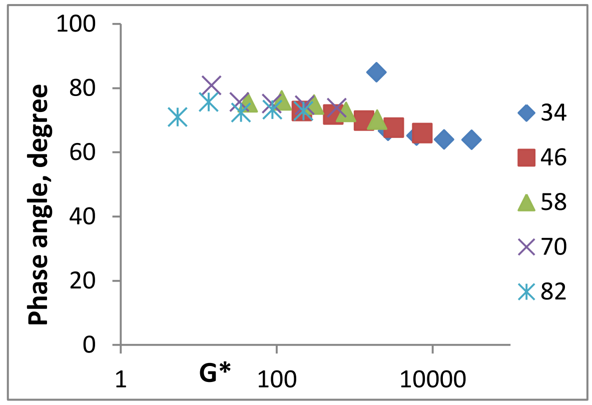




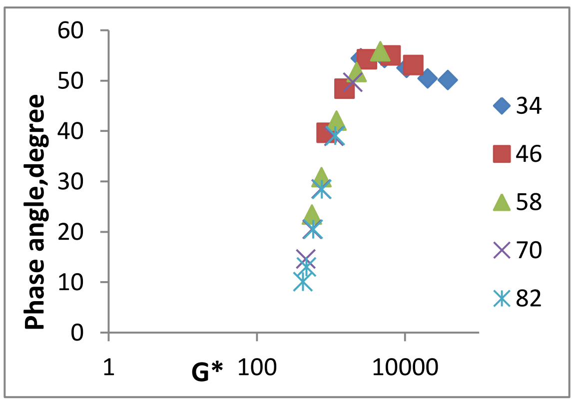
Black space diagram for Unaged: E



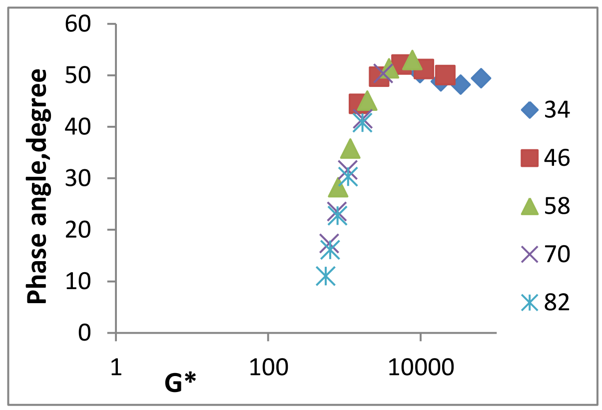
Black space diagram for Unaged: F



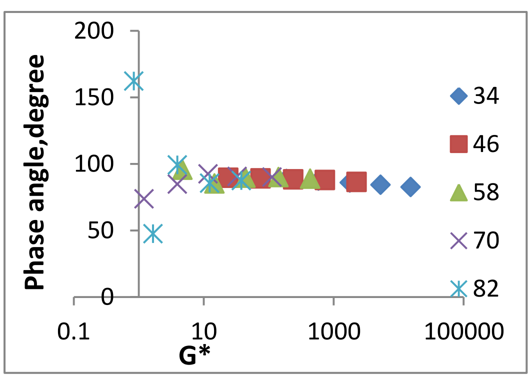
Black space diagram for Unaged: P



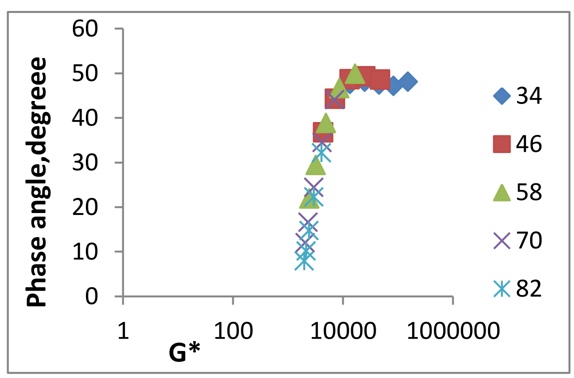
Black space diagram for Unaged: H



Black space diagram for Unaged: N



Black space diagram for Unaged: O



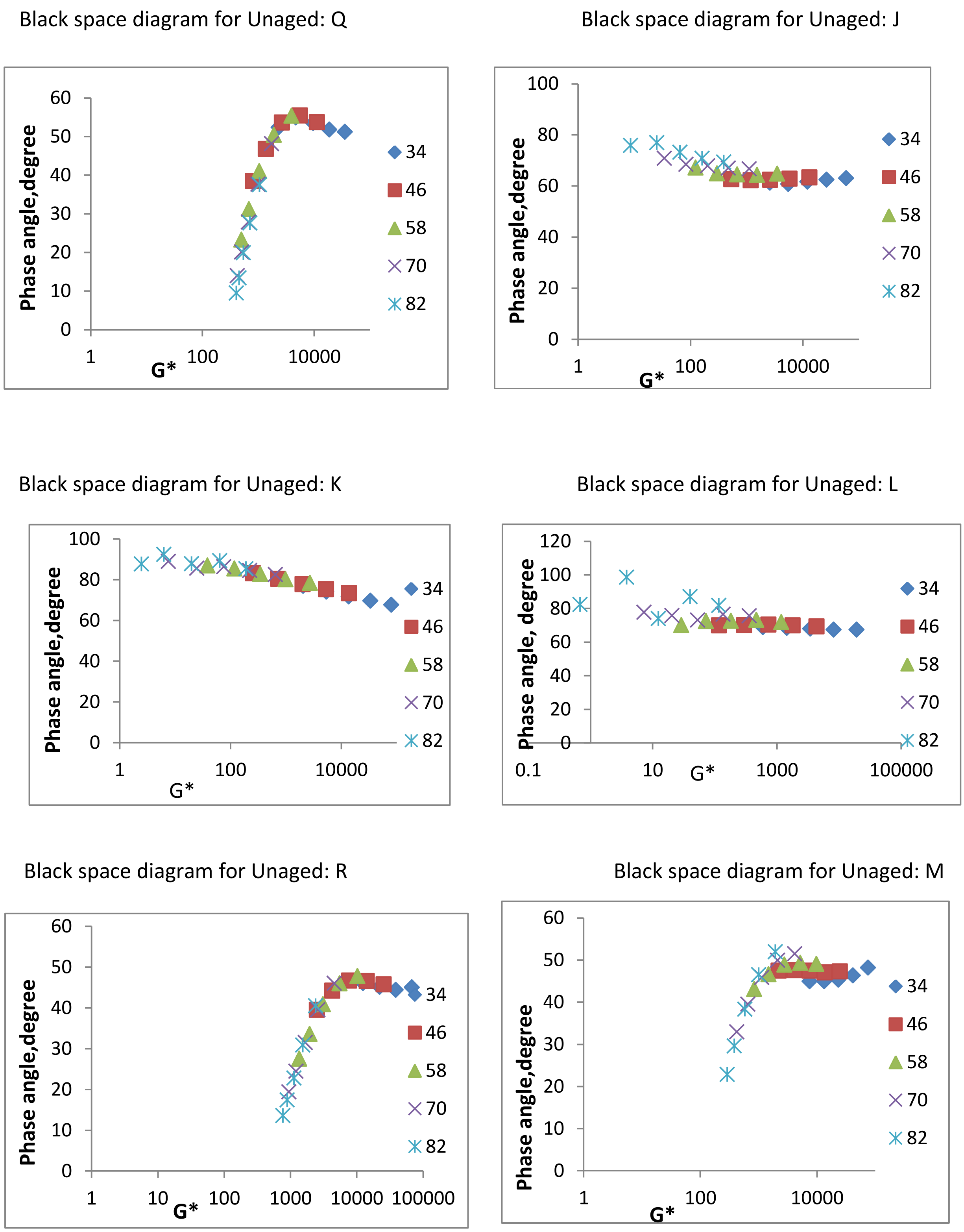
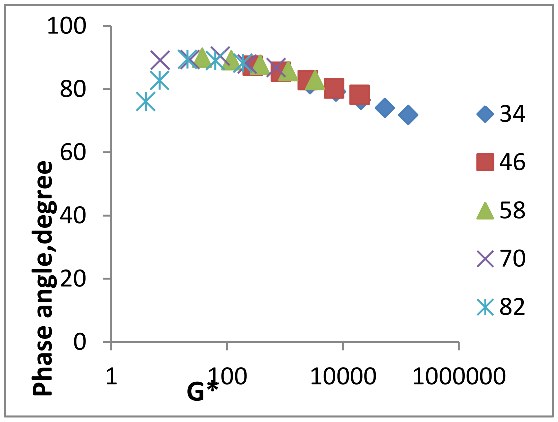
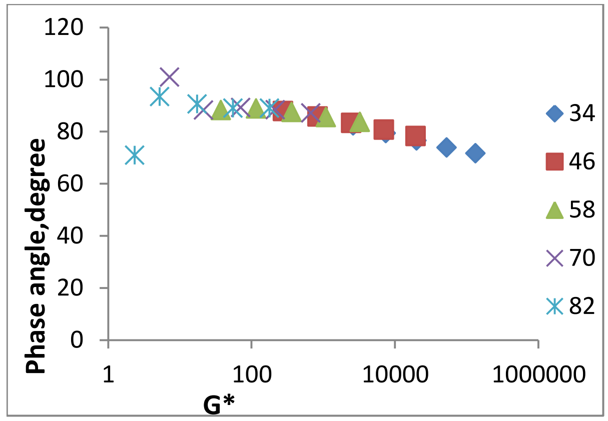


Figure 4.5: Black space diagram for unaged binders of regular asphalt cement, warm asphalt cement asphalt rubbers and warm asphalt rubbers.

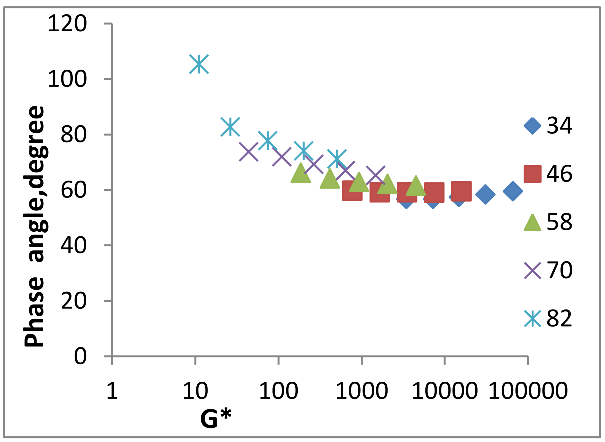
Black space diagram for RTFO: G



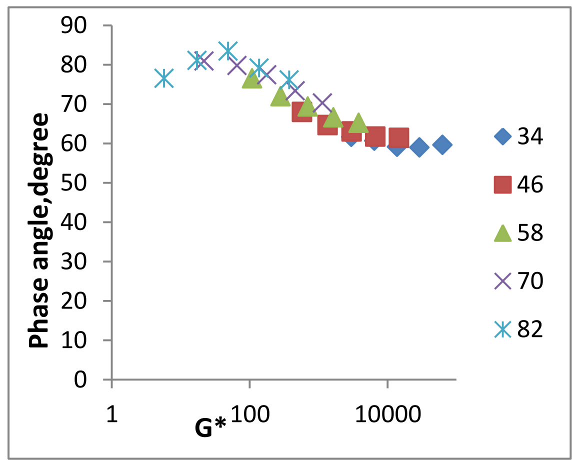
Black space diagram for RTFO: I



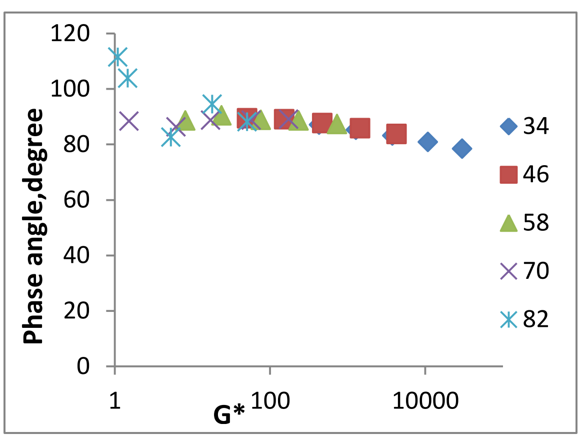
Black space diagram for RTFO: E



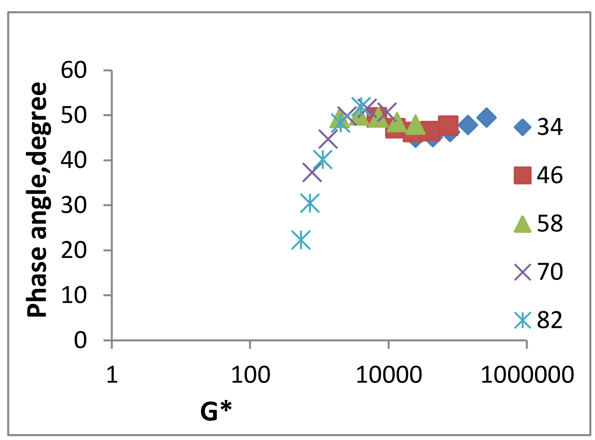
Black space diagram for RTFO: F



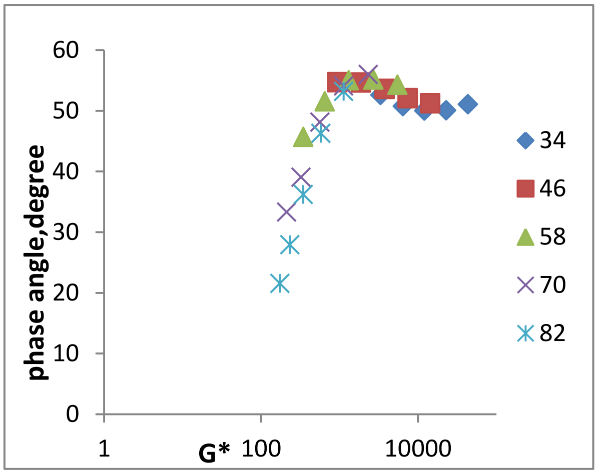
Black space diagram for RTFO: P



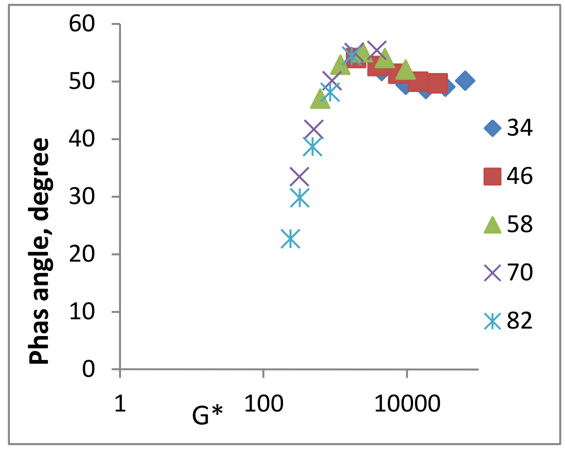
Black space diagram for RTFO: H



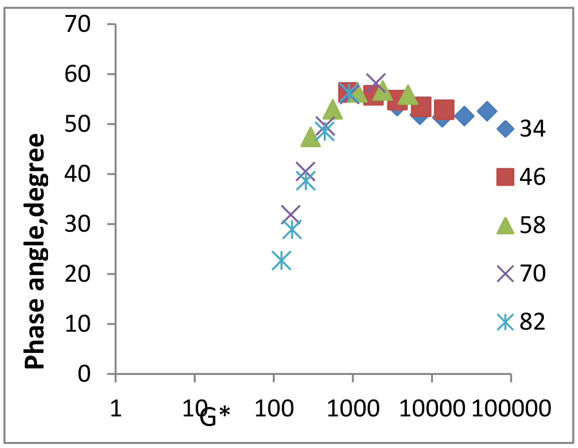
Black space diagram for RTFO: N



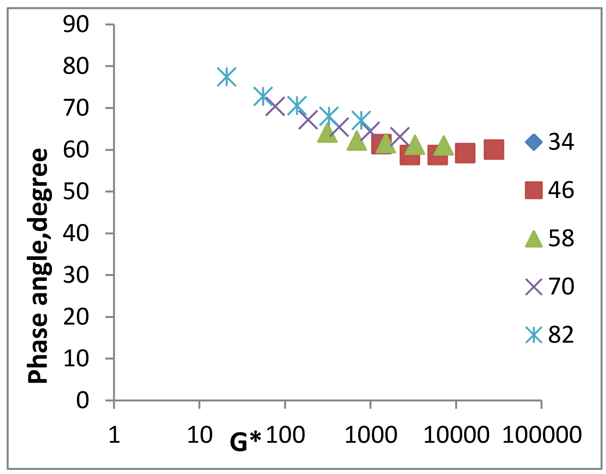
Black space diagram for RTFO: O



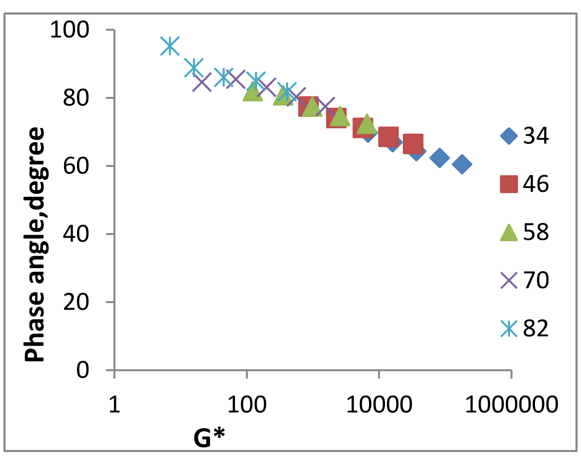
Black space diagram for RTFO: Q



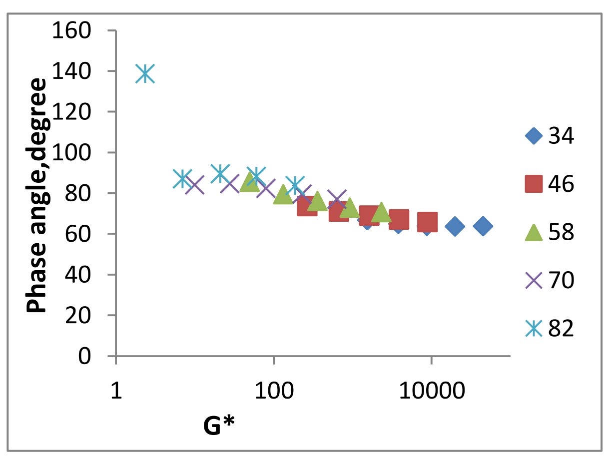
Black space diagram for RTFO: J



Black space diagram for RTFO: K



Black space diagram for RTFO: L



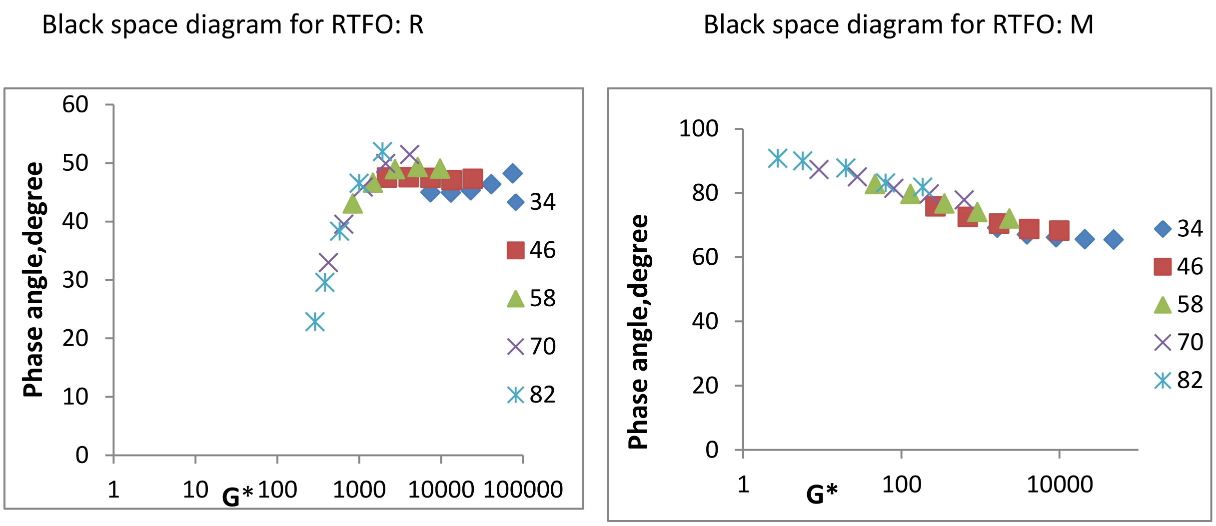
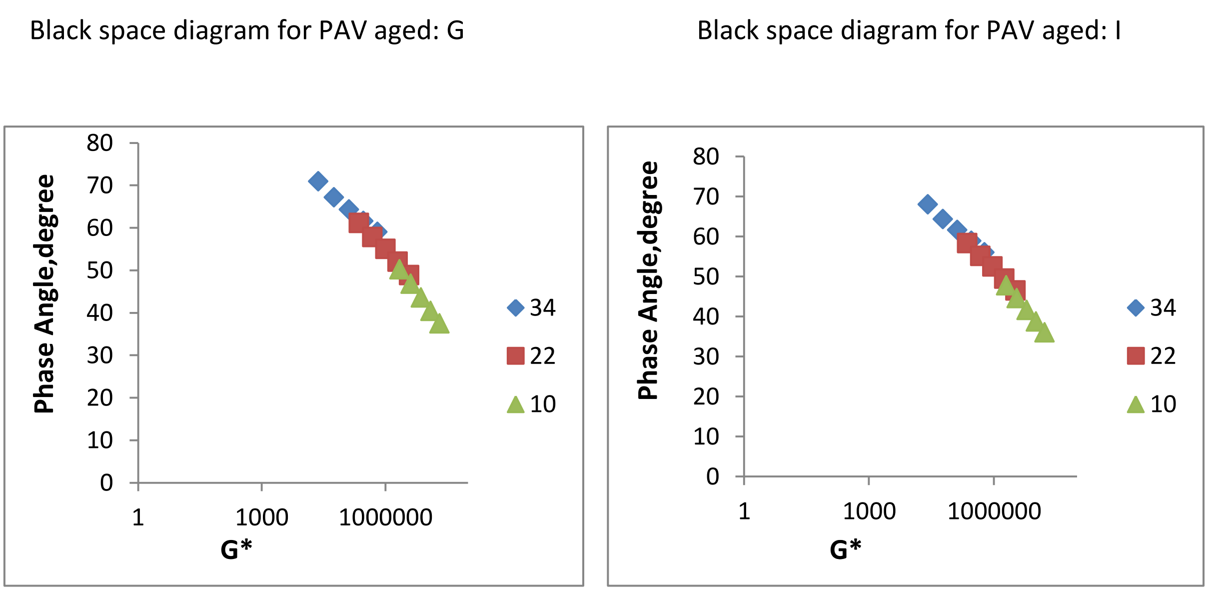
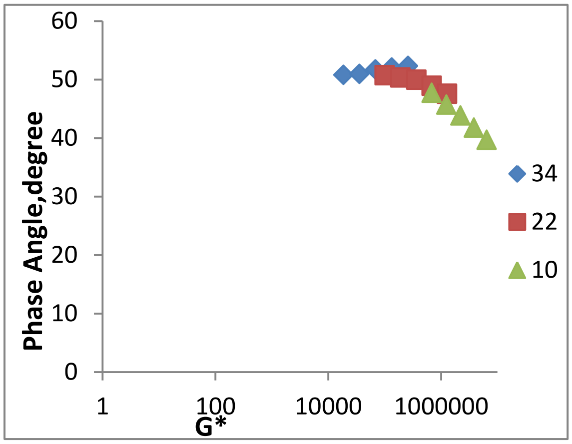


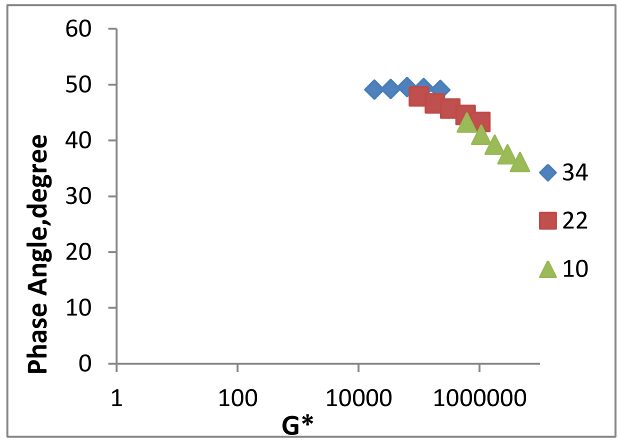
Figure 4.6: Black space diagram for RTFO aged binders of regular asphalt cement, warm asphalt cement, asphalt rubbers and warm asphalt rubbers.



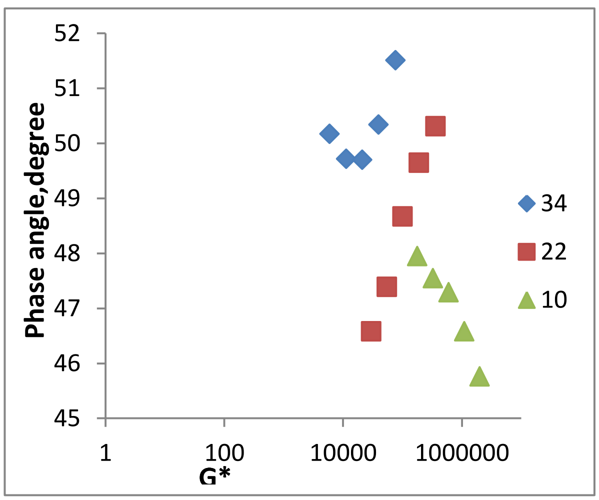
Black space diagram for PAV aged: E



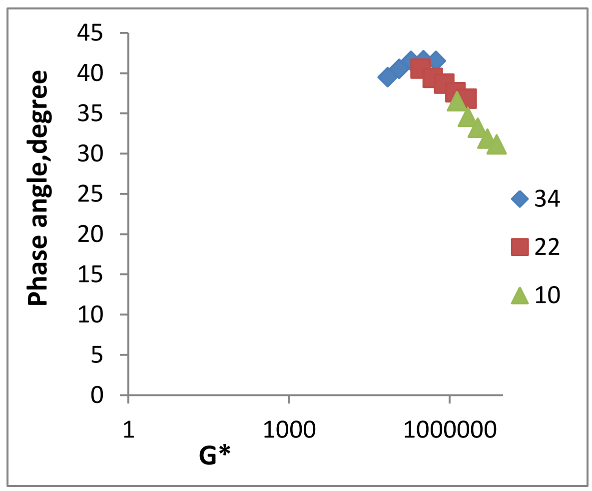
Black space diagram for PAV aged: F



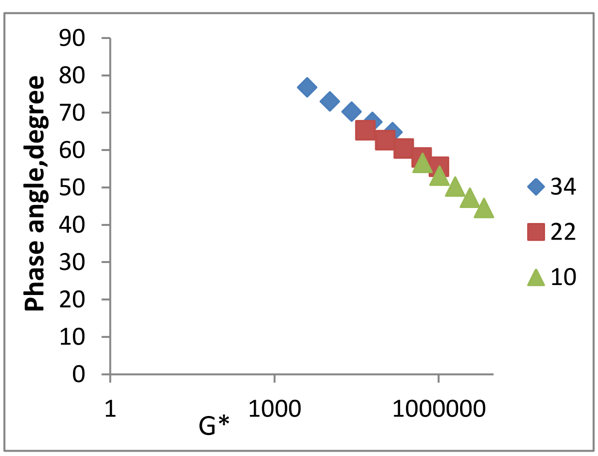
Black space diagram for PAV aged: P



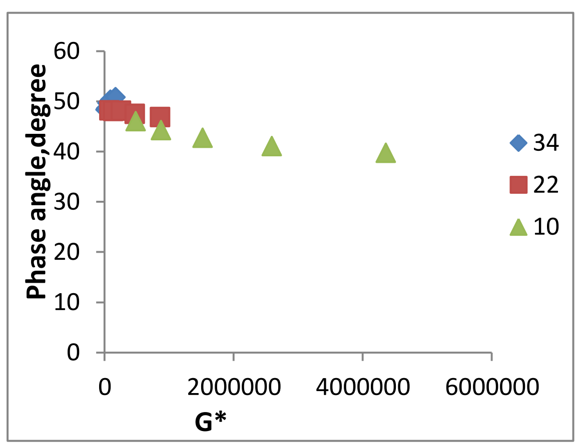
Black space diagram for PAV aged: H



Black space diagram for PAV aged: N



Black space diagram for PAV aged: O



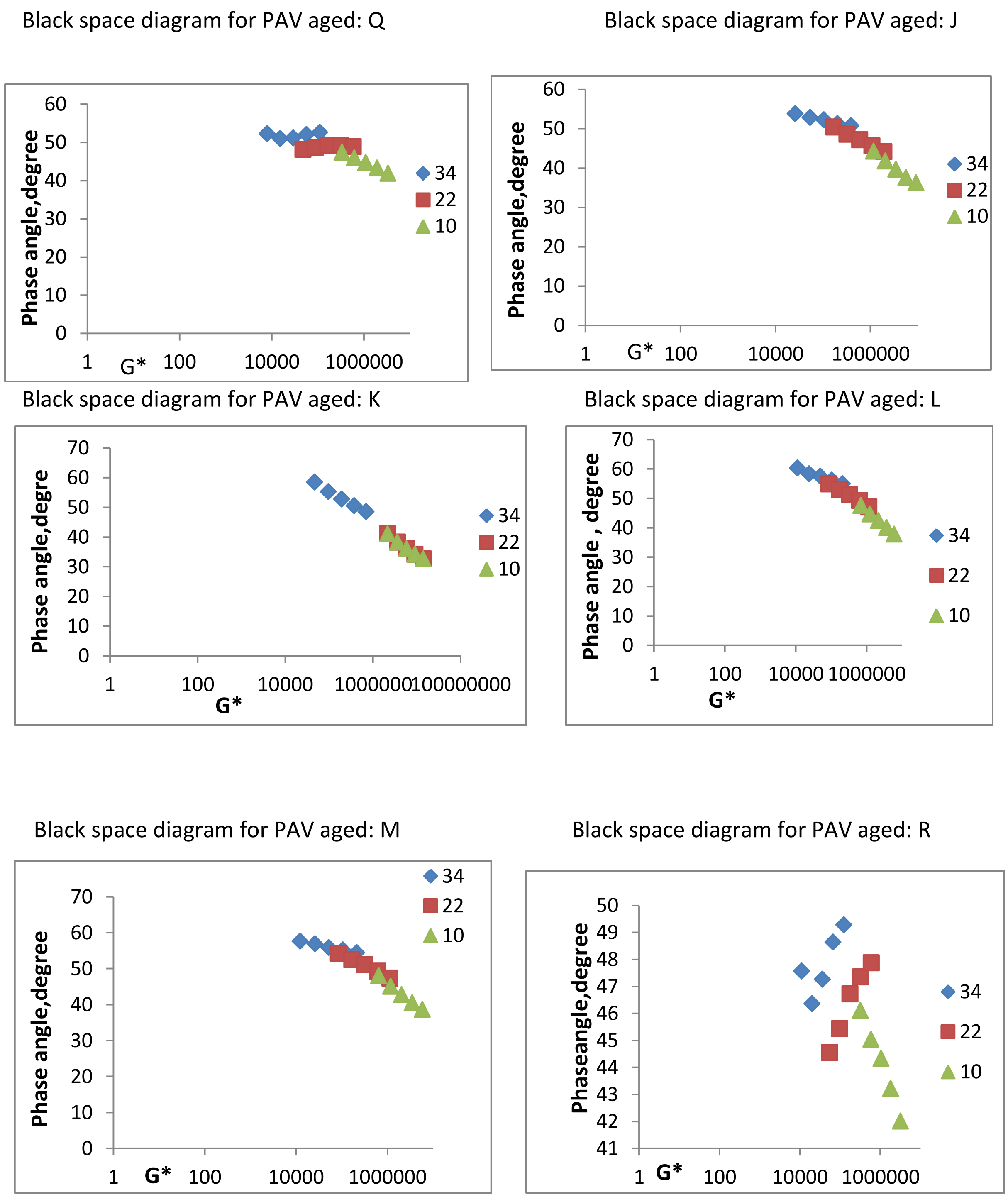


Figure 4.7: Black space diagram for PAV aged binders of regular asphalt cement, warm asphalt cement asphalt rubbers and warm asphalt rubbers.

By observing the Black space diagram for unaged samples, it is seen that most of the samples are rheologically simple except samples E and L. The complex behaviour of samples E and L may be due to the addition of polymer to these samples. The phase separation takes place in these samples due to the polymers. Similarly, it can be observed from the Black space diagram that the phase angle decreases with complex shear modulus at higher temperature for warm asphalt rubbers (P-R) showing their good performance at higher temperature. These binders would perform very well in service and have high resistance towards rutting.

It should be noted that rutting resistance of asphalt binders is largely determined by the structure of the aggregate skeleton or mixture design. It is expected that asphalt binders used for construction of road pavement be viscoelastic at all temperatures, however this does not usually take place at extreme low and high temperatures. On the basis of the viscoelastic character of an asphalt binder, it is expected that asphalt binders should have lower values of phase angle at higher temperatures and higher values at lower temperatures to perform better in service. For the samples E, F, K and L in the black space diagram, it is observed that phase angle increases and complex modulus (measure of stiffness) decreases at higher temperature. This could be due to the fact that at higher temperature, the asphalt binder shows the shear thinning type of behaviour and tends to be more viscous fluid resulting an increase in phase angle at higher temperature. Further, it can be noticed in black space diagram that the samples G, F, N, J and K showed rheological simple behaviour at lower temperature and deviated from rheologically simple behaviour at higher temperature. This might be due to the phase separation at higher temperature. As can be seen from black space diagram in Figure 4.7 for PAV aged residues (representing the aging that occurs in the field after 5-10 years in service), samples O, P and R shows the rheologically complex behaviour after long term aging. This could be due to the phase separation as result of oxidation of the asphalt binder during long- term aging.

4.3 Double Edge Notched Tension (DENT) Test Analysis

The double-edge-notched tension test was developed to measure the strain tolerance and the failure properties of asphalt cement in the ductile state by using PAV residue. This method successfully can rank asphalt binders in terms of performance and to differentiate superior performing binders from inferior performing. In this method, all the asphalt binders underwent the same ductility test by pulling of a notched asphalt binder in a water bath at isothermal condition, i.e. at 15 °C, and loading rate of 50 mm/min until failure occurred according to the LS-299 standard. It was observed from this test that the tests for different ligament lengths were similar but were different for each binder as shown in the raw data in Figure 4.8, which represents the duplicates force against displacement curves for a series of DENT samples with ligament lengths from 5 to 15 mm with a difference of 5 mm from each other. The different shape of curves for different binders could be due to different modification methods by which samples were prepared that lead to different binders like elastic or rubbery, stiff binders or soft binder.

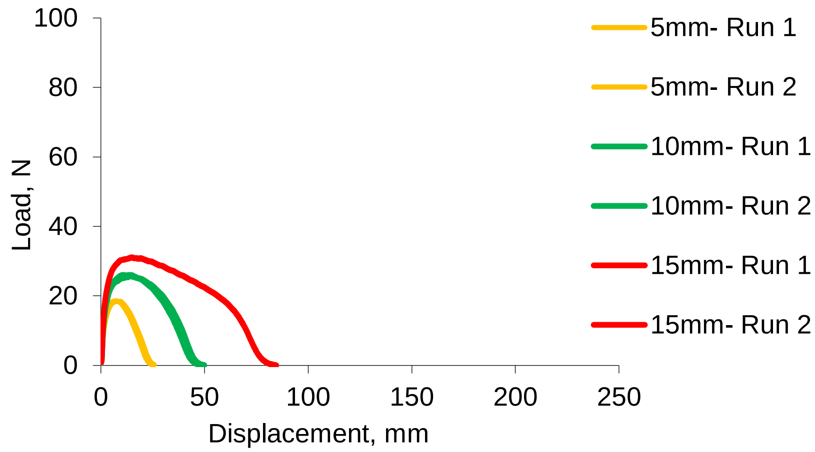


Figure 4.8: Representative Force-Displacement Data for the DENT Test (Sample I).

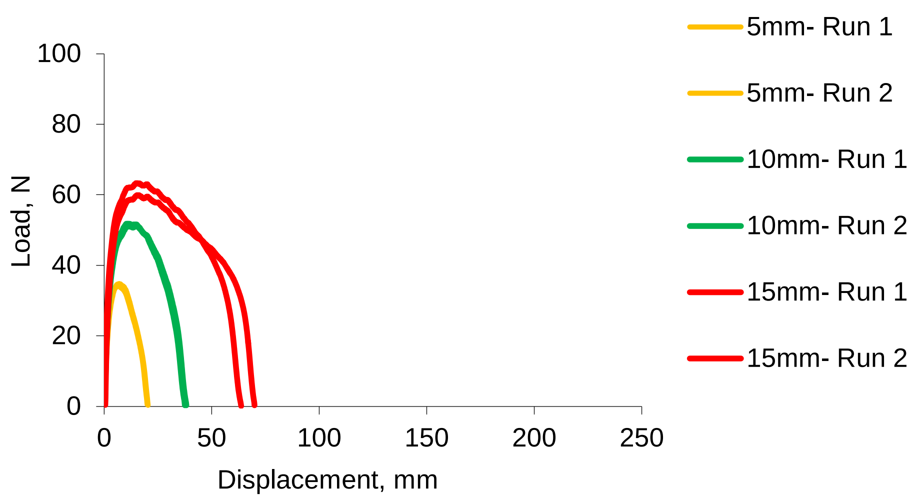


Figure 4.9: Representative Force-Displacement Data for the DENT Test (Sample J).

Since all the samples usually exhibit the same pattern of reproducibility, except a few samples (J, K and G), Figure 4.8 acts as representative of all other samples and Figure 4.9 is representative for samples J, K and G. From the results obtained on force versus displacement curves for each binder tested using the DENT test, it was found that there was a similar pattern of deformation at different ligament lengths from 5 mm to 15 mm. It shows that all the specimens had a similar sequence of stretching, yielding and tearing which is the necessary requirement for the essential work of fracture (EWF) to be completed.

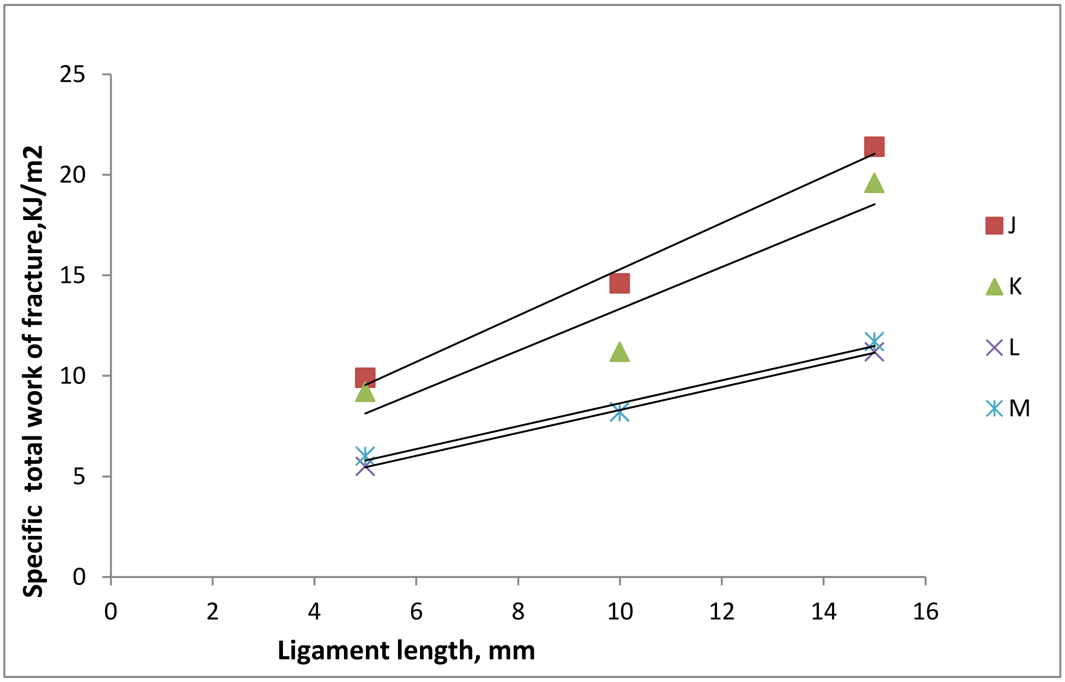


Figure 4.10: Essential work of fracture versus ligament length for warm asphalt cement (J-M).

Further, the essential work of fracture model assumes that ligaments yields fully prior to fracture or failure occurred which is seen in the flat shape of force-displacement curves in the Figures 4.8 and 4.9 which is considered to take place during the sudden drop of force at the end of each test [12]. The integrated area under the force-displacement curve gives the total work of fracture which is the sum of the essential and the plastic works of fracture. By plotting the specific total work of fracture against the ligament length, L, a linear graph is formed as shown in Figure 4.10 with the intercept at L = 0 gives the essential work of fracture and the slope gives the product of the plastic zone shape factor and the essential plastic work of fracture, respectively. The regression coefficient of these straight lines range from 0.94 to 0.99, which shows the accuracy of this method.

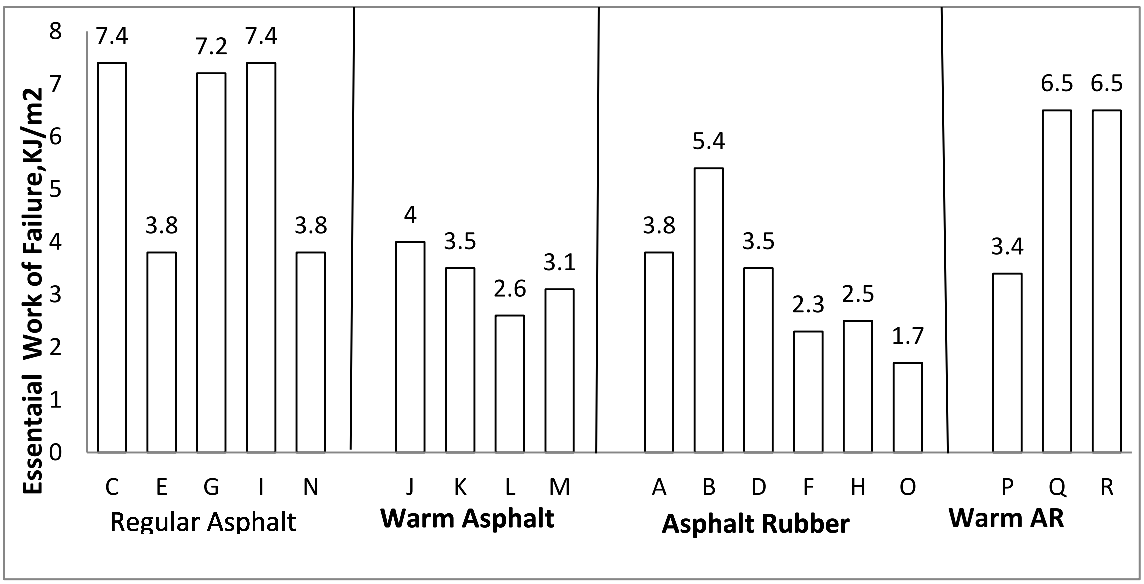


Figure 4.11: Essential work of failure for regular asphalts, warm asphalts, asphalt rubber and warm asphalt rubbers.

The essential work of fracture is considered as a material property that does not depend on geometry of binders in the sense of performance grading of asphalt but the plastic work of fracture is not material property and it related to the mixture design, asphalt binder and air void. If the asphalt mixture is rich in asphalt binder, it contains higher plastic work of fracture and this type of binder is less sensitive to cracking [12]. So, it is expected that high value of essential work of fracture and plastic work of fracture for good resistance towards pavement distress [12]. Figure 4.11 shows the essential works of fracture of regular asphalt cements, warm asphalts, asphalt rubbers and warm asphalt rubbers. It can be observed that the specific essential work of fracture is comparatively high for samples C, G, I, Q and R indicating that they have high strain tolerance to resist low temperature cracking and fatigue cracking and they perform very well in service. The low value of essential work of fracture for samples L, F and O indicates they have low strain tolerance and they could show poor performance in service. The remaining binders are in borderline in terms of strain tolerance perspective and performance point of view. The other important parameter used to distinguish superior from inferior binder is crack tip opening displacement (CTOD). It is defined as the ratio of the essential work of fracture to the net section stress of the 5 mm ligament length specimen which is used to predict the fracture characteristics of the asphalt binder. It indicates the limit by which the asphalt binder can stretch before it fails. It measures the strain tolerance in the ductile state that would occur during ductile fracture in the hot mix asphalt (HMA). The CTOD has minimum requirement in Ontario's LS specification based on the base asphalt's low temperature grade. Basically, CTOD should be 10 mm for asphalt cement of PG-28, 14 mm for PG-34 and 18 mm for PG-40. The summary of CTOD values for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber are shown in Figure 4.12. It can be observed from the data presented in Figure 4.12 that CTOD values varies from low of 4 for field blend asphalt to high of 22 for warm asphalt rubber. The data in Figure 4.12 are interesting in several ways. First, most of binders except C, G, O, Q and R are either slightly acceptable or deficient from a strain tolerance perspective. These materials are either too stiff or lack the necessary cohesive strength at 15 °C and therefore fail to provide a sufficient CTOD. Among warm asphalt rubbers, sample P ranks just below the 18 mm required for a PG -40 grades while sample R ranks well above the 14 mm required of a PG -34 grades showing high strain tolerance and high performance at low temperature. The very high values of CTOD for binders C and R might be due to the addition of small amount of tri-block polymer styrene butadiene styrene (SBS) in these binders [4]. However, it is likely that the benefit of a high CTOD through SBS modification is only short-term because the LS-299 testing is done on PAV residue and that the regular AASHTO R-28 protocol fails to reproduce aging that occur in SBS polymer in service [4].

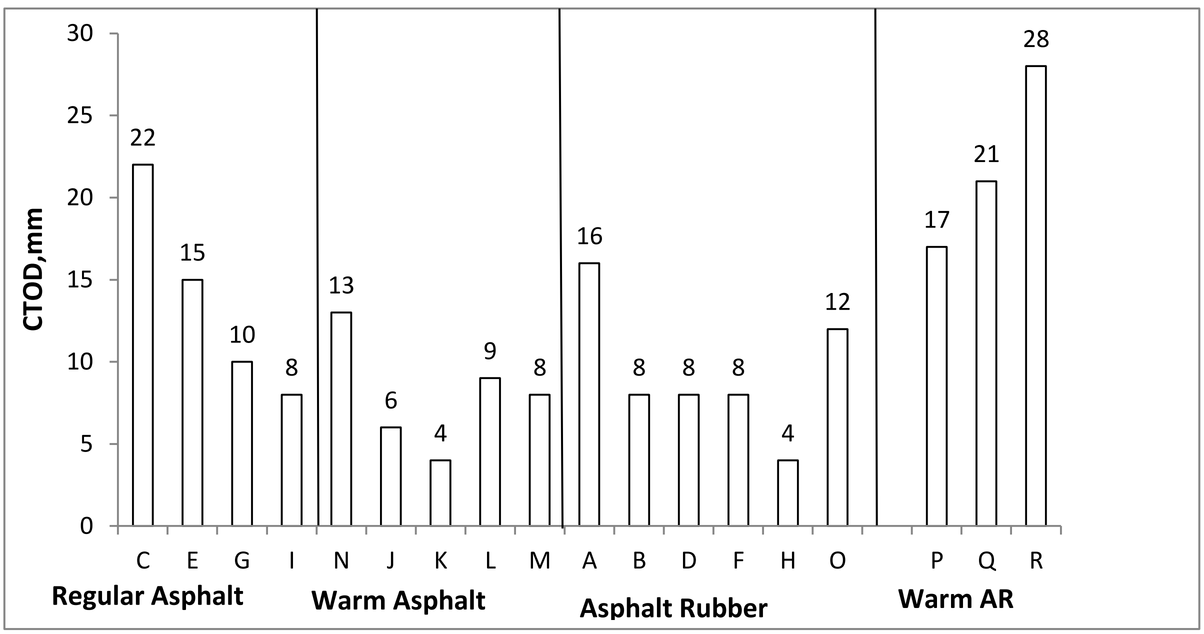


Figure 4.12: Crack tip opening displacement of regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber.

Also, it can be noted in the Figure 4.8 that sample K of warm asphalt cement and sample H of asphalt rubber have lowest CTOD (i.e. CTOD = 4) instead of minimum 10 and 14 according to LS-299 standard specification. It is understood that these binders would show very low strain tolerance and crack significantly at low temperature showing very low durability.

4.4 Regular Bending Beam Rheometer (BBR) Test Analysis

The regular BBR grades for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber were determined according to AASHTO M320 standard test method. The test was done by conditioning the asphalt beams at 10 °C and 20 °C above the pavement lowest design temperature and the creep stiffness (S(t)) and slope of the creep stiffness master curve (m(t)-value) were determined using three point bending.

The limiting temperature at which S (60 s) = 300 MPa, and m (60 s) = 0.3 were determined and the warmest of these two temperatures was used as minimum performance grade temperature of the binder. The findings are summarized in Figure 4.13.

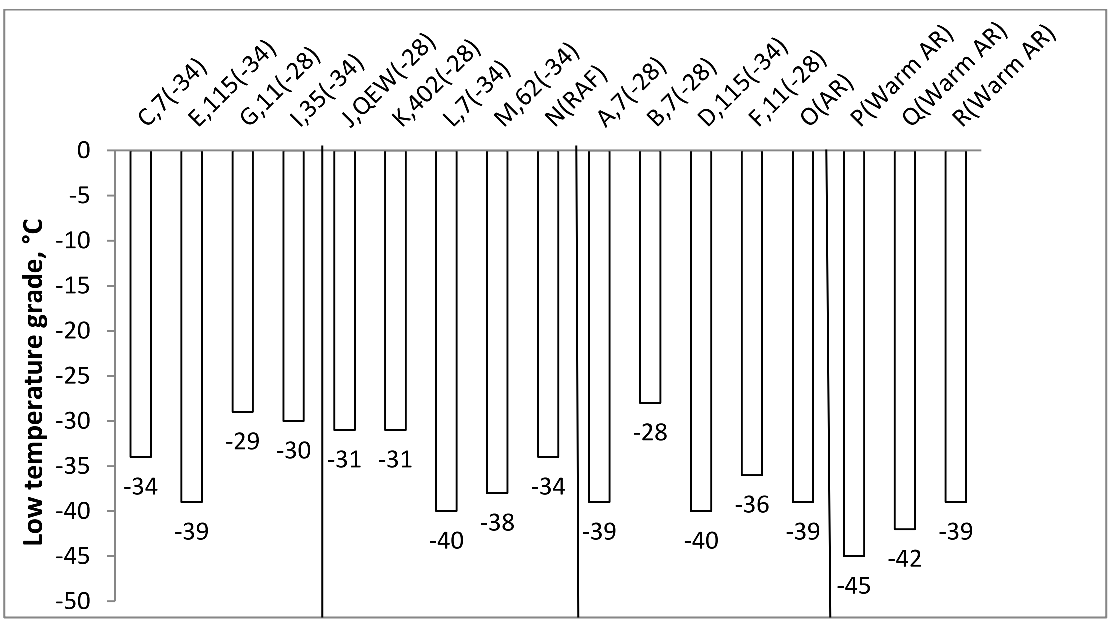


Figure 4.13: Regular BBR grade for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber.

The number in the bracket represents the minimum PG temperature according to Superpave grade and the minimum PG temperature for samples O-R is not shown. From the data presented in Figure 4.13, it is observed that most of binders have grade lower than their respective of minimum performance grade temperature.

This shows that these binders should be free from any form of distress. However, the binders G and I of regular asphalt showed the limiting temperature grade warmer than minimum performance grade temperature so it is understood that these binders may crack significantly at low temperature. Also, the binders C of regular asphalt and B of asphalt rubber retained the same minimum temperature grade with minimum performance grade temperature. It is seemed that these samples did not show any negative impact in the performance. Further, it can be noted that warm asphalt rubbers showed very high limiting temperature grade though their minimum performance grade temperature are unknown and they are likely to show good durability at low temperature.

4.4.1 Superpave Grade and Grade Span

The Superpave grades and grade span for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber are summarized on Table 4.1. The grade span is the difference of high temperature grade and low temperature grade of a particular binder. For good quality asphalt, it should have high grade span so that it can be used wide range of temperature. The binder with very low grade span cannot be used in the climate of wide range of temperature.

The results are summarized in Table 4.1 shows that all the asphalt rubber and warm asphalt rubber have high grade and grade span as compared to their control binder. These binders can be used in the climatic region of very high and very a low temperature. Gain in grade span due to crumb rubber modifier ranges from low of 10 °C for sample F to a high of 41 °C for sample H compared to their control.

Table 4.1: AASHTO M30 Grades and Grade Spans (XX-YY) for different asphalt cements.

Sample	Highway	Composition	High Grade,°C	Intermediate Grade, °C	Low Grade, °C	Grade Span, °C
A	7	TB	70	3	-39	109
B	7	FB	84	8	-28	112
C	7	AC	58	11	-34	92
D	115	FB	102	6	-40	142
E	115	AC	67	4	-39	106
F	11	TB	64	4	-36	100
G	11	AC	61	17	-29	90
H	35	FB	93	7	-38	131
I	35	AC	60	16	-30	90
J	QEW	Warm AC	71	11	-31	102
K	402	Warm AC	67	14	-31	98
L	7	Warm AC	59	7	-40	99
M	62	Warm AC	59	7	-38	97
N	-	RAF	51	9	-34	85
O	-	AR	75	5	-39	114
P	-	Warm AR	74	0	-45	119
Q	-	Warm AR	71	3	-42	113
R	-	Warm AR	84	3	-39	123

Note: AC = control asphalt cement; AR = asphalt rubber; FB = field blend AR; TB = terminal blend AR; QEW = Queen Elizabeth Way; and RAF = roofing asphalt flux (soft AC from Cold Lake).

This could be due to the modification of binder with smaller particle size to bigger size of crumb rubber. The field blend binders from Highways 115 and 35 have comparatively higher grade span as compared to other binders prepared in the laboratory. This is likely that laboratory formulated binders were modified with CRM of 30 mesh. These binders could be used in the climate of wide range of high and low temperature as compared to formulated binders.

4.4.2 Bending Beam Rheometer Grading of Modified PAV residue

According to LS-299 protocol, the PAV condition was altered by changing the PAV conditioning time from 20 hours to 40 hours keeping the sample weight at 50 g to allow more time for aging in order to see correctly the effect of physical aging on the performance of asphalt.

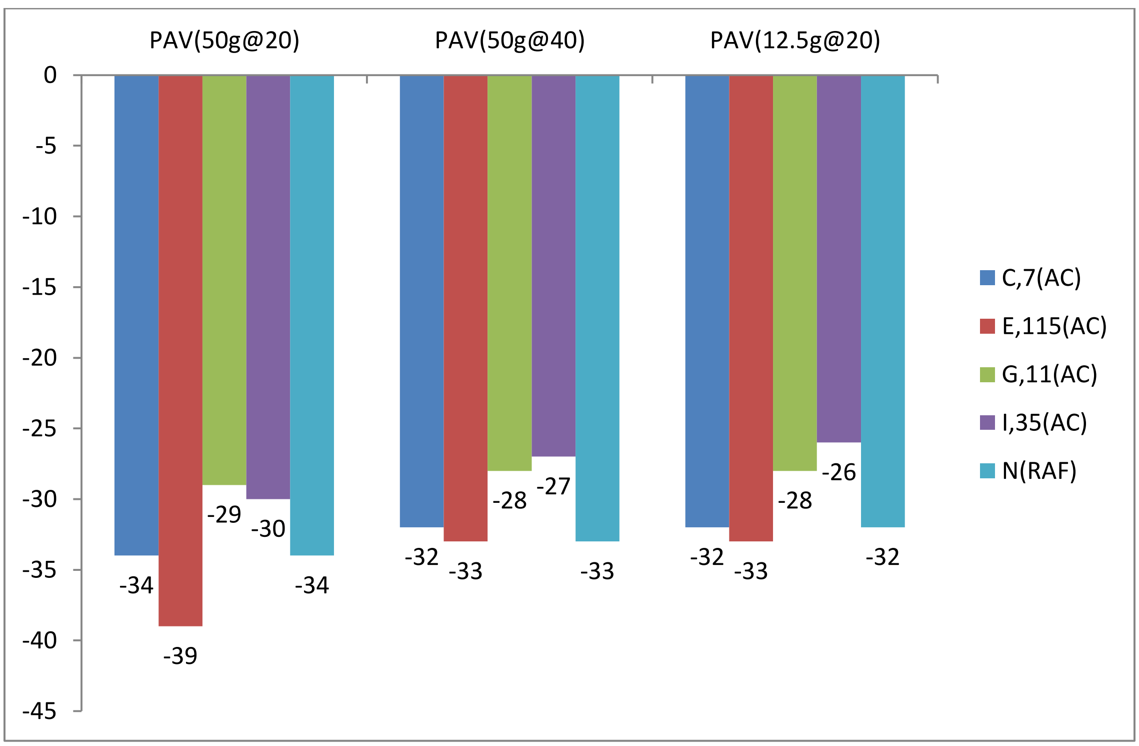


Figure 4.14: Low temperature grade for regular asphalt cement due to modified PAV.

Another way of aging was done by reducing the sample weight from 50 g to 12.5 g keeping the test time to 20 hour to see the effect of reduction of sample size on the aging process. The results are summarized in Figure 4.14 to 4.17.

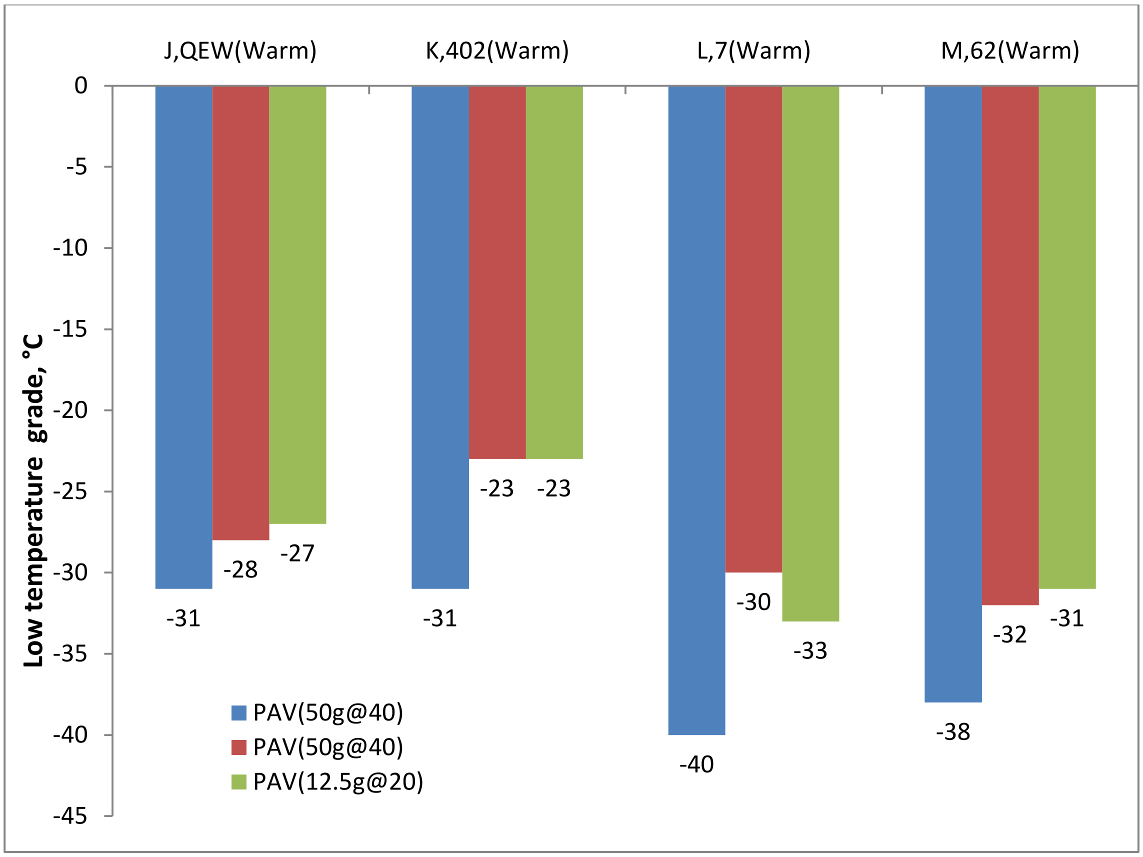


Figure 4.15: Low temperature grade for warm asphalt cement due to modified PAV.

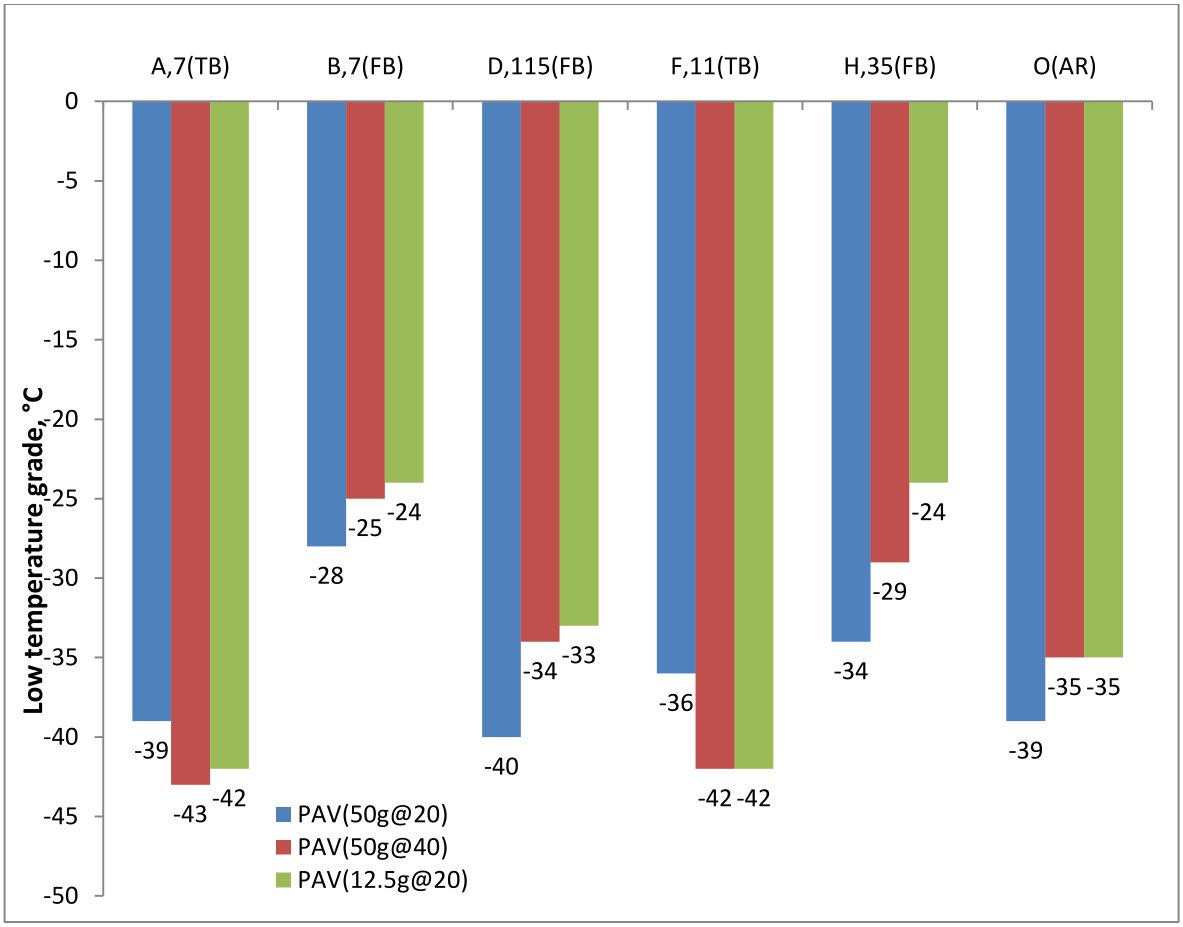


Figure 4.16: Low temperature grade for asphalt rubber due to modified PAV.

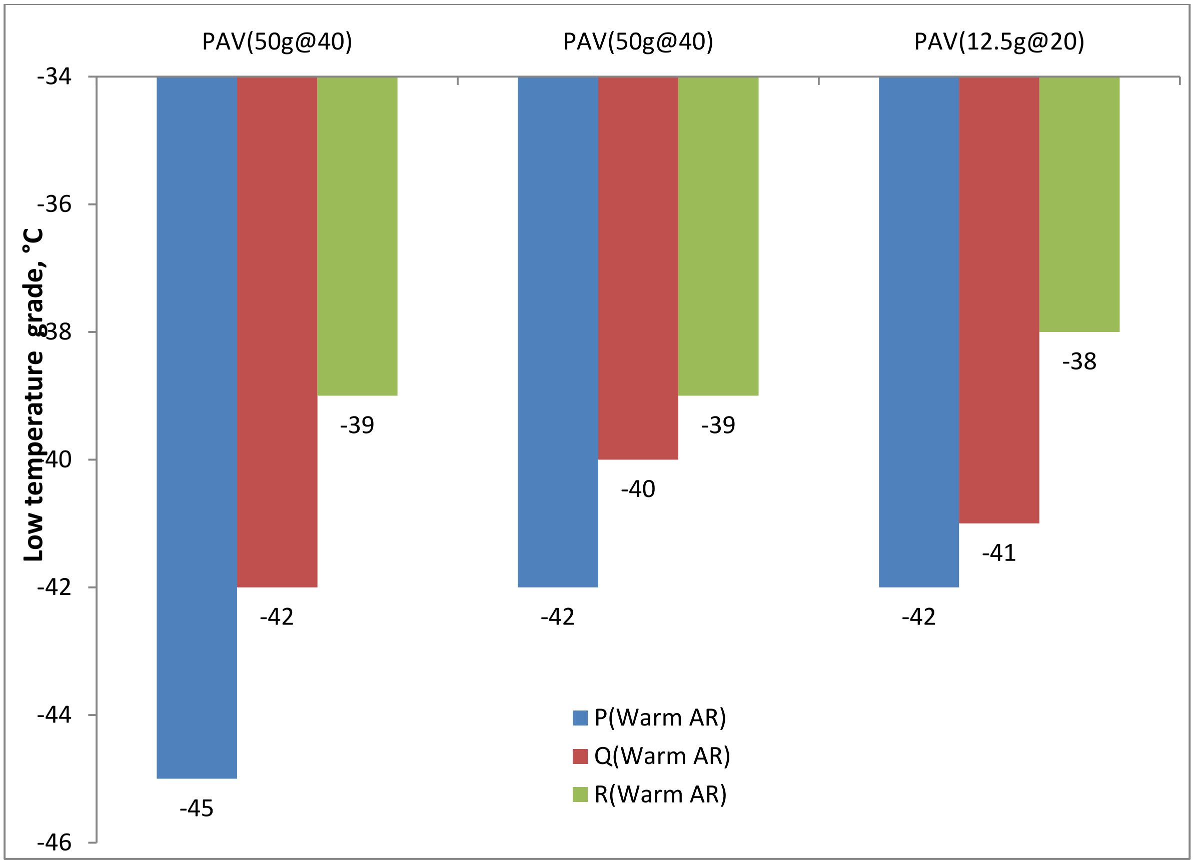


Figure 4.17: Low temperature grade for warm asphalt rubber due to modified PAV.

It can be observed from Figure 4.16 that the two terminal blend asphalt samples A and F gained -3 °C to -6 °C in their low temperature grade under the longer time or thinner film as used in modified PAV. This could be due to the presence of high natural rubber or polyisoprene content from the truck tire rubber. The polymer breaks down rapidly upon oxidation in the PAV. Fragments produced through oxidation would be able to soften the binder and then increase the relaxation rate (m-value). We need to study more whether this could be beneficial at cracking performance for this section or not. The data also shows that field blend sample H from the trial on Highway 35 lost 10 °C when aged in a thinner film. This rapid loss in grade indicates that this binder shows low durability and likely shows premature and excessive cracking at low temperature. The other interesting thing in the data is that the control binders C, G, I and J as well as binders formulated in the laboratory N-R all showed an average grade loss of only 2 °C due to modified PAV aging and hence would likely show very low cracking in PG-28 and PG-34 climatic regions. The data also show that a number of commercial binders rapidly lost grade after aging with modified PAV. Both the samples D and E lost around 6 °C. This would typically reduce the confidence that no damage occurs in a given winter from around 98 % to around 50 %. Also, it can be seen data in the Figure 4.16 that three of four commercial warm asphalt cement samples lost 8 °C, 10 °C and 7 °C when aged for longer time or thinner films indicating low durability and are likely to show premature and excessive cracking. This poor performance of these binders could be due to the presence of warm mix additives with inferior technologies such as air blowing, waste engine oil, or combination of both.

Figure 4.18 summarizes the comparison between the grade loss in longer time (50 g @ 40 hr) and thinner film (12.5 g @ 20 hr) according to LS-299 standard method of modified PAV. From the data presented in the Figure 4.18, it is obvious that most of samples after aging in longer time either lost equal grade or one degree more than the aging in thinner film. This may be due to the more oxidative hardening in aging for longer time. However, samples L of warm asphalt cement and sample O of warm asphalt rubber lost 1-3 °C more in thinner film than aging for longer time. This might be due to the presence of additives with other inferior technologies such as air blowing or waste engine oil.

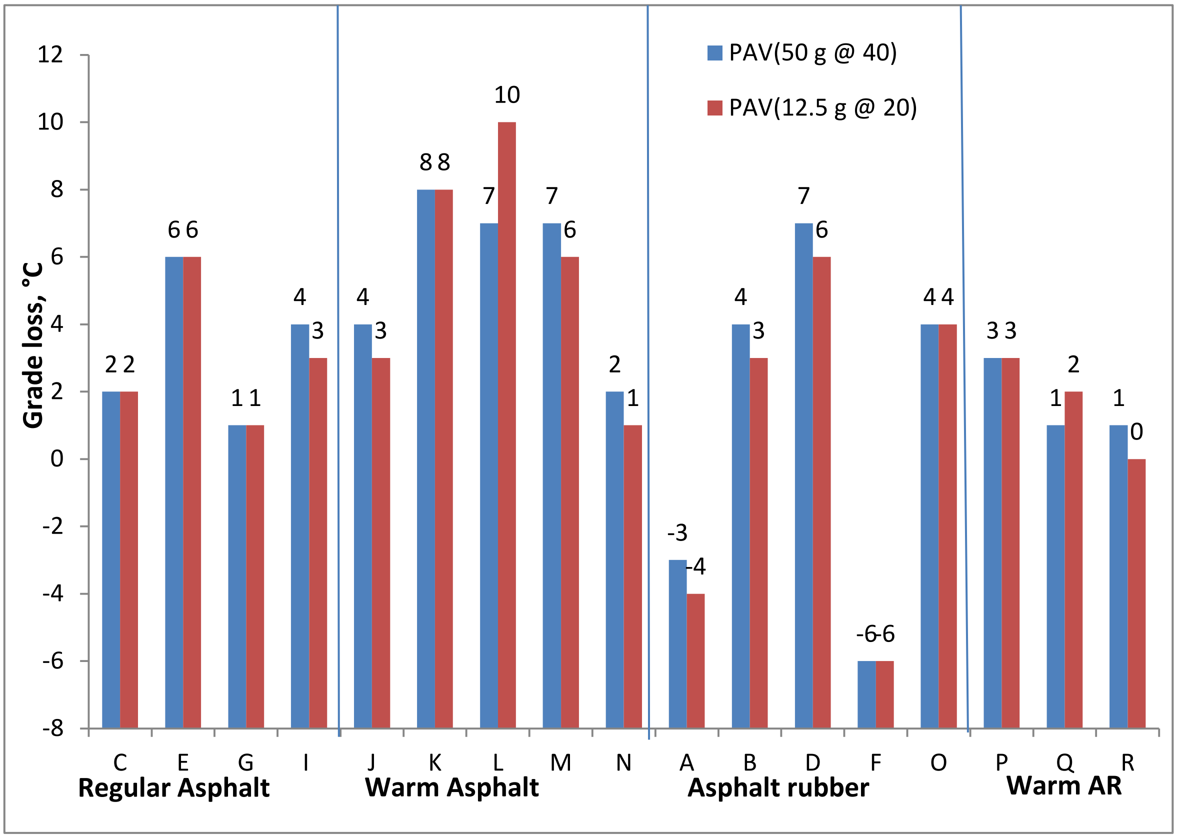


Figure 4.18: Comparison of Grade loss between PAV (50 g @ 40 hr) and PAV (12.5 g @ 20 hr).

4.4.3 Extended BBR Data Analysis

The asphalt samples after regular BBR test were further tested according to Ontario's extended BBR protocol as described in the MTO standard test methods LS-308. This gives the significant differences in the performance pavements in low temperatures. The asphalt binders were conditioned for 72 hours not just for one hour at isothermal conditions, i.e. at temperature 10 ^{o}C and 20 ^{o}C above the pavement minimum low temperature grade and the binders were tested in the same temperature. The limiting temperature where m (60) = 0.3 and S (60) = 300 MPa were determined according to AASHTO M320 standard protocol and the warmer temperature among the two limiting temperatures gives the minimum performance grade temperature of the pavement. Thus, the grades and grade losses were also determined after 72 hours of conditioning. These grades and grade losses indicate the performance of pavements. This method provides the minimum level of quality and durability and it provides vast improvement over AASHTO M320 specification. The extended BBR results for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber are given in Tables 4.2-4.5.

Table 4.2: Limiting temperature and limiting grade at different conditioning temperature for regular asphalt cement

Samples	Conditioning Temperature, °C	Limiting Temperature at which m=0.3 , °C		Limiting Temperature at which S=300, °C		Limiting Grade, °C	
		1 h	72 h	1 h	72 h	1 h	72 h
E	-14	-38.7	-33.4	-38.7	-33.9	-38.7	-33.4
	-24	-39.2	-38.2	-38.7	-36.4	-38.1	-32.2
C	-8	-37.2	-32.7	-34.6	-33.6	-34.6	-32.7
	-18	-34.8	-30.2	-34.3	-30.2	-34.3	-30.2
G	-8	-31.1	-30.2	-29.6	-28.6	-29.6	-28.6
	-18	-30.3	-26.6	-36.2	-29.2	-29.2	-26.6
N	-14	-35.7	-32.6	-34.1	-33.9	-34.1	-32.6
	-24	-34.7	-31.0	-34.1	-33.9	-34.1	-31.0

The eBBR grade and grade loss are provided in Figures 4.19 and 4.20 respectively. It has known that the grade loss is a good indicator of pavement performance. A lower grade loss indicates a low possibility or lower tendency to physical hardening at the conditioning temperature. The pavements with higher grade losses perform poorly in service while those with low grade lose perform better in service. According to LS-308 specification, the grade for contract climatic condition should meet after three days of conditioning and the grade loss should be less than 6 °C compared to one hour conditioning. So, the binders with grade loss than 6 °C are considered as good and those with more than 6 °C are considered poor for durability perspective.

Tables 4.2-4.6 show the limiting temperature at which m = 0.3, limiting temperature at which S = 300 MPa and limiting grade of regular asphalt cements at two different conditioning temperatures. It is observed that limiting temperature for all binders become warmer at 72 hour conditioning than one hour conditioning and further, it is noticed that the binders reduced their performance at lower temperature conditioning (-20 °C) than higher temperature conditioning (-10 °C). This is due to the more physical hardening of the binder at lower temperature and longer time. Further, it is noticed for all samples that the value of limiting grade is seen slightly higher at high conditioning temperature (-10 °C) than at low conditioning temperature (-20 °C). It can also be seen from the Table 4.2 that the sample G and N lose only 3 °C after three days conditioning whereas the samples C and sample E lost 4-6 °C after three days conditioning. This shows that samples G and N perform better than other regular asphalts and they have high durability. Table 4.3 shows that data for extended BBR for warm asphalt cement. It is observed that the samples J and K lost only 5 °C after three days conditioning which shows that these binders have no significant cracking at low temperature. The sample L lost 6 °C that means it is either borderline or may be fail test according to LS-299 specification and comparing to these data sample L lost 8 °C which indicates this sample has several cracking problem and is likely show low durability in performance perspective.

Table 4.3: Limiting temperature and limiting grade at different conditioning temperature for warm asphalt cement.

Samples	Conditioning Temperature, °C	Limiting Temperature at which m = 0.3 , °C		Limiting Temperature at which S = 300MPa, °C		Limiting Grade, °C	
		1 h	72 h	1 h	72 h	1 h	72 h
J	-8	-30.7	-25.4	-36.3	-34.3	-30.7	-25.4
	-18	-30.7	-22.6	-35.4	-34	-30.7	-26.4
K	-8	-29.7	-28.5	-33.5	-32.7	-29.7	-28.5
	-18	-30.6	-25.6	-32.9	-30.6	-30.6	-25.6
L	-14	-40.0	-36.2	-40.4	-39.7	-40	-36.2
	-24	-40.5	-32.4	-40.0	-37.6	-40	-32.4
M	-14	-39.2	-35.7	-38.3	-37.1	-38.3	-37.1
	-24	-39.5	-32.1	-37.7	-35.1	-37.7	-32.1

The data in the Table 4.4 shows the limiting temperature and limiting grade for all the samples of asphalt rubber. From these data, it is seen that most of the samples lost more than 6 °C and failed to meet the criteria set out in LS-308 specification and the sample D lost as much as 12 °C after three days conditioning indicating lack of durability which would likely show premature and excessive cracking. As we know an error of 6 °C reduces the confidence that no damage occur by 50 % so, an error of 12 °C reduces the confidence that no damage occur nearly to zero %. Further, results in Table 4.4 show that even the high quality Cold Lake roofing flux modified with 18 % CRM (Sample O) suffers too much from physical hardening with a three day loss of 8 °C. Thus, it is obvious that all these samples have serious problem in durability point of view.

Table 4.4: Limiting temperature and limiting grade at different conditioning temperature for asphalt rubber

Samples	Conditioning Temperature, °C	Limiting Temperature at which m = 0.3 , °C		Limiting Temperature at which S = 300 MPa, °C		Limiting Grade, °C	
		1 h	72 h	1 h	72 h	1 h	72 h
A	-8	-40.5	-37.8	-42.4	-42.7	-40.5	-37.8
	-18	-39.3	-32.3	-42.1	-41.4	-39.3	-32.3
B	-8	-27.1	-21.9	-34.4	-35.8	-27.1	-21.9
	-18	-27.7	-22.2	-34.1	-33.5	-27.7	-22.2
D	-14	-35.2	-28.1	-43.3	-42.4	-35.2	-28.1
	-24	-40.2	-28.5	-42.1	-40.2	-40.2	-28.5
F	-14	-37.7	-33.8	-42.9	-42.3	-37.7	-33.8
	-24	-35.6	-30.1	-41.7	-41.1	-35.6	-30.1
O	-14	-37.4	-33.6	-37.4	-33.6	-37.4	-33.6
	-24	-38.8	-30.6	-38.8	-30.6	-38.8	-30.6

Table 4.5 shows the data for extended BBR results obtained for warm asphalt rubber which contain the limiting temperature and limiting grade for all the samples of warm asphalt rubber. Based on the data shown in Table 4.5, it is observed that these samples lost 4 -7 °C indicating

satisfactory to significant cracking problem. However, these binders were modified with naphthenic oil so this reduces the physical hardening to some extent and can be used in the climatic region of PG-28 and PG-34.

Table 4.5: Limiting temperature and limiting grade at different conditioning temperature for warm asphalt rubber

Samples	Conditioning Temperature, °C	Limiting Temperature at which m = 0.3 , °C		Limiting Temperature at which S = 300MPa, °C		Limiting Grade, °C	
		1 h	72 h	1 h	72 h	1 h	72 h
P	-14	-43.4	-40.9	-42	-40.9	-42.0	-40.9
	-24	-44.9	-38.2	-44.8	-38.2	-44.8	-38.2
Q	-14	-41.0	-45.5	-41.9	-40.7	-41.0	-40.7
	-24	-42.9	-35.8	-41.7	-39.7	-41.7	-35.8
R	-14	-39.8	-38.8	-41.4	-40.9	-39.8	-38.8
	-24	-39.0	-35.4	-41.1	-39.1	-39.0	-35.5

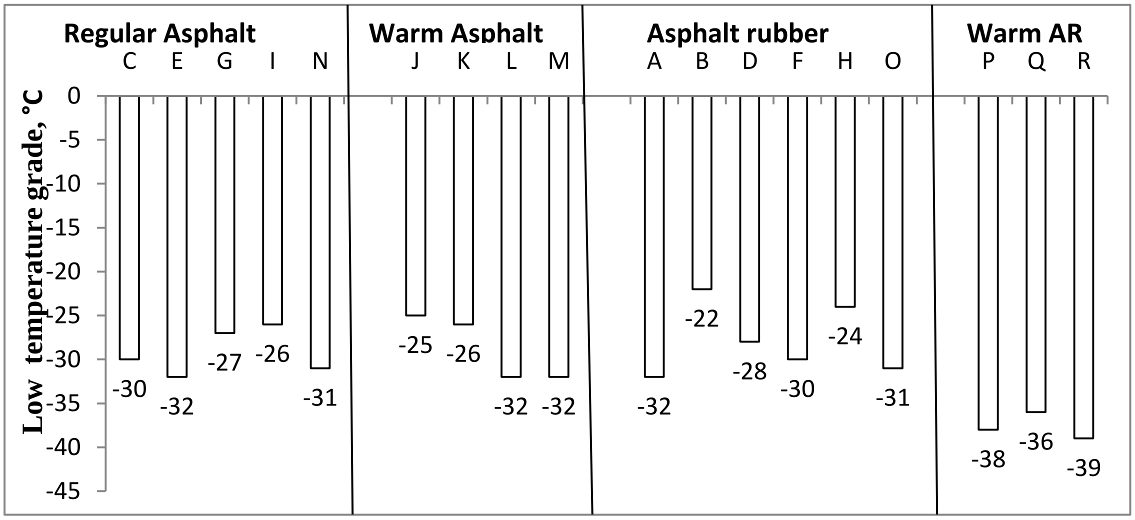


Figure 4.19: eBBR grades according to LS-308 for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber.

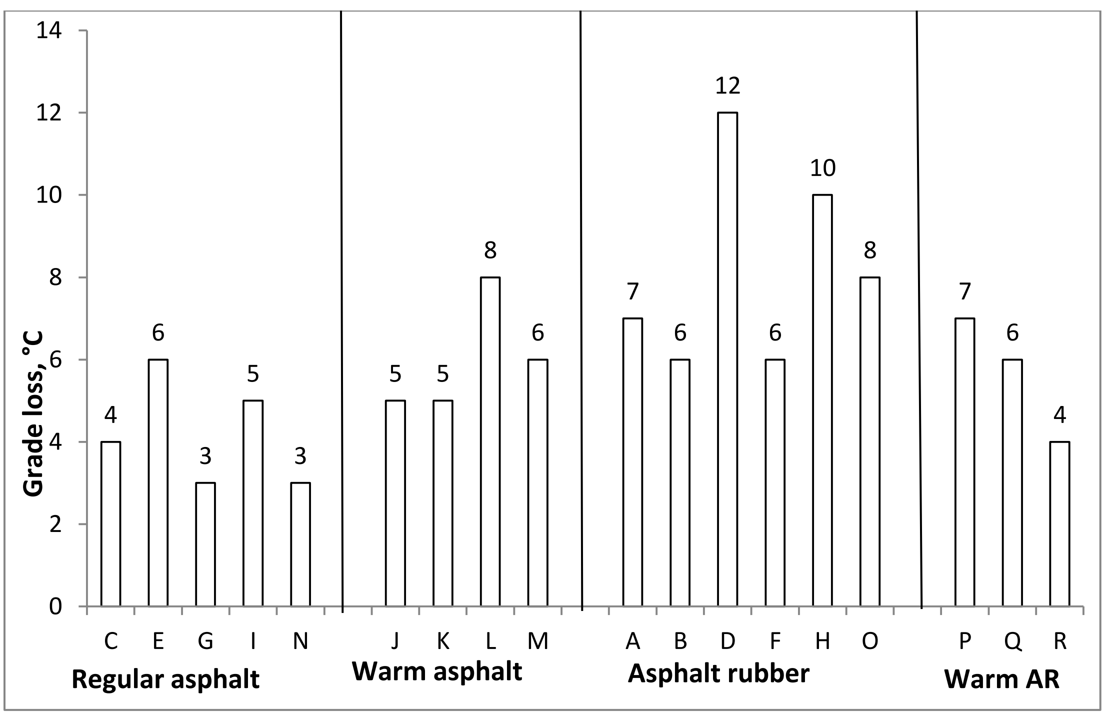


Figure 4.20: Low temperature grade loss according to LS-308 for regular asphalt cement, warm asphalt cement, asphalt rubber and warm asphalt rubber.

Figures 4.19 and 4.20 contain the eBBR grade and grade loss for all the binders tested according to LS-308 protocol. On the basis of data presented in the Figures 4.19 and 4.20, it is obvious that all the samples of asphalt rubber which have highest grade loss rank worst and regular asphalt which have low grade loss rank best in the performance perspective. The other samples of warm asphalt and warm asphalt rubber are in between in the ranking in term of performance perspective because their grade loss is on the average less than 6 °C.

4.4.4 Correlation between chemical hardening and physical hardening

For straight or regularly modified AC of approximately equal grade, it has been found that the grade loss due to extended BBR, i.e. LS-308 grade is highly correlated with the grade loss due to modified PAV, i.e. a doubling of the PAV aging time as shown in Figure 4.21. The correlation coefficients for these straight lines are pretty close to each other. Figure 4.21 shows that on increasing grade loss due to modified PAV increase the grade loss due to extended BBR and vice-versa. Further, it is seen that any shift of 3 °C results these two straight lines coincide with each other as studied in field blend AR. This could be due to different mechanisms of physical and chemical hardening in AR compared to those in straight and regularly modified AC.

Asphalt rubber contains 18 % crumb rubber modifier along with extender oil. Extender oils swell the rubber particles and help to disperse them in asphalt. The extender oils are likely released during high temperature mixing in the asphalt rubber and these oils are likely reabsorbed only slowly during low temperature conditioning. This would result in a physical hardening effect. For example, the RAF with 18 % CRM lost 8 °C.

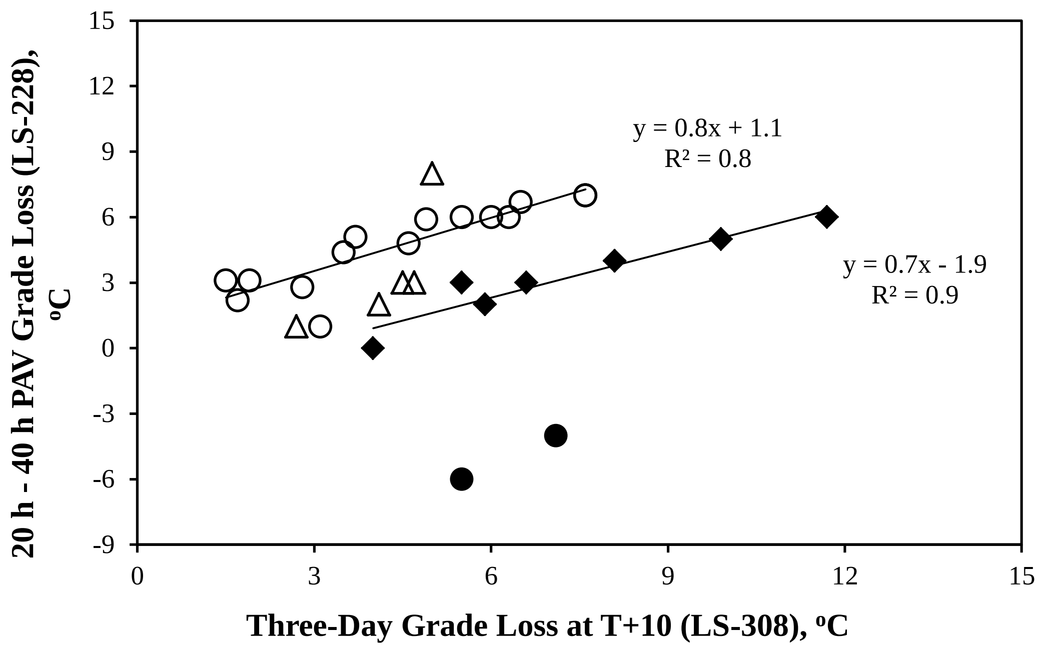


Figure 4.21: Chemical hardening (LS-228) versus physical hardening (LS-308) in various asphalt cements, warm asphalt cements, and asphalt rubbers. Note: Open symbols are for straight and regularly modified AC from test sections and a number of regular contracts including those from a northern Ontario trial. Circles are for PG -34 while triangles are for PG -28. Filled circles are for TB AR and filled diamonds are for FB AR.

In contrast, physical hardening in straight and regularly modified asphalt cement only involves the wax and resin fractions that slowly precipitate onto the asphaltene network. This results in the formation of rigid amorphous solid and stiffens the material [4]. In addition, the significant increase in viscosity of field blend asphalt rubber due to PAV aging hinders the diffusion of oxygen and thus slows the aging process.

CHAPTER 5

Summary and Conclusions

Based on the background, experimental procedures, results and discussions of this thesis, the following summary and conclusions are given:

- The Black space diagram shows that all the tested asphalt samples are rheologically simple except E and L. These two samples are rheologically complex. This may be due to the addition of polymer to these samples which causes phase separation
- The asphalt samples tested in this study showed a range of tendencies for chemical and physical hardening. Most commercial warm and AR binders failed AASHTO M320 specification criteria for low temperature performance after modified PAV aging or extended conditioning in the BBR test.
- 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 and hence it serves as an improvement over the regular BBR method because it has been seen that it penalizes the poor performing asphalt more than good performing asphalt. The grade loss for the extended BBR after 72 hours of conditioning at -10 °C ranged from 1-2 °C for samples G and N (good performing asphalt) respectively to 12 °C for sample D (poor performing asphalt).

The grade losses for sample G and N indicate that these samples have a low affinity for reversible aging at lower temperatures. The difference of grade loss of more than 10 °C in sample D indicates lack of durability and that will likely lead to premature and excessive cracking. This will reduce the confidence that no damage occur by nearly 100 % for a typical Ontario climatic condition.

- Increasing the PAV aging time to 40 h with constant weight or reducing the weight per pan to 12.5 g would provide significant improvements to the current RTFO/PAV protocol which uses 20 h and 50 g.
- The commercial binders also possessed low strain tolerances as measured with the double-edge-notched tension test protocol. The CTOD embodied in the MTO LS-299 has been able to distinguish superior performing binders from inferior performing binders, and it has also been able to rank the performance of the asphalt cements with maximum accuracy. The double-edge-notched tension (DENT) test thus appears to do a reasonable job at separating the superior binders from inferior ones.
- The poor performance of some of these commercial samples may be due to the use of inferior modification technologies such as air blowing, waste engine oils, etc. or combination of both. Asphalt cements modified with above technology (e.g., waste engine oils, air blowing etc.) can pass the current specification methods under AASHTO M320 but these materials were unable to give good performance and failed to provide a good return on investment. Pavements that are modified in the above manners were found to crack prematurely or excessively. This is likely 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 oxidation in service.

- Rubberized asphalt samples failed likely due to oils being absorbed by the crumb rubber modifier and thus destabilize the colloidal structure of the base asphalt cement.
- Superior chemical and physical aging performance can be obtained through a selection of appropriate ingredients and by an adjustment of the low temperature grade through the addition of naphthenic oils. Strain tolerance can be improved by the addition of a small amount of high vinyl SBS copolymer.

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