



# Value-Added Opportunities for Conventional and Atypical Asphalt Binders and Asphaltenes Derived from Alberta Oil Sands in Road Construction

Alberta Innovates File AI 2515

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## CLEAN RESOURCES FINAL PUBLIC REPORT

### 1. PROJECT INFORMATION:

<b>Project Title:</b>	<b>Value-Added Opportunities for Conventional and Atypical Asphalt Cements and Asphaltenes Derived from Alberta Oil Sands in Road Construction</b>
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## EXECUTIVE SUMMARY

The main objective of the research described herein was to demonstrate how the use of Alberta oil sands-derived bitumen is able to produce superior-quality, regular and atypical asphalt cements (binders), for use in the construction and rehabilitation of flexible pavements.

A total of over 40 different binders were obtained, either from commercial sources or purposely distilled from crude bitumen, to supply regular and atypical materials for this project. Binders sourced from Alberta (5 crude producers, 25 binders) were compared with materials from China (5 sources), Europe (10 sources), India (1 source), and the Middle East (3 sources). Testing included standard and enhanced binder tests as well as a limited number of advanced asphalt mix tests.

The research has demonstrated value-added opportunities for Alberta oil sands-derived binders and asphaltenes in global road construction. The current global binder market approaches 100 million tonnes worth \$50+ billion. For conventional and atypical Alberta binders, it was demonstrated that they provide superior performance in terms of cracking resistance when compared with materials sourced from elsewhere. The superior performance comes for the most part from their exceptionally low wax contents in combination with moderate to high asphaltene contents, providing high quality and durability. Only binder obtained from a Laguna, Venezuela crude was found to have similarly desirable and balanced properties.

Asphaltenes obtained from solvent de-asphalting (SDA) operations can be used in value-added applications to upgrade asphalt cements for enhanced, long-life pavement designs. Reclaimed asphalt pavement-derived binder (RAP) was used in conjunction with atypical binders from Alberta possessing a low grade span to provide economical designs with improved life cycle costs and environmental benefits.

The end users of the research products/outcomes will be transportation agencies from around North America and beyond. By using Alberta oil sands-derived asphalt cements, municipal, provincial, and state highway agencies will be able to appreciate enhanced performance and reduced life cycle costs for both newly constructed and rehabilitated flexible asphalt pavements. Highways constructed with superior asphalt binders low in wax and high in phase stability provide enhanced quality and durability with less need for maintenance.

A first project goal was to compare a wide range of Alberta oil sands-derived asphalt binders with those sourced from elsewhere. The appraisal was in terms of performance-based properties relevant to the four main pavement distresses — namely rutting, fatigue cracking, thermal cracking, and moisture damage. It has been shown that when enhanced binder grading methods are utilized — based on the measurement of relaxation properties such as m-value and phase angle of equilibrated binders — that most Alberta sources outperform those from elsewhere. This is due to the low amount of wax found in the Alberta crudes leading to homogeneous and durable materials. Currently used Superpave™ specifications fail to reveal these benefits due to ranking protocols that test binders under non-equilibrium conditions providing an unfair advantage to inferior quality materials.

A second goal was to investigate the use of solvent de-asphalting (SDA) residues for the upgrading of atypical asphalt binders to meet commercial Superpave grades, and to produce materials with higher stiffness for base and binder course pavement layers. It has been shown that small quantities of SDA can upgrade atypical binders with low grade spans to commercial grades. Add too much asphaltene and the grade spans will increase at the cost of a loss in durability.

A third project goal was to develop and investigate innovative, atypical binders which can handle higher binder replacement levels from RAP without detrimental effects on performance. Such novel compositions will benefit from reduced cost as compared to regular hot mix asphalt (HMA) containing little or no RAP. The materials investigated included atypical binders with low grade spans from Athabasca and Peace River sources and were found to produce rheological performance properties nearly identical to those obtained for straight Cold Lake binder yet when modified with up to 20 percent RAP. As such these materials will be used at a lower cost and are anticipated to produce flexible pavements with enhanced life cycles.

A final project goal was to demonstrate that most Alberta derived materials from Athabasca, Cold Lake and Peace River sources can perform well in asphalt mixture tests. Hamburg testing was conducted to assess both rutting and moisture resistance. It was found that binders are easily modified with anti-strip additives to improve moisture resistance and with reclaimed polyethylene terephthalate (PET) fiber to considerably improve rutting and cracking resistance. The aggregate gradation and quality was also found to have an overbearing impact on rutting performance.

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## **A. INTRODUCTION**

### **Sector Introduction**

The asphalt paving industry in North America and beyond uses asphalt cement — also known as “asphalt binder”, “bitumen” or simply “binder” — for the construction of high quality flexible pavements. A typical pavement consists of several layers of hot-mix asphalt (HMA) that is compacted to an air void content anywhere between 4 and 8 percent by volume. Binder contents typically range from 5 percent by weight of the aggregate for dense graded designs to around 7 percent for gap graded designs. The aggregate gradation and binder content are adjusted in order ensure that the aggregate is not overfilled, as this would invariably lead to flushing and early rutting in service. An appropriately under-filled sand or stone skeleton provides the load bearing and spreading capacity to prevent the subgrade from being overloaded.

Binders provide flexibility and resistance to moisture to obtain sufficient durability and a resultant long life cycle. Pavement thickness is largely determined by the load bearing capacity of the underlying layers (lower stiffness subgrades require thicker pavement structures), and the traffic level and percentage of commercial trucks (heavier traffic requires thicker pavement structures).

In Ontario and much of the rest of Canada and the northern USA, typical pavement designs consist of two layers of HMA for a total thickness of around 9-10 cm. In and around urban areas this can increase to three layers at around 15 cm, while on busy parts of divided freeways it can go as high as six layers for a total thickness of around 30 cm or more. Typical life cycles range from 10-15 years for the busiest of highways, to over 40 years for secondary roads in residential areas with very low traffic and/or for municipalities that are strapped for funds.

The approximate consumption of asphalt binder in Ontario is on the order of 1 million tonnes annually, in North America it is close to 30 million tonnes, whereas worldwide it approaches 100 million tonnes. Hence, asphalt road construction is a major end use application for the heavy crude oil fraction.

### **Knowledge and Technology Gaps**

Pavement design is largely an empirical exercise with historical expertise setting the limits for pavement thickness, aggregate type and gradation, and binder grade (stiffness, pliability and adhesive qualities). Attempts have been made to provide a mechanistic design protocol but these have often had mixed success with resultant premature failures being regularly reported in both the scientific and popular media. Current North American asphalt cement production practices are largely guided by a set of specifications known as Superpave. The main problem with the specification relates to the fact that binders of identical grades can perform vastly different. Superpave has proven to be entirely a purchase specification, i.e. one has to have trust in the seller. Material properties measured as part of Superpave have little to do with long-term pavement performance and therefore the use of this specification involves a great deal of risk for buyers. In order to address these problems, our research over the last 15-20 years at Queen’s University, in collaboration with several Ontario user agencies, has been focussed on

developing specification methods that reward better quality and durability. These enhanced methods clearly show the benefits of Alberta oil sands-derived binders.

For illustration, trial sections constructed by the Ministry of Transportation of Ontario (MTO) in northern Ontario, and designed with asphalt binders of identical PG 64-34 Superpave grades, have performed on opposite sides of the spectrum. Figure 1 shows representative photographs for Section 1 and Section 4 on a Highway 655 trial north of Timmins, Ontario, as these appeared in September 2019.



**Figure 1.** Comparison of best Section 1 (left) and worst Section 4 (right) performing PG 64-34 binders on Highway 655. Note: Construction was completed in late 2003 and photographs taken 16 years later.

The image on the left is for Section 1 made with superior quality Lloydminster base asphalt modified with a very small quantity of reactive ethylene terpolymer (1.0 percent RET) to a grade of PG 64-34. In contrast, Section 4 on the right was made with styrene-butadiene-styrene (SBS) modified asphalt cement tainted with recycled engine oil bottoms (REOB) to produce an identical PG 64-34 grade under Superpave. The reason for why the SBS/REOB-tainted material performed so much worse relates to the insufficient conditioning at both high and low temperatures during specification grading. High temperature aging in the pressure aging vessel (PAV) is used in the specification protocol to mimic long-term chemical aging during construction and service. Low temperature conditioning in the bending beam rheometer (BBR) is used to mimic exposure to cold temperatures during winter months. Binders with inferior performance attributes are at an advantage under the Superpave specifications as they would age (deteriorate) more during extended conditioning compared to superior materials. It has been reported that, until recently, about 50 percent of asphalt cement sold in Ontario was tainted with REOB, while for the USA it was around 30 percent. It is unclear what current contamination levels are today.

Figure 2 shows September 2019 images of Section 9, constructed on Highway 655 in 2007 with superior Cold Lake base asphalt cement modified with 0.3 weight percent of recycled PET fibers on the hot mix asphalt (HMA). Figure 3 shows representative images of a full scale contract constructed in 2011 on Highway 17 north of Algonquin Park in northeastern Ontario. The sections on Highway 655 are exposed to temperatures below  $-40^{\circ}\text{C}$  on regular occasions. The Highway 17 contract was exposed to an air temperature of  $-37^{\circ}\text{C}$  once but gets below  $-30^{\circ}\text{C}$  regularly.



**Figure 2.** Representative photographs of 12-year-old, PET fiber-modified Cold Lake asphalt Section 9 on Highway 655 north of Timmins, Ontario (base asphalt is 300/400 pen or PG 46-34).



**Figure 3.** Representative photographs of an 8-year-old, 10 km long contract on Trans-Canada Highway 17 north of Algonquin Park constructed with base asphalt cement produced by a Montreal, Quebec refinery using crude oil derived from Alberta oil sands.

Another problem directly linked to this issue is the fact that mixes containing RAP are overrated due to the inability of the Superpave bending beam rheometer (BBR) protocol to detect the presence of poor compatibility. RAP contents of up to 20 and 30 percent can readily pass regular BBR criteria for which the samples are only conditioned at cold temperatures for a single hour, but fail the extended 72-hour cooling protocol with losses of up to one or sometimes even two full grades (6 °C-12 °C). A grade loss of 6 °C typically reduces the confidence that a particular road is not exposed to damaging temperatures in a given winter from the design of 98 percent to around 50 percent. Hence, grade losses in the 6 °C-12 °C range can have devastating consequences for life cycles.

Figure 4 provides representative photographs for stretches of Princess Street in downtown Kingston, Ontario. The two images on the left are from three blocks of reconstructed pavement where the upper binder course was allowed to contain up to 15 percent of RAP. No RAP was permitted in the surface lift. This section of pavement is 10 years old today. In contrast, on the right are two representative images of the remaining seven blocks of pavement constructed 8-9 years ago. For this part of the contract the extended 72-hour cooling protocol for extracted and recovered binder properties was used for

acceptance. It is obvious that the way in which binders are tested and specified for acceptance into a contract can have an important impact on long-term pavement performance. This particular example shows how small quantities of RAP in lower pavement layers can have significantly detrimental impact on long-term pavement performance if the base binder is not adjusted to accommodate reclaimed material.



**Figure 4.** Representative photographs of a 10-year-old stretch of Princess Street in downtown Kingston, Ontario (two left images) and an adjacent 9-year-old stretch (two right images).

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## B. PROJECT DESCRIPTION

### Knowledge and Technology Description

As stated earlier, asphalt binders are currently graded in most of North America according to Superpave methodology which was developed in the late 1980s through research funded by the United States government. Superpave grades asphalt binders according to their high-, intermediate- and low-temperature rheological properties, producing binder grades denoted by PG XX-II-YY. At high temperatures, the performance grade, PG XX, is determined by the temperature where the complex modulus,  $G^*$ , divided by the sine of the phase angle,  $\delta$ , reaches a limit of 1.0 kPa for the unaged binder or 2.2 kPa for the Rolling Thin Film Oven (RTFO) residue. The lower of the two limiting temperatures sets the grade, HTPG XX. At intermediate temperatures, the performance grade, ITPG II, is set at the temperature where the  $G^*$  times sine  $\delta$  reaches 5.0 MPa, with lower grade temperatures being more desirable to avoid cracking. It has been found that the ITPG II fails to correlate with fatigue cracking performance largely due to the fact that it confounds the energy lost through viscous and damage mechanisms. At low temperatures, the grade, LTPG YY, is set at the warmer of the temperatures where the creep stiffness in bending after 60 seconds of loading,  $S(60\text{ s})$ , reaches 300 MPa, or where the slope of the creep stiffness master curve, the so-called  $m(60\text{ s})$ -value, reaches 0.300. The LTPG YY has failed to correlate to thermal cracking performance largely because it is determined after conditioning for only a one hour period at 10 °C above the low temperature pavement design temperature.

The fact that Alberta oil sands produce top quality asphalt cements is well known among the oil companies in the business of selling asphalt. However, as all the major oil companies are largely transnational operations that span the globe, it has not ever been easy to have local crudes be displaced by heavy crude from Alberta, given the fact that more than just asphalt is produced in a refinery. Hence, it is important to generate additional knowledge about the superior quality attributes of Alberta asphalt cements and publicize this through conference presentations, journal articles, and visits to user agencies. It will largely be the user agencies that have to drive up the demand and price of superior quality asphalt binder from Alberta.

The objectives and scope of the research reported here is to increase the knowledge base on the use of Alberta asphalt in various climates around the world in the short term. Regular Superpave and enhanced grades for Alberta binders are contrasted with those for materials sourced from around the world. Alberta-derived material properties are contrasted against those obtained for competing sources in the context of binder replacement through the recycling of RAP and the use of SDA asphaltenes. Finally, superior performance of Alberta-derived asphalt cements is demonstrated in mixture tests under controlled environments.

### Performance Metrics

The project has generated a significant amount of new knowledge related to the superior performance properties of asphalt derived from Alberta oil sand crudes.

Selected results have been published in three conference publications (2019 and 2020 *Proceedings of the Canadian Technical Asphalt Association Annual Meeting*, Montreal and Regina), and an additional five top-quality, refereed journal publications (*Fuel* and *Construction and Building Materials*). It is anticipated that an additional six to eight refereed journal publications will follow (four are nearly ready for submission while additional material related to aligned mastic test results is available for several more publications).

The project has trained a significant number of highly qualified personnel. In total, three postdoctoral fellows, two M.Sc., and seven B.Sc. students have worked on the research from the beginning of 2019 till the end of 2020. Aligned research on asphalt mastics has involved an additional two Ph.D. students at Tallinn University of Technology in Estonia.

The project has developed a new variable temperature Fourier-transform infrared (VT-FTIR) spectroscopic technique and has taken another look at both modulated differential scanning calorimetry (MDSC) and solid state nuclear magnetic resonance spectroscopy (NMR) for the accurate and practical measurement of solid wax at cold temperatures in asphalt binder. The measurement of wax provides insights into long-term pavement performance and thermoreversible aging effects that degrade stress relaxation properties (m-value and phase angle). Because Alberta-sourced binders are low in wax this will benefit the oil sands industry. Wax content determination and eventual specification provides a competitive advantage for Alberta binders.

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## C. METHODOLOGY

All experimental work in this project was conducted in the asphalt laboratories at Queen's University in Kingston, Ontario. The laboratory is well equipped for the testing and evaluation of straight and modified asphalt binder for road construction. Methods employed included those that were specifically developed to resolve the shortcomings associated with the Superpave methodology (EBBR, DENT and phase angle measurements). Figure 5 provides images of all binder instrumentation that was used in the work while Figure 6 provides images for the mixture instrumentation.



Rolling Thin Film Oven (RTFO)



Pressure Aging Vessel (PAV)



Bending Beam Rheometer (BBR)



Dynamic Shear Rheometers (DSR)



Modulated Differential Scanning Calorimeter (MDSC)



Double-Edge-Notched Tension (DENT) Tester



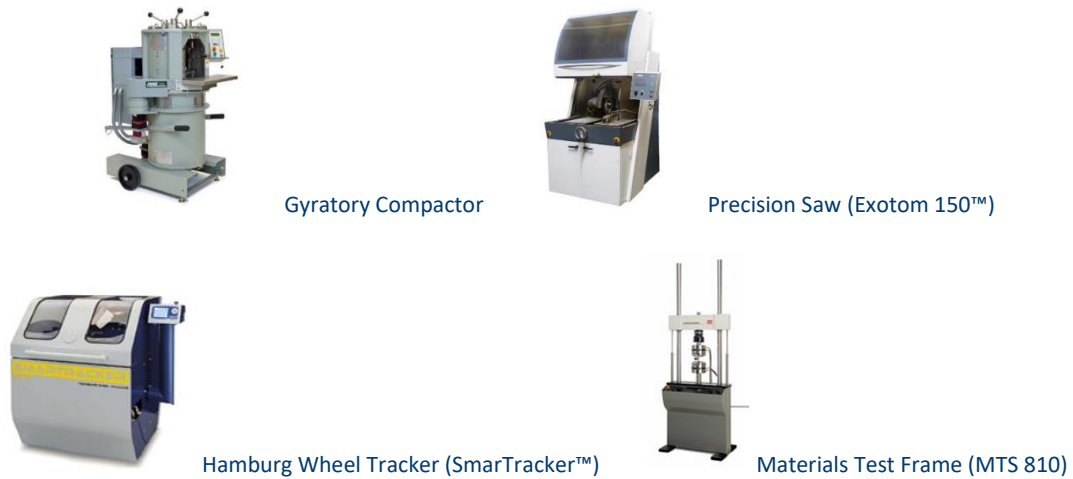
Nuclear Magnetic Resonance (NMR) Spectrometer



Fourier-Transform Infrared (FTIR) Spectrometer

**Figure 5.** Equipment used to assess binder quality and durability.





**Figure 6.** Equipment used to assess mixture performance.

Asphalt binder tests were conducted according to standard procedures as described in the following AASHTO test standards:

- American Association of State, Highway and Transportation Officials, AASHTO T 240, Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test) (2010).
- American Association of State, Highway and Transportation Officials, AASHTO R28, Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV) (2010).
- American Association of State, Highway and Transportation Officials, AASHTO M320 - Performance-Graded Asphalt Binder, 2010.
- American Association of State, Highway and Transportation Officials, AASHTO TP 113-15, Determination of Asphalt Binder Resistance to Ductile Failure Using the Double-Edge-Notched Tension (DENT) Test. AASHTO, Washington, D.C., 2015.
- American Association of State, Highway and Transportation Officials, AASHTO TP 122-16, Determination of Performance Grade of Physically Aged Asphalt Binder Using Extended Bending Beam Rheometer (BBR) Method. AASHTO, Washington, D.C., 2016.

Asphalt mixture tests were conducted according to standard procedures with minor modification to loading rate for the semi-circular bend test. Details can be found in the following publications:

- American Association of State, Highway and Transportation Officials, AASHTO TP 124 Standard Method of Test for Determining the Fracture Potential of Asphalt Mixtures Using the Flexibility Index Test (FIT), 2019.
- American Association of State, Highway and Transportation Officials, AASHTO T 324 Standard Method of Test for Hamburg Wheel-Track Testing of Compacted Asphalt Mixtures, 2019.

## D. PROJECT RESULTS

On a task by task basis the following has been accomplished:

### **Task 1 – Material Sourcing**

A large number of bitumen and asphalt cement samples were obtained from suppliers and user agencies from around the world. Table 1 provides a listing of the Alberta crude oil (bitumen) sources for which samples were obtained while Tables 2 and 3 provide a listing of all asphalt cement samples sourced directly from Canada and abroad, respectively. Note that the supplier’s name and crude source information was subjected to confidentiality agreements and/or was uncertain for a number of the materials.

Six of the bitumen samples from Table 1 were distilled to one, two or three different cut points to produce the first set of asphalt binders as listed in Table 4. An additional 10 asphalt binders were prepared from the remaining two Alberta oil sands-derived bitumen sources (five cut points each). The testing of these last 10 binders concluded the regular binder testing part of this project.

**Table 1. Listing of All Crude Oils Sourced from Alberta**

Bitumen Source	Comment
<i>Athabasca, Alberta</i>	<i>Steam-Assisted Gravity Drainage (SAGD)</i>
<i>Peace River, Alberta</i>	<i>Cyclic Steam Stimulation</i>
<i>Athabasca, Alberta</i>	<i>Conventional Oil Recovery (Water Flooding)</i>
<i>Cold Lake, Alberta</i>	<i>Cold Production of Heavy Oil with Sand (CHOPS)</i>
<i>Athabasca, Alberta</i>	<i>Open-Pit Mining/High-Temperature PFT</i>
<i>Athabasca, Alberta</i>	<i>Steam-Assisted Gravity Drainage (SAGD)</i>
<i>Athabasca, Alberta</i>	<i>Steam-Assisted Gravity Drainage (SAGD)</i>
<i>Cold Lake, Alberta</i>	<i>Steam-Assisted Gravity Drainage (SAGD)</i>

**Table 2. Listing of All Asphalt Cements Sourced from Alberta, Ontario and Quebec**

Sample	Crude Source	Grade	Comment
<b>1</b>	<i>Western Canadian</i>	<i>58-28</i>	<i>Produced with Alberta crude in Ontario.</i>
<b>1</b>	<i>Western Canadian</i>	<i>58-28</i>	<i>Produced with Alberta crude in Quebec.</i>
<b>2</b>	<i>Western Canadian</i>	<i>58-28</i>	<i>Unknown source of asphalt cement sourced in Ontario.</i>
<b>3</b>	<i>Cold Lake</i>	<i>46-34</i>	<i>Produced with Cold Lake crude in Alberta refinery.</i>
<b>4</b>	<i>Cold Lake</i>	<i>58-28</i>	<i>Produced with Cold Lake crude in Alberta refinery.</i>
<b>5</b>	<i>Cold Lake</i>	<i>64-22</i>	<i>Produced with Cold Lake crude in Alberta refinery.</i>

**Table 3. Listing of All Asphalt Cements Sourced from Around the Globe**

Sample	Bitumen Source	Grade
1	Middle Eastern	VG 10
2	Middle Eastern	VG 10
3	Middle Eastern	VG 20
4	Unknown	70 pen
5	Unknown	PG 76-28
6	Unknown	70 pen
7	Unknown	70 pen
8	Unknown	70 pen
9	Russian	70-100 pen
10	Russian	160-200 pen
11	Russian/Offshore	70-100 pen
12	Russian/Offshore	160-200 pen
13	Venezuelan	70-100 pen
14	Venezuelan	160-220 pen
15	Russian	70-100 pen
16	Russian	160-200 pen
17	Kuwait	50-70 pen
18	Kuwait	150-220 pen
19	Kuwait	250-330 pen
20	Offshore	70-100 pen
21	Offshore	50-70 pen

Note: Listed grades were as reported by the suppliers.

**Table 4. Distillation of Alberta Oil Sands-Derived Crudes**

Source	Distillation Yield, %	XX-YY Grade, °C
<i>Athabasca</i>	34.6	53-32
<i>Peace River</i>	40.7	52-30
<i>Athabasca</i>	41.2	48-37
<i>Cold Lake</i>	26.8	47-37
<i>Athabasca</i>	44.5	51-30
<i>Athabasca</i>	31.1	58-26
<i>Athabasca</i>	15.2	50-32
<i>Athabasca</i>	22.0	63-26

### **Task 2 – Spectroscopy**

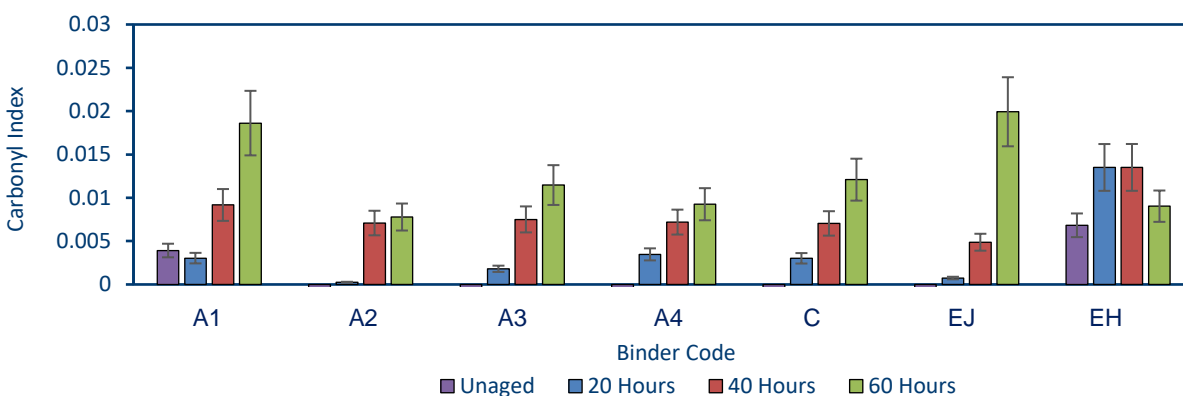
A large number of Alberta and Northern European binders were investigated with both infrared (FTIR) and nuclear magnetic resonance (<sup>1</sup>H-NMR) spectroscopy. The focus of this task was to obtain a better understanding of how the various laboratory aging protocols affect the formation of carbonyl and sulfoxide functional groups and how these in turn affect rheological performance. Athabasca, Venezuelan, and Polish binders were aged at two different film thickness (1.5 mm and 4.5 mm) for two different times (20 h and 60 h) in the PAV.

Liquid cell FTIR data showed that the absolute methylene peak area stays relatively constant for extended times in the PAV (20 and 60 hours, 100 °C and 2.08 MPa) compared to the unaged binder. Table 5 provides the changes in peak area for all investigated binders. A rough estimate for the error in these measurements would be plus or minus 20 percent. The highest change was found for one of the binders from Athabasca. Hence, it is suggested that the methylene peak can be used as an internal standard for FTIR spectra taken in the solid state on KBr disks.

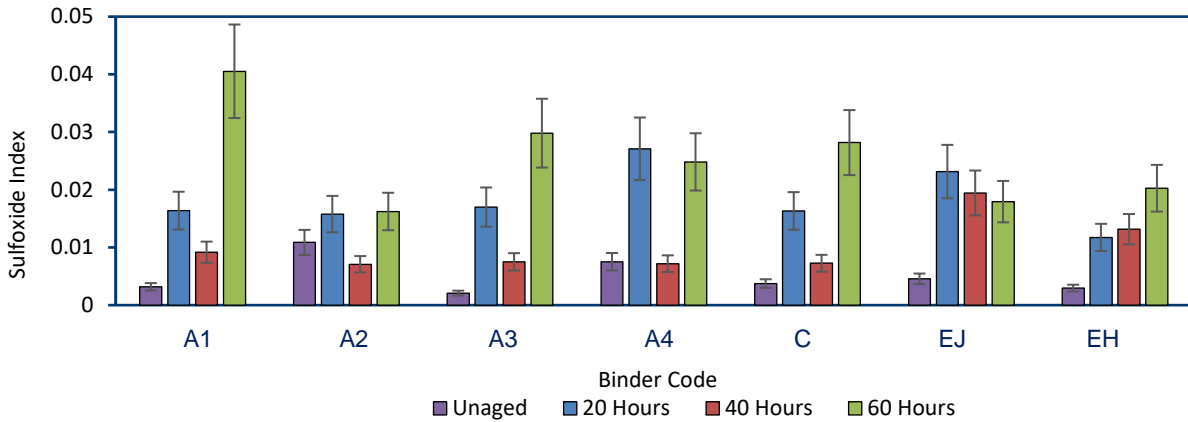
**Table 5. Absolute CH<sub>2</sub> Peak Area Percent Change**

Sample	UN to 20 h PAV @ 1.5 mm, %	UN to 60 h PAV @ 4.5 mm, %
<i>Polish</i>	3	3
<i>Venezuelan</i>	2	1
<i>Athabasca 1</i>	3	2
<i>Athabasca 2</i>	4	9
<i>Athabasca 3</i>	5	2
<i>Cold Lake</i>	11	15
<i>Athabasca 4</i>	21	6

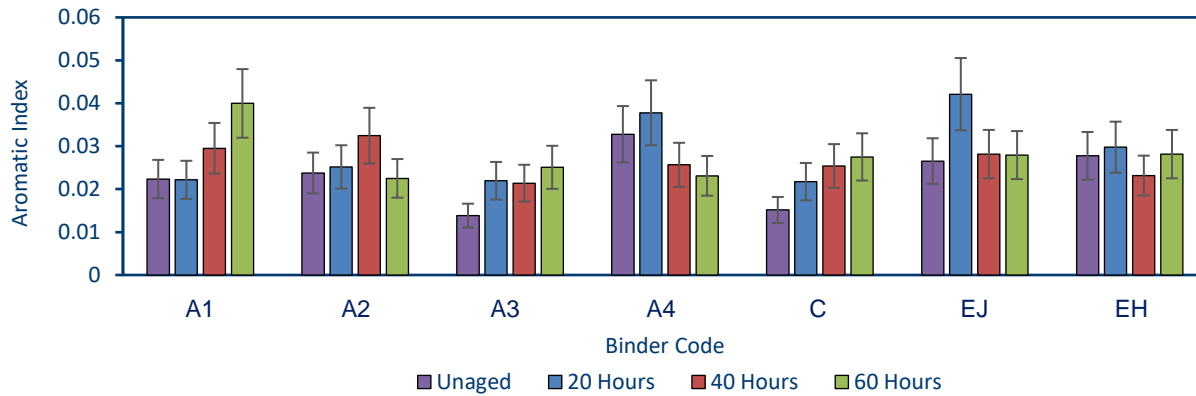
A large number of samples were analyzed for carbonyl, sulfoxide, and aromatic peaks by using measurements through liquid cells and solid films on KBr disks. The findings for the solid film spectra are provided in Figures 7-9.



**Figure 7.** Carbonyl indices for Athabaskan (A1-A4), Cold Lake (C), Polish (EH) and Venezuelan (EJ) binders after PAV conditioning for 0, 20, 40, and 60 hours.



**Figure 8.** Sulfoxide indices for Athabasca (A1-A4), Cold Lake (C), Venezuelan (EJ) and Polish (EH) binders after PAV conditioning for 0, 20, 40, and 60 hours.



**Figure 9.** Aromaticity indices for Athabasca (A1-A4), Cold Lake (C), Venezuelan (EJ) and Polish (EH) binders after PAV conditioning for 0, 20, 40, and 60 hours.

The above spectroscopic data show that the Athabasca binders chemically age in a similar fashion to the Polish and Venezuelan materials. Differences between the sources are not very significant and the degree of oxidation appears to be mainly influenced by the time in the PAV. The Venezuelan binder EJ starts with a small carbonyl peak of uncertain origin. It is possible that the contractor added a fatty acid amine-based antistripping additive. These are known to slowly evaporate from hot asphalt cement, which could explain the decrease in the carbonyl peak with increased aging times. NMR spectroscopic testing of these materials presented similar findings in that the binders do not appear to differ by especially significant amounts.

### **Task 3 – Standard Aging**

The purposely distilled materials as well as all the binders acquired from commercial sources (Tables 2 and 3) were aged according to standard rolling thin film oven (RTFO) and pressure aging vessel (PAV)

protocols. A total of 42 binders were aged and tested for low-, intermediate- and high-temperature performance properties according to standard Superpave and enhanced Ontario testing protocols.

#### ***Task 4 – Performance Grading***

Binders aged under Task 3 were tested according to regular AASHTO M 320 (Superpave) and enhanced AASHTO TP 122-16 (extended bending beam rheometer (EBBR)) and AASHTO TP 113-15 (double-edge-notched tension (DENT)) specifications. Binders were analyzed according to their limiting phase angle temperatures and grade spans. Important results are provided in Table 6 and Figures 10-16.

Binders 1-10 and 33-42 were produced entirely with Alberta crudes from single sources, while 11 and 13 were made with Alberta crude, likely predominantly Cold Lake and/or Western Canadian Select, transported by pipeline to refineries in Ontario and Quebec. Binder 12 was obtained from a contractor who could have blended materials produced in various locations, including the USA, or may have bought this from a local refinery. Binders 14-16 were made using exclusively Middle Eastern crude. Binders 17-22 were made in China and India with what likely were blends of various crudes from the Middle East, China and likely other sources. Binders 23-26, 28, 29, 31 and 32 were likely made with blends of crudes from Russia, the Middle East and possibly other sources. Binders 27 and 30 were made with 100 percent Venezuelan crude in Sweden. Binders 33-38 were made from a single Cold Lake crude (bitumen source A) from a second supplier while binders 39-42 were made from a single Athabaskan crude (bitumen source B) from the same supplier.

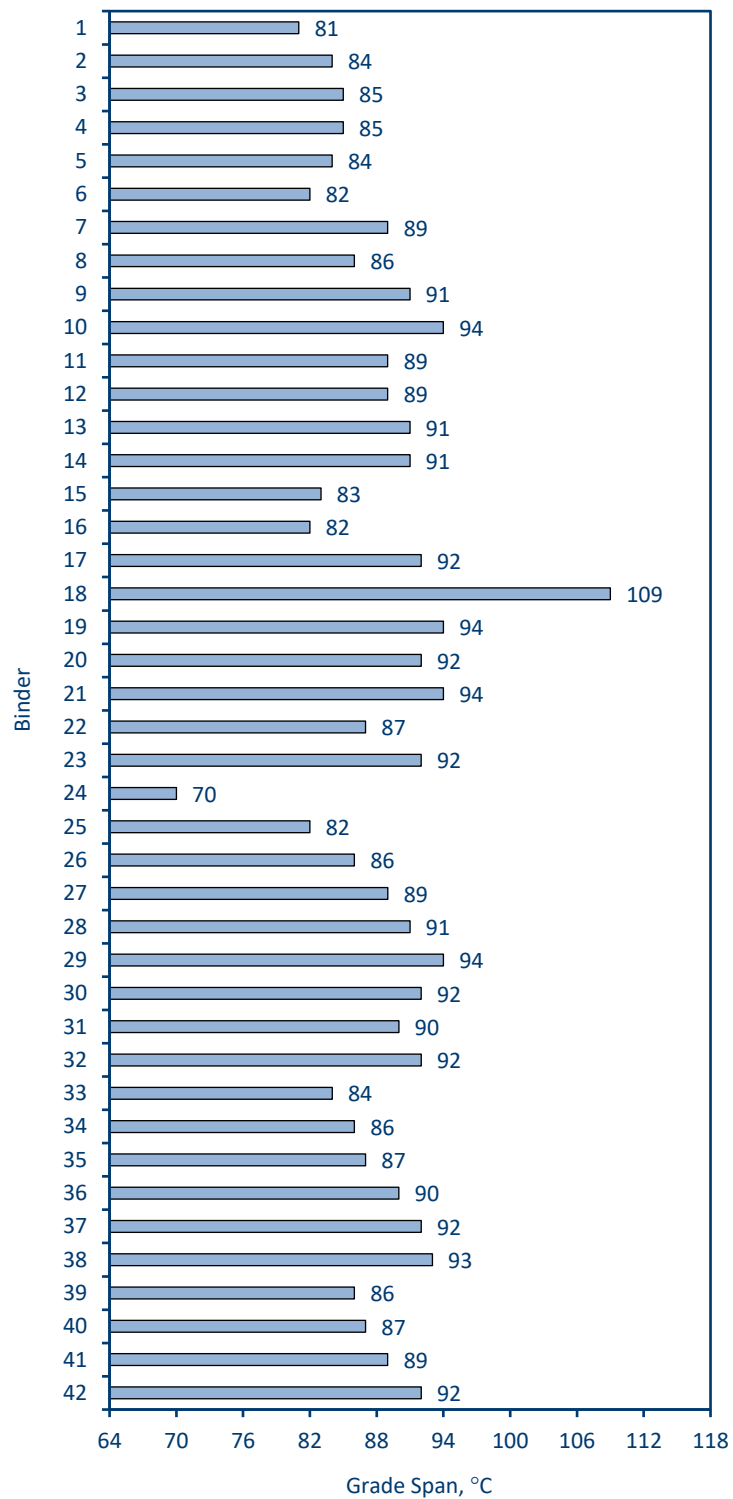
The AASHTO M 320 grade spans XX-YY are presented in Figure 10. Excluding the two outliers, the average grade span is 88.2 °C with a standard deviation of  $\pm 4.0$  °C. The Alberta oil sands-derived materials 1-13 and 33-42 rank reasonably well yet not spectacularly in the regular Superpave grading. However, real world experience has shown that this measure must lack accuracy given the fact that binders of nearly identical grade can perform so differently. This discordance between grade and actual performance is largely due to the fact that Superpave lacks a solid measure of phase stability (durability). Hence, it is useful to consider the grade losses after storage for long periods at cold and warm temperatures.

The results for binders 33-38 (bitumen source A) and 39-42 (bitumen source B) show that the grade span for these materials increases with higher cut points for the distillation. It is likely that both quality and durability improves somewhat with harder binders as the softer material contains a significant amount of low molecular weight aromatics. The lower molecular weight aromatics fraction makes the material softer but can also make it more prone to oxidative hardening.

Extended BBR losses are given in Figure 11, which shows that the materials from Alberta are generally more stable and are thus expected to be less sensitive to cold temperature cracking. The average loss for Alberta binders 1-13 and 33-42 is 1.9 °C, the average for 27 and 30 made from Venezuelan crude is 1 °C, while for the remaining 17 binders from around the globe it is 4.1 °C. While these do not appear to be very different, individual variances can be significant and this in turn explains why certain pavements do much better than others. Laguna, Venezuelan binder is generally considered to be the best in the world but with the political uncertainty in the country its ability to reliably produce this crude oil is uncertain.

**Table 6. Performance-Based Properties for Asphalt Binders**

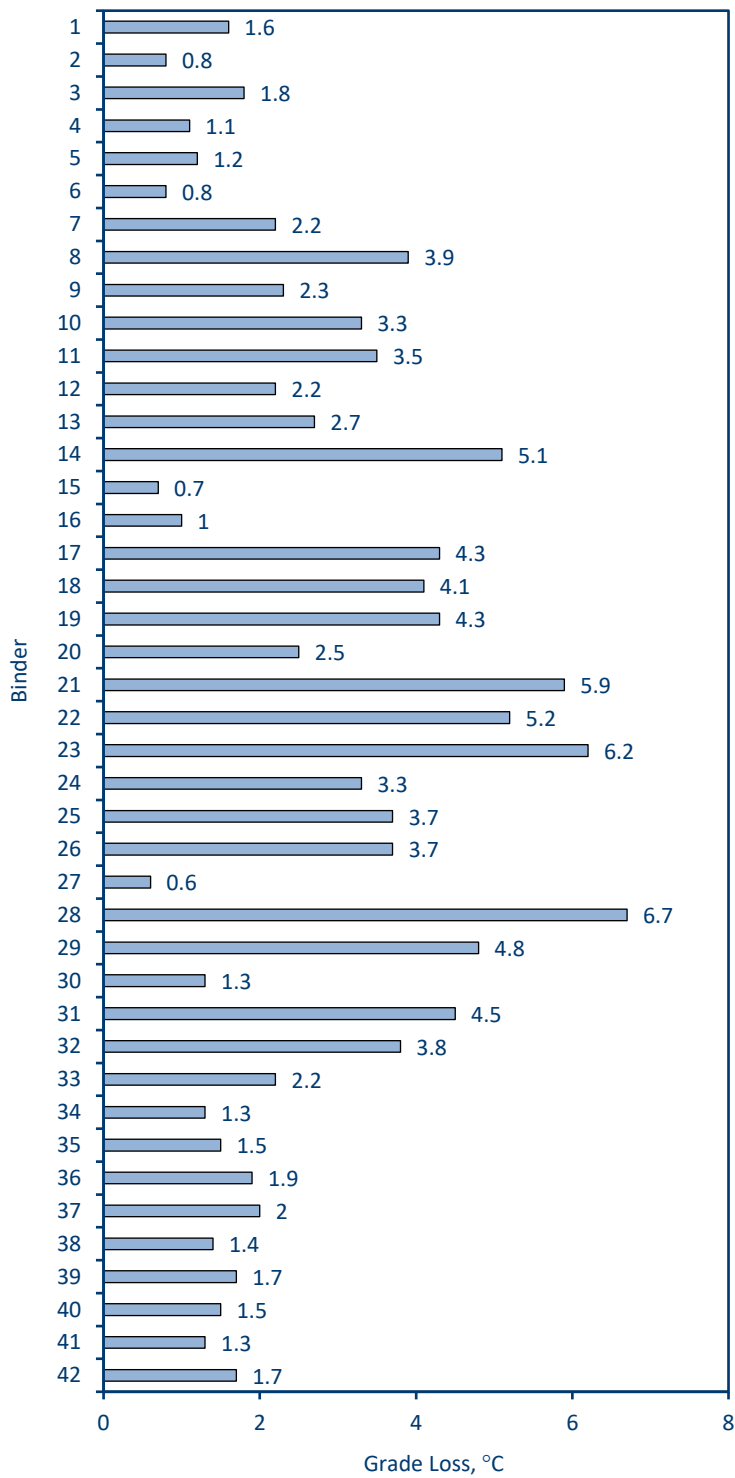
Sample	Binder Source	Superpave Grade, °C	AASHTO TP 122-16 EBBR, °C		AASHTO TP 113-15 DENT CTOD, mm
			Grade	Loss	
1	Alberta	51-15-30	-28.7	1.6	22.8
2	Alberta	58-20-26	-24.6	0.8	21.2
3	Alberta	53-15-32	-29.7	1.8	17.7
4	Alberta	48-7-37	-34.3	1.1	27.9
5	Alberta	47-6-37	-35.6	1.2	17.2
6	Alberta	50-12-32	-31.5	0.8	22.3
7	Alberta	63-20-26	-23.9	2.2	17.3
8	Alberta	50-10-36	-33.3	3.9	13.8
9	Alberta	60-17-31	-28.4	2.3	12.2
10	Alberta	66-23-28	-24.3	3.3	10.7
11	Ontario	59-17-30	-26.2	3.5	11.4
12	Ontario	60-20-29	-26.7	2.2	10.9
13	Quebec	61-19-30	-26.6	2.7	13.3
14	Kuwait	67-26-24	-19.4	5.1	10.1
15	Kuwait	56-15-27	-26.1	0.7	16.7
16	Kuwait	52-11-30	-29.2	1	20.6
17	China	66-24-26	-21.9	4.3	9.5
18	China	80-20-29	-24.5	4.1	19.8
19	China	68-24-26	-22	4.3	10.1
20	China	67-23-25	-22.2	2.5	10.8
21	China	68-23-26	-19.5	5.9	10.7
22	India	60-20-27	-21.3	5.2	14.9
23	Poland	65-24-27	-20.4	6.2	9.4
24	Poland	51-17-29	-25.7	3.3	26.7
25	Poland	53-15-29	-25.1	3.7	17.9
26	Belarus	55-11-31	-27.2	3.7	17
27	Sweden	58-12-31	-30.6	0.6	22.5
28	Poland	63-18-28	-21.6	6.7	12.3
29	Belarus	63-14-31	-25.8	4.8	12.5
30	Sweden	66-20-26	-24.9	1.3	14.2
31	Spain	63-21-27	-22.8	4.5	10.5
32	Spain	66-21-26	-22.1	3.8	10
33	Alberta	43-3.5-41	-38.7	2.2	24.5
34	Alberta	50-7.2-36	-34.3	1.4	18.4
35	Alberta	54-13-33	-31.6	1.3	18.1
36	Alberta	58-15-32	-30.1	1.5	12.2
37	Alberta	62-16-30	-28.5	1.9	15
38	Alberta	64-19-29	-26.6	2.0	11.5
39	Alberta	55-13-31	-29.0	1.7	15.9
40	Alberta	57-14-30	-28.6	1.5	17.9
41	Alberta	60-17-29	-27.5	1.3	13.4
42	Alberta	65-21-27	-25.2	1.7	Brittle Failure



**Figure 10.** AASHTO M 320 grade spans for investigated binders.

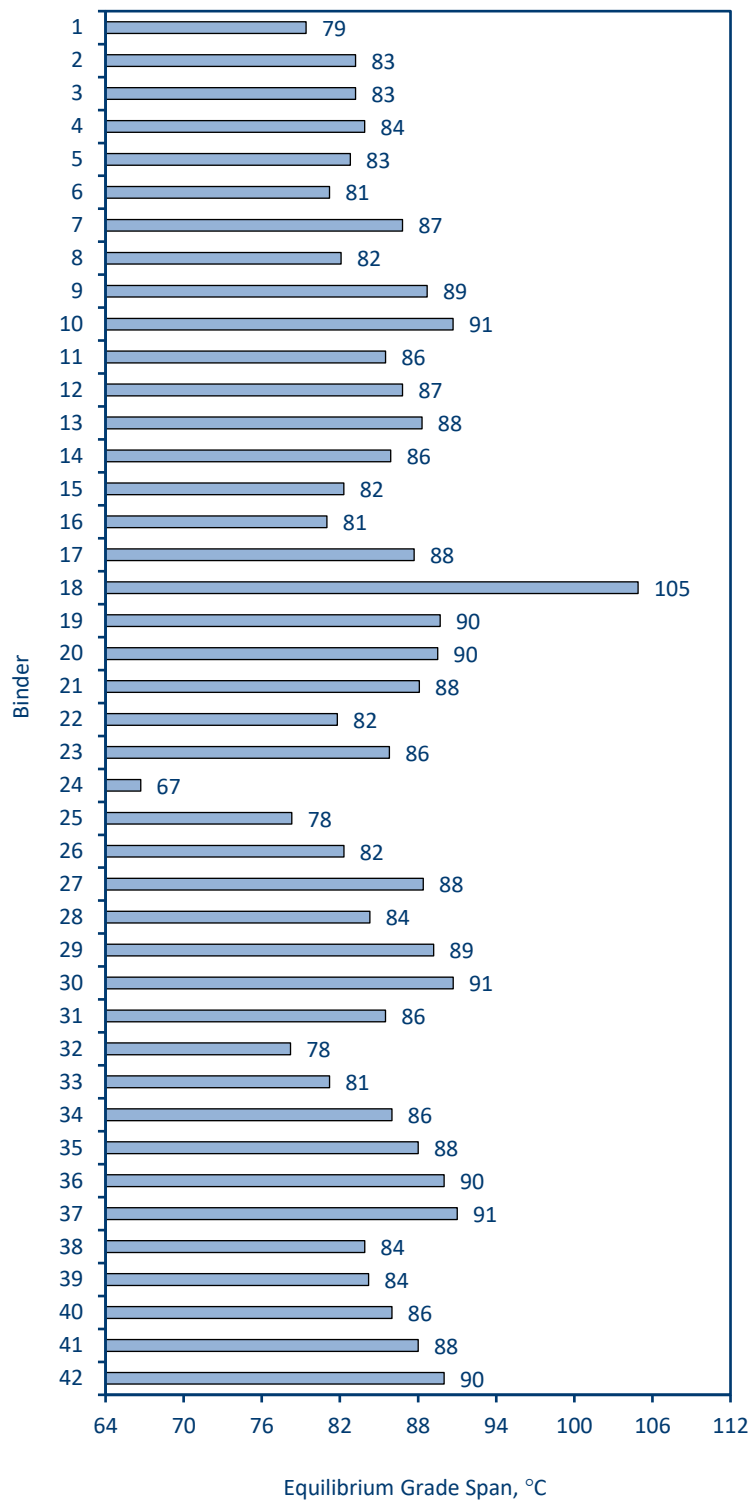
Note: Binder 18 was modified with a SBS polymer to increase its high temperature performance grade to 80 °C and a resulting grade span of 109 °C. Average grade span for all but outliers 18 and 24 is 88.2 °C with a standard deviation of  $\pm 4.0$  °C.





**Figure 11.** Extended BBR grade losses for investigated binders.

Note: A grade loss of 6 °C typically brings the confidence level that a given pavement is not exposed in any given year to damaging cold temperatures from the intended 98 percent to around 50 percent.



**Figure 12.** Grade span after 72 hours of cold conditioning.

Note: Average for all but the two outliers is 85 °C, with a standard deviation of ± 3.6 °C.

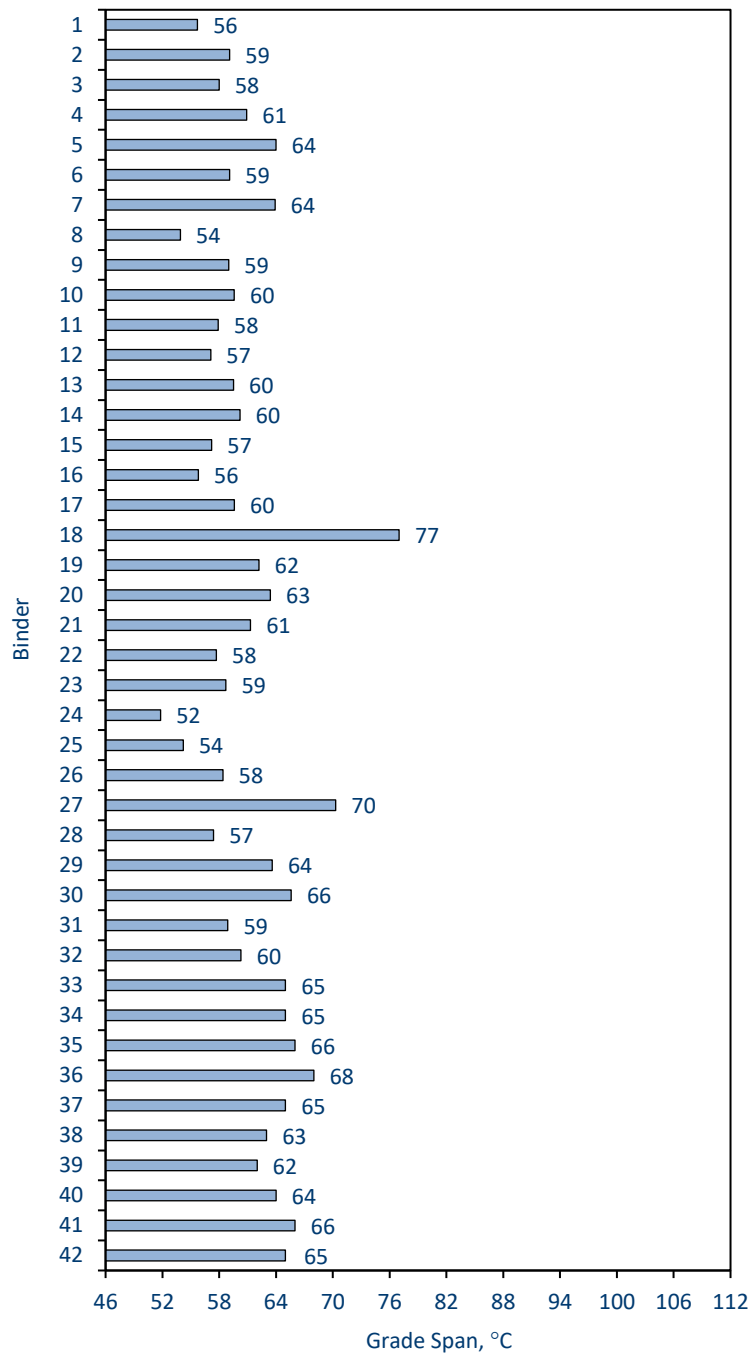
Figure 12 provides the grade spans for all binders after 72 hours of cold conditioning. It shows the benefit for the Alberta binders 1-7 and 32-42, which now come much closer to the materials from Northern Europe and Asia. The average for all but the two outliers is 85 °C, with a slightly lower standard deviation of  $\pm 3.6$  °C. The reduced variability between the sources reflects the fact that after equilibration at cold temperatures the performance grades are largely set by the BBR m-value.

As these are all hydrocarbon-based materials, it is not surprising that most materials grade close to the average. That said, the harder binders Cold Lake 10, Venezuelan 30 and Albertan 37 retain a healthy advantage with the highest equilibrated grade spans of 91 °C for the straight run materials. Other Alberta sources will likely also be able to reach such performance levels if distilled to higher cut points as softer materials typically show lower spans. However, this research had a second focus of using asphaltenes and RAP in more sustainable ways, hence softer binder grades were chosen as starting points.

A likely more accurate way to look at the useful temperature interval for asphalt binders is to consider the limiting 30° phase angle temperature, as it has been shown to correlate well (and perhaps better than the EBBR limiting temperature) with cracking severity. Figure 13 shows the relative differences between binders investigated for this project. It is clear that for the straight run materials, binders 27 and 36 perform best, with respective grade spans of 70 and 68 °C. These are binders from Venezuela and from Cold Lake, Alberta. Such materials are high in naphthene aromatics and contain a moderate amount of asphaltenes. As such they have good grade spans and suffer little from the effects of cold conditioning.

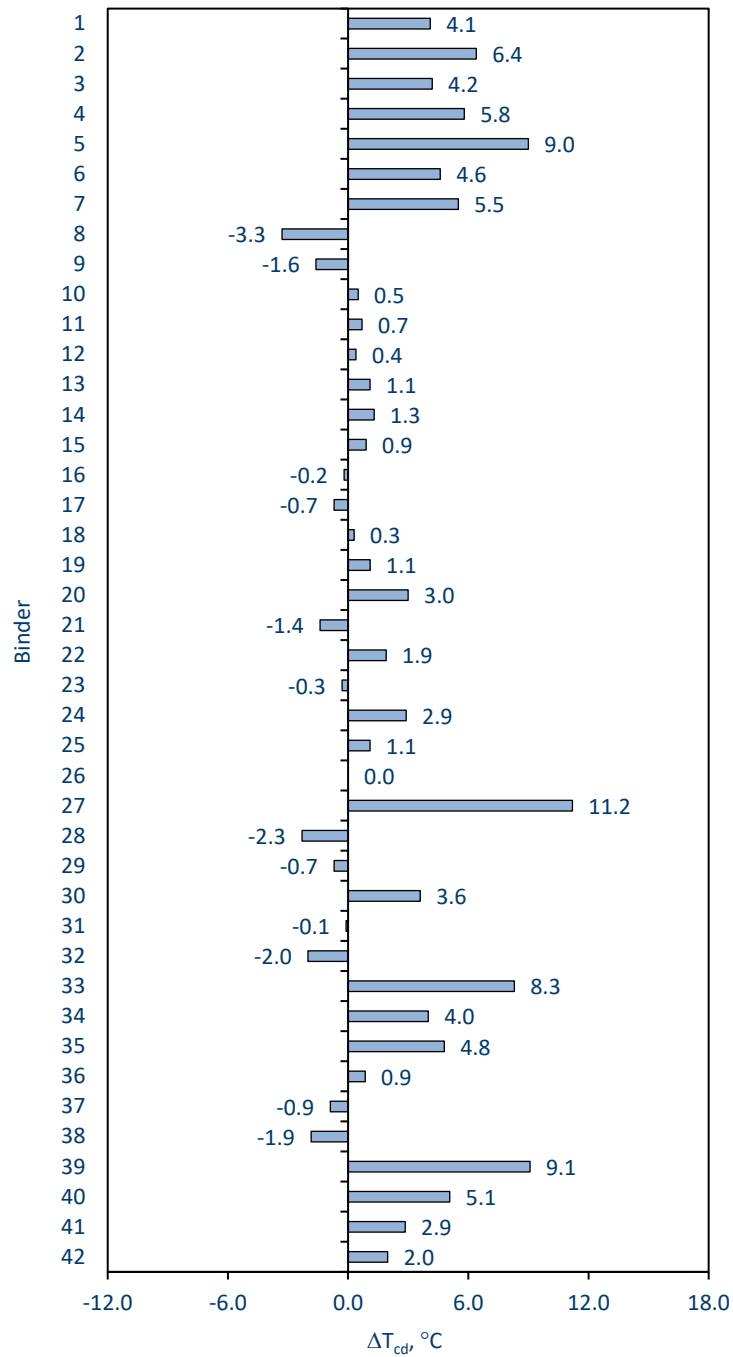
The difference in the temperature where the phase angle reaches 30° and the complex shear modulus reaches 60 MPa can be used as a measure of the curvature in the rheological master curve and phase stability. Figure 14 shows the  $\Delta T_{cd}$  temperature intervals (note that c stands for critical and d stands for DSR). Positive values for  $\Delta T_{cd}$  represent binders that are more tolerant of aging at both low and high temperatures while negative values reflect less tolerance to aging. These materials are high in naphthene aromatics and low in asphaltenes. So, when additional asphaltenes are formed during oxidative aging, or when resins precipitate during cold conditioning, the binder will not as quickly transition from the sol-type to the gel-type state and pavement cracking is delayed. Once more it is noted that the Venezuelan and Alberta binders outperform the others.

As softer binders typically have lower grade spans compared to harder materials, it is also helpful to present the data from Table 6 in a different format. Figures 13 and 14 show grading box plots for a select group of materials investigated (others are provided in Progress Report 1). Those that are located towards the upper left corner show the highest grade spans, while those that are in the lower right corner rank lowest under Superpave and Ontario grading protocols. It is obvious that the materials from Cold Lake perform amongst the best for all binders tested. The two Ontario and single Quebec binders grade below the green line but still make a good PG 58-28 grade under Superpave. Using the EBBR protocol they miss the -28 °C limit by a few degrees, which is an issue the suppliers would have to address in the months and years to come. It is noteworthy that the Athabasca binder (bitumen source B) grades almost the same as the Cold Lake materials (bitumen source A and Stratcona) when improved specification tests are used as in Figures 11 and 14. The binders that show the lowest grade spans in both Figures 13 and 14 have been modified with added asphaltenes and were investigated for recycling to investigate whether they produce end products that come closer to the top performers from Cold Lake and Venezuela.



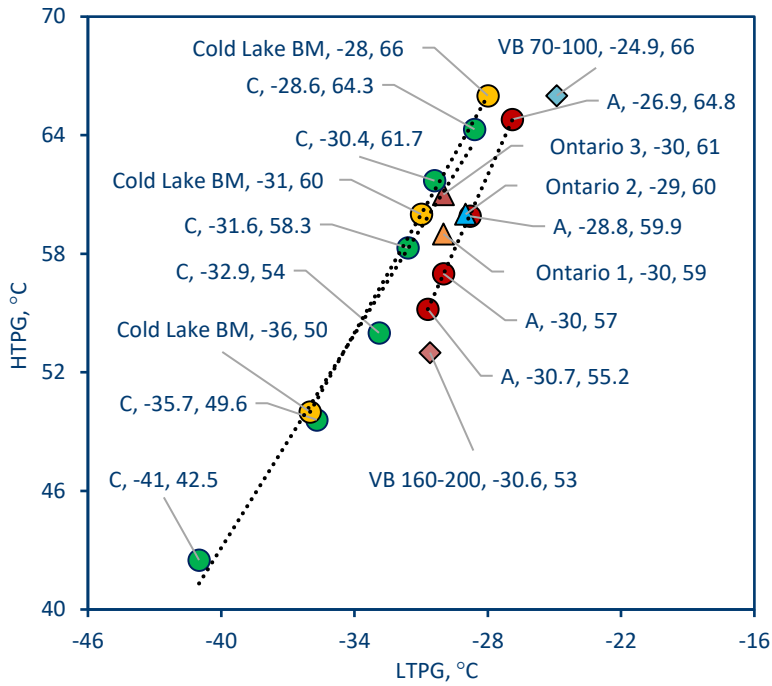
**Figure 13.** Grade intervals based on limiting 30° phase angle.

Note: Average for all but the two outliers is 61 °C, with a standard deviation of ± 3.8 °C.



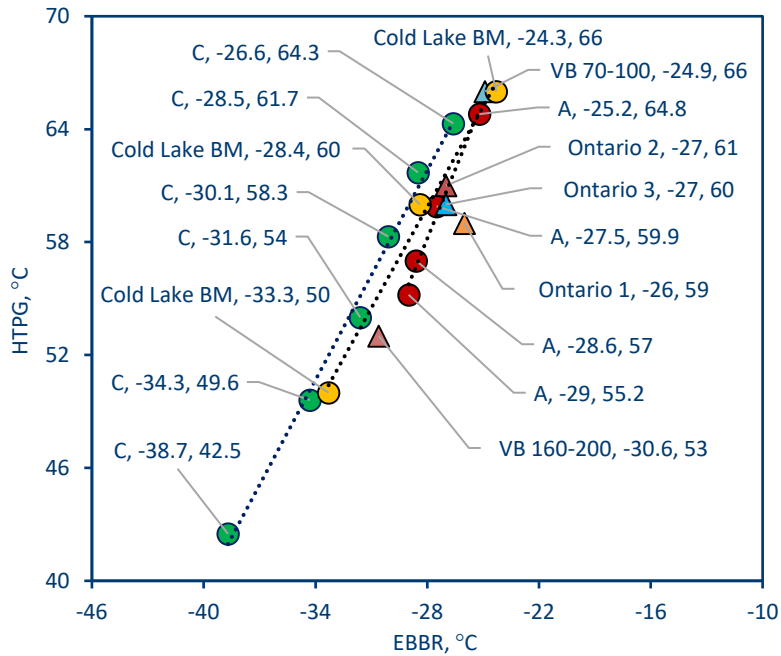
**Figure 14.**  $\Delta T_{cd}$  for limiting phase angle of  $30^\circ$  and limiting complex modulus of 60 MPa.

Note: A positive  $\Delta T_{cd}$  reflects a more stable binder while a negative value implies a less stable one.



**Figure 15.** Superpave box plot for selected binders (HTPG XX versus LTPG YY).

Note: BM = Cold Lake benchmark from Alberta, VB for Venezuelan binder, C for Cold Lake binders 33-38, A for Athabasca binders 39-42.



**Figure 16.** Performance grading box plot for all selected binders (HTPG versus EBBR LLTG).

Note: BM for Cold Lake benchmark from Alberta, VB for Venezuelan binder, C for Cold Lake binders 33-38, A for Athabasca binders 39-42.

### ***Task 5 – Modified Aging***

In addition to the standard PAV aging as discussed under Task 2, a group of 26 binders were also aged under modified PAV conditions of thinner films and longer times. Research has shown that by reducing the film thickness in the PAV or extending the aging time that inferior binders can lose a significant amount from their low temperature limiting grade. It was found that waxy and unstable binders suffer more from thinner films and aging for longer times compared to non-waxy binders.

Aging of smaller quantities in tins at a film thickness of 3.2 mm for periods of 20 hours, 40 hours and 60 hours and at 1 mm for 20 hours was conducted in the PAV. The residues so obtained were investigated with both DSR and FTIR. Results generally confirmed earlier findings that gel-type binders suffer more from extended aging and aging in thinner films as they are somewhat protected in the regular PAV protocol that uses rather thick 3.2 mm films. Thicker films allow for the formation of a thin layer of gelled binder that protects the underlying material from further oxidation through the formation of a diffusion barrier. A number of results are included in the CTAA conference paper presented in fall of 2020 and the complete data set is included in the M.Sc. thesis of Oluwatobi.

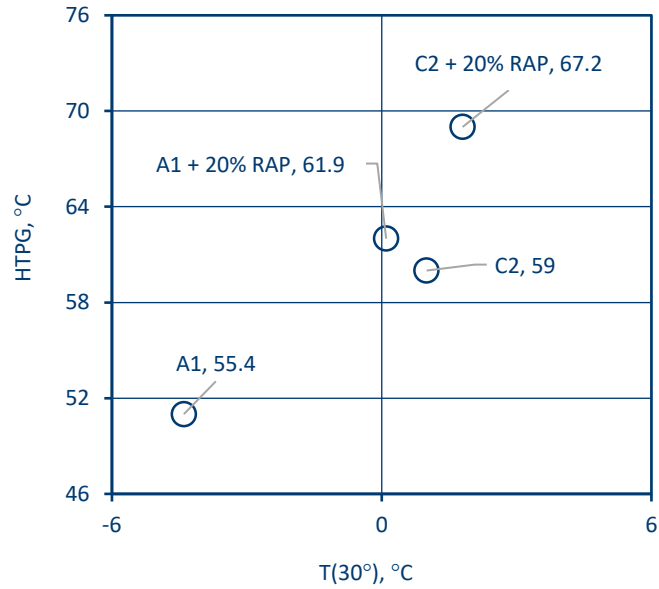
### ***Task 6 – RAP Tolerance Testing***

Both Athabasca and Cold Lake binders were modified with 20 and 40 percent of RAP from two different sources (Highway 7 and Highway 403) as well as recycled asphalt shingles (RAS) from a single source. These compositions were aged for 20 and 40 hours in the PAV prior to testing by FTIR and DSR. It appeared that both base binders were relatively tolerant of the RAP obtained from Highway 7. In contrast, they were found to be less tolerant of the RAP from Highway 403 and the RAS, as both of these were more severely oxidized. A number of results are included in a CTAA conference paper presented in fall of 2020 and the complete data set is included in the M.Sc. thesis of Nawarathna.

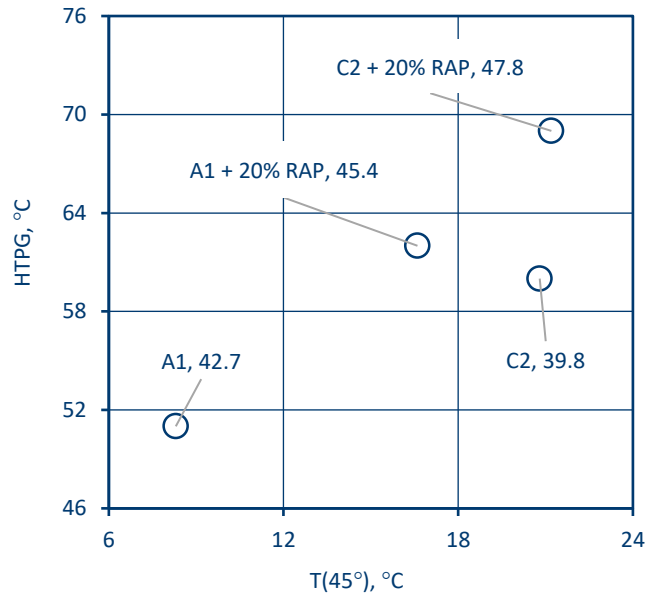
The grade span for the Cold Lake 58-28 binder reaches to very high values as the starting level is significantly higher than what it is for the Athabasca material. It will likely turn out that more moderate grade spans are desirable and that therefore the Athabasca material will do better in terms of overall pavement performance.

Figures 17 and 18 provide key results for grade spans based on Superpave high temperature performance grade, HTPG XX, and limiting phase angle grades, T(30°) and T(45°). The data shows that based on the HTPG and T(30°) performance measures, an Athabasca binder A1 modified with 20 percent RAP performs almost as good as a straight run PG 58-28 Cold Lake binder C2. The C2 modified with the same amount of RAP will possess a grade span that is significantly greater and may therefore be less flexible and more susceptible to load induced cracking.

Based on the HTPG and T(45°) performance measures, the A1 + 20 percent RAP composition will perform slightly better than the straight PG 58-28 Cold Lake C2 binder and nearly as good as the C2 + 20 percent RAP. However, there could also be subtle differences in terms of long-term chemical aging tendencies so the only way to get a higher level of confidence in this is to conduct real world pavement trials with test sections that compare these compositions side by side. Such pavement trials would need to be designed on a stretch of highway with appropriate climate and traffic level.



**Figure 17.** Performance grade box plot for HTPG versus T(30°) for straight and RAP-modified binders.



**Figure 18.** Performance grade box Plot for HTPG versus T(45°) for straight and RAP-modified binders.

**Task 7 – SDA Tolerance Testing**

Alberta base asphalt cements were modified with various quantities of asphaltenes (SDA residue). Two Cold Lake (C1 and C2) and four Athabaskan base asphalt binders (A1-A4) were modified with 5, 10 and 20 percent SDA residue. The materials were aged for various lengths of time in the PAV prior to testing in the FTIR, DSR and modulated differential scanning calorimeter (MDSC).



Table 7 provides the DSR grading results that show HTPG XX, ITPG II, T(45°) and T(30°) as well as grade span (HTPG XX – T(30°)). Compared to the data in Figure 13, it is obvious that the SDA residue can increase the grade span to levels well above those for straight binders. However, whether these compositions will show better performance in service will also depend on the chemical aging and ductile strain tolerance. The compositions from Table 7 were also investigated according to FTIR and MDSC protocols and the detailed results were published in a third CTAA conference publication presented in the fall of 2020.

**Table 7. SDA Modified Binder Properties**

Binder	HTPG XX, °C	ITPG II, °C	T(45°), °C	T(30°), °C	Span, °C
C1	47.4	7.4	6.7	-13.4	61.7
C1 + 5 % SDA	55.3	3.2	12.6	-9.6	58.5
C1 + 10 % SDA	60.1	14.8	18.0	-3.6	74.9
C2	61.7	17.3	18.0	0.0	61.7
C2 + 5 % SDA	66.2	19.5	23.2	3.2	69.4
C2 + 20 % SDA	77.1	29.2	34.7	11.8	88.9
A1	48.8	9.8	1.8	-17.0	65.8
A1 + 5 % SDA	53.9	10.4	6.2	-11.3	65.2
A1 + 10 % SDA	59.6	14.3	16.6	-4.2	63.8
A2	56.5	15.6	9.6	-4.6	61.1
A2 + 5 % SDA	59.3	19	15.1	-0.4	59.7
A2 + 20 % SDA	70.7	29	29.1	9.6	80.3
A3	56.1	15.7	10.8	-4.8	60.9
A3 + 5 % SDA	59.3	17.1	15.0	-3.4	62.7
A4	48.4	7.7	2.5	-17.1	65.5
A4 + 5 % SDA	51.6	7	3.8	-15.4	67.0

Note: Span is based on HTPG XX – T(30°) and T(30°) is measured on PAV residue.

### **Task 8 – Mixture Preparation**

A significant number of purposely distilled binders from Table 4 and three commercial Cold Lake binders were used to prepare a large number of asphalt mix specimens for testing in the SCB test (Task 9) and Hamburg wheel-tracking machine (Task 10). Two different mix designs HL-1 and FC-2 were used for the Hamburg testing program in order to show the effect of aggregate type and gradation on performance-based properties. The scope of the mixture testing program was significantly limited due to the Covid-19 related restrictions placed on laboratory access.

### **Task 9 – Mixture Fracture Testing**

Asphalt mixtures were aged in an oven for 16 hours at 140 °C, compacted to an air voids content of 4 percent, and loaded in an SCB fixture to failure at 20 °C and –10 °C (Figure 19). The aging step has shown to be equivalent to approximately 20-40 hours of PAV aging of the asphalt binder.

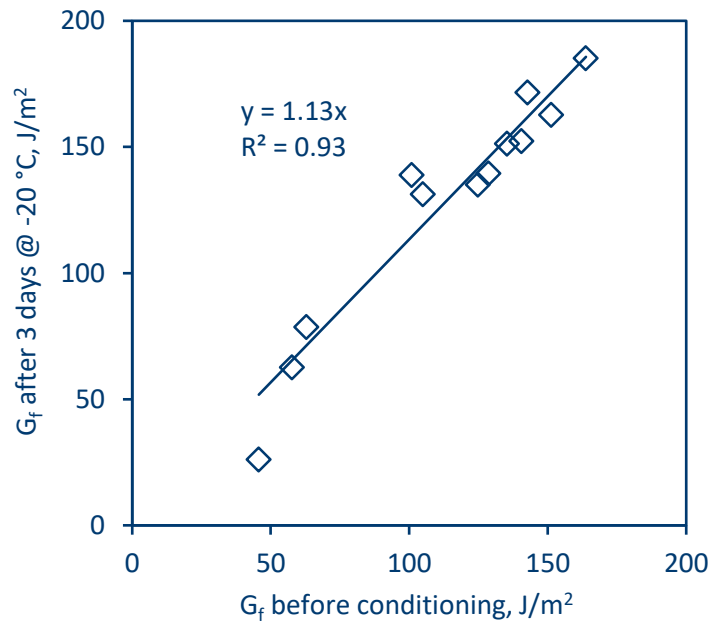


**Figure 19.** Semicircular bending (SCB) test with crack mouth opening displacement (CMOD) gauge.

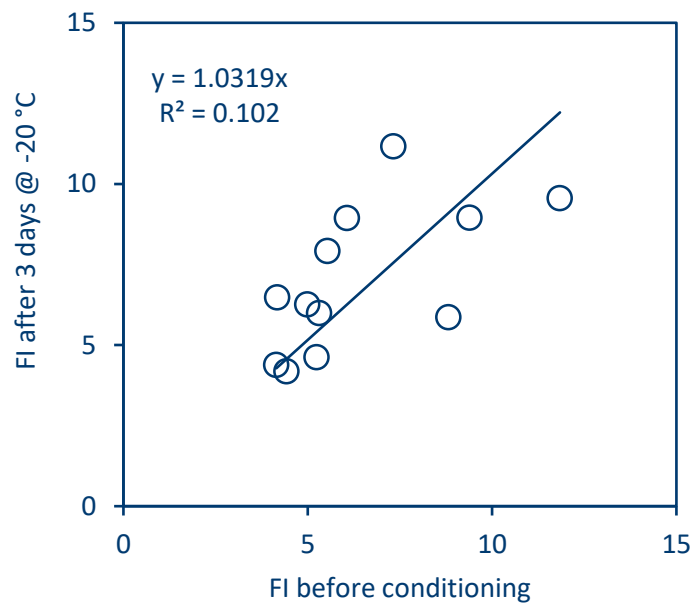
The SCB test was conducted according to procedures as embodied in AASHTO TP 124 *Standard Method of Test for Determining the Fracture Potential of Asphalt Mixtures Using the Illinois Flexibility Index Test (I-FIT)*. The AASHTO TP 124 standard specifies a loading rate of 50 mm/min, which is rather high and therefore fails to differentiate between binders. Hence, for this project a much slower rate of 0.5 mm/min was used in all experiments. This slower rate should be more discriminating and provides results dependent on the binder properties at longer loading times, more relevant to the actual pavement condition in service, rather than those at short loading times in brittle failure.

The parameters measured in the notched SCB test include fracture energy (area under the force versus displacement curve) and Illinois flexibility index (FI). The FI parameter relates to the measured properties in the SCB test as follows:  $FI = A (G_f/|m|)$ , where  $G_f$  is the fracture energy (area under the curve),  $|m|$  is the absolute value of the post-peak slope for the force versus displacement curve, and  $A$  is a scaling coefficient of 0.01. Higher values of  $G_f$  and FI are generally considered more desirable for the control of pavement cracking.

The effect of cold conditioning was investigated on the performance properties in the SCB test at room temperature as well as cold temperatures. A number of commercial mixes obtained from a US DOT agency were tested for comparison purposes. Cold conditioning for three days appears to make binders considerably stiffer when tested at room temperature and this effect is likely dependent on the base asphalt source (quality and durability), with Alberta oil sands-derived binders outperforming other lesser quality materials (see Table 6 and Figure 11). However, the results from this series of experiments, as shown in Figures 20 and 21, do not show much change in the SCB test before and after cold conditioning. This is likely due to the fact that the test is done at a high rate of loading to take the sample to failure within a reasonable timeframe, rather than keep it at a set load to induce creep fracture at much lower stress levels over significantly longer times as occurs in service. The data in Figure 21 also show that the determination of FI varies by a significant amount and this is likely due to the fact that only duplicates were measured with limited repeatability in the parameter.



**Figure 20.** Effect of cold conditioning for 3 days at  $-20\text{ }^\circ\text{C}$  on fracture energy at  $+20\text{ }^\circ\text{C}$ .



**Figure 21.** Effect of cold conditioning for 3 days at  $-20\text{ }^\circ\text{C}$  on flexibility index at  $+20\text{ }^\circ\text{C}$ .

Recycled polyethylene terephthalate (PET) fibers were used to toughen the mixtures at both ambient and low temperatures and to improve the rutting resistance at high temperatures. With regards to the SCB performance, the PET fibers have a major positive impact at only 0.3 weight percent on the mixture (approximately \$12 added cost per tonne of HMA). As shown in Tables 8-11, the fracture energy is significantly improved for most compositions at both temperatures and the flexibility index is particularly increased at room temperature.

**Table 8. Effect of Binder Type and Mixture Composition on Normalized Fracture Energy ( $G_f$ ) at 20 °C**

Mixture Type	Sample 1	Sample 2	Average $G_f$ , J/m <sup>2</sup>	Standard Deviation
CL64-22	111	170	140	29
CL64-22-PET	223	251	237	14
CL58-28	123	87	105	18
CL58-28-PET	115	108	111	3.9
CL46-34	43	83	63	20
CL46-34-PET	50	51	51	0.3
A3	45	34	40	5.1
A3-PET	127	125	126	1.2
A4	5.2	4.7	4.9	0.3
A4-PET	19	12	15	3.2
PR4	47	57	52	4.9
PR4-PET	66	67	67	0.5
PR5	21	24	22	1.3
PR5-PET	71	76	74	2.7
A5	63	52	58	5.6
A5-PET	24	28	26	2.4

Note: CL = Cold Lake, A = Athabasca, and PR = Peace River.

The change in fracture properties was determined with only two duplicate specimens for each composition. Hence, for some of these results the confidence level is rather low. However, it is fair to conclude that the overall effect of base asphalt binder is significant at room temperature. Softer binders typically possess less strength and therefore show lower fracture energies. The Athabaskan and Peace River materials ranked among the lowest cut points and grade spans and therefore performed less impressively compared to the harder Cold Lake materials with PG 58-28 and PG 64-22. However, the addition of PET fibers was able to improve the fracture energy dramatically in some instances (A3 and PR5), while it did not seem to have the same effect for others (CL46-34, PR4, A5). Further work will most likely shed light on what compositions are most desirable to obtain high fracture toughness mixtures. Polymer modification of some of these softer materials will also likely be able to bridge the gap in toughness. At -10 °C the binders have all become stiffer and stronger and with it the effect of the PET fibers becomes more significant and remains largely positive.

**Table 9. Effect of Binder Type and Mixture Composition on Fracture Energy ( $G_f$ ) at  $-10\text{ }^\circ\text{C}$**

Mixture Type	Sample 1	Sample 2	Average $G_f$ , J/m <sup>2</sup>	Standard Deviation
CL64-22	117	101	109	7.9
CL64-22-PET	178	161	169	8.3
CL58-28	186	228	207	21.0
CL58-28-PET	300	263	281	18.5
C6	169	163	166	2.8
C6-PET	271	224	248	23.6
A6	297	371	334	37.0
A6-PET	296	415	356	59.7
PR5	418	333	375	42.4
PR5-PET	454	425	439	14.1
A3	204	157	181	23.4
A3-PET	425	303	364	61.0

Note: CL = Cold Lake, A = Athabasca, and PR = Peace River.

**Table 10. Effect of Binder Type and Mixture Composition on Flexibility Index (FI) at  $20\text{ }^\circ\text{C}$**

Mixture Type	Sample 1	Sample 2	Average FI	Standard Deviation
CL64-22	6	5	5	0.5
CL64-22-PET	29	19	24	4.6
CL58-28	5	4	4	0.3
CL58-28-PET	50	42	46	4.3
CL46-34	6	5	6	0.9
CL46-34-PET	25	18	21	3.8
A3	9	5	7	1.9
A3-PET	39	37	38	1.1
A4	10	7	9	1.3
A4-PET	20	27	24	3.8
PR4	7	7	7	0.1
PR4-PET	45	46	45	0.5
PR5	6	5	5	0.5
PR5-PET	29	19	24	4.6
A5	5	4	4	0.3
A5-PET	50	42	46	4.3

Note: CL = Cold Lake, A = Athabasca, and PR = Peace River.

**Table 11. Effect of Binder Type and Mixture Composition on Flexibility Index (FI) at -10 °C**

Mixture Type	Sample 1	Sample 2	Average FI	Standard Deviation
CL64-22	0	0	0	0.0
CL64-22-PET	1	0	1	0.1
CL58-28	0	0	0	0.0
CL58-28-PET	1	1	1	0.1
C6	0	0	0	0.0
C6-PET	0	0	0	0.1
A6	0	0	0	0.0
A6-PET	0	2	1	0.7
PR5	5	4	4	0.4
PR5-PET	7	7	7	0.1
A3	0	0	0	0.0
A3-PET	1	1	1	0.0

Note: CL = Cold Lake, A = Athabasca, and PR = Peace River. Most tests at -10 °C ended in brittle failure hence it was impossible to calculate the FI.

For the flexibility index it is noteworthy that at room temperature the changes are all very significant and positive. The binders are soft and lack strength on their own, so addition of thin, ductile fibers is able to spread the load and improve post-peak load performance. At -10 °C on the other hand, brittle failure appears to be unavoidable for most if not all systems investigated. Somewhere in between room temperature and -10 °C, the performance for the fiber-modified mixes will be significantly superior and this will most likely benefit thermal cracking performance in a big way. Figure 2 shows how this works in the real world as test section 9 in northeastern Ontario shows remarkable performance after 12 years of service under extreme climate and traffic conditions. It was constructed with a Cold Lake PG 46-34 base asphalt binder.

### **Task 10 – Moisture Sensitivity Testing**

A significant number of asphalt specimens were compacted to an air voids content of 7 percent and loaded in the Hamburg wheel track testing machine until failure or 20,000 passes (see Figures 22 and 23). For the Hamburg test there are no acceptance criteria in Canada as no jurisdiction currently uses the test. Hence, data in this study were compared to the performance of Cold Lake base asphalt as it is known to perform to satisfaction (e.g., see Figure 3). Dry specimens were not found to show significant rutting so for most compositions only wet tests were conducted. A number of wet specimens showed severe stripping (see Figure 23). A total of 44 different Hamburg tests were conducted. Nearly all the Alberta oil sands-derived binders appear to perform similarly to Cold Lake material and to satisfaction.

High temperature stability and water sensitivity are important performance indicators of asphalt mixtures. Insufficient high temperature stability of the asphalt mixture will lead to rutting distress that can significantly affect driving comfort and safety. Rutting usually happens at higher temperatures during

summer and many countries and regions use a test temperature of 50 °C to represent typically unfavorable high temperature conditions. Testing at a single temperature allows agencies to obtain relative rankings for rutting resistance of the different asphalt mixtures and makes a Hamburg specification easy to implement. However, using a single testing temperature cannot represent all the environmental conditions, especially for cold regions like those encountered around Canada. There are currently no specifications for the Hamburg test in Canada. Hence, the results obtained here can only be considered preliminary and for relative comparison between different binders with known performance (Cold Lake commercial grades) and unknown performance (Athabasca sources purposely distilled).

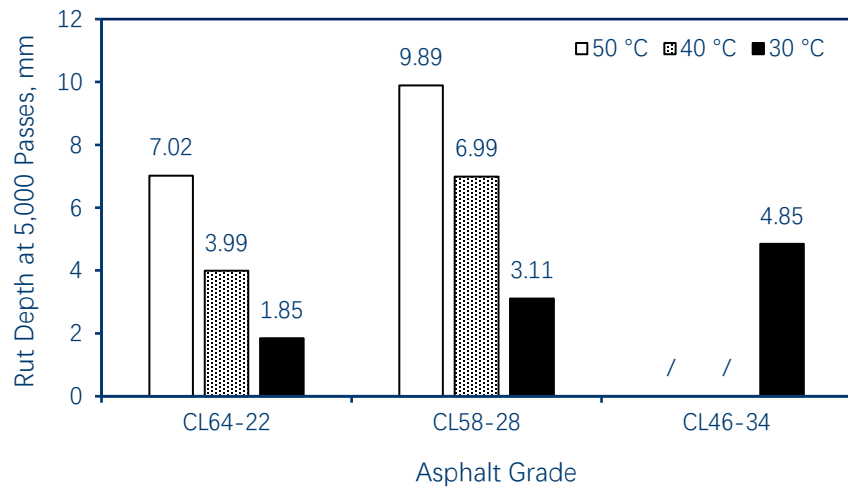


**Figure 22.** InstronTek’s SmarTracker™ Hamburg wheel tracking machine.

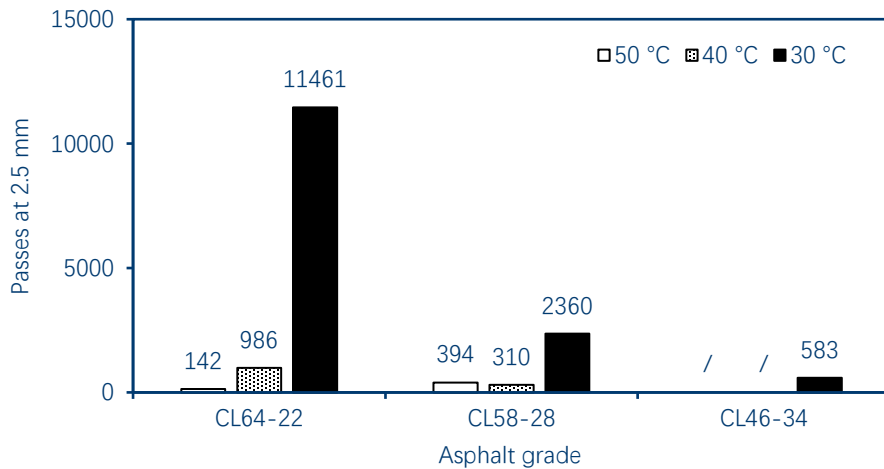


**Figure 23.** Dry and Wet Hamburg Specimens after 20,000 Cycles.

From various Hamburg tests on different binders it shows that, as expected, the rut depth at 5,000 passes decreases with an increase of the high temperature grade of asphalt binder. A decrease in the testing temperature also decreases the rut depth in the asphalt mixture. Similar trends are observed for passes at 2.5 mm and 5 mm rut depth. For the stripping inflection point, a higher performance grade asphalt usually shows better moisture resistance. Decreasing the testing temperature will increase the moisture stability for the asphalt mixture. Since the Cold Lake PG 46-34 binder is too soft at temperatures of 50 °C and 40 °C, it is unable to maintain enough strength to perform the Hamburg test. Hence, results for Cold Lake PG 46-34 at 50 °C and 40 °C are not shown here.

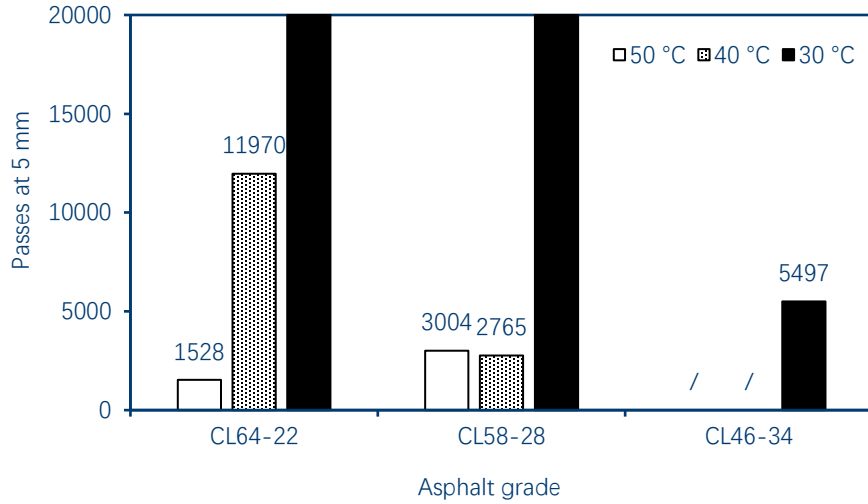


**Figure 24.** Rut depth at 5,000 passes of different grade asphalt under different temperature.

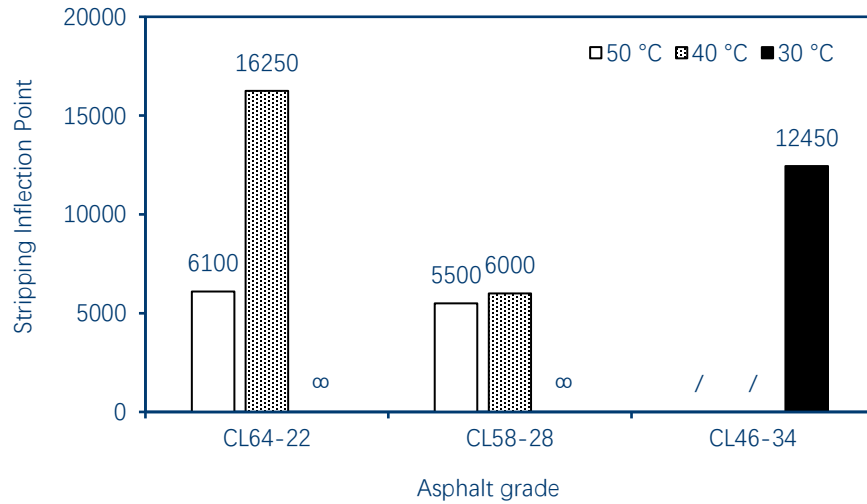


**Figure 25.** Passes at 2.5 mm of different grade asphalt under different temperature.



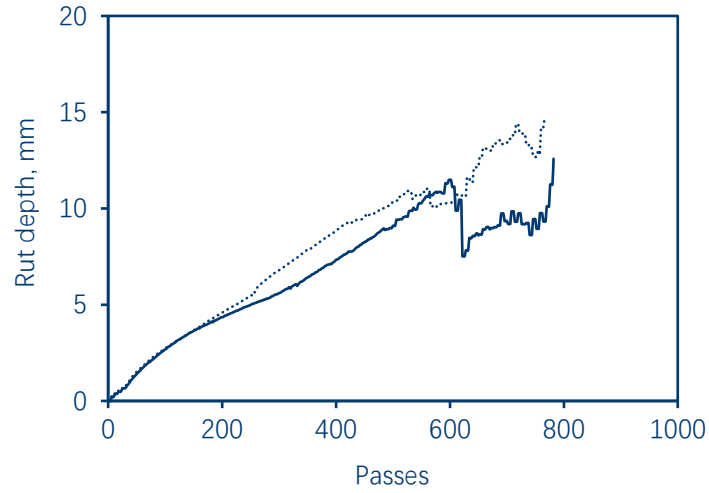


**Figure 26.** Passes at 5 mm of different grade asphalt under different temperature.

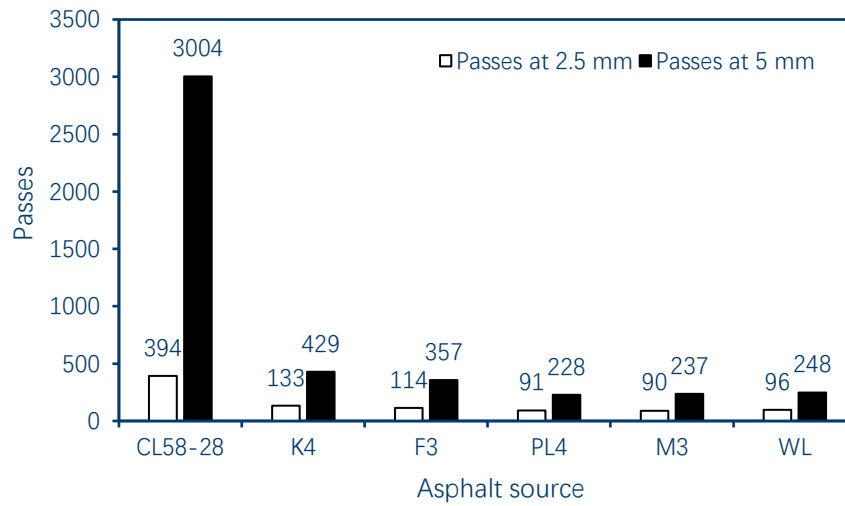


**Figure 27.** Stripping inflection point of different grade asphalt under different temperature.

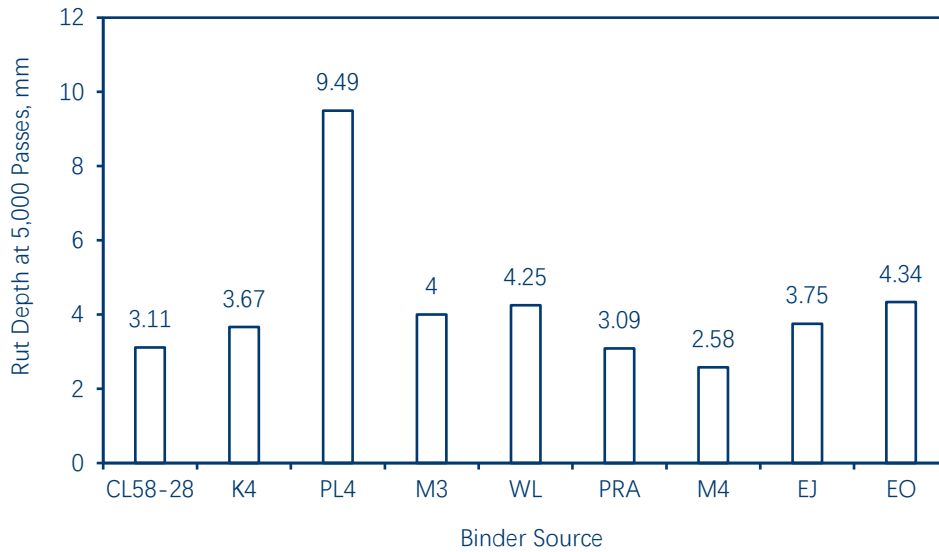
It was not possible to obtain the rut depth at 50 °C for any of the atypical binder samples since they achieve the maximum allowed depth before 5,000 passes. In addition, it was also impossible to obtain stripping inflection points (SIP) as there appeared to be no change in slope prior to the maximum rut depth was reached. A representative result for an Athabasca sample at 50 °C is shown in Figure 28. When the testing temperature was reduced to 30 °C, no stripping inflection points were observed for either commercial binders or any of the unconventional binders.



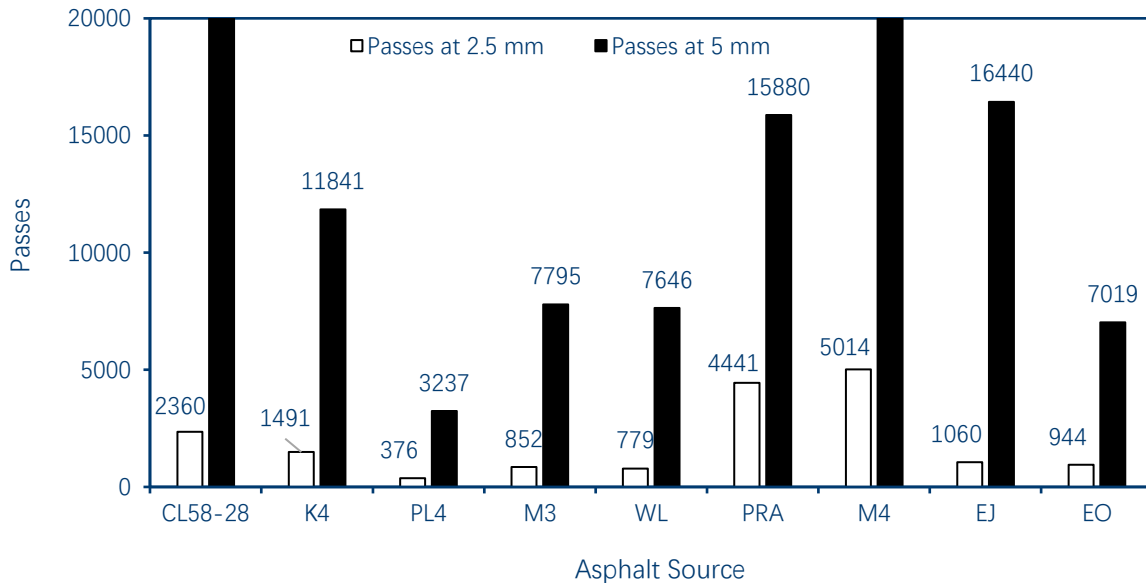
**Figure 28.** Duplicate wet Hamburg results at 50 °C for Athabasca binder.



**Figure 29.** Number of passes for different binders at 50 °C.

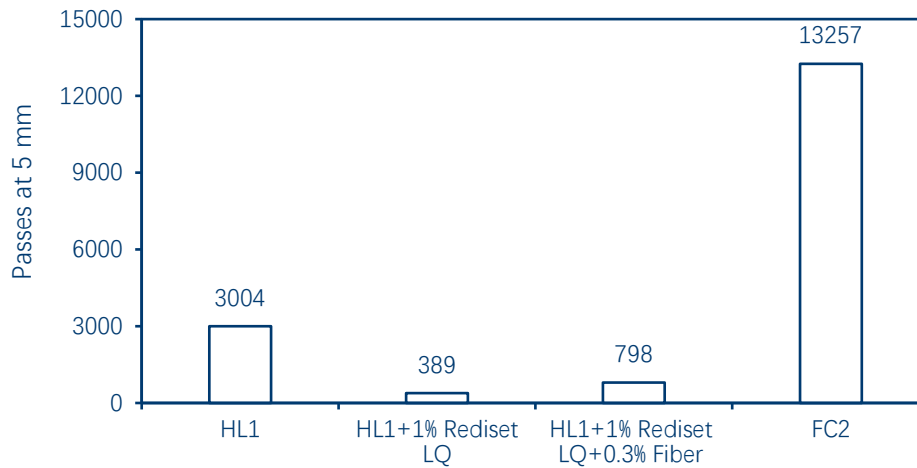


**Figure 30.** Rut depth at 5,000 passes for different binders at 30 °C.



**Figure 31.** Number of passes for different binders at 30 °C.

Rediset LQ antistripping and PET fiber additives were investigated in order to better control the moisture and rutting distresses with varied results. Figure 32 shows the findings for a number of systems tested at 50 °C. It is obvious from these data that the aggregate type and gradation can be of paramount importance. These issues can be addressed through the selection of appropriate aggregate and likely the differences between various designs are less pronounced at lower temperatures. It is obvious from the results that a test temperature of 50 °C is too high for the soft binder grades investigated.



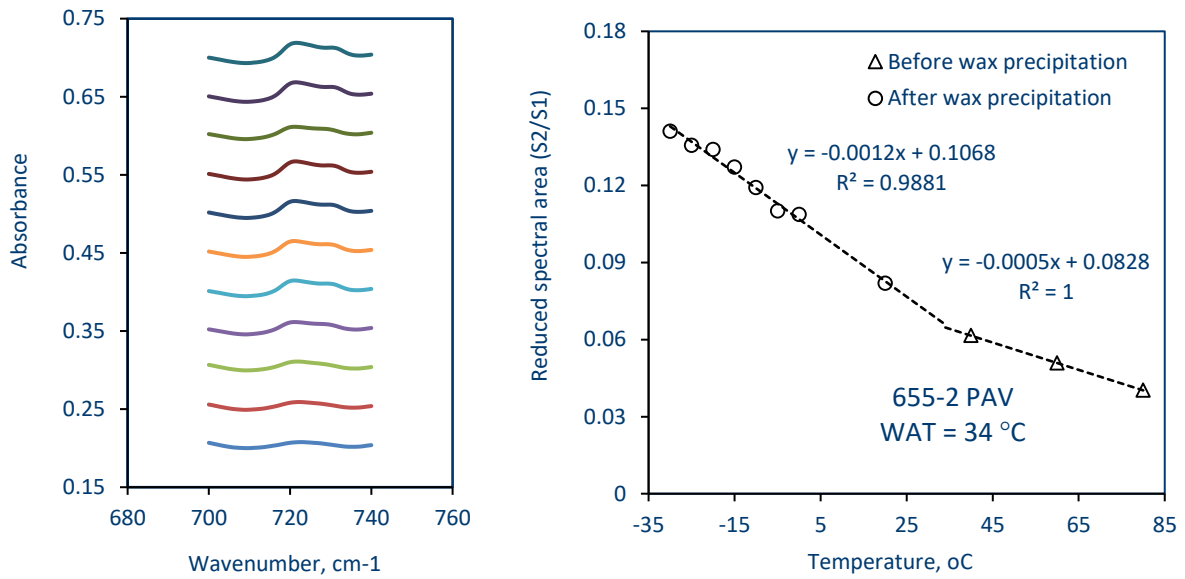
**Figure 32.** Passes at 5 mm curves of Cold Lake PG 58-28 binder with and without fiber at 50 °C.

***Task 11 – Development of a VT-FTIR Technique to Measure Wax Precipitation in Asphalt Binder***

A final task related to the detection of wax precipitation temperatures, and to quantify the amount of precipitated wax at various temperatures for a large range of the binders from Table 6 and various additional sources.

Figure 33 shows how the infrared spectral area between 700  $\text{cm}^{-1}$  and 740  $\text{cm}^{-1}$  changes with temperature and how a plot of the reduced spectral area changes rather abruptly at around 34 °C for this particular binder. The main results for wax contents, determined from the difference between the reduced spectral area, as measured versus the extrapolated value from the amorphous phase prior to wax precipitation, is given in Figure 34.

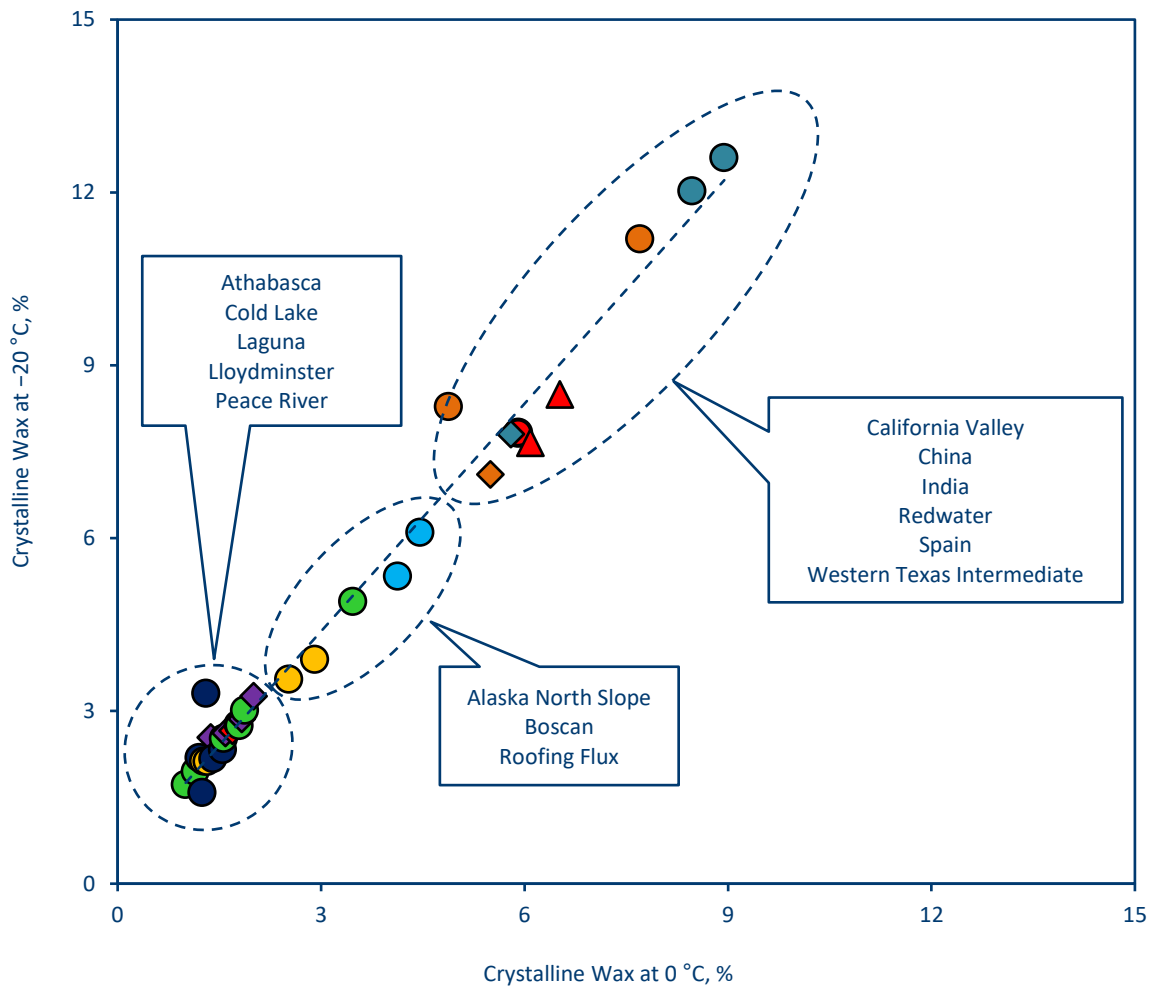
The data in Figure 34 show that the binders obtained from Athabasca, Cold Lake, Peace River and Venezuela are all very low in wax at both room temperature and below freezing. A large number of additional data have been obtained using the newly developed VT-FTIR method and one paper was published in Fuel and another in Construction and Building Materials. A third has been submitted for publication in Thermochemica Acta and another two are in preparation.



**Figure 33.** FTIR analysis of wax precipitation in commercial binder from a Highway 655 trial.

Note: Eleven spectra were taken after equilibration at 80, 60, 40, 20, 0, -5, -10, -15, -20, -25 and -30 °C.

Wax appearance temperature (WAT) is determined from where liquid and solid/liquid lines meet.



**Figure 34.** Crystalline wax content for various binders sourced from around the world.

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## E. KEY LEARNINGS

The key learnings from the research can be summarized as follows:

- 1) Asphalt binders produced from Athabasca, Cold Lake and Peace River crude bitumen perform similarly to commercial binders obtained from around the world under standard specification protocols. Useful temperature intervals (grade spans) are similar to those obtained for binders from China, India, the Middle East, Russia, the United States, and Venezuela (see Table 6 and Figure 10).
- 2) Grade spans typically increase for harder binders distilled to higher cut points. Softer grades from Athabasca and Peace River sourced materials are atypical in that they produce rather low grade spans. However, such atypical binders were found to be largely free of solid waxes, and therefore desirable for the use in designs with RAP and SDA waste streams. These combinations would lead to less need for virgin materials, lower costs, and thus improved sustainability for the oil sands and road construction industries.
- 3) Alberta oil sands-derived binders obtain a competitive advantage once they are graded more accurately according to enhanced protocols such as AASHTO TP 113-15 DENT, AASHTO TP 122-16 EBBR and limiting phase angles (see Table 6, Figures 11-16). The implementation of new and improved specifications is imperative for the design of longer life pavements that benefit from the use of Alberta binders.
- 4) Enhanced specification protocols need to assess not only binder quality (grade span) but also durability (effects of extended conditioning at both cold and hot conditions). Asphalt binder freshly poured at high temperatures and cooled for only a short period of time at room temperature is at non-equilibrium. Long-term conditioning allows for the crystallization of solid waxes and this can change rheological and failure properties. Cracking resistance will be degraded for binders that are high in solid wax. Alberta oil sands-derived binders are at an advantage when tested under equilibrium conditions as they are low in wax and therefore will suffer little from cold temperature conditioning.
- 5) Asphalt mixture tests have shown that atypical binders with low grade spans can be made to perform to satisfaction by selecting an appropriate mix design and/or by adding recycled PET fibers to the mixture. Such PET fibers are able to toughen the material to significantly enhance cracking and rutting resistance.

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## F. OUTCOMES AND IMPACTS

The following refereed journal articles, conference publications, student theses, and webinar presentation have resulted from the work conducted during the project:

### Refereed Journal Publications (Published)

1. E. Diak, E. Beier, S.A.M. Hesp. Development of a Butt Joint Test for the ductile performance grading of asphalt binders. *Construction and Building Materials*, 243 (2020) 118195.
2. H. Ding, S.A.M. Hesp. Quantification of crystalline wax in asphalt binders using variable-temperature Fourier-transform infrared spectroscopy. *Fuel*, 277 (2020) 118220.
3. H. Ding, S.A.M. Hesp. Variable-temperature Fourier-transform infrared spectroscopy study of wax precipitation and melting in Canadian and Venezuelan asphalt binders. *Construction and Building Materials*, 264 (2020) 120212.
4. K. Lill, A.N. Khan, K. Kontson, S.A.M. Hesp. Comparison of performance-based specification properties for asphalt binders sourced from around the world. *Construction and Building Materials*, 261 (2020) 120552.
5. H. Ding, S.A.M. Hesp. Another look at the use of modulated differential scanning calorimetry to study thermoreversible aging phenomena in asphalt binders. *Construction and Building Materials*, 267 (2021) 121787.

### Refereed Journal Publications (Submitted or Soon to be Submitted)

1. L. de Loë, S.A.M. Hesp. Large Amplitude Oscillatory Shear testing for the performance grading of straight and polymer-modified asphalt binders. Submitted to *Construction and Building Materials*, November 21, 2020.
2. J. Kovinich, S.A.M. Hesp, H. Ding. Modulated scanning calorimetry study of wax-doped asphalt. Submitted to *Thermochimica Acta*, December 22, 2020.
3. H. Ding, S.A.M. Hesp. Fast quantification of wax-based additives in asphalt binder to balance sustainability and pavement performance. To be submitted, 2021.
4. H. Ding, S.A.M. Hesp. Variable-temperature Fourier-transform infrared spectroscopy investigation of asphalt binders sourced from various crude oils. To be submitted, 2021.
5. J. Kovinich, A. Kuhn, A. Wong, S.A.M. Hesp, H. Ding. Wax in asphalt: a comprehensive review of important issues. To be submitted, 2021.
6. J. Kovinich, A. Kuhn, A. Wong, S.A.M. Hesp, H. Ding. The optimal binder. To be submitted, 2021.

### Conference Publications (Non Refereed)

1. K. Lill, K. Kontson, A. Khan, P. Pan, S.A.M. Hesp. Comparison of physical and oxidative aging tendencies for Canadian and Northern European asphalt binders. *Proceedings of the Annual Meeting of the Canadian Technical Asphalt Association*, 2019.



2. A. Khan, C. Lemaitre, S.A.M. Hesp. Impact of asphaltene addition on performance-based rheological and failure properties of Alberta oil sands-derived asphalt binders. *Proceedings of the Annual Meeting of the Canadian Technical Asphalt Association*, Regina, 2020.
3. H. Ding, S.A.M. Hesp, A. Khan, C. Nawarathna, L.M. Young. Thermoreversible and oxidative aging effects on properties of soft Alberta oil sands-derived, Russian and Venezuelan asphalt binders. *Proceedings of the Annual Meeting of the Canadian Technical Asphalt Association*, Regina, 2020.

#### Masters Theses

1. C. Nawarathna. Investigation of Atypical Asphalt Binder for the Sustainable Recycling of Reclaimed Asphalt Pavement, May 2021.
2. O. Oluwatobi. Investigation of Aging Behaviour of Asphalt Binders Sourced from Around the World, May 2021.

#### Webinar Presentation

1. S.A.M. Hesp. Value-Added Opportunities for Asphalt Binders Derived from Alberta Oil Sands, April 20, 2019. Presentation is posted on the Alberta Innovates website under the following link: [https://albertainnovates.ca/wp-content/uploads/2020/04/Simon\\_Hesp\\_Presentation.mp4](https://albertainnovates.ca/wp-content/uploads/2020/04/Simon_Hesp_Presentation.mp4).

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## G. RECOMMENDATIONS AND NEXT STEPS

It is imperative to better educate large numbers of user agencies that are currently under-designing their asphalt pavements for cracking due to the effects of oxidative and thermoreversible aging. Once users obtain a better understanding of why certain asphalt binders perform much better than others, they will naturally gravitate towards the implementation of enhanced binder performance grading tests. The tests we developed through long-term research efforts in this area are ready to be implemented once agencies become more aware of the life cycle benefits.

There is over 25 years of history on convincing users and producers alike to adopt enhanced specifications with success in several large jurisdictions in Ontario. This has been a slow process since technical staff working at these user agencies are often overworked and underpaid. Hence, they rather stick with the status quo than implement major changes in specifications with uncertain outcomes.

It is therefore important to take such staff by the hand and show them—at no risk or financial cost to them—how they are currently designing in a sub-optimal fashion their pavements with grade losses due to thermoreversible aging ranging from 6 °C to 12 °C. Once they are made aware of their pavements being seriously under-designed, the next step is to show them how to do things better.

By selecting straight asphalt binder from Cold Lake it will be possible to obtain significantly enhanced life cycles due to insignificant effects of cold conditioning. If they insist on the use of RAP then atypical binders such as those listed in Table 6 will be used to produce binders with balanced properties in order not to over-design for rutting resistance at the expense of cracking resistance.

Finally, agencies that over the next 2-3 years will gain a better understanding of how to properly design their pavements will be asked to facilitate the construction of a significant number of reconstruction and rehabilitation projects, where side-by-side the benefits of Alberta oil sands-derived binders will be demonstrated against the use of conventional materials and designs.

It is anticipated that with 20-30 of such full scale demonstration projects around North America and beyond the future for the use of Alberta produced binder in asphalt pavement construction will be bright.

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## **H. KNOWLEDGE DISSEMINATION**

Three conference presentations and one webinar were presented on the knowledge generated from the project. In addition, a large number of peer reviewed journal articles were published and will be published in the months to come. All papers are in top engineering journals (e.g., Fuel, Construction and Building Materials, Thermochemica Acta) that are widely distributed around the world.

It is anticipated that with further funding the outcomes from this research will be directly taken to a large number of user agencies in North America and beyond.

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## I. SUMMARY AND CONCLUSIONS

This project has investigated how Alberta oil sands-derived asphalt binders compare to materials produced from crude oils produced around the world. Asphalt binder and mixture rheological and failure properties were used to provide a comprehensive assessment of performance-related properties. Higher quality and durability materials will provide improved life cycle costs and promote long-term sustainability for both the road building and oil sands industries.

Given the results and discussions presented, the following conclusions are offered:

1. Asphalt binders from a wide range of Alberta oil sands crudes are competitive within the global market based on their regular Superpave grading performance.
2. Cold Lake crudes provide binders with grade spans that are consistently higher than those of nearly all competing materials. Only binders distilled from Laguna, Venezuela crude oil provide similarly wide useful temperature intervals.
3. Athabasca and Peace River crudes provide atypical binders with somewhat lower grade spans, largely due to their lower asphaltene contents. Such atypical binders are promising materials for use in conjunction with SDA and RAP waste streams. Combining these will provide materials of similar performance to those obtained from straight Cold Lake at a lower cost.
4. Enhanced grading protocols, that test binders under equilibrium conditions, show that Athabasca and Peace River materials provide similar performance to those obtained from Cold Lake and Venezuelan crudes.
5. A novel variable-temperature Fourier-transform infrared spectroscopy method was developed to measure solid wax content as a function of temperature in thin binder films. Binders obtained from Alberta oil sand crudes are exceptionally low in solid wax, providing them with a competitive advantage in terms of long-term performance.
6. Performance deficits for heavy traffic designs are easily overcome through the use of (1) improved mix designs with more angular aggregate to prevent rutting, (2) moisture resistant aggregate to prevent stripping, and (3) the addition of recycled PET fibers to prevent rutting and cracking.
7. It was found that the addition of only 0.3 weight percent of PET fibers increased fracture properties by enormous amounts. A single 12-year-old pavement trial section constructed with soft Cold Lake asphalt binder on Highway 655 in far northern Ontario demonstrates that this is a viable approach to the construction of long-life, sustainable pavements.

It is important that the knowledge generated through this research project be taken directly to a large number of user agencies. This will allow the benefits of straight Alberta oil sands-derived binder to be demonstrated in real-world road construction around North America and beyond.