EFFECTS OF WARM MIX ADDITIVES AND DISPERSANTS ON RHEOLOGICAL, AGING AND FAILURE PROPERTIES OF ASPHALT CEMENTS

by

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Abstract

Existing specifications for asphalt cement employ insufficient aging and conditioning times prior to testing and low strains during the actual test which are insufficient to predict asphalt performance, especially if the materials are modified with additives such as those used for warm mix technology. However, slightly modified protocols, like increasing the conditioning time in the bending beam rheometer (BBR) test and increasing the aging duration in the pressure aging vessel (PAV), predict asphalt performance better than the current Superpave™ specification. These improved protocols are published as new test standards through the collaborative effort between the Ontario Ministry of Transportation and Queen's University.

In this study, the effects of warm mix and other additives on rheological, aging and failure properties are investigated. The properties are measured by regular tests and by modified protocols. The latter include the extended BBR test (LS-308) and the double-edge-notched tension (DENT) test (LS-299). Changes in ductile strain tolerance within base asphalts due to the various additives as measured with the DENT test were found to be very significant. The DENT results like essential work of fracture, w_e, plastic work of fracture term, βw_p, and critical crack tip opening displacement, CTOD, are usually helped to correlate with the cracking distress survey results of the pavement in service. The addition of amide and polyethylene waxes risks increasing the cracking susceptibility in the pavement. They show a negative effect on strain tolerance in the ductile state, which is likely to show up as premature and/or excessive cracking in service which is similar to their physical hardening behavior from low temperature grading and extended BBR testing.

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Abbreviations and Acronyms

AASHTO	American Association of State and Highway Transportation Officials
AC	Asphalt Cement
ASTM	American Society for Testing and Materials
BBR	Bending Beam Rheometer
CTOD	Crack Tip Opening Displacement, m
DENT	Double-Edge-Notched Tension
DSR	Dynamic Shear Rheometer
eBBR	Extended Bending Beam Rheometer
EWF	Essential Work of Fracture
HMA	Hot Mix Asphalt
LS	Laboratory Standard Test Method
LTPPBind®	Long Term Pavement Performance Binder Selection Software
m(t)	Slope of the Creep Stiffness Master Curve (m-value)
S(t)	Time-dependent Flexural Creep Stiffness, MPa
MPa	Mega Pascal (Pa)
MTO	Ministry of Transportation of Ontario
NSERC	Natural Sciences and Engineering Research Council of Canada
PG (PGAC)	Performance Grade (Performance Graded Asphalt Cement)
PMA	Polymer Modified Asphalt
PPA	Polyphosphoric acid
SBR	Styrene-Butadiene-Rubber
SBS	Styrene-Butadiene-Styrene
SHRP	Strategic Highway Research Program
TFOT	Thin Film Oven Test
PAV	Pressure Aging Vessel
PI	Penetration Index
RTFO	Rolling Thin Film Oven
SUPERPAVE^{TM}	SUperior PERforming Asphalt PAVEment
WEO	Waste Engine Oil
3D	Three-Dimensional

Symbols

A	Temperature susceptibility parameter (Penetration test)
a	Length of a sharp crack, m
b	Beam width, 12.5mm
B	Specimen Thickness, m
G*	Complex shear modulus,
G'	Storage modulus
G"	Loss modulus
h	Beam thickness, 6.25mm
K	Stress intensity factor, N.
L	Ligament length
L	Distance between beam supports, 102 mm (BBR)
N	Number of load repetitions

P	Load applied, N (BBR)
t	Loading time, s
T	Temperature, K
W_e	Essential fracture energy, J
w_e	Specific essential work of fracture, $J.m^{-2}$
W_p	Plastic or non essential work of fracture, J
w_p	Specific plastic work of fracture, $J.m^{-2}$
W_t	Total energy, J
w_t	Specific total work of fracture, Jm^{-2}
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/m^2)

Chapter 1

INTRODUCTION

1.1 Asphalt Origin, Chemistry and Applications

As shown in Figure 1.1, asphalt cement is a semi-solid material with both solid and liquid characteristics depending on the time scale of the observation. It is generally used in road construction as a binder for aggregates and sand while in the roofing industry, it is mixed with fine aggregate and fibers to produce a waterproofing membrane. The American Society for Testing and Materials (ASTM) defines asphalt as " a subclass of bitumen which occurs in nature or is obtained in petroleum processing" [1]. The name, asphalt or asphalt cement (AC) is generally used in North America. But it is called bitumen in Europe and in most eastern countries. The word asphalt has a different meaning in Europe, where it is used to describe the asphalt mix, i.e. the mixture of binder and the aggregate forming the road. In North America, asphalt or asphalt cement is used to indicate the binder only.

Figure 1.1: (A) Asphalt and (B) Asphalt mix (asphalt mixed with aggregates) [85].

The American Association of State Highway Transportation Officials (AASHTO) defines binder as an "asphalt based cement that is produced from petroleum residues either with or without the addition of non-particulate organic modifiers" [2]. Asphalt has a composition of mainly carbon (82-88%) and hydrogen (8-11%). Heteroatoms like sulphur (0-6%), oxygen (0%-1.5%) and nitrogen (0-1%) are also present. Finally, it also has small amounts of vanadium, nickel and traces of other metals. The molecular structure of asphalt can be explained by its SARA fractions. SARA refers to Saturates, Aromatics, Resins and Asphaltenes.

Natural asphalt is available as lake asphalt, rock asphalt, Gilsonite and tar. Artificial asphalt, typically paving grade, is about 85% produced from crude oil as the vacuum residue of the petroleum distillation process [4]. Asphalt is modified by blending, air blowing and adding additives like acids, bases, mineral fillers, and polymers, to improve its properties to achieve the desired performance.

Today, 95% of the 100 million tonnes of asphalt that is produced world-wide is used for road construction in the form of asphalt mixes [3]. It is also used for roofing, emulsions, paints, damp proofing, water proofing buildings and structures, disinfectants, fence post coatings, mulches, tree paints and adhesives applications.

1.2 Asphalt Pavements

Generally road pavements are of two types: rigid pavements (PCC - Portland cement concrete) and flexible pavements (asphalt mixes). Asphalt pavement concrete is also called hot mix asphalt (HMA) pavement. Generally, asphalt mixes are produced by heating the asphalt cement to about 160°C and blending it at about 5 weight percent with aggregate. An

asphalt pavement design involves the thickness design of granular bases, binder courses, and surfaces courses [5], as shown in Figure 1.2, with each one performing a specific function to help the pavement resist various forms of distress.

Surface Course ~ 40 mm
Binder Course ~ 50 mm
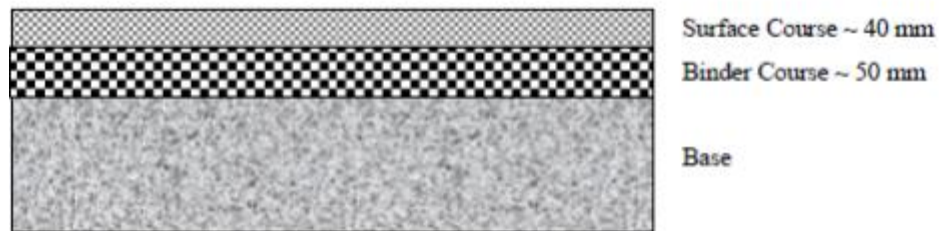
Base

Figure 1.2: Typical design of a two lift asphalt pavement on top of a granular base [5].

Pavement design methods are used to do a structural evaluation process needed to ensure the distribution of traffic loads such that stresses and strains developed in all levels of the pavement and sub-grade remain within the load bearing limits of the materials [6].

1.3 Asphalt Distresses

Asphalt pavement failures through rutting, fatigue cracking, low temperature cracking, and moisture damage are caused by rain, sunlight, oxygen, chemicals, thermal stresses, distortions, and disintegrations, with the application of traffic load repetitions over a period of time.

1.3.1 Rutting

Rutting is caused by permanent deformation within the asphalt surface layers as shown in Figure 1.3(a). Rutting typically occurs at high temperatures by unrecoverable strain accumulated from repeated loads to the asphalt pavement. The viscosity and thermal history of the asphalt are important factors in the rutting behavior. The rutting resistance factor,

G*/sinδ (where G* is the complex modulus and δ is the phase angle) helps to predict the rutting behavior and it can be measured by using a dynamic shear rheometer (DSR).
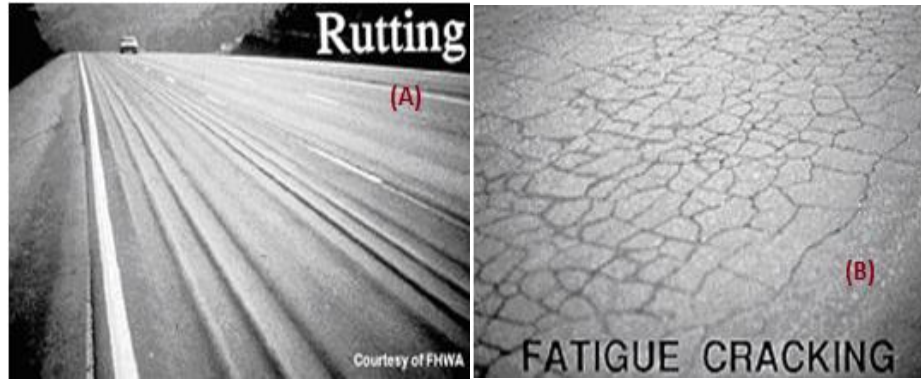
Figure 1.3: (A) Severe rutting and (B) Fatigue cracking [87]

1.3.2 Fatigue Cracking

Fatigue cracking occurs as shown in Figure 1.3(b) due to cycling loading by vehicles under moderate and low temperature conditions which induces stress that is more than the fatigue limit of the pavement. Fatigue distress is also known as alligator cracking since its pattern resembles the skin of an alligator. Potholes develop in fatigue cracking by the action of traffic loads which separate the pavement surface from the underlying layers [7]. The fatigue cracking resistance factor, G*sinδ (where G* is the complex modulus and δ is the phase angle) helps to predict the fatigue behavior and it can also be measured by using a dynamic shear rheometer (DSR).

1.3.3 Thermal Cracking

Thermal cracking, also known as low temperature or transverse cracking, is shown in Figure 1.4(a). Stresses induced in the layers of asphalt under low temperature conditions like those

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frequently encountered in Canada causes low temperature shrinkage followed by thermal cracking. Initially, single-event cracking forms when a drop below the temperature limit causes stress build-up above the strength of the mix. Secondly, thermal fatigue cracking resulted by the loss of strength of asphalt mix as a result of repeated thermal stresses below the temperature limit. Finally, mixed load/thermal distress cracking occurs by freeze/thaw cycling during spring in addition to heavy traffic loading [8].

Figure 1.4: (A) Thermal cracking and (B) Moisture damage [87]

1.3.4 Moisture Damage

Stripping of the asphalt cement from the aggregate and a lack of cohesion between the layers causes moisture damage as shown in Figure 1.4(b). It happens by the presence of temperature, moisture, poor construction materials and traffic loads. It reduces the strength of the asphalt, lack of bonding between the layers, and freezing of the individual aggregate particles, which leads to failure of the pavement [9]. Anti-stripping agents, proper drainage and proper compaction can avoid moisture damage.

1.3.5 Asphalt Durability

Durability is defined as the change of asphalt physical properties due to aging. The factors causing loss of durability are age hardening through oxidation, volatilization, polymerisation, thixotropy, syneresis, separation, short term and long term aging [10]. Generally, It is not possible to directly measure durability but we can simulate aging and measure the properties with standard physical tests like viscosity, DSR, BBR, and direct tension test (DTT).

1.4 Performance Grading

Since there is not enough chemical knowledge to account for asphalt properties, mainly physical or mechanical testing methods are used to estimate their performance because fundamental asphalt failure cannot be chemically investigated. So, methods are developed to classify asphalts to know their properties and predict their performance by conventional testing methods and more recently, Superpave^{TM} methods.

1.4.1 Conventional Testing

Conventional testing is generally used to classify asphalt grades. Penetration grade, oxidized grade, hard grade, and cutback grade, are terms used under conventional specifications. Conventional tests are the penetration test at 25°C, softening point test, and viscosity tests, like dynamic viscosity test at 60°C, kinematic viscosity test at 135 °C, Fraass breaking point test, specific gravity test, storage stability test, ductility test, force-ductility test, elastic recovery test, and aging like thin film oven test (TFOT), rolling thin film oven test (RTFO), and PAV.

1.4.2 Superpave^{TM} Testing

Conventional test methods are generally empirical so they are not always effective at predicting the performance of asphalt in the field. In 1987, the Strategic Highway Research Program (SHRP) undertaken in the USA developed the Superpave^{TM} testing methods. These methods were supposed to allow for the construction of SUperior PERforming PAVEments. This method helped to classify grades based on their performance with simulated aging and physical properties determinations. It involved aging like RTFO and PAV followed by measuring of properties in the DSR and BBR.

1.4.3 Ontario Ministry of Transportation (MTO) Test Standards

It is important to consider physical aging [11] and not only the properties such as creep and stress relaxation as per the AASHTO M320 (American Association of State Highway Officials) specification. The Superpave^{TM} specification tests are done on asphalt cements that are insufficiently aged and conditioned and are tested under low strain conditions to predict asphalt performance. The collaboration work of the Ontario Ministry of Transportation with Queen's University has developed the following set of new and improved test methods:

1. Extended Bending Beam Rheometer (eBBR) test (LS-308) [12];

2. Double-Edge-Notched Tension (DENT) test (LS-299) [13]; and

3. Modified Pressure Aging Vessel protocols (LS 228) [14].

These methods have shown to be significantly more effective in terms of asphalt performance prediction than the Superpave^{TM} methods. Poor quality asphalts are penalized during aging for longer times, or in thinner films, and/or in the presence of moisture compared to good asphalts which are much less affected [15]. So, these new test methods

make it easier to select quality asphalts rather than to guess, as is done with the Superpave specification.

1.5 Scope and Objectives

Asphalt pavement contractors prefer to lay the paving with less energy consumption and easier way of compaction particularly in the colder season or northern regions like Canada. This issue motivated asphalt manufactures to develop warm mix asphalt technology which involves the addition of additives to the asphalts.

This technology helps to reduce the compaction temperature of the pavements by as much as 30°C to 50°C by lowering the high shear viscosity during compaction, by providing lubrication, and/or the stabilization of limited amounts of air bubbles in the hot asphalt cement. These additives are based on waxes and/or surfactants and other proprietary modifiers. Generally, these additives are able to help improve the useful temperature interval in service, phase stability, durability by reduced hardening, and also fit in the Superpave™ specification. They are also expected to improve binder performance by providing good cracking resistance, moisture resistance, and good adhesion and cohesion within the pavements layers [16].

But, the existing specification follows insufficient aging and conditioning time and low strain testing methods to predict asphalt performance. However, slightly modified protocols, like increasing the conditioning time in the BBR test and increasing the aging duration in the PAV, will predict asphalt performance better than the current Superpave™ approach. These protocols are published as new test standards through the collaborative effort between the Ontario Ministry of Transportation and Queen's University.

In this study, the effect of warm mix and a host of other additives on rheological, aging and failure properties are investigated. The properties are measured by regular tests and by modified new protocols such as the extended BBR test (LS-308) and the DENT test (LS-299).

Chapter 2

BACKGROUND

2.1 Asphalt Modifications

Asphalt is a colloidal dispersion of asphaltenes peptized by non-polar materials which are generally referred to as resins [17, 18]. There are two types of asphalts: those with highly aromatic maltene fractions and asphaltenes that are well dispersed are called sol-type asphalt while the other are called gel-type asphalts where the aromatic maltene fractions are unable to sufficiently disperse the asphaltenes. Schematic representations of both sol-type and gel-type asphalts are provided in Figure 2.1 Sometimes the asphaltenes agglomerate into connected structures and form a continuous network. Sol-type asphalts have better physical properties and resistance to cracking than gel-type [19, 20].
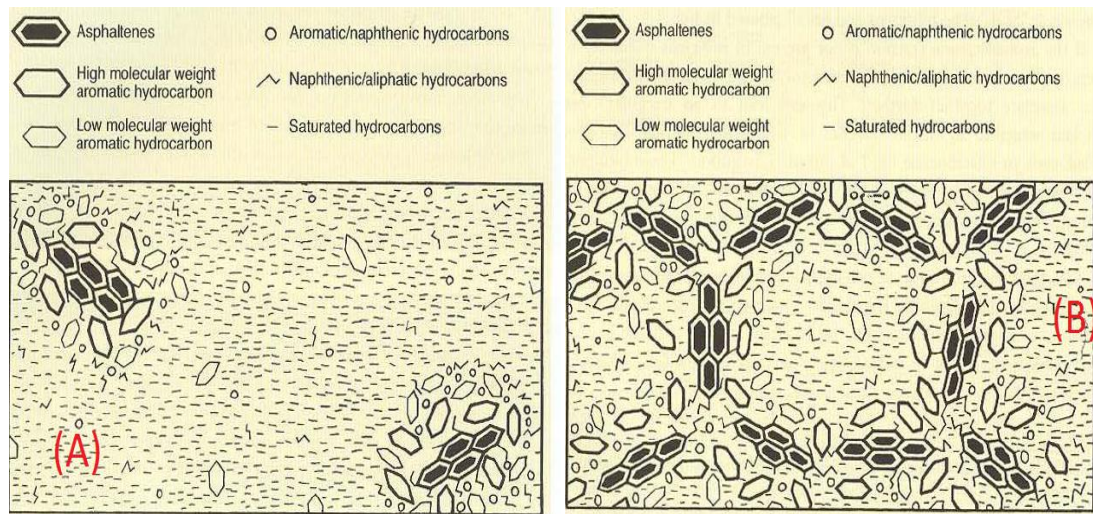
Figure 2.1: (A) Sol-type and (B) Gel-type asphalts [6].

In the United States of America and Canada asphalt is manufactured by the refining of crude oil which involves basic unit operations like atmospheric and vacuum distillation. Various grades of asphalt are produced by various processes like blending, air blowing, solvent deasphalting, solvent extraction, emulsification and modification etc., as shown in Table 2.1. What process is used for the production of asphalt depends on the crude sources and a host of other considerations.

Table 2.1: Various Methods used to Produce Asphalt [53]

S.No	Production/process	Base Material	Product
1	Atmospheric and Vacuum Distillation	Asphalt-based Crude or Crude Mix	Asphalt Cement
2	Blending	Hard and soft asphalt cements and petroleum distillates	Asphalt Cements of intermediate consistency cutback asphalts
3	Air blowing	Asphalt flux	Asphalt cements, roofing asphalt, pipe coating, special membranes
4	Solvent Deasphalting	Vacuum Residuum	Hard asphalt
5	Solvent Extraction	Vacuum Residuum	Asphalt Components (Asphaltenes, Resin, Oils)
6	Emulsification	Asphalt, Emulsifying agent and water	Emulsified Asphalts
7	Modification	Asphalt and Modifiers (Polymers, Chemicals etc.)	Modified asphalts

Generally, asphalt is modified to meet the special requirements of pavements ease of road constructions, reduce the maintenance frequency, improve the useful temperature interval in service, phase stability, reduce the cost, durability by reduced hardening and also fit in the specifications like Superpave™, etc. They are also expected to improve binder performance

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by providing good cracking resistance, moisture resistance, and good adhesion and cohesion within the pavement layers [16].

Asphalt pavement contractors prefer to construct the pavement with less energy consumption and easier way of compaction particularly in the colder season or northern regions like Canada. This issue has motivated asphalt manufactures to develop warm mix asphalt technology, which involves the addition of additives to the asphalts which will be discussed in detail later. The various types of asphalt modifiers and their examples are as shown in the Table 2.2.

Table 2.2: Asphalt Modifier Types [6].

S.No	Type of Modifier	Example
1	Thermoplastic elastomers	Styrene-butadiene-styrene (SBS)
		Styrene-butadiene-rubber (SBR)
		Styrene-ethylene-butadiene-styrene (SEBS)
		Ethylene-propylene-diene terpolymer (EPDM)
		Natural rubber
		Crumb tire rubber
		Polybutadiene (PBD)
		Polyisoprene
2	Thermoplastic polymers	Ethylene vinyl acetate (EVA)
		Ethylene butyl acrylate (EBA)
		Atactic polypropylene (APP)
		Polyethylene (PE)
		Polypropylene (PP)
3	Thermosetting polymers	Epoxy resin
		Polyurethane resin
4	Chemical modifiers	Organo-metallic compounds
		Sulphur
		Lignin
5	Fibres	Cellulose
		Alumino-magnesium silicate
		Glass fibre
		Asbestos
		Polyester
		Polypropylene
6	Adhesion improvers	Organic amines
		Amides
7	Anti-oxidants	Amines
		Phenols
		Organo-zinc/organo-lead compounds
8	Natural asphalts	Trinidad Lake Asphalt (TLA)
		Gilsonite
		Rock asphalt
9	Fillers	Carbon black
		Hydrated lime
		Lime
		Fly ash

2.2 Warm Mix Technology and Additives

Hot mix asphalt (HMA) is widely used as the primary paving material in road construction in North America. It involves the mixing of aggregate and asphalt binder by the application of heat. The HMA process produces emissions of particulate matter such as dust, smoke, exhaust vapor, and other gaseous pollutants. So, asphalt manufactures are trying to decrease the mixing and compaction temperatures of the mixes, without affecting the mix properties, in order to reduce emissions from the HMA process, and also to reduce energy requirements [71]. Today, Warm Mix Asphalt (WMA) is used to reduce the mixing temperatures to anywhere from 100 to 140°C compared to mixing temperatures of 150 to 180°C for regular hot mix asphalt [67]. It involves three major methods which are based on foaming, water bearing agents, and special bitumen additives.

The foaming process makes small steam bubbles inside the asphalt binder. It increases the volume of the asphalt binder to be coated onto the aggregate and sand. It results in an increase of asphalt wettability and also lower high shear viscosities. WAM foam is a type of foaming process. This process was patented and developed jointly by Shell Global Solutions and Kolo Veidekke in Norway. This process helps to produce the asphalt mixture at temperatures between 100°C and 120°C. It also helps to compact it at 80 to 110°C [68]. WAM foam involves two steps. In the first step, a softer binder is mixed with aggregates to provide good fluidity at lower temperatures. In the second step, the harder binder is foamed. This foamed asphalt is mixed with the aggregates pre-mixed with the softer binder.

Secondly, water-bearing agents are added with the asphalt in the mixing process which releases the chemically bound water. Aspha-Min® is a type of water-bearing agent

which is a sodium aluminum silicate, hydro-thermally crystallized into a fine powder. Heated aggregate and asphalt turns the released water into a finely dispersed steam which helps to improve compaction properties like workability and compactibility of the mixture at lower temperatures by the reduction of about 20 to 30°C [69].

In the third method, special additives are used to reduce the viscosity of the asphalt. They are usually paraffins with long chain hydrocarbons which are not supposed to change the asphalt properties. These paraffins are generally soluble in the asphalt above temperatures of 80 to 120°C to reduce the viscosity of asphalt. Sasobit® is a type of paraffin which has a long chain aliphatic hydrocarbon with chain lengths of 40 to 115 carbon atoms, melts in the asphalt binder at temperatures of 85 to 115°C to reduce the mixing and handling temperatures by 30 to 50°C [70]. They are manufactured from coal gasification by using the Fischer-Tropsch process.

In this study, asphalt cements are modified with warm mix additives, asphaltene dispersants, a paraffin inhibitor, RAP additives and rejuvenators. Recycled Asphalt Pavement (RAP) can be added 50 to 60% to the asphalt after drying and heating to remove the moisture. If RAP is severely oxidized, the rejuvenating agent is added to the mixture [6]. In 1960, asphalt rejuvenator Reclamite® came on the market. Rejuvenators were produced as emulsions generally 60-65% residual. They are typically cationic emulsions which contain maltenes and saturates (light fractions). The function of a rejuvenator is to soften the oxidized asphalt pavement surface. It also helps to flux the asphalt and extend the life of the pavement surface by adjusting properties of the asphalt mixture [72]. The figure 2.2 shows the various products used in warm mix technologies [73].

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WMA processes	Product	Company	Description	Dosage of additive	Country where technology is used	Production temperature °C (or reduction range)
Foaming processes						
Water-containing	Aspha-Min®	Eurovia and MHI	Water-containing technology using zeolites	0.3% by total weight of the mix	USA, Germany, France, worldwide	(20–30 °C)
Water-containing	Advera®	PQ Corporation	Water-containing technology using zeolites	0.25% by total weight of the mix	USA	(10–30 °C)
Water-based	Double Barrel Green	Astec	Water-based foaming process	2% water by mass of bitumen; anti-stripping agent	USA	116–135 °C
Water based	Ultrafoam GX	Gencor industries	Water-based foaming process	1–2% water by mass of bitumen	USA	Not specified
Water-based	LT Asphalt	Nynas	Foam bitumen with hydrophilic additive	0.5–1 % by mass of bitumen	Netherlands and Italy	90 °C
Water-based	WAM-Foam	Shell and Kolo-Veidekke	Soft binder coating followed by foamed hard binder	2–5 % water by mass of hard binder	Worldwide	100–120 °C
Water based	Low Energy Asphalt	LEACO	Hot coarse aggregate mixed with wet sand	3% water with fine sand	USA, France, Spain, Italy	<100 °C
Water based	Low Emission Asphalt	McConnaugh–ay Technologies	Hot coarse aggregate mixed with wet sand, combined with chemicals	3% water with fine sand; 0.4% bitumen weight	USA	90 °C
Water based	LEAB	Royal Bam Group	Direct foam with binder additive. Mixing of aggregates below water boiling point	0.1% of bitumen weight of coating and adhesion additive	Netherlands	90 °C
Organic						
FT Wax	Sasobit	Sasol	Fischer-Tropsch wax	Approx. 2.5% by weight of binder in Germany; , 1.0–1.5%, in the U.S.A.	Germany as well as 20 other countries	(20–30 °C)
Montan Wax	Asphaltan B	Romonta GmbH	Refined Montan wax with fatty acid amide for rolled asphalt	2.0–4.0% by mass of bitumen	Germany	(20–30 °C)
Fatty Acid Amide wax	Licomont BS	Clariant	Fatty acid amide	3.0% by mass of bitumen	Germany	(20–30 °C)
	3E LT or Ecoflex	Colas	Proprietary	Yes, but not specified	France	(30–40 °C)
Chemical						
Chemical	Evoterm Technologies	Mead Westvaco	Chemical packages, with or without water	0.5% of mass of bitumen emulsion. Emulsion contains 70% of bitumen	USA, France, Worldwide	85–115 °C
Chemical	Cecabase RT	CECA	Chemical package	0.2–0.4% by mixture weight	USA, France	(30 °C)
Chemical	Rediset	Akzo Nobel	Cationic surfactants and organic additive	1.5–2% of bitumen weight	USA, Norway	(30 °C)
Chemical	Revix	Mathy-Ergon	Surface-active agents, waxes, processing aids, polymers	Not specified	USA	(15–25 °C)
Chemical	Iterlow T	IterChimica		0.3–0.5% by mass of bitumen	Italy	120 °C

Figure 2.2: Processes and products used in warm mix technologies [73].

Asphaltene dispersants are generally organic molecules similar to resins. They are used to modify the viscosity of the asphalt to ease processing. Paraffin inhibitors are used to prevent further buildup of the viscous paraffin wax from agglomerating in the asphalt.

2.3 Conventional Testing Methods

The main aim of asphalt cement testing is to determine some kind of property of the material and to correlate this with field performance. Since multiple types of asphalts are produced, so it is important to classify the different grades. Here specification plays the major role to set the standards and testing methods to address these issues. Generally, testing helps to address two main things like quality control to meet the specification requirements and research and development to know more fundamental rheological and mechanical properties and to develop new grades and binders.

Conventional testing is generally used to classify asphalt grades. Penetration grade, oxidized grade, hard grade, and cutback grade are based on conventional testing methods as per their specifications. Basically these tests are to know about the response of asphalt when it is subjected to stress. This response depends on the testing conditions like temperature and loading time, etc. The generally used conventional tests are the penetration test at 25°C, softening point test, and viscosity tests, like dynamic viscosity test at 60°C, kinematic viscosity test at 135°C, Fraass breaking point test, specific gravity test, storage stability test, ductility test, force ductility test, elastic recovery test, and aging like thin film oven test (TFOT), rolling thin film oven test (RTFO), and PAV. From a quality control perspective, the repeatability and reproducibility of the results are really important.

2.3.1 Penetration Test

Penetration and softening point tests are very important and are the oldest tests to validate asphalt and for their classifications too [6]. Generally there are many standards like American

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Society for Testing and Material (ASTM), European standards (CEN), Indian Standards (IS), which explain the procedures to do testing.

As per the ASTM method [22], the penetration test is the measurement of the distance moved by the specified dimension needle into the asphalt under the known load (100 g) at constant temperature (25°C) for a specified time (5s). Usually three individual penetration measurement readings are recorded for each sample and their difference should not exceed the specified limit. The average of three measurements are reported to the nearest whole number. The lower value of penetration measurements represents the harder asphalt and vice versa. Since this test is empirical, it has to be carried out exactly the same way and same testing conditions at every time. The penetration-temperature relationship [23] is given by the following equation 1:

$$\log P = AT + K \tag{1}$$

where P is the penetration at temperature T, K is a constant, and A is the temperature susceptibility. The value of A defines the Penetration Index (PI), which is used to evaluate stiffness of the binder at any temperature:

$$A = PI = \frac{\log (Pen @ T1) - \log (Pen @ T2)}{T1 - T2} \tag{2}$$

where, T1 and T2 were two different temperatures at which penetration tests are done.

The penetration test set up is shown in the Figure 2.3. PI values are used to predict the stiffness of the asphalt and thus the resistance to deformation.
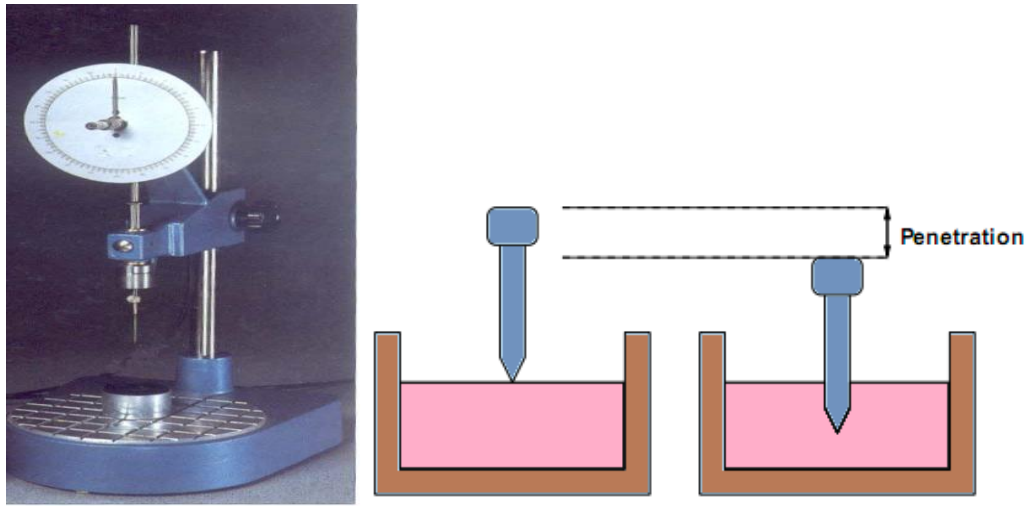
Figure 2.3: Penetration test [24].

2.3.2 Ring and Ball Softening Point Test

The Ring and Ball (R&B) test is also called Softening Point test. The softening point test set up is shown in Figure 2.4. As per ASTM D36-95 method [25], asphalt is poured in two shouldered brass rings and allowed to solidify. Then steel balls (3.5 g) are placed centrally on the top of asphalt which was poured in the rings. Then it is heated at a constant rate (5°C per minute) in a liquid bath inside the beaker. Water is used as medium at the temperature range of 30 to 80°C, glycerin is used as the heating medium at the temperature range of 80 to 157°C, and ethylene glycol is used as medium at the temperature range of 30 to 110°C. The mean of the temperatures at which the two rings soften and alow each ball to pass through asphalt, to fall a distance of 25 mm is noted as softening point. The difference between the each ball temperature readings should not exceed the specified value. The stirrer is employed depending on the test standards. This method is used to classify the asphalts based on the tendency of the material to flow at elevated temperature encountered in service.
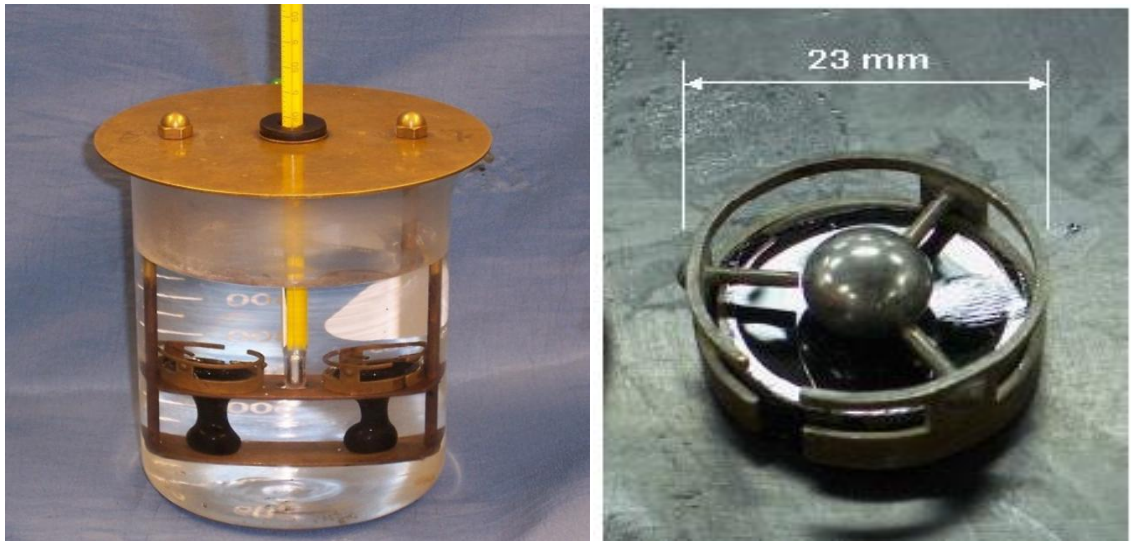
Figure 2.4: Softening point test set up [26].

2.3.3 Viscosity Test

Viscosity is a fundamental property and a measure of a fluid's resistance to flow. It describes the internal friction of a moving fluid. Asphalts are graded according to their viscosity like VG-10, VG-20, VG-30, and VG-40, by viscosity measurements particularly in eastern countries like India based on the specifications of Indian Standards IS-73-1992 (viscosity at 60°C), along with other qualification tests like specific gravity, water content, ductility, loss on heating and Fraass breaking point, etc. Asphalt absolute viscosity is typically measured at 60°C, which is related to the maximum pavement surface temperature during summer. The kinematic viscosity of a liquid at 135°C is related to mixing and lay down temperatures of the pavement and it is the ratio of the absolute (or dynamic) viscosity and the density of the liquid at the temperature of measurement [27]. Asphalt viscosities are usually measured by Brookfield viscometers and capillary viscometers based on the specification requirements [28] as shown in Figure 2.5. Temperature-viscosity graphs are used to estimate mixing and

compaction temperatures for the asphalt mix design. The Bitumen Test Data Chart (BTDC) is also used to predict mixing and compaction temperatures based on the correlation between penetration, softening point, Fraass breaking point, and Brookfield viscosity test results.

Figure 2.5: Brookfield viscometer [29] and Capillary viscometer [30].

2.3.4 Ductility Test

Ductility testing determines the cohesive strength of asphalt cement at low temperatures. As per ASTM D113-07 method [31], samples are prepared in dumb-bell shape by pouring in molds and allowing solidification. Then, samples are conditioned at test temperatures like 10°C, 15°C and 25°C, etc., which depends on the hardness of the asphalt which was measured by the penetration test. Then samples are stretched at a constant speed like 50 mm per minute up to the failure point when the sample breaks inside the water bath at constant temperature. The failure distance is noted as the ductility as shown in Figure 2.6. The test is stopped at a point or thread until rupture occurs at the point where the thread has practically

no cross-sectional area. Average of three normal tests of the sample is reported as the ductility.
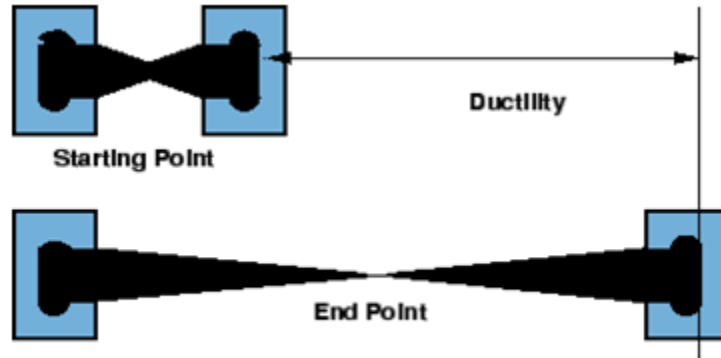
Figure 2.6: Ductility Test [32].

2.3.5 Force - Ductility Test

Force - ductility is the European version of a conventional ductility test. We measure the distance in the normal ductility test. At the end of test after stretching, it does not help to differentiate the cohesive strength of the thin thread of conventional asphalt with thick thread of polymer-modified bitumen (PMB). Force-ductility test is used to measure the force required to stretch the asphalt. Then the graph is plotted as force versus strain. The cohesive energy is calculated as the area under the force-distance curve [33].

2.3.6 Asphalt Cement Durability and Aging Indices

Durability is the determination of change of asphalt physical properties due to aging which is called age hardening. The factors that cause age hardening are oxidation, volatilization, polymerisation, thixotropy, syneresis, separation, short term and long term aging [10]. It is difficult to measure durability but it can be done by simulated aging and measure the properties before and after aging with standard physical tests like viscosity, DSR, BBR and

Direct Tension Test (DTT). Asphalt has the tendency to undergo the hardening by the influence of atmosphere. The main factors for aging are,

- Oxidation or oxidative hardening or age hardening

- Loss of volatiles

- Steric or physical hardening

- Exudative hardening, etc [21].

Organic molecules in the asphalt react with environmental oxygen. This oxidation causes structural and compositional changes for the asphalt molecules called oxidative or age hardening. This hardening happens at slow rate in pavement and causes cracking. It also happens at the time of asphalt mixing with aggregates at elevated temperatures. Volatilization happens during hot mixing and pavement construction. Physical hardening occurs at less than $0°C$ for long period of time which causes asphalt to shrink and harden. Transfer of oily components from asphalt to aggregates causes exudative hardening which depends on the porosity of aggregates and nature of asphalt [34]. Hesp et al. stated wax content in the asphalt causing physical hardening by the formation of asphaltene clusters[58].

Aging is determined by measuring the change in physical properties like stiffness, viscosity and penetration with time. Aging index is the ratio of two values like stiffness, viscosity or penetration measured at different times and it is not a fundamental parameter. For example, aging index based on viscosity (η_a/η_o) is the ratio of viscosity of aged asphalt, η_a, to the viscosity of original asphalt, η_o. Figure 2.7 shows the aging index graph.

Aging tests like TFOT, RTFO and PAV are used to identify the volatility and susceptibility to oxidation. TFOT is the short term aging. TFOT oven is designed to heat the

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thin layer of samples in the cylindrical pan at 163°C for 5 hours. After aging, mass change is calculated and physical properties change measured [35]. RTFO and PAV are described in the Superpave^{TM} testing standards.
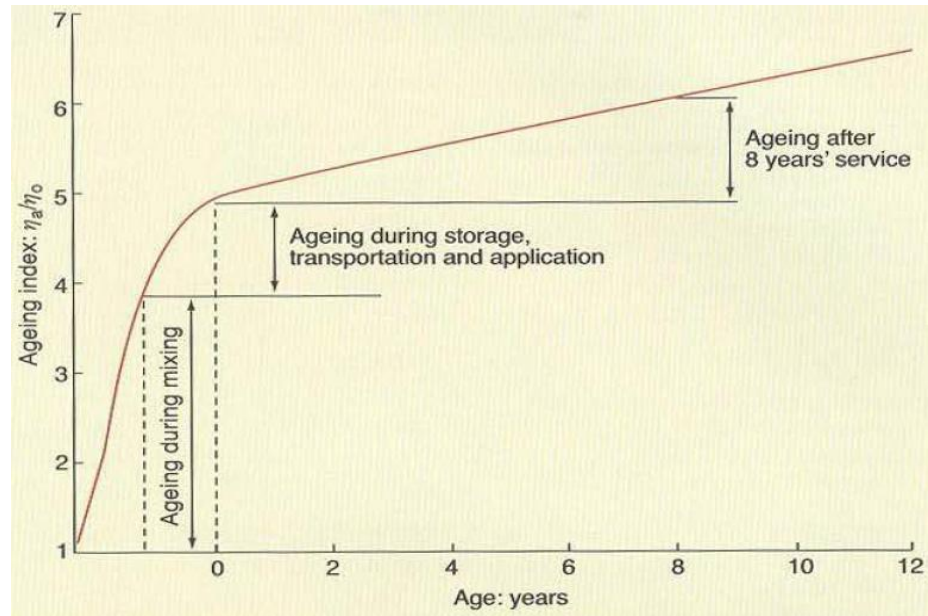
Figure 2.7: Aging of asphalt during mixing, storage, transportation, application and in service [6].

2.4 Superpave^{TM} Testing

Asphalt grading is mainly designed to know the high and low temperature conditions at which it performs without failure or damage. The high temperature grade is related to rheological properties at the average highest 7-day surface temperature for an average summer in the contract location. The high temperature performance also depends on the aggregate properties like size, gradation, content, etc. The low temperature grade is related to the one-in-fifty-year lowest pavement surface temperature during winter. The rheological properties of DSR are related to rutting while BBR properties are related to cracking.

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Long Term Pavement Performance Binder selection software (LTTPBind®) is used to find these surface temperatures from a weather database. The surface temperatures are calculated using a calibrated equation which relates the air to surface temperatures based on the latitude of the geographical location. Generally, a 98% confidence limit is recommended to prevent thermal cracks, deformations and damage [5].

Conventional tests are generally empirical so they are not effective at predicting the performance of asphalt in the field. In 1987, the Strategic Highway Research Program (SHRP) undertaken in the USA developed the Superpave^{TM} testing methods. These methods were developed to allow for the construction of SUperior PERforming PAVEments. This method was supposed to help the grades classification based on their performance with simulated aging and physical properties determinations. Table 2.3 shows the equipment used in Superpave^{TM} testing and their purposes.

Table 2.3: Superpave^{TM} Binder Test Equipments [36].

S.No	Equipment	Purpose
1	Rolling Thin Film Oven (RTFO)	Simulate binder aging (hardening) characteristics.
2	Pressure Aging Vessel (PAV)	
3	Dynamic Shear Rheometer (DSR)	Measure binder stiffness and elasticity properties at high and intermediate temperatures (G*, δ).
4	Rotational Viscometer (RV)	Measure binder viscosity at high temperatures.
5	Bending Beam Rheometer (BBR)	Measure low temperature stiffness and failure properties.
6	Direct Tension Test (DTT)	

Aging tests like TFOT, RTFO and PAV are used to identify the volatility and susceptibility of asphalt to oxidation. Superpave testing involved aging of RTFO and PAV followed by a

measure of the properties in the DSR and BBR. In RTFO aging (short term aging), thin films of asphalt are exposed to heat and air flow. The oven is preheated at an aging temperature of 163°C. Samples of 35 g are poured into each RTFO cylindrical bottle. The bottles are placed in the carriage which rotates at a rate of 15 rpm for 85 min under an air flow of 4000 ml/min as shown in Figure 2.8. After aging, the mass change is calculated and the physical properties changes are measured [37].

Rolling Thin Film Oven

Figure 2.8: Rolling Thin Film Oven (RTFO) [38].

Long-term asphalt binder aging is used to simulate the effects 8 to 10 years pavement service by using the pressure aging vessel (PAV) as shown in Figure 2.9. It was adopted by SHRP researchers [57]. The vessel is designed to heat samples of 50 g, which are kept in a pan at temperatures of 90, 100, or 110 °C, as shown in the table, under a pressure of 2,070 kPa for 20 hours. The temperatures are fixed based on the climate as shown in Table 2.4.

Table 2.4: PAV Test Temperatures [56].

Temperature	Simulation
194°F (90°C)	cold climate
212°F (100°C)	moderate climate
230°F (110°C)	hot climate

After aging, samples are degassed in a 170°C vacuum oven for 30 min under 15 kPa pressure. Then the physical properties are measured [39]. After a month to five years of service, many pavements are damaged or cracked, even though the materials passed through the above mentioned standard test methods [49]. So, the long-term aging process should be improved to become more efficient and precise to avoid pavement failures especially for cold weather.

Figure 2.9: Pressure Aging Vessel (PAV) and Vacuum Degassing Oven [40].

2.3.1 Dynamic Shear Rheometer (DSR) Method

Based on the Superpave^{TM} test methods, asphalt grades are specified as PG (or PGAC) XX-YY, where XX represents the high temperature working limit by Dynamic Shear Rheometer (DSR) test and -YY the low temperature limit by Bending Beam Rheometer (BBR) test. DSR also known as dynamic rheometer or oscillatory shear rheometer or parallel plate rheometer is shown in Figure 2.10. It determines the rheological properties like complex shear modulus, G* (elastic component), and phase angle, δ (viscous component), of fresh, RTFO, and PAV-aged asphalt samples, for intermediate and high temperature performance grading [41].
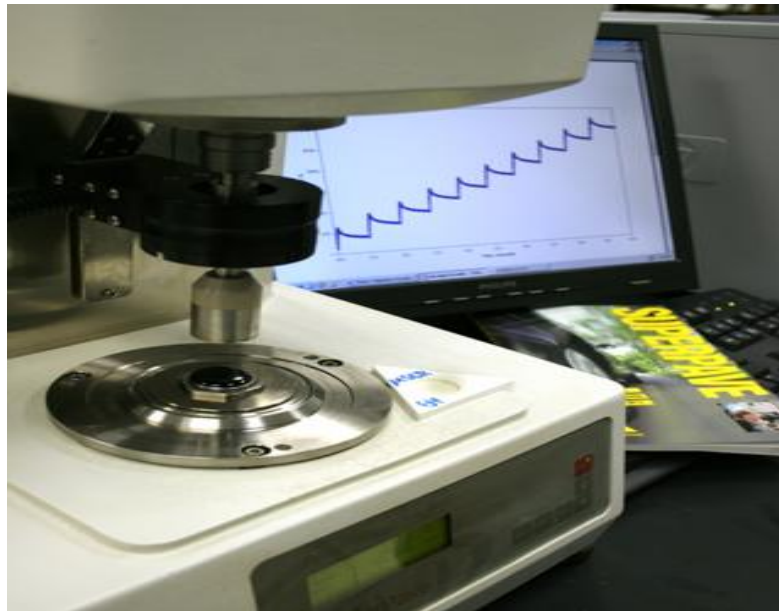

Figure 2.10: Dynamic Shear Rheometer (DSR) [43].

DSR is used to measure the torque and angle of rotation to calculate the shear stress and shear strain as follows [42]:

The oscillatory strain, γ,

$$\gamma = \gamma_o \sin wt \tag{3}$$

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where, γ_o is the peak shear strain and w is the angular velocity in radian/second

The shear stress, τ,

$$\tau = \tau_o \sin (wt + \delta) \tag{4}$$

where, τ_o is the peak shear stress and δ is the phase shift angle.

Then, Complex Shear Modulus can be determined as $G^* = \tau_o / \gamma_o$ (5).

Complex shear modulus, G* determines the resistance of material to the deformation under applied shear stress. The phase angle, δ is related to the time lag between the applied stress and the resulting strain, and it can be expressed as the ratio of viscous or loss modulus , G", to the storage or elastic modulus, G'.

The fresh and RTFO samples are used to find the high temperature grade that is supposed to control rutting. A 25 mm diameter plate geometry and a 1 mm gap is maintained to run the test. Sample is poured in a silicon mold and solidified before loading to the bottom plate. Trimming tool is used to trim the excess sample. Then testing parameters like frequency, which is speed of oscillation (one cycle), generally 10 radians/second, loading time, and test temperature, etc., are given to the software to run the testing. Strain values should be small and remain in the linear viscoelastic range. The rheometer produces the results automatically by using the software. As per the AASHTO specification, the high temperature PG grades decided on the following values, G*/sin$\delta \geq$ 1.00 kPa for fresh and G*/sin$\delta \geq$ 2.20 kPa for RTFO.

The PAV samples are used to find the intermediate temperature grade for the control of fatigue. An 8 mm diameter plate geometry and 2 mm gap is maintained to run the fatigue

grading test. Sample is poured in the silicon mold and solidified before loading to the bottom plate. Trimming tool is used to trim the excess sample. Then testing parameters like frequency which is speed of oscillation (one cycle), generally 10 radians/second, loading time and test temperature, etc., are given to the software to run the testing. Strain values should be small and remain in the linear viscoelastic range. The rheometer produces the results automatically by using the software. As per the AASHTO specification, the intermediate temperature PG grades decided on G*sin δ less than 5000 kPa for PAV [41]. Finally, DSR measurements at high temperatures are related to rutting and at intermediate temperatures are related thermal cracking.

2.3.2 Bending Beam Rheometer (BBR) Method

The BBR measures the binder stiffness by the deflection or creep at constant load and temperature, that stiffness is used to predict the low temperature cracking as shown in Figure 2.11. The PAV-aged asphalt binders are heated and poured it into the mold. After cooling for 45 to 60 minutes, trimming has to be done before conditioning in an ethanol bath. After 1 hour of thermal conditioning, the asphalt beam is placed on the supports to apply a three point load. After the application of preload of 35 mN, a seating load of 980 mN is applied for 1 second, and allowed for a 20 second recovery period. The graph of load and deflection versus time is plotted continuously. The rheometer software produces the results automatically. The creep stiffness (S), which is the binder resistance to creep loading, and creep rate (m), which is the asphalt stiffness change with time during the application of load, are measured by using the BBR instruments.

The deflection of the beam is recorded when load is applied during this period, and creep stiffness of the asphalt can then be calculated by the following equation:

$$S(t) = PL^3 / 4bh3\delta(t) \qquad (6)$$

where: $S(t)$ = creep stiffness at time, t

P = applied load, 100 g

L = distance between beam supports, 102 mm

b = beam width, 12.5 mm

h = beam height, 6.25 mm

$\delta(t)$ = deflection at time, t

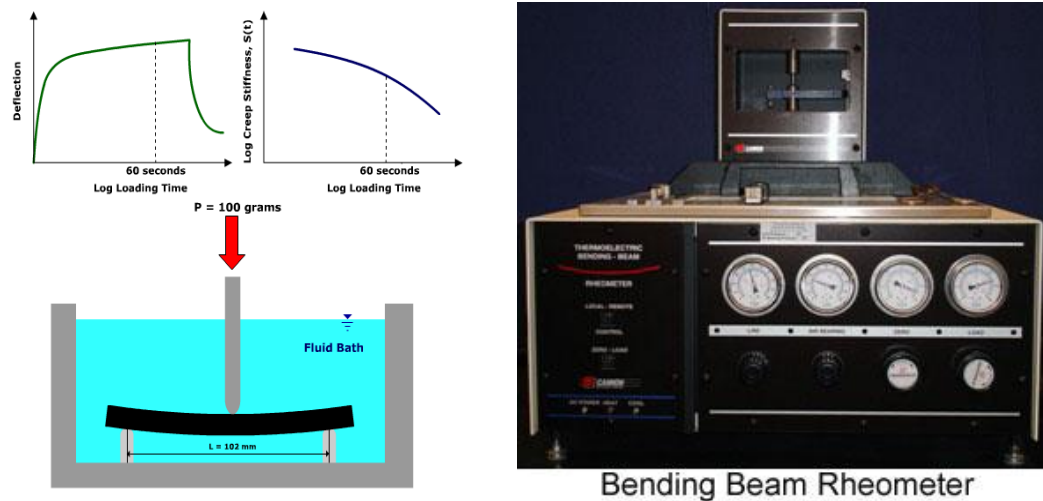**Figure 2.11: Bending Beam Rheometer (BBR) [32].**

As per the Superpave binder specification, the bending beam rheometer test is to be conducted at 10°C above the expected minimum pavement temperature, T_{min}, which is approximately equal to its stiffness at T_{min} after 2 hours loading time, which is related to low temperature cracking potential. The Superpave binder specification requires the stiffness at

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the test temperature after 60 seconds to be less than 300 MPa, to control low-temperature cracking. The slope of the log stiffness versus log time curve gives the m-value at a specified time. As per the AASHTO specification, the low temperature PG grades are decided based on the limiting temperature where stiffness < 300 MPa or m-value > 0.300 for the PAV residue [44]. A higher m-value indicates that asphalt creep at a faster rate to reduce the thermal stress and in turn desirable to reduce low-temperature cracking [42]. Absence of sufficient ageing and high strain testing in existing methods created the need for improved test methods [5].

2.4 Improved Ministry of Transportation of Ontario (MTO) Test Methods

It is important to consider physical aging [11] and not only properties such as creep and stress relaxation as per AASHTO M320 (American Association of State Highway Officials) specification, which only conditions the samples for one hour prior to testing. The Superpave specification follows insufficient aging and conditioning time and low strain testing methods to predict asphalt performance. The collaboration work of the Ontario Ministry of Transportation with Queen's University has developed the following set of new test methods:

      1. Extended Bending Beam Rheometer (eBBR) test (LS-308);

      2. Double Edge Notched Tension (DENT) test (LS-299); and

      3. Modified Pressure Aging Vessel protocols (LS-228).

      These methods have shown to be more effective in terms of asphalt performance prediction than Superpave methods. Poor quality asphalts are penalized more during aging for longer times, or in thinner films, and/or in the presence of moisture than good asphalts [15]. So, these new test methods make it easier to select quality asphalts rather to guess as is currently done with the Superpave specification approach.

2.4.1 Extended Bending Beam Rheometer (eBBR) Method LS-308

Low-temperature physical hardening of asphalt cement is different at different temperatures and can increase over time. Unlike the one hour conditioning in the regular BBR test, one day of conditioning and also three days of conditioning in the extended BBR can cause severe grade losses in the asphalt especially in poor quality asphalt cement which contains large quantities of wax and unstable asphaltene dispersions. In the extended BBR the conditioning temperatures T_1 and T_2 are fixed as follows:

$T_1 = T_{design} + 10$ and

$T_2 = T_{design} + 20$.

LS-308 conditions at +10 °C and +20 °C above the actual grade temperature (i.e. pavement design temperature) for periods of 1 h, 24 h, and 72 h to simulate the effect of extended exposure to two different cold temperatures [12]. Exact grade of pass and fail temperatures are determined according to AASHTO M320 criteria by interpolation which involves plotting the grade on a semi-logarithmic scale [54]. This method helps to get a high degree of confidence to prevent thermal cracking [5].

2.4.2 Double-Edge Notched Tension (DENT) Test LS-299

The test is conducted after thermal conditioning to determine the essential work of failure, the plastic work of failure, and an approximate critical crack tip opening displacement (CTOD), at a specified temperature and rate of loading [13]. Samples are prepared in double-edge-notched shape with ligaments, i.e., distances between two opposing notches of 5 mm, 10 mm, and 15 mm in length, by pouring in molds and allowed to solidify. Conditioning was done at room

temperature and in a water bath for 24 hours at the test temperature like 15 or 25ºC before testing. Then samples are pulled until they fails in a water bath as shown in Figure 2.12.
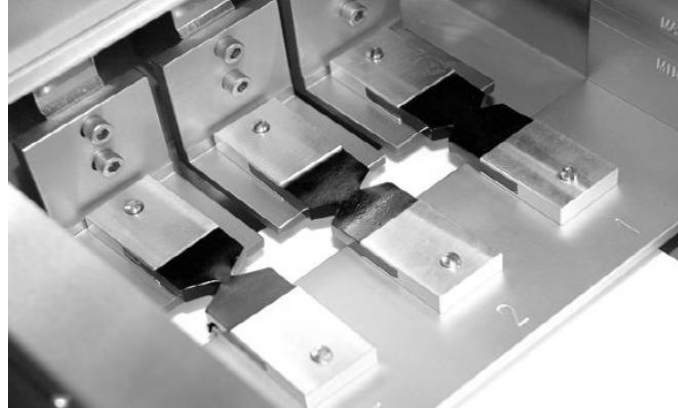
Figure 2.12: DENT test setup [55].

The DENT test is based on the Essential Work of Fracture (EWF) method, which is a thermodynamic analysis and was initially proposed by Cotterell and Reddel [45], further studies were carried out by Mai et al. [46]. In this method, the assumption is made that the total work of failure (W_t) is the sum of an essential work of failure (W_e) and a non-essential work of failure (W_p).

W_t can be determined from the total area under the force-displacement curve:

$$W_t = W_e + W_p \tag{7}$$

Essential work of failure (W_e) and non-essential work of failure (W_p) can be determined by the following equations:

$$W_e = w_e \text{ x } LB \tag{8}$$

$$W_p = w_p \text{ x } \beta L^2 B \tag{9}$$

Where,

w_e = specific essential work of fracture (J/ m^2),

w_p = specific plastic work of fracture (J/ m^2),

L = the ligament length in the DENT specimen (m),

B = the thickness of the sample (m) and

β = the shape factor of the plastic zone, which is geometry dependent.

If we substitute equations 8 and 9 in 7,

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

By dividing equation (10) by the cross-sectional area of the plastic zone (LB),

$$\frac{W_t}{LB} = w_t = w_e + w_p \beta LB \tag{11}$$

where, w_t = specific total work of fracture (J/ m^2).

Specific total work of failure, w_t, versus the ligament length, L in equation (11), gives a straight line with slope and intercept on the w_t axis. The w_e helps to predict fatigue cracking resistance and to determine the CTOD [47]:

$$\delta_t = \frac{w_e}{\sigma_n} \tag{12}$$

where, δ_t = the crack tip opening displacement parameter (m), and

σ_n = the net section stress or yield stress (N/m^2), determined from the 5 mm ligament length of the DENT mould.

High correlations exist between the CTOD and the fatigue properties of the asphalt cement. CTOD can be used to rank the performance and determine a high correlation with cracking distress.

2.4.3 Modified Pressure Aging Vessel Method LS-228

This method is used to accelerate the aging of PAV, because the existing PAV does not help efficiently to predict fatigue and thermal cracking [48-52].

It involves three methods, namely A, B and C. In method A, there is no testing modifications with AASHTO R28. In Method B, the film thickness is reduced to approximately 0.8 mm by reducing the weight of 50 +/- 0.5 grams to 12.5 +/- 0.5 grams. The presence of moisture is achieved by loading one empty TFOT stainless steel pan with 50 grams of distilled water. In Method C, aging time increased from 20 hrs to 40 hrs with the presence of moisture by loading one empty TFOT stainless steel pan with 50 grams of distilled water. The standard film thickness is maintained [14]. Once aging is done, the physical property changes will be measured.

Poor quality asphalts are penalized during aging for longer times, or in thinner films, and/or in the presence of moisture compared to good asphalts [15]. So, this new test method make life easier to find good quality asphalts.

Chapter 3

MATERIALS AND EXPERIMENTAL PROCEDURES

3.1.1 Materials

The asphalt cement materials used in this study were distilled from various commercial crude sources like Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster by major oil refiners. They were obtained from refineries in Alberta, New Jersey, and Texas, respectively, as shown in Table 3.1.

Table 3.1: Asphalt Sources

No.	Asphalt Cement	Source
1	Boscan	NuStar, New Jersey
2	Cold Lake	Imperial Oil, Alberta
3	Lloydminster	Husky, Alberta
4	Western Texas	Alon, Texas

Boscan asphalt has a higher asphaltene content than others. So, it is expected to have a better PG temperature span to increase in stiffness but also more chances of hardening to occur. Western Texas Intermediate has a higher wax content than others. So, it is expected to undergo more physical hardening. Change in source and composition result in changes in physical properties like viscosity, penetration, softening point, ductility, aging, and rheological properties [59]. The performance grading (PG) of asphalt is shown in Table 3.2. As per the AASHTO specification, the high temperature PG grades are decided based on the following values, $G^*/\sin\delta \geq 1.00$ kPa for fresh and $G^*/\sin\delta \geq 2.20$ kPa for RTFO residue, the intermediate temperature PG grades is decided on $G^* \sin\delta$ less than 5000 kPa for PAV

residue, and the low temperature PG grade is decided on stiffness < 300 MPa and m-value > 0.300 for PAV residue [44].

Table 3.2: Superpave Grades of Asphalt Cements

Asphalt Cement	Superpave Grades and Grade Span, °C			
	Low	Intermediate	High	Span
Boscan	-27	18	70	97
Cold Lake	-28	21	67	95
Lloydminster	-28	19	66	94
Western Texas	-24	28	66	90

The asphalts were blended in small quantities with different modifiers as per Table 3.3. Mixing is done by using a standard laboratory mixer. Mixing is done at temperatures between 150°C and 160°C. The amount of additive used for each formulation varied between 0.75 and 1.5 percent by weight. The additive level was kept at the maximum which was specified by the respective suppliers. Blending or mixing is done as per the instructions provided by the suppliers of the materials.

3.1.2 Modifiers

Asphalt properties change with crude or the refinery processes. Usually all the crudes are not so good, and also lot of difficulties in refining process to achieve the required grade or property of asphalt [60]. So, asphalt producers decided to introduce the additives to modify the properties. Air blowing process is applied to harden the asphalt. Fluxing agents or diluents oils are added to make the asphalt softer. Polyphosphoric acid (PPAs) is used to increase the pavement service temperature range with required performances. Polymers are added to improve the quality of asphalt in various ways by helping to ease the paving and

providing good mechanical and cracking resistance [62, 63]. Polymer modified asphalt (PMA) mainly used in places like airports, intersections of busy streets, vehicle weighing stations, and parking lots with high stress [64]. And other additives like mineral acids, bases, and fillers, etc., are also added to asphalt depending upon their requirements.

Asphalt is modified to meet the special requirements of pavements like ease of road constructions, reduce the maintenance frequency, improve the useful temperature interval in service, phase stability, reduce the cost, durability by reduced hardening and also fit in the specifications like Superpave. They are also expected to improve binder performance by providing good cracking resistance, moisture resistance, and good adhesion and cohesion within the pavements layers [16].

Asphalt pavement contractors prefer to lay the paving with less energy consumption and easier way of compaction particularly in the colder season or northern regions like Canada. This issue motivated asphalt manufactures to develop warm mix asphalt technology, which involves the addition of additives to the asphalts. The warm mix additives, dispersants, adhesion promoters and other additives which are commercial and developmental products, were added with the above mentioned asphalts as shown in Table 3.3. They were obtained from Akzo Nobel, Nalco, and King Industries. The additives listed in Table 3.3 were blended with asphalt cements listed in Table 3.1 in small quantities using a standard laboratory mixer at temperatures between 150°C and 160°C. The amount of additive varied between 0.75 and 1.5 percent by weight, which was kept maximum as specified by the respective suppliers for each formulation and labeled as Dispersant 1 or D1 etc. The blending instructions and conditions were followed as per the instructions of material suppliers.

Table 3.3: Additives and Compositions

S.No	Description	Label	Conc. (%)
1	Rheology Modifier / Surfactant	A or Dispersant 1 (D1)	1.5
2	Amine	B or Dispersant 2 (D2)	0.75
3	Polymer	C or Dispersant 3 (D3)	1.5
4	Amine	D or Dispersant 4 (D4)	0.75
5	Complex Mixture	E or Dispersant 5 (D5)	1.5
6	Amide Wax	F or Dispersant 6 (D6)	1
7	Polyethylene Wax	G or Dispersant 7 (D7)	1

3.2 Experimental Procedures

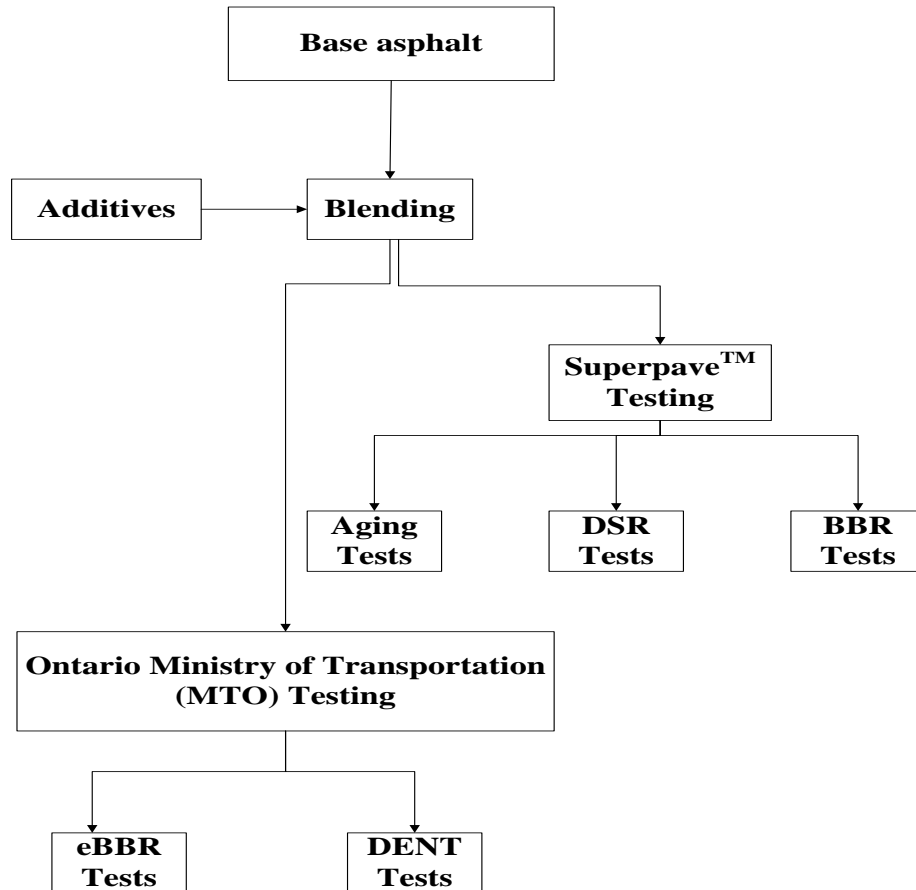The following Figure 3.1 shows the experimental plan for this study.

Figure 3.1: Experimental plan for this study.

3.2.1 Regular DSR and BBR Testing According to AASHTO M320

DSR measurements are related to rutting and thermal cracking at high and low temperatures, respectively. Based on the Superpave test methods, asphalt grades are specified as PG (or PGAC) XX-YY, where XX represents the high temperature working limit by Dynamic Shear Rheometer (DSR) test and -YY the low temperature limit by Bending Beam Rheometer (BBR) test.

Complex shear modulus, G^* determines the resistance of material to the deformation under applied shear stress. The phase angle, δ, is related to the time lag between the applied stress and the resulting strain and it can be expressed as the ratio of viscous or loss modulus, G'', to the storage or elastic modulus, G'. The following Table 3.4 shows the typical DSR test parameters

Table 3.4: DSR Test Parameters [55].

S.No	Test Parameter	Value
1	Total test time	2 hours
2	Soak period at each temperature	10 min
3	Measurement period	10 sec
4	Temperature range	-10 to 82 °C
5	Oscillation frequency	0.1 rad/s
6	Shear strain	0.10 %

The sample is heated in oven until sufficiently fluid at a temperature of 163°C for conventional asphalt to obtain a pouring consistency. It is poured into the silicone mold which is carved to different diameters, to obtain pellets of asphalt as shown in Figure 3.2. Once the instrument and computer are switched on, the proper geometry is selected as shown in Table 3.5. The zero gap setting is done by moving the spindle in the moveable plate

downwards until it touches the fixed plate, at that position the system detects and makes as zero when the plates are just touching together.

Table 3.5: DSR Test Geometry.

Asphalt Condition	Spindle Geometry	Measuring Gap
Neat	25 mm	1 mm
RTFO aged	25 mm	1 mm
PAV aged	8 mm	2 mm

Trimming temperature is set in the system, the sample is placed on the bottom plate. Then the excess sample is trimmed at trimming position with movement of top plate by a hot spatula. Then plates are reached to the measuring gap position which is mentioned in the method. Then testing is done at the required strain, frequency and measuring points details in the testing window as per the method.
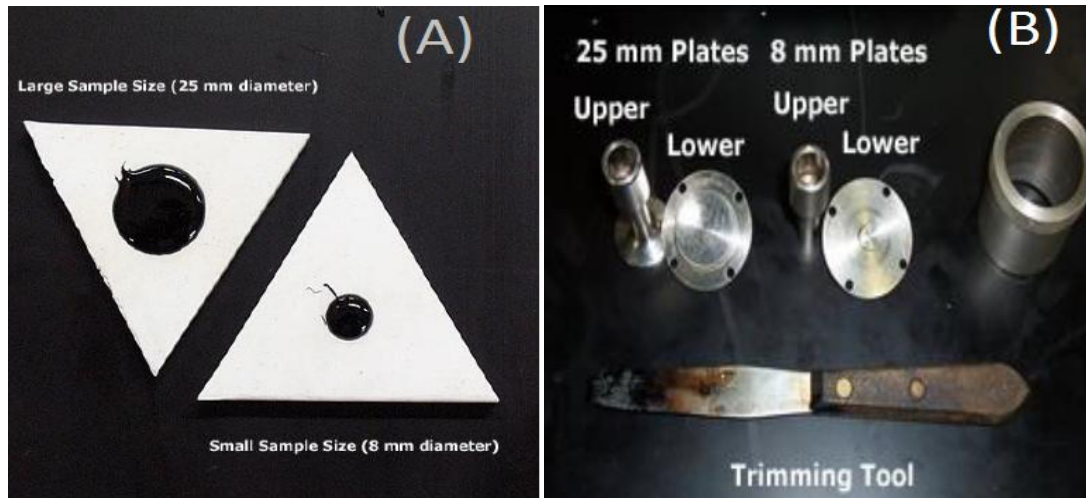

Figure 3.2: DSR sample (A) and Spindles (B) [61].

The fresh and RTFO samples are used to find the high temperature grade (XX) for determining the rutting. The 25 mm diameter spindle geometry and 1 mm gap is maintained to run the test. Sample is poured in the silicon mold and solidified before loading to the bottom plate. Trimming tool is used to trim the excess sample. Then testing parameters like frequency, which is speed of oscillation (one cycle), generally 10 radians/second, loading time, and test temperature, etc., are given to the software to run the testing as shown in Table 3.4. Strain values should be small and remain in the linear viscoelastic range. The rheometer produces the results automatically by using the software. As per the AASHTO M320 specification, the high temperature PG grades are decided based on the following values, $G^*/\sin \delta \geq 1.00$ kPa for fresh and $G^*/\sin \delta \geq 2.20$ kPa for RTFO.

The PAV samples are used to find the intermediate temperature grade for determining the fatigue. The 8 mm diameter spindle geometry and 2 mm gap is maintained to run the test. Sample is poured in the silicon mold and solidified before loading to the bottom plate. Trimming tool is used to trim the excess sample. Then testing parameters like frequency which is speed of oscillation (one cycle), generally 10 radians/second, loading time and test temperature, etc., are given to the software to run the testing. Strain values should be small and remain in the linear viscoelastic range. The rheometer produces the results automatically by using the software as per the AASHTO specification, the Intermediate temperature PG grades decided on $G^*.\sin \delta$ less than 5000 kPa for PAV [41].

Template is used to find the exact temperature. The test frequency and temperature, the values of complex shear modulus ($|G^*|$), in Pa, to three significant figures, values of $|G^*|\sin \delta$ and $|G^*|/\sin\delta$ were reported.

Bending Beam Rheometer (BBR) Method

BBR measures the asphalt stiffness by the deflection or creep at constant load and temperature, that stiffness is used to predict the low temperature cracking. Generally PAV-aged asphalt cements are used for testing. They are heated and poured into a mold. After cooling for 45 to 60 minutes, trimming has to be done before conditioning at the bath temperature. After 1 hour of thermal conditioning, asphalt beam is placed on the supports to apply three point load. After the application of preload of 35 mN, a seating load of 980 mN applied for 1 second and allowed for a 20 second recovery period. The graph of load and deflection versus time is plotted continuously. The rheometer software produces the results automatically. The creep stiffness (S), which is the binder resistance to creep loading, and creep rate (m), which is the asphalt stiffness change with time during the application of load, are measured by using BBR. The BBR mold and test set up is shown in the following Figure 3.3.
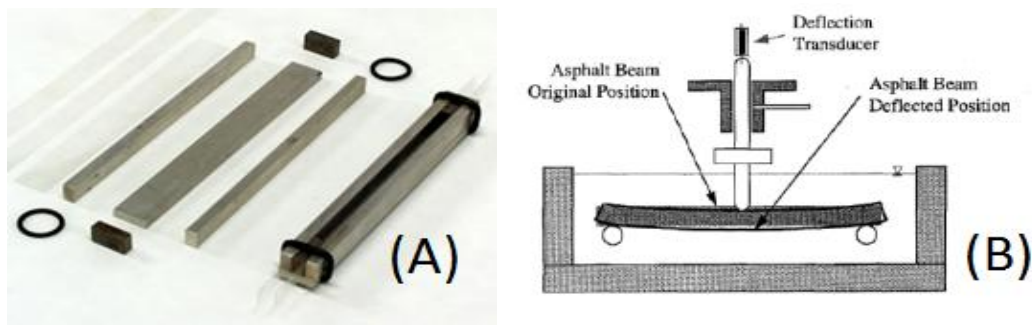

Figure 3.3: BBR mold (A) and test set up (B) [65,66].

As per Superpave binder specification, the bending beam rheometer test is to be conducted at 10 °C above the expected minimum pavement temperature, T_{min}, which is approximately equal to its stiffness at T_{min} after 2 hours loading time, which is related to low-

temperature cracking potential. The Superpave binder specification requires the stiffness at the test temperature after 60 seconds to be less than 300 MPa to control low-temperature cracking. Slope of the log stiffness versus log time curve gives the m-value at a specified time. As per the AASHTO specification, the low temperature PG grades decided on stiffness < 300 MPa and m-value > 0.300, for PAV residue [44]. A higher m-value indicates that asphalt creep at a faster rate to reduce the thermal stress and in turn desirable to reduce low-temperature cracking [42]. Absence of a proper chemical ageing method, true failure tests (BBR and DSR determines the rheological properties at low strain regime but thermal cracking is a high strain phenomenon), and insufficient physical aging (i.e., 1 h conditioning of the asphalt according to the AASHTO M320), suggested the need for improved test methods [5].

3.2.2 Extended BBR Testing According to LS-308

LS-308 conditions at + 10 °C and + 20 °C above the actual grade temperature (i.e., pavement design temperature) for periods of 1 h, 24 h, and 72 h, to simulate the effect of extended exposure to two different cold temperatures [12]. Exact grade of pass and fail temperatures are determined according to AASHTO M320 criteria by interpolation which involves plotting the grade on a semi-logarithmic scale [54]. This method helps to get a high degree of confidence to prevent the thermal cracking [5].

3.2.3 Ductile Failure Testing According to LS-299

The test is conducted after thermal conditioning to determine the essential work of failure, the plastic work of failure, and an approximate critical crack tip opening displacement (CTOD), at a specified temperature and rate of loading [13].

Samples are prepared in dumbbell shape with ligaments (i.e., distances between two opposing notches) of 5 mm, 10 mm and 15 mm in length, by pouring in molds and allowed to solidify as shown in Figure 3.4. Conditioning was done at room temperature and in the water bath for 24 hours at test temperature like 15 or 25ºC before testing. Then, samples are pulled until it fails in a water bath. This test describes of determining the ductility of asphalt measured by the distance to which it will elongate before breaking when two ends of a briquette specimen of the material are pulled apart at a specified speed (5 cm/min) and at a specified temperature.
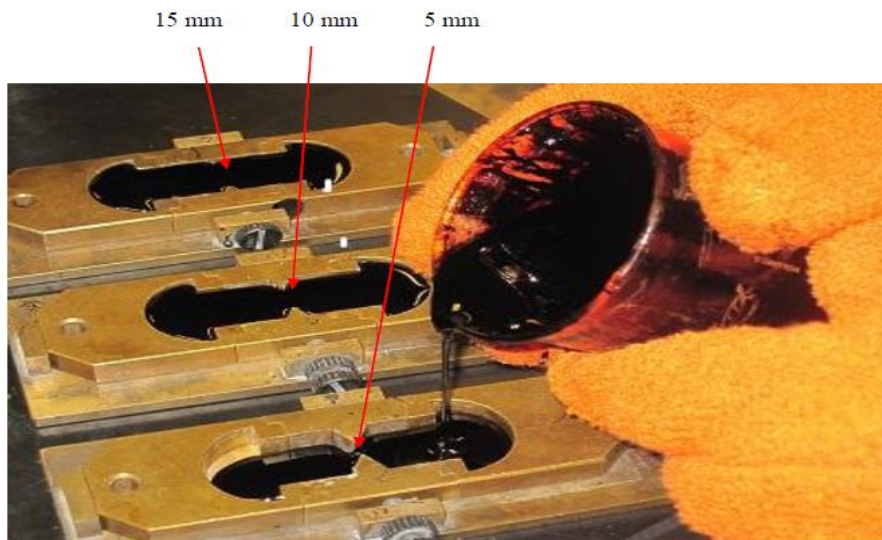

Figure 3.4: DENT sample preparation [7].

The sample is heated carefully in a covered container to prevent local overheating until it has become sufficiently fluid to pour. The brass molds are assembled on base plates. Thin film sheet and coating of thin layer of a mixture of talc powder with glycerol or grease is used on the molds and plates to prevent the test material from sticking on the mold. Sample is stirred and rapidly poured in the mold. Pouring has to be done in a stream back and forth

from end to end until the mold is more than level full. Care should be taken to avoid the disarranging of mold parts which will distort the shape of the sample during the filling of the mold. Then it is cooled to room temperature for 35 minutes. The excess material is trimmed with a hot spatula to make the molds just level full. The trimmed specimen is placed in the water bath at the specified test temperature for 24 h prior to testing. The sample is removed from the plate by a shearing action between specimen and plate, avoiding any bending of the test specimen. Then side pieces of mold are removed carefully without distortion or fracture the specimen. The rings at each end of the clips are attached to the pins or hooks in the testing machine. The two clips are pulled apart at a uniform speed as specified until the specimen ruptures or reaches the length limitations of the testing machine. The computer measures the force and distance to produce rupture or final length and plots the graph.

The essential work of failure (we) helps to predict fatigue cracking resistance and to determine the CTOD [47]. High correlations exist between the CTOD and the fatigue properties of the asphalt cement was investigated by Queen's University and the US Federal Highway Administration which has an accuracy of 85 percent to predict fatigue cracking and ductile failure properties of pavement [5]. CTOD can be used to rank the performance and determine a high correlation with cracking distress.

Chapter 4

RESULTS AND DISCUSSIONS

4.1 Dynamic Shear Analysis

4.1.1 High Temperature Grading

All the Cold Lake, Boscan, Western Texas, Lloydminster binders and their compositions with various warm mix additives were aged in both the rolling thin film oven (RTFO) and pressure aging vessel (PAV) as per standard protocols. All the Superpave^{TM} grading properties and other properties of interest were determined on the residues obtained from RTFO and PAV. All the unaged and RTFO residues were tested by using a TA Instruments AR2000ex DSR to determine their high temperature grades. Similarly, intermediate grades were determined for the PAV residue in the DSR. The table 4.1 shows the sample labels with their description and concentration which helps to understand the graphs.

Table 4.1 Sample labels with its description and concentration.

S.No	Description	Label	Conc. (%)
1	Unmodified Asphalt Cement	U or Unaged (fresh)	-
2	Rheology Modifier / Surfactant	A or Dispersant 1 (D1)	1.5
3	Amine	B or Dispersant 2 (D2)	0.75
4	Polymer	C or Dispersant 3 (D3)	1.5
5	Amine	D or Dispersant 4 (D4)	0.75
6	Complex Mixture	E or Dispersant 5 (D5)	1.5
7	Amide Wax	F or Dispersant 6 (D6)	1
8	Polyethylene Wax	G or Dispersant 7 (D7)	1

The PG grades for the unaged materials were determined in order to compare these with the values for the respective RTFO and PAV residues as per the AASHTO M320 protocol [35-37, 41, 44]. This comparative analysis helps for an assessment of how the various additives affected chemical aging. Figures 4.1 to 4.4 show that high temperature grade difference between unaged and RTFO samples vary only by 1-3°C for all Cold Lake, Boscan, Western Texas Intermediate, Lloydminster compositions.
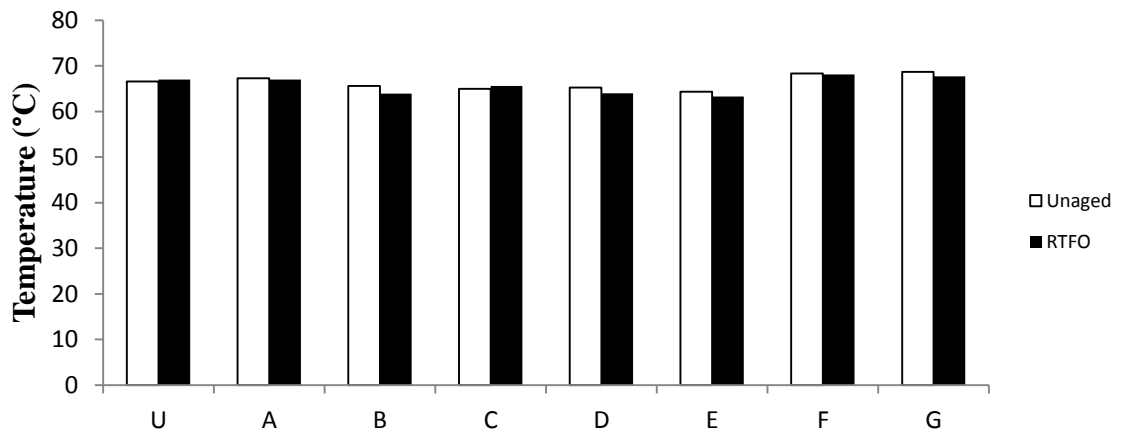

Figure 4.1: High temperature grades for Cold Lake samples.
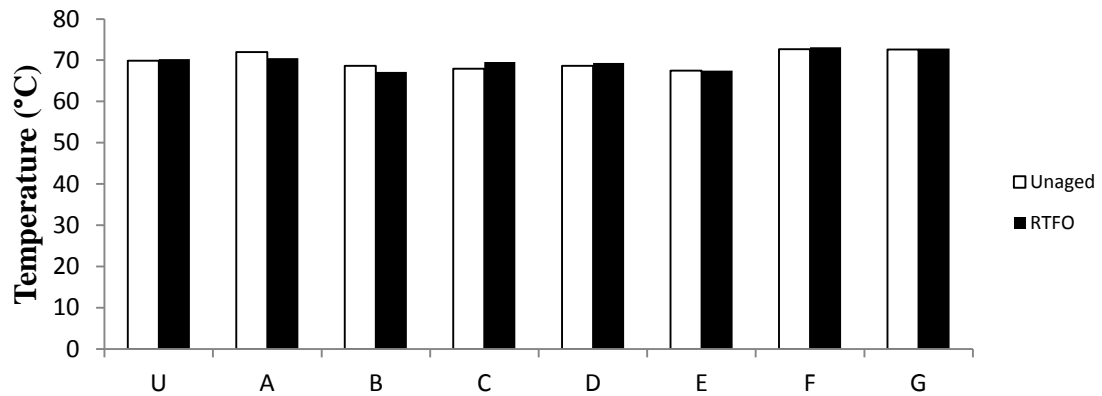
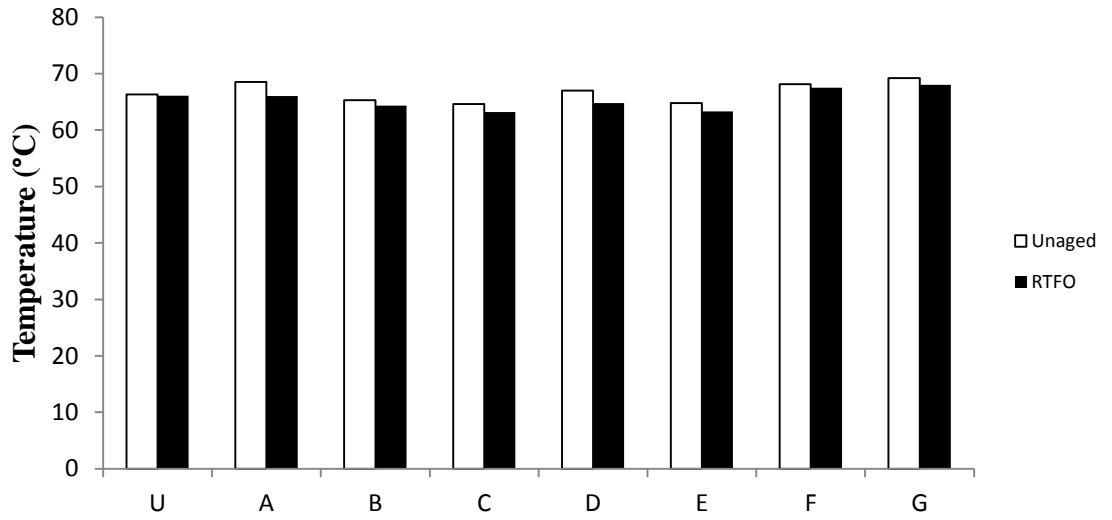**Figure 4.2: High temperature grades for Boscan samples.**

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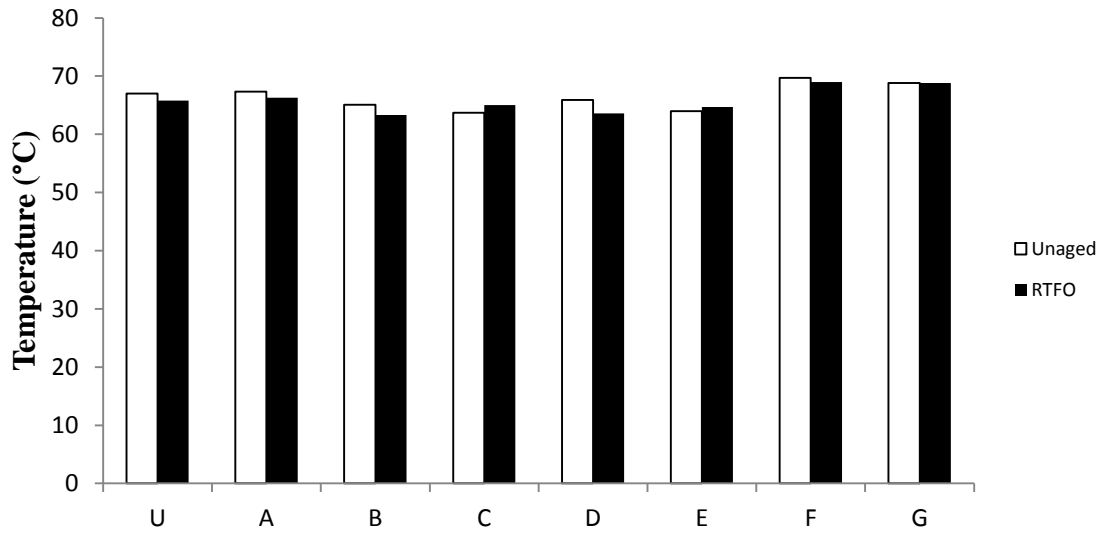**Figure 4.3: High temperature grades for Western Texas Intermediate samples.**

Figure 4.4: High temperature grades for Lloydminster samples.

4.1.2 Intermediate Temperature Grading

The PAV samples were used to find the intermediate temperature grades for control of fatigue distress. As per the AASHTO specification, the intermediate temperature PG grade is determined by the temperature at which G*.sin δ is less than 5000 kPa for a PAV residue [41]. The Intermediate grade temperatures are investigated as per Superpave[TM] specifications for all Cold Lake, Boscan, Western Texas, Lloydminster binders and their compositions as provided in Figure 4.5.
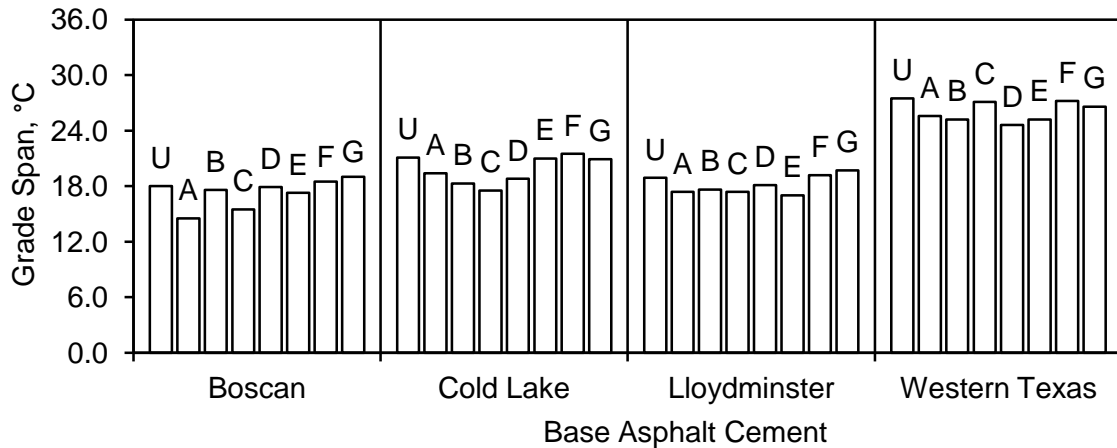

Figure 4.5: Intermediate temperature grades for all Cold Lake, Boscan, Western Texas Intermediate, Lloydminster binders and their compositions.

It is clear from the findings that there are only slight differences of about 2 °C between the straight and modified materials. It shows that the grade span is not affected much and the additive might not show a significant effect in this respect due to the fact that modification levels are only 0.75 to 1.5%. However, these same additives may increase or decrease the grade at higher concentrations.

4.1.3 Black Space Diagrams

Rheology is the study of the deformation and flow of materials. Rheological studies are used to determine the nature of asphalt cement in terms of homogeneous (single phase) or heterogeneous (multiple phase) systems. In this study, DSR is used to find the Black space diagram apart from PG grading, it helps to determine whether asphalt cement is rheologically simple or complex. In Black space diagrams, if there is a smooth progression of curves at various temperatures without any overlaps, the asphalt cement is said to be rheologically simple, whereas if there is no smooth progression, with overlaps and discontinuities at different temperatures, the material is considered to be rheologically complex. Rheologically simple materials are generally found to be homogenous single phase systems whereas rheologically complex materials are oftentimes multiple phase systems with more than one type of flow mechanism and phase transition.

A Black space diagram is obtained by plotting the phase angle versus the complex modulus in a log scale graph plotted according to a frequency sweep done at various temperatures. In our study, the frequency sweep is done from 0.1 to 10 rad/s at various temperatures from 34 to 82°C. Phase angle values at high temperatures show the viscous behavior of binders, which in turn relates to their rutting behavior. Complex modulus (stiffness) values at low temperatures shows the elastic behavior of binders, which in turn related to cracking behavior.

Figure 4.6 shows a typical Black space diagram for unaged and unmodified Cold Lake sample by the frequency sweep done at various temperature from 34 to 82°C. It is clear

that the data follows a smooth line within the Black space and this suggests that the material
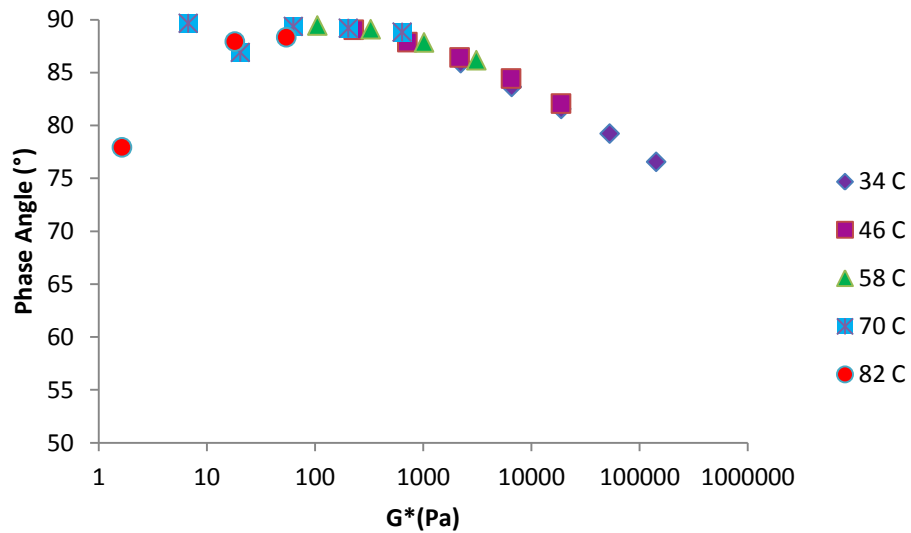
is rheologically simple.

Figure 4.6: Black space diagram for Unaged and Unmodified Cold Lake sample.

Figure 4.7 to 4.10 show the Black space diagrams for unaged Cold Lake, Boscan, Western

Texas Intermediate, and Lloydminster binders and their various compositions. At low

temperatures all the samples behave similarly but at high temperatures the Cold Lake + 1.0 %

Dispersant 6 shows a slightly different behavior. It shows very low phase angle

comparatively with other additives which is a typical behavior of amide wax modified

materials. So it is likely due to the wax that has gelled this asphalt cement.
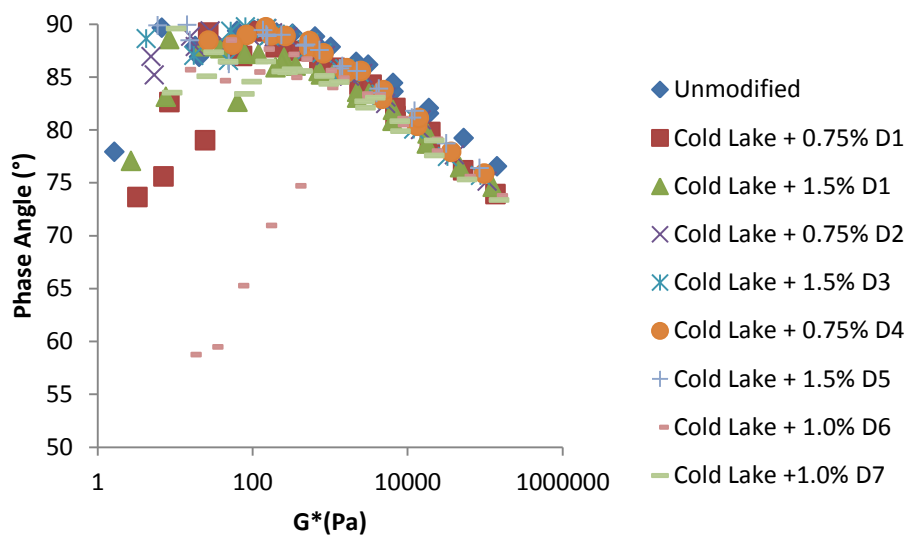
Figure 4.7: Black space diagram for unaged Cold Lake samples with various modifiers.
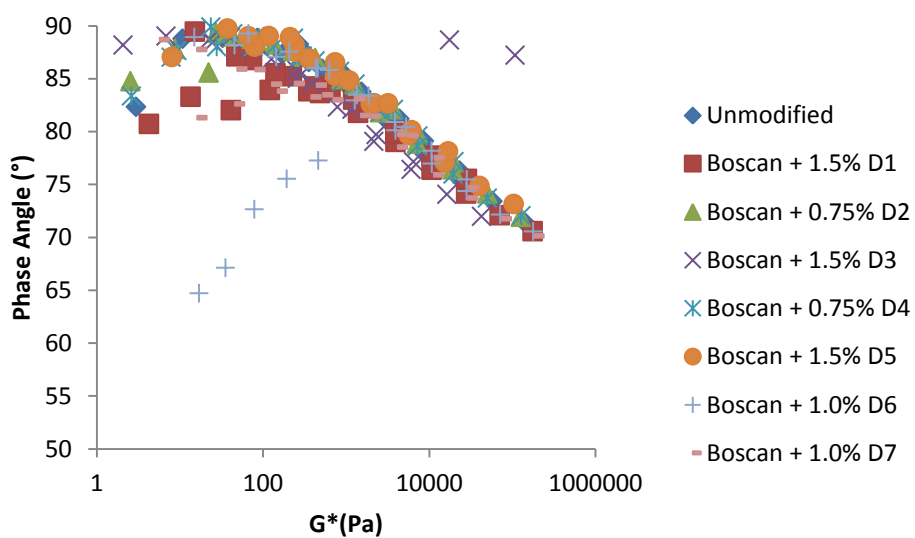

Figure 4.8: Black space diagram for unaged Boscan samples with various modifiers.
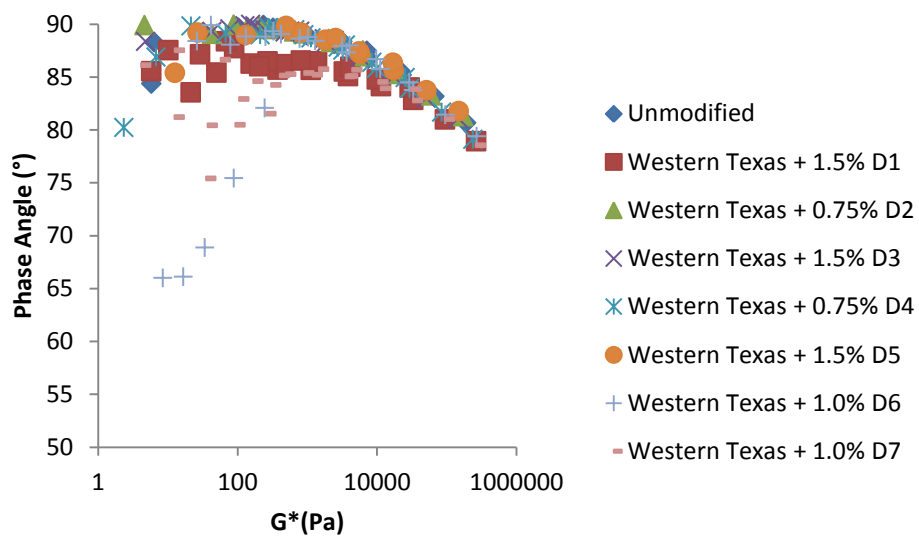
Figure 4.9: Black space diagram for unaged Western Texas Intermediate samples with various modifiers.
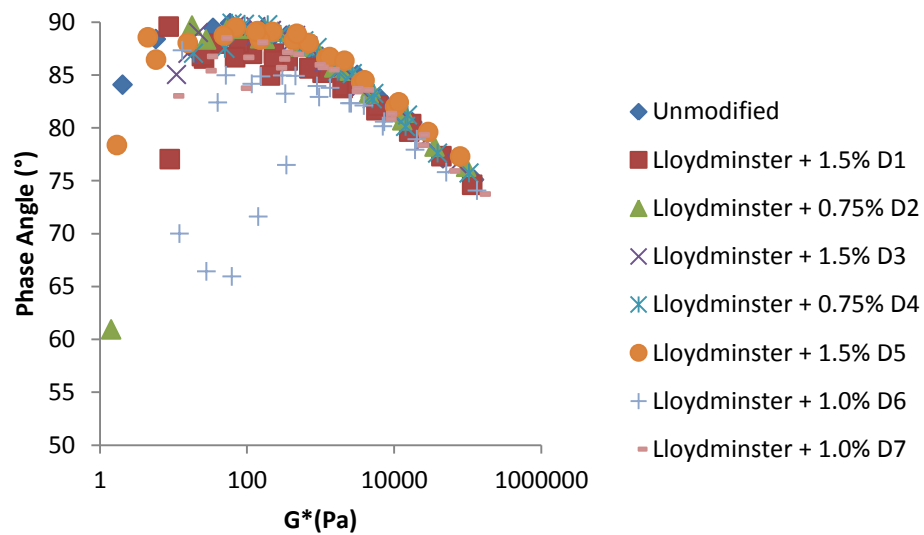

Figure 4.10: Black space diagram for unaged Lloydminster samples with various modifiers.

Figures 4.11 to 4.14 show the Black space diagrams for unaged, RTFO and PAV aged Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions with various additives. At low temperature all the samples behave similarly but at high temperature, Cold Lake + 1.0 % Dispersant 6 shows a slightly different behavior. It can be seen that most are rheologically simple asphalt cements but some show complex behavior. There is a smooth transition between the curves of different temperatures without any overlaps between the curves in rheologically simple binders and a less than smooth transition in rheologically complex binders as shown in the Black space diagrams.
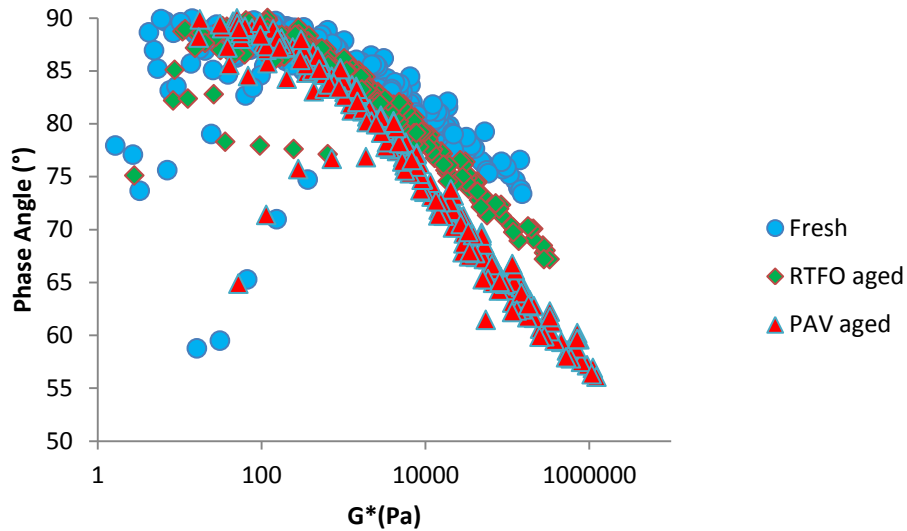

Figure 4.11: Black space diagrams for Unaged, RTFO and PAV-aged Cold Lake samples with various modifiers.
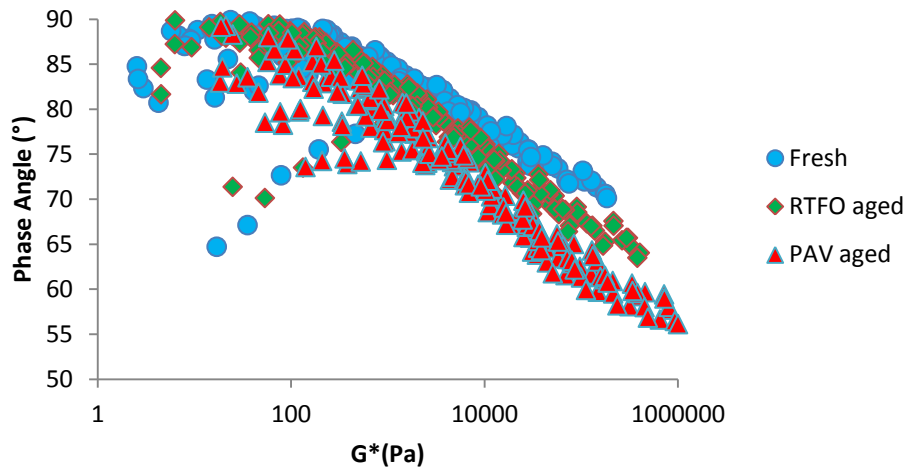
Figure 4.12: Black space diagrams for Unaged, RTFO and PAV-aged Boscan samples with various modifiers.
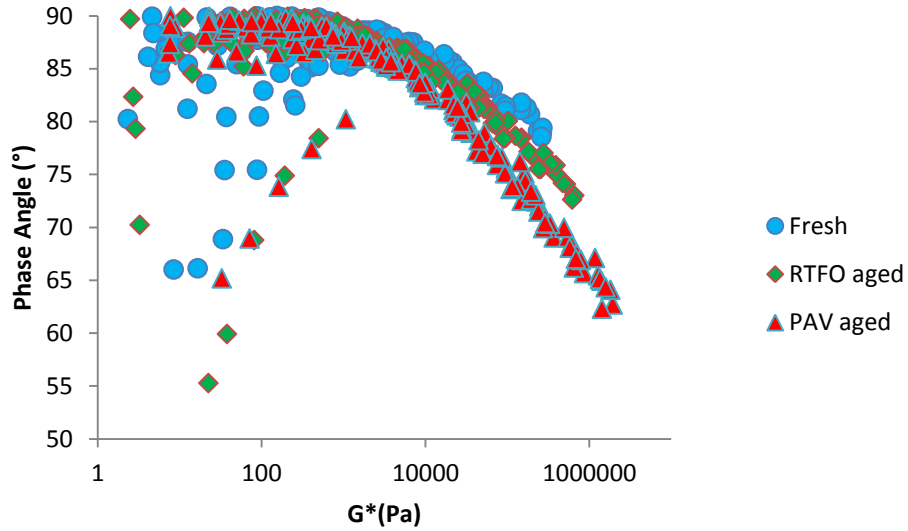

Figure 4.13: Black space diagrams for Unaged, RTFO and PAV-aged Western Texas Intermediate samples with various modifiers.
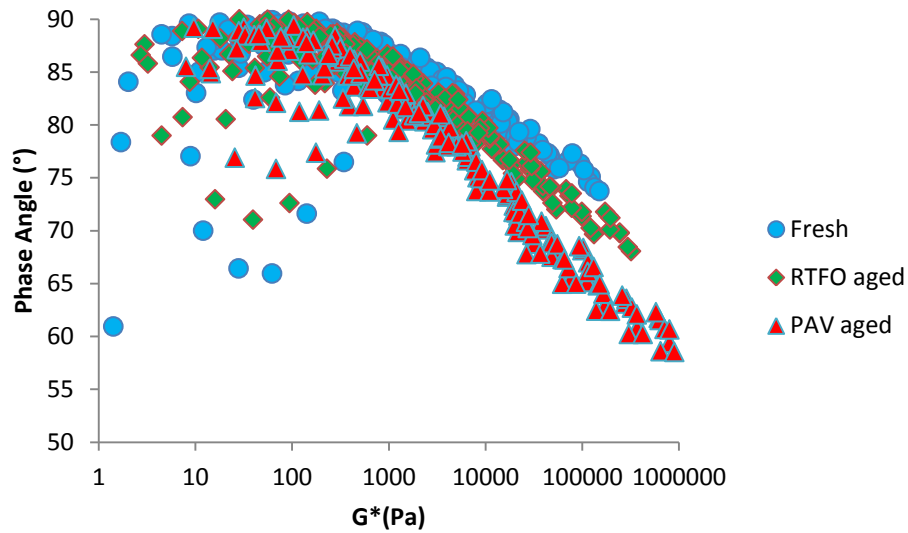
Figure 4.14: Black space diagram for Unaged, RTFO and PAV-aged Lloydminster samples with various modifiers.

The asphalt pavement performance is related to the physical and rheological properties. Those properties depend on their viscoelastic behavior. This viscoelastic behavior is to be maintained at all temperatures for the better asphalt cement pavement performance on road, which is very difficult at extreme low and high temperatures. Lower values of phase angle at higher temperatures and higher values at lower temperatures results in better viscoelastic character to the asphalt cement. It is noted in Black space diagram, phase angle values are high at lower frequencies. The reason for that behavior is explained as the sample has more time to dissipate an amount of the applied stress as flow and delaying the response to stress and vice versa at higher frequencies. Phase angle values are higher at higher frequencies than at lower frequencies for stiffer materials. Finally, the Black space diagram is a graphical tool for determining the changes in the phase angle and complex modulus.

4.2 Regular BBR Analysis

As per the Superpave binder specification, the bending beam rheometer test is to be conducted at 10°C above the expected minimum pavement temperature, T_{min}, which is approximately equal to its stiffness at T_{min} after 2 hours loading time, which is related to the low temperature cracking potential. As per the AASHTO M320 specification, the low temperature PG grades are decided based on the limiting temperature where stiffness < 300 MPa or m-value > 0.300 for the PAV residue [44]. A higher m-value indicates that asphalt creep at a faster rate to reduce the thermal stress and this is desirable to reduce low-temperature cracking [42].

4.2.1 Low Temperature Grades

The Low temperature grades are investigated as per the Superpave specifications for all Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions which are provided in Figures 4.15 to 4.18
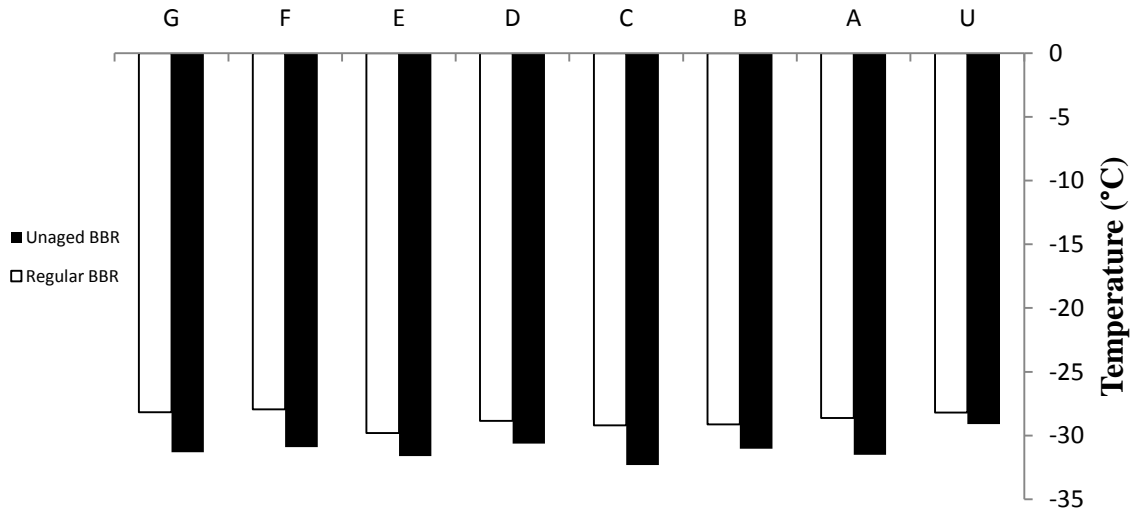

Figure 4.15: BBR low temperature grades for Cold Lake samples.

Figure 4.15 shows that the temperature differences between unaged and regular BBR on PAV residues varies only slightly by about 1-3 °C in the low temperature grading in Cold Lake and its various compositions. Figure 4.16 shows that temperature differences between unaged and regular BBR grades on PAV residues vary by about 2-4 °C in the low temperature grading in Boscan and its compositions.
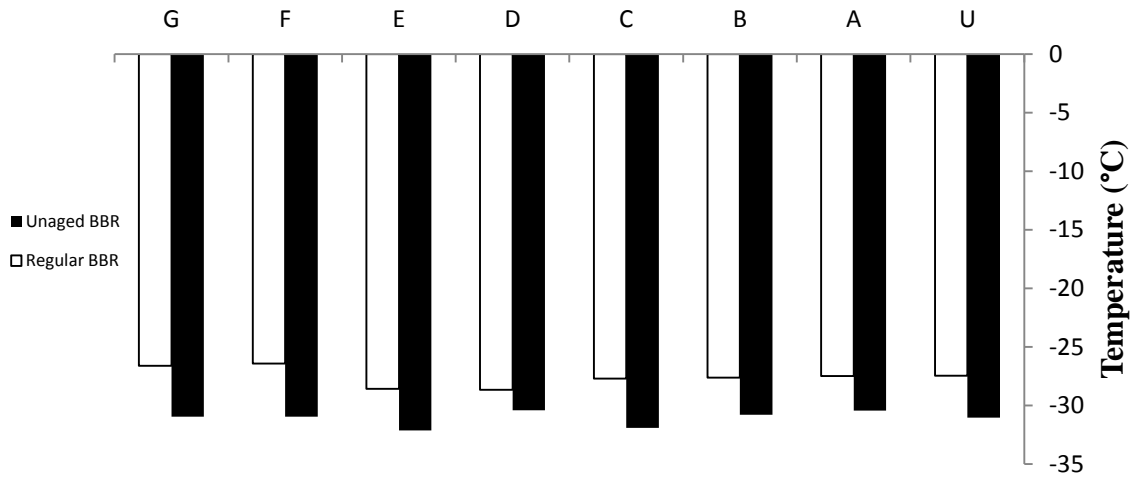

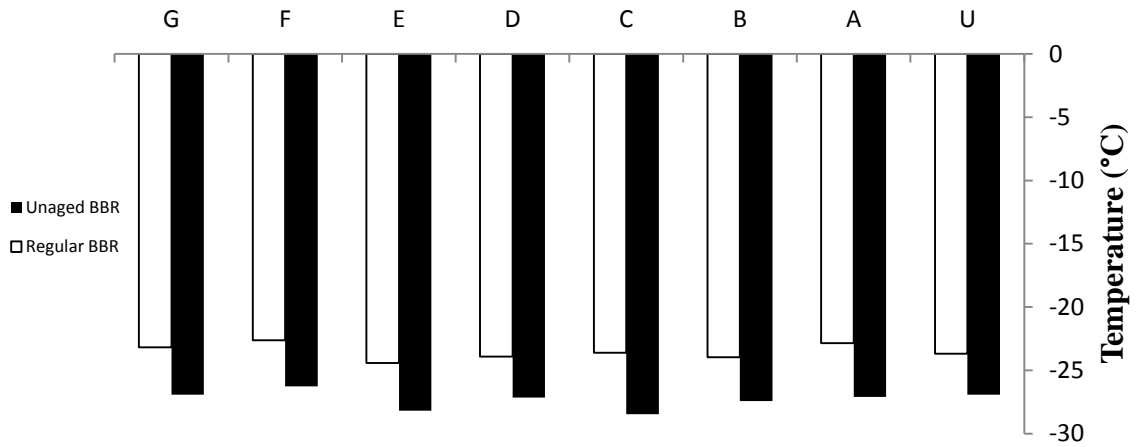**Figure 4.16: BBR low temperature grades for Boscan samples.**

Figure 4.17: BBR low temperature grades for Western Texas Intermediate samples.

Figure 4.17 shows that the temperature differences between unaged and regular BBR grades on PAV residues vary by about 2-4 °C in the low temperature grading in Western Texas Intermediate and its compositions except for Dispersant 3 which deviates by about 5°C.
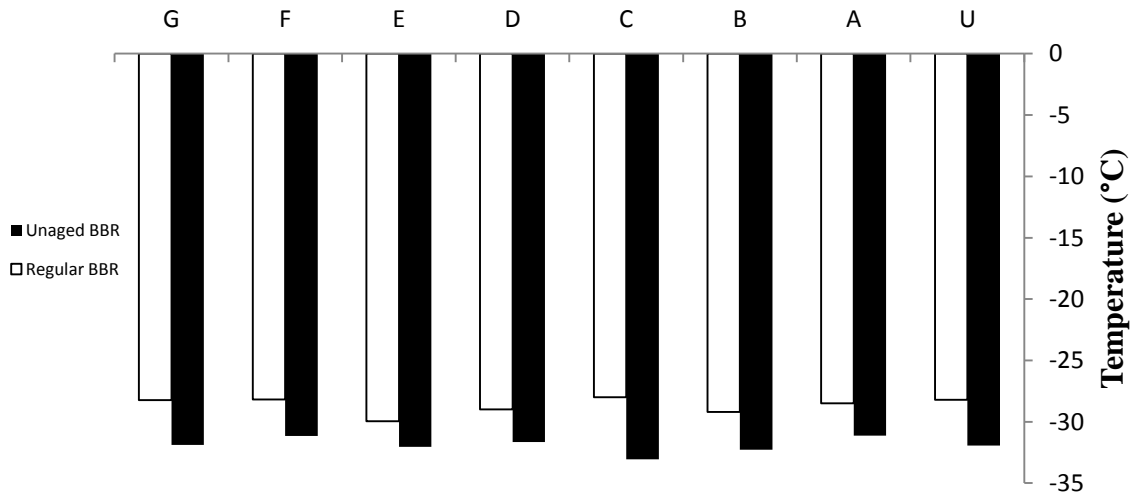
Figure 4.18: BBR low temperature grades for Lloydminster samples.

Figure 4.18 shows that temperature difference between unaged and regular BBR varies about 2-4°C in the low temperature grading in Lloydminster and its compositions except the Dispersant 3 which deviates about 5°C.

Figures 4.15 to 4.18 shows that the temperature difference between unaged and regular BBR vary by only about 1-4 °C in the low temperature grading in all Cold Lake, Boscan, Western Texas, Lloydminster and its compositions except the Dispersant 3 which deviates about 5°C in Western Texas Intermediate and Lloydminster.

4.2.2 Grade Spans

The grade spans are investigated as per Superpave specifications for the all Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions, which are provided in Figure 4.19. It is clear from the findings that there are only slight differences about 2 °C between the straight and modified materials. Changes in high temperature grades often come with similar changes in low temperature grades. The figure shows that the grade span is not affected much and the additive might not show a significant effect in this respect due to the fact that modification levels of only 0.75 to 1.5% were used. However, these additives may increase or decrease the grade at higher concentrations.
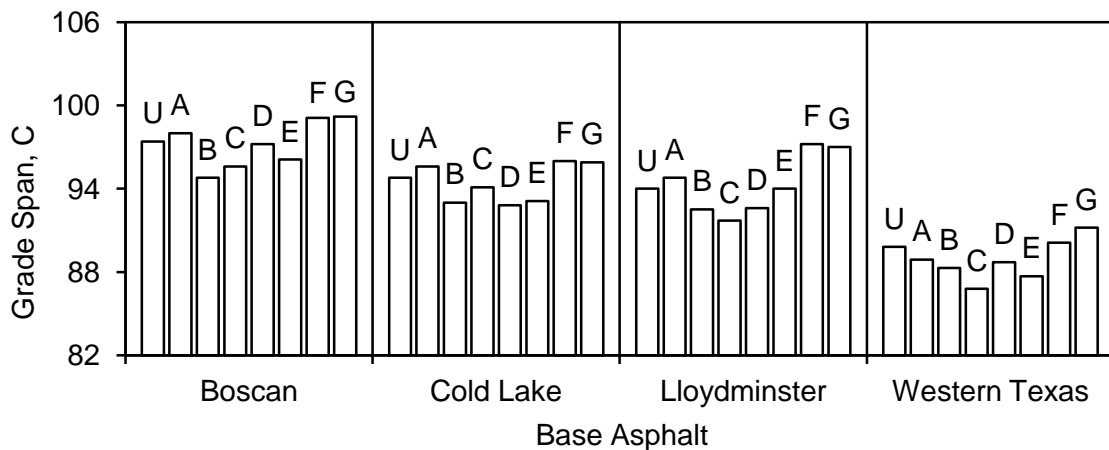

Figure 4.19: Grade span from high temp PG with low temp PG of BBR for all Cold Lake, Boscan, Western Texas, Lloydminster binders and its compositions.

4.2.3 Grade Losses

Chemical aging tendency was investigated by measuring the change in the low temperature grades from unaged to PAV residue to know the effect of various additives. If there is a huge

change, it will result in a greater tendency towards chemical oxidation and a loss in durability. Durability is explained as the ability of a binder to maintain or retain its useful properties. Durability is a desirable attribute which is not considered in the current Superpave specifications. Figure 4.20 shows the grade losses for unmodified as well as modified systems of all Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster samples. So there are no major differences in the above data. The unmodified Cold Lake sample shows the least susceptible behavior where the Lloydminster modified with 1.5% of additive C shows the most susceptible behavior. The grade loss difference between these two is 4.2°C, which may cause significant low temperature cracking as it is a catastrophic phenomenon.
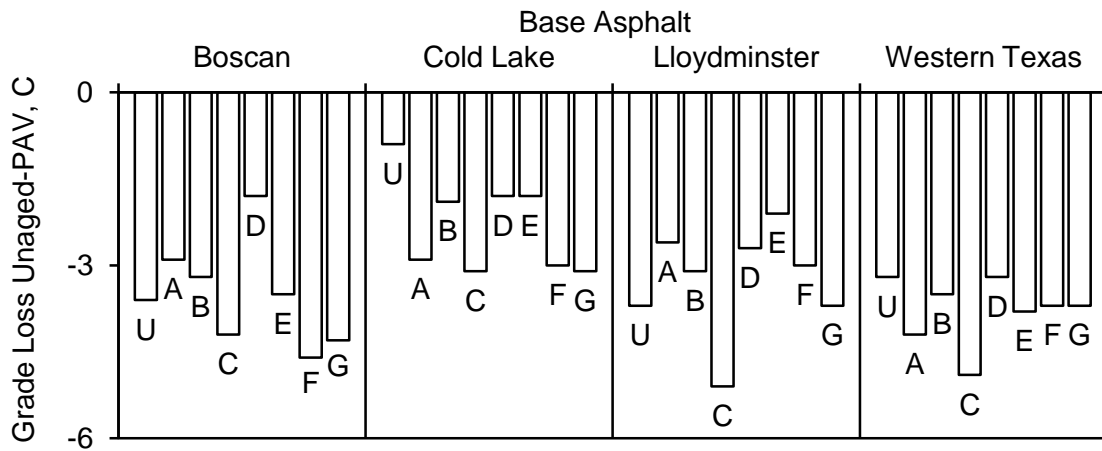

Figure 4.20: Grade losses due to RTFO / PAV treatment for all Cold Lake, Boscan, Western Texas, Lloydminster binders and its compositions.

If there is a deficit of 6°C for the low temperature grade, it will typically reduce the confidence level that a pavement is not exposed to damage in a given year from the intended 98% to around 50% [81].

4.3 Extended BBR Analysis

LS-308 conditions at +10°C and +20°C above the actual grade temperature (i.e. pavement design temperature) for periods of 1 h, 24 h, and 72 h to simulate the effect of extended exposure to two different cold temperatures [12]. Exact grades of pass and fail temperatures are determined according to AASHTO M320 criteria by interpolation which involves plotting the grade on a semi-logarithmic scale [54]. This method helps to get a high degree of confidence to prevent thermal cracking [5].

4.3.1 Low Temperature Grades

In this study we investigated the effect of various additives on the tendency for physical hardening, which is explained as a volume relaxation or shrinkage at cold temperatures accompanied by slow stiffening of the asphalt cement and a loss in relaxation ability [75]. The main difference between physical hardening and chemical hardening is the reversible nature for physical hardening and the irreversible nature for chemical hardening. It has been hypothesized from few asphalt literature that certain additives improve the asphaltene colloidal stability in the maltene phase [75-80]. Due to this reason, physical hardening is very important to study with these additives to know if there is any positive or negative effect on the hardening tendency during cold conditioning. The following figures show the low temperature grades and the worst three day grade losses after conditioning of PAV residues at both -8°C and -18°C according to Ontario's extended BBR protocol [12]. Figure 4.21 shows that temperature differences between unaged and extended BBR varies about 6°C in the low temperature grading in Cold Lake and its compositions except for Dispersant 6 and 7 which deviates about 8-10 °C. Figure 4.22 shows that temperature

differences between unaged and extended BBR vary by about 8-9 °C in the low temperature

grading in Boscan and its compositions.
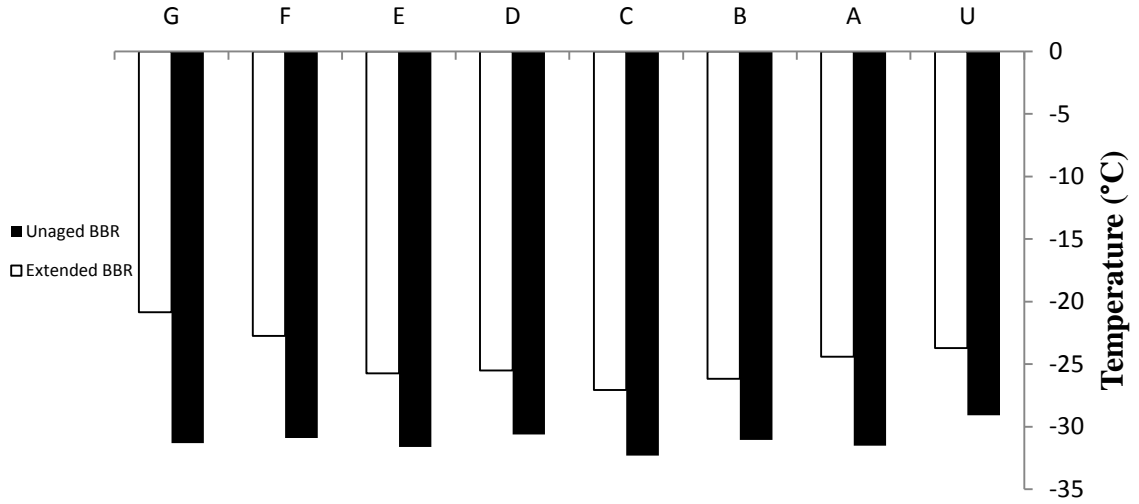
Figure 4.21: Extended BBR low temperature grade for Cold Lake samples.
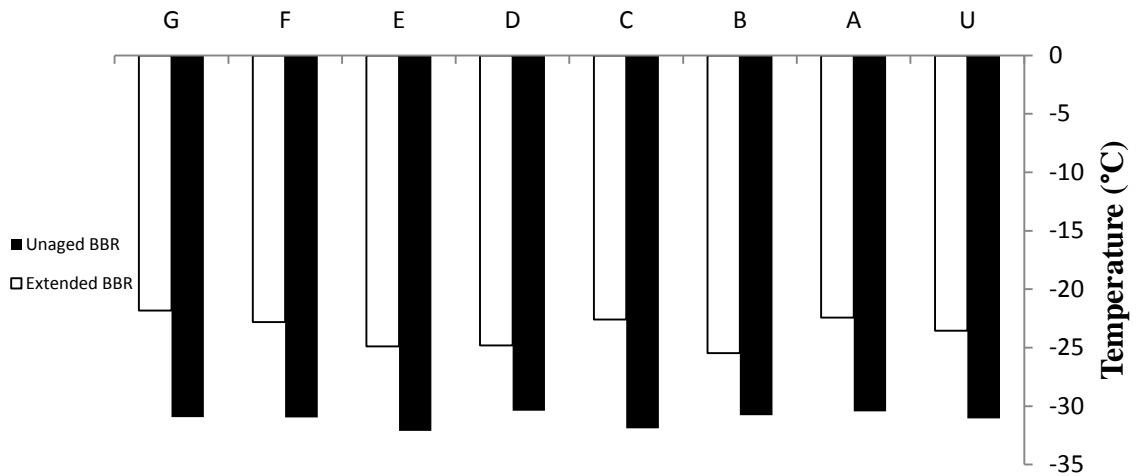
Figure 4.22: Extended BBR low temperature grades for Boscan samples.

Figure 4.23 shows that temperature differences between unaged and extended BBR vary by

about 9-10°C in the low temperature grading in Western Texas Intermediate and its

compositions. Figure 4.24 shows that temperature differences between unaged and extended BBR vary by about 6°C in the low temperature grading in Lloydminster and its compositions except the Dispersant 3 which deviates about 8°C.

Figure 4.23: Extended BBR low temperature grade for Western Texas samples.

Figure 4.24: Extended BBR low temperature grades for Lloydminster samples.

4.3.2 Grade Spans
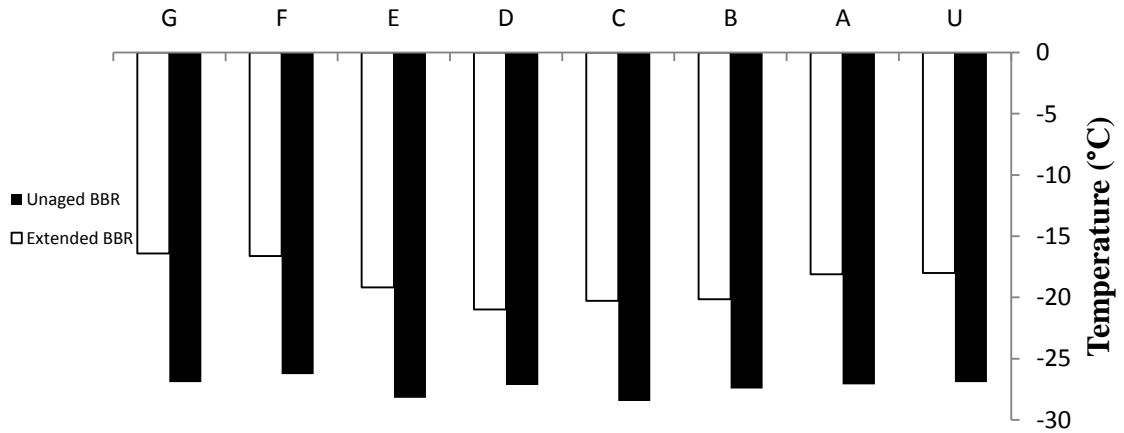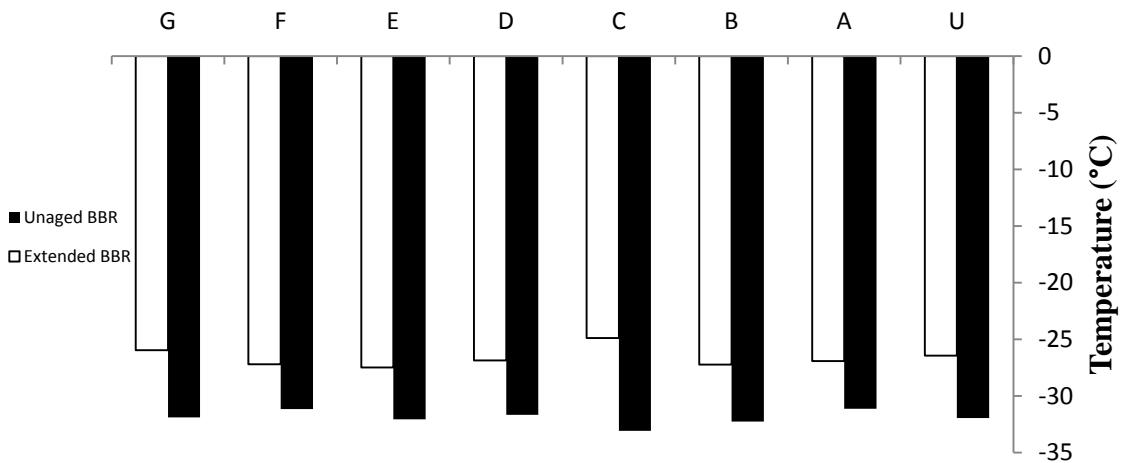
The grade spans are investigated as per the Superpave specifications for all Cold Lake, Boscan, Western Texas, Lloydminster binders and its compositions with high temp PG and

low temp PG of extended BBR are provided in Figure 4.25. It is clear from the findings that there are major differences in grade span for Western Texas Intermediate and its compositions compared to the others.
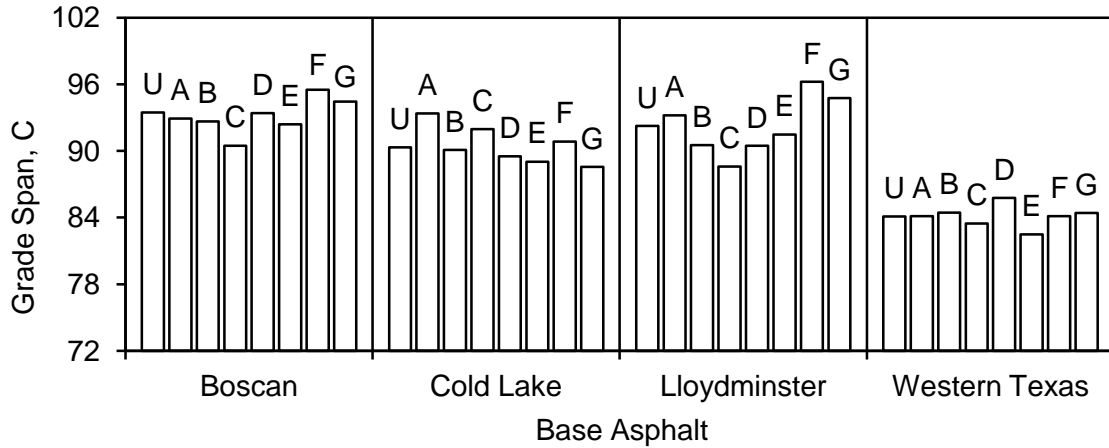
Figure 4.25: Grade spans from high temp PG with low temp PG of extended BBR for Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions.

4.3.3 Grade Losses

There are some general differences noted among all the samples. Figure 4.26 shows that Lloydminster base asphalt cement has the better performance than the other three asphalts. Lloydminster base asphalt has the average loss average loss of 2°C is less than half of that for the others which all lose around 4°C. However, the losses of these all materials after three days of cold conditioning show relatively lower than the materials tested in our laboratory over the last 10 years is about 5.8°C, with some materials losing more than 10°C [82]. Generally, the base asphalts will suffer in physical hardening when they are low in asphaltenes (e.g., California Valley, Peace River), or rich in compatible naphthenes

(cycloalkanes) and low in linear alkanes (paraffins) (e.g., Bow River, Cold Lake, Lloydminster) [82, 83].
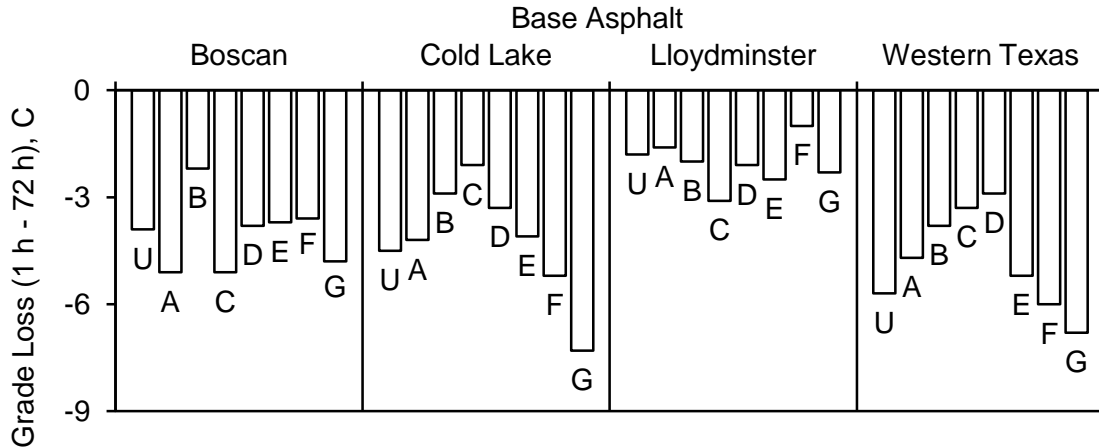
Figure 4.26: Grade losses due to physical hardening at -8 °C and -18 °C for 72hours for all Cold Lake, Boscan, Western Texas, Lloydminster binders and its compositions.

Wax F showed the ability to increase the hardening tendency among all the additives. Waxes are expected to immobilize the amorphous phase. Therefore they increase the hardening rate and consequent stiffness [82]. So it can be concluded that care has to be taken when a warm mix additive is selected since along with improved constructability, increased low temperature cracking could be an unwanted side effect. The results of a large investigation on premature and excessive low temperature cracking in eastern Ontario pavements helps us to understand the findings of Figures 4.25 and 4.26. The following Figure 4.27 shows the effect of physical hardening on thermal cracking tendency after 72 h of storage at -10°C for asphalt cements recovered from 20 eastern Ontario paving contracts [74].

The graph of cracking severity versus grade loss explains that the increase of grade loss by more than 3°C from physical hardening affects the asphalt cement enough to increase

cracking severity to show premature and excessive cracking in early life. In contrast, binders that lost less than 3°C after three days of conditioning all produced pavements with little or no cracking after 7-14 years of service.
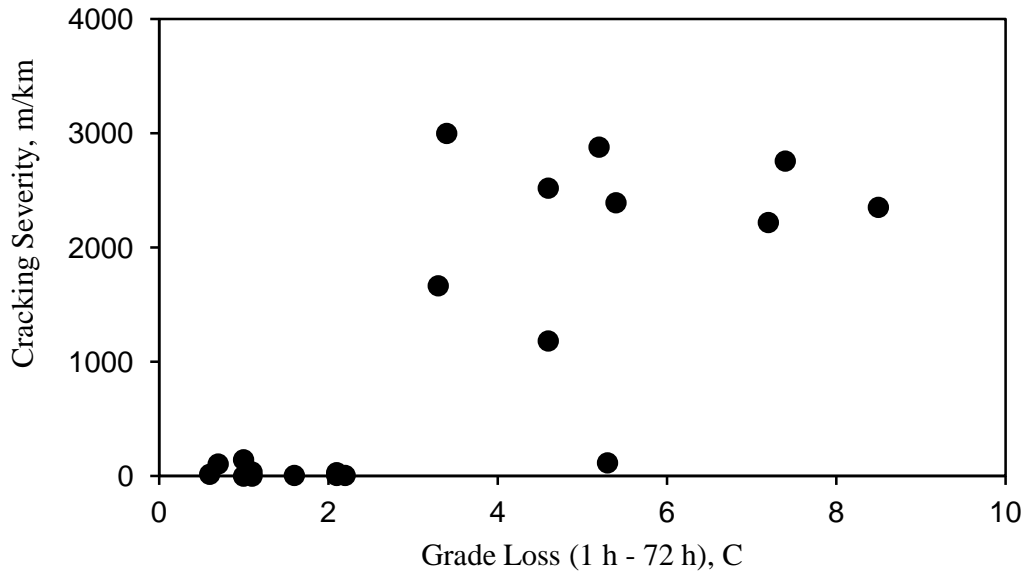
Figure 4.27: Physical hardening on thermal cracking tendency after 72 h of storage at -10°C for asphalt cements recovered from 20 eastern Ontario paving contracts [74].

4.4 Double Edge Notched Tension (DENT) Testing

Samples are prepared in dumbbell shape with ligaments, i.e., distances between two opposing notches of 5 mm, 10 mm, and 15 mm in length, by pouring in molds and allowed to solidify. Conditioning was done at room temperature and in a water bath for 24 hours at the test temperature like 15 or 25°C before testing. Then samples are pulled until they fail in a water bath. Figure 4.28 shows that raw force-displacement traces for duplicate DENT tests on Cold Lake unmodified samples at 50 mm/min and 25°C. Figure 4.29 shows that determination of the essential work of failure for Cold Lake unmodified sample at 50 mm/min and 25°C.
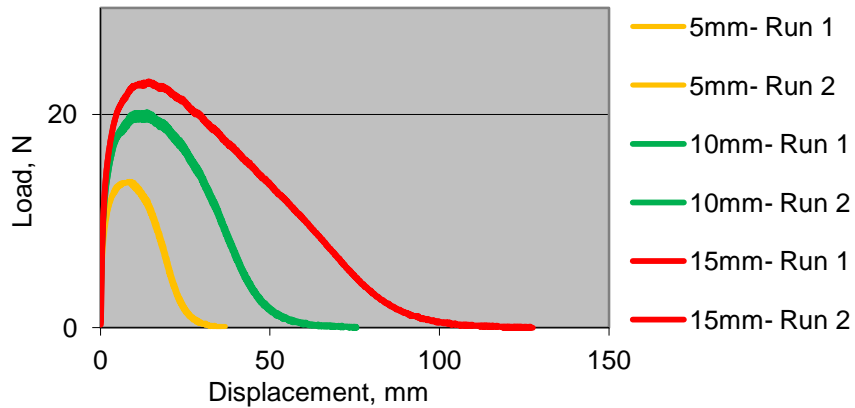
Figure 4.28: Raw force-displacement traces for duplicate DENT tests on Cold Lake unmodified samples at 50 mm/min and 25°C.
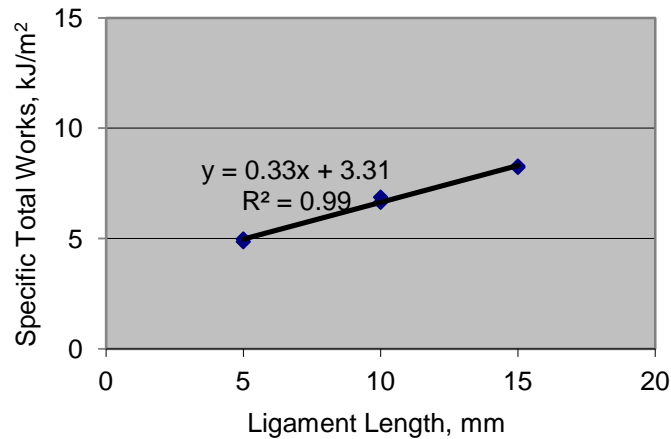
Figure 4.29: Determination of the essential work of failure for Cold Lake unmodified sample at 50 mm/min and 25°C. Note: Essential work of failure = 3.31 kJ.m^{-2}, CTOD = 12.13 mm.

The intercept for this graph provides an accurate essential work of failure and this is then used together with the peak loads in the 5 mm ligament specimens to calculate a critical crack tip opening displacement. The graph clearly shows the highly reproducible results and accuracy of works of failure, essential works of failure, and CTODs.

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4.4.1 Essential Works of Failure (W$_e$)

The essential work of fracture is a material property, because it is not depend on the geometry of the asphalt cement sample. Generally, the EWF model is used to determine the resistance of fatigue cracking and low temperature cracking distresses in asphalt cement pavement. Usually, this EWF model concept is suitable to apply when the essential work of fracture values and the plastic work of fracture values are relatively high, since pavement cracking will occur only under flexure when the strain tolerance of the pavement is exceeded [5]. Western Texas Intermediate and its compositions has the higher essential works of failure (w$_e$) than other base asphalt cements as shown in the Figure 4.30.
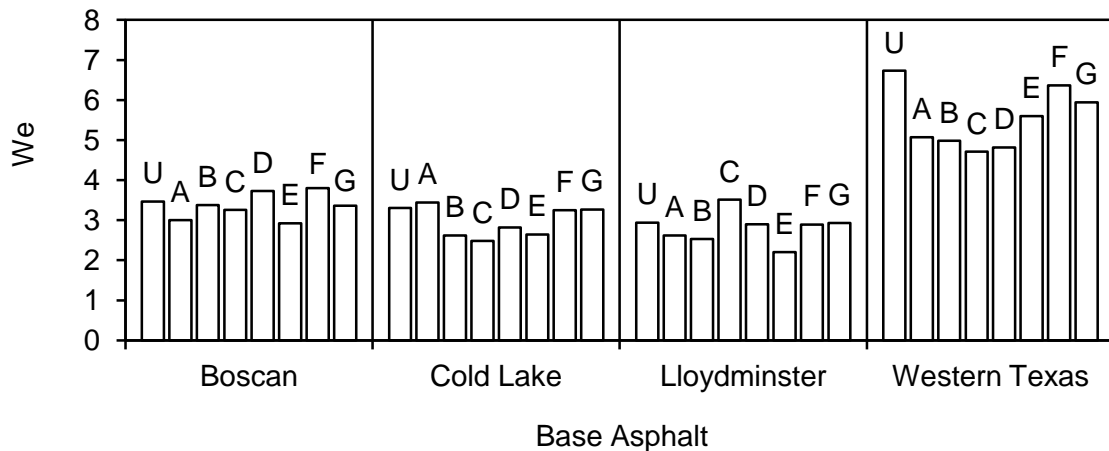
Figure 4.30: Essential works of failure (w$_e$) for all Cold Lake, Boscan, Western Texas, Lloydminster binders and their compositions.

4.4.2 Plastic Works of Failure (βw$_p$)

The β factor that describes the shape of the plastic zone where the non-essential work is dissipated which can have different values depending on the geometry of the plastic zone. However, it should be noted that β is only of secondary importance since we are most

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interested in the essential work and CTOD. If asphalt mixture has higher asphalt binder content, usually will have higher plastic work of fracture, which in turn makes it less sensitive to fatigue and low temperature cracking. It happens by the result of the higher strain tolerance deposited in them due to the higher binder content [84]. Western Texas Intermediate and their compositions have the higher plastic works of failure (βwp) than other base asphalt cements as shown in the Figure 4.31. Lloydminster and its compositions have the lower plastic works of failure (βwp) than other base asphalt cements.

Figure 4.31: Plastic works of failure (βwp) for all Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions

4.4.3 Approximate Critical Crack Tip Opening Displacements

High correlations exist between the CTOD and the fatigue properties of the asphalt cement. CTOD can be used to rank the performance and determine a high correlation with cracking distress. The effect of various additives on high strain failure properties like critical crack tip opening displacement (CTOD) are also measured apart from the low strain rheological properties. CTOD explains about how easily cracks propagate under ductile conditions

during spring thaw and other periods of increased loading and strain [47]. Experimental part explains about the determination of CTOD. Figure 4.32 shows the CTOD (mm) investigation for all Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions.

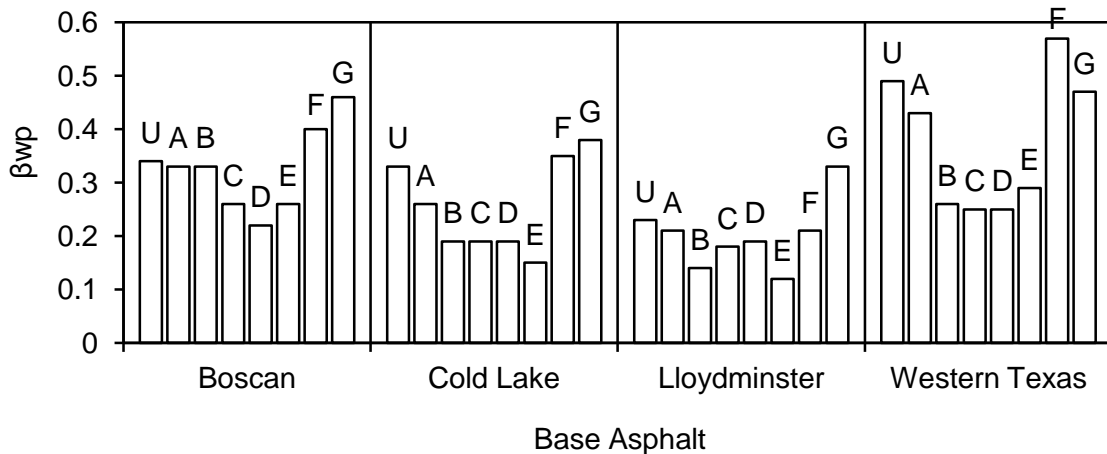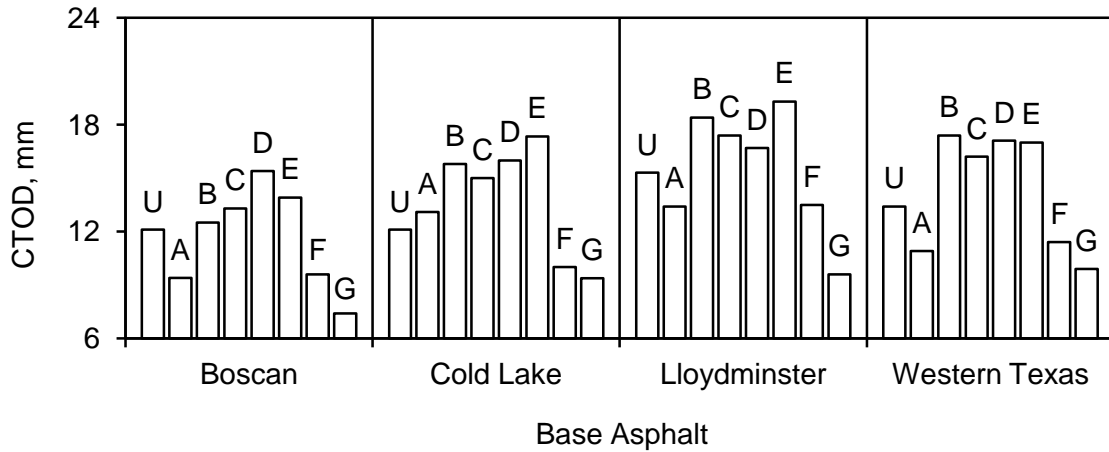**Figure 4.32: CTOD (mm) for all Cold Lake, Boscan, Western Texas Intermediate, and Lloydminster binders and their compositions.**

It shows the major differences for all asphalts than Superpave grades comparatively. Waxes F and G has shown the negative effect on the strain tolerance in the ductile state, which is likely to show up as premature and/or excessive cracking in service [47,48,74] and which is similar to their physical hardening behavior from low temperature grading and extended BBR testing. But asphalt rejuvenator E appear increased the CTOD without affecting the Superpave grade span or on the chemical and physical hardening tendencies [86].

Figure 4.33 shows the cracking distress versus the CTOD for recovered asphalt cements from 20 eastern Ontario paving contracts [74]. It supports the findings of CTOD with investigation of Ontario contracts as discussed earlier.

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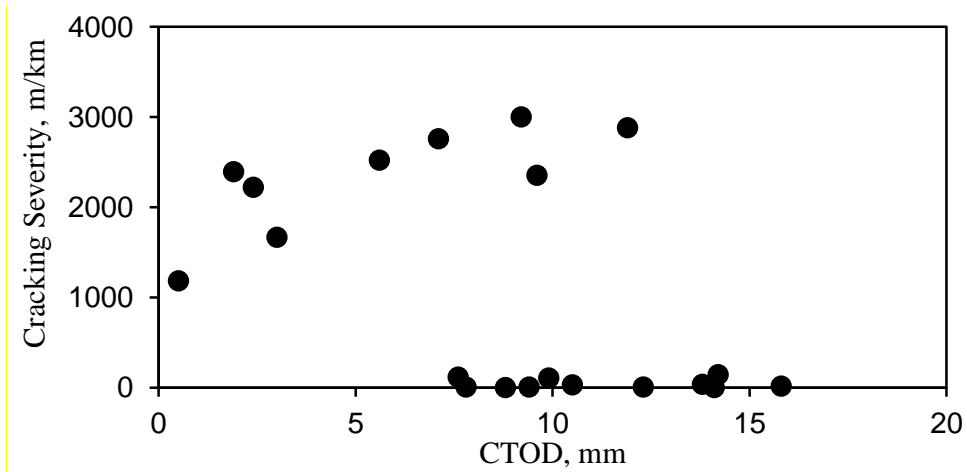
Figure 4.33: Cracking distress versus the CTOD for recovered asphalt cements from 20 eastern Ontario paving contracts [74].

It explains that there is a general tendency for asphalt cements with high CTODs to show little or no cracking in service and majority of severely cracked contracts show low CTODs. Some materials have relatively high CTODs with significant cracking have likely failed due to a deficit in their low temperature grade.

Chapter 5

SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

5.1 Summary and Conclusions

1) The changes in grade span temperature between different base asphalt cements were found to be very significant. It is clear from the findings that there are differences in high and intermediate temp PG grading in straight and modified materials. Changes in high temperature grades often come with similar changes in low temperature grades.

2) The changes in grade span temperature within base asphalts due to the additives were found to be significant. These additives will increase or decrease the grade more effectively at higher concentrations.

3) The effect of additives on chemical and physical hardening tendencies were found to be significant. Amide wax showed the ability to increase the hardening tendency among all the additives. So it can be concluded that care has to be taken when a warm mix additive is selected since along with improved constructability, increased low temperature cracking could be an unwanted side effect.

4) The Black space diagrams shows that most of asphalt cements are rheologically simple by single phase system. Few asphalt cements are rheologically complex binders by heterogeneous phase system. The addition of polymers and waxes to these binders, causes phase separation of the asphalt cement as shown in Cold Lake + 1.0 % Dispersant 6 (Amide Wax) shows the slightly different behavior than others at high temperature.

5) The changes in ductile strain tolerance within base asphalts due to the various additives were found to be very significant. The DENT test results provided a reasonable correlation with cracking distress in service.

6) The approximate CTOD provides an improved and fundamental specification parameter for fatigue. Asphalt rejuvenator E increased the CTOD without affecting the Superpave grade span or the chemical and physical hardening tendencies.

7) The CTOD is used to distinguish and rank the superior performing binders from inferior performing binders especially with the various additives modified asphalt binders with high level of accuracy.

8) The double-edge-notched tension test results like the essential works of fracture, w_e and the plastic works of fracture term, βwp and the critical crack tip opening displacement, CTOD usually helps to correlate with the distress survey results of the pavement in service.

9) The addition of amide and polyethylene waxes risks increasing the cracking susceptibility in the pavement performance. They have shown the negative effect on the strain tolerance in the ductile state, which is likely to show up as premature and/or excessive cracking in service [47, 48, 74] which is similar to their physical hardening behavior from low temperature grading and extended BBR testing.

5.2 Recommendations and Further Work

1) This study suggests the following further work and recommendations based on the results and discussions: Asphalt mixture tests should be conducted in order to understand the effect of various warm mix additives added with base asphalts

cements on the long term pavement performance. And also the effect of different aggregates from different sources should be investigated to understand the impact of aggregate types on performance.

2) More asphalt pavement trials should be constructed to investigate the effect of various warm mix additives added with base asphalts cements on pavement service. Such trials help to understand the various distress mechanisms like thermal cracking, rutting, fatigue cracking, and moisture damage on these additives modified pavements.

3) The implementation of the Ontario Ministry of Transportation's test methods like LS-308 and LS-299 will help to validate the warm mix additives modified asphalt cements to enhance the long term performance of the pavements in service and also to reduce the cost of pavement reconstruction, resurfacing and rehabilitation in colder regions such as Canada and northern parts of the United States.

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