Waste engine oil residue in asphalt cement

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ABSTRACT: The use of waste engine oil residues obtained from the recycling of motor oils in asphalt cement has been reported. The detection of zinc and molybdenum indicates the presence of this modifier. A purported benefit is an increase in the grade span through a partial precipitation of the asphaltene fraction when the base asphalt is blended with this largely paraffinic material. Unfortunately, the precipitation does not stop at the desired grade, but continues at an accelerated rate for a long time. Hence, pavements constructed with such binders are often under-designed and show a tendency to crack prematurely and excessively. A case study of two stretches of the Trans-Canada Highway north of North Bay, Ontario and west of Cochrane, Ontario is presented. The North Bay pavement was newly constructed on a granular base in the summer of 2000. It showed widespread cracking distress after only 5-7 years of service. Recovered asphalt cement revealed the presence of zinc and molybdenum explaining the premature failure. The Cochrane pavement was constructed in 1999 and has so far remained free of any distress. It likely used superior quality western Canadian asphalt cement and was found to be free of any deleterious contaminants. The objective of this research project was to investigate and document the danger of using Superpave™ specifications that promote waste engine oil residues and other similar low cost modifiers in asphalt cement.

1 INTRODUCTION

1.1 *Low temperature specification test development*

Early work performed at the Royal Dutch Shell research laboratories in Amsterdam considered a range of properties for the control of cold temperature cracking in asphalt pavements (penetration, ring and ball softening point, ductility, tensile strain to failure, etc). Pioneering studies published by Van der Poel (1954, 1955) and Heukelom (1966) led to the development of methods for the control of transverse cracks that were largely based on limiting the binder stiffness after a set period of loading. Krom and Dormon (1963) were the first to propose a binder specification scheme that limits the binder stiffness at specific loading times and temperatures to control cracking. Heukelom (1966) tested a wide range of binders and found that there was a high correlation between binder stiffness and actual failure properties. Hence, he concluded that "Van der Poel's stiffness concept has provided a valuable means of simplifying the description of not only rheological, but also fracture properties of asphalt cements and asphalt mixtures." The stiffness properties were predicted using Van der Poel's nomogram from the measurement of largely empirical properties such as penetration, penetration index, and ring and ball softening point, as measured at or slightly below ambient temperatures on unaged binders. Following the Shell studies, a large number of researchers focused their attention on measuring stiffness at fixed loading times to control thermal cracking. However, what few realized is that the original studies at Shell largely relied on unmodified and unaged materials, that there was a reasonable degree of scatter, and the early published work was largely silent on how isothermal conditioning at low temperatures affects stiffness and relaxation ability of the asphalt cement.

1.2 *Superpave™ grading*

During the 1970s and 1980s it became apparent that empirical properties such as penetration and ring and ball softening point were no longer able to control thermal and traffic induced cracking. The United States' Strategic Highway Research Program (SHRP) of the late 1980s had as one of its main mandates the development of an improved asphalt cement specification system. In line with the early Shell work, SHRP investigators first proposed a limiting stiffness temperature to control thermal cracking but later augmented it with a lower limit on the logarithmic creep rate or m-value (Anderson and Kennedy (1993)). Various limits on the m-value were proposed and in the end a minimum of 0.3 found its way into the specification.

A weakness of the SHRP Superpave specification was that it relied entirely on a single Canadian field study by Readshaw (1974) who reported that an upper 200 MPa limit on creep stiffness after 2 hours of loading would largely prevent thermal cracking. He never considered the m-value though, which, in today's specification controls the grade for the large majority of asphalt cements. The m-value was introduced by the SHRP team to reduce the confounding effect of fatigue on low temperature cracking (Anderson and Kennedy (1993)). Given the fact that certain commercial binders have limiting m-value temperatures that are as much as 12°C warmer than their limiting stiffness temperatures, one would expect fatigue not to be a significant problem with the current specification in place across much of North America.

1.3 *Waste engine oil modification of asphalt cement*

Since the implementation of Superpave in North America, a number of patents and publications have disclosed relatively inexpensive ways to increase the grade span through the addition of various chemical agents (Kriech and Wissel (1989), Kamel and Miller (1991), Johnson and Juristovski (1995), Memom et al. (1995), Bonemazzi and Giavarini (1999), Hayner (1999), Collins and Jones (2000), Giavarini (2000), Moore et al. (2000), Hagens et al. (2004), others). The large majority of these rely on the formation of gel-type binders by reducing the solubility of the asphaltene fraction in the maltene continuous phase. The binders produced are characterized by a low stiffness, high zero shear viscosity, significant degrees of non-Newtonian flow, and delayed elasticity. At low temperatures such binders are less able to relax thermal stresses and are thus more prone to cold temperature cracking (McLeod (1972)).

The addition of waste engine oil residue stands out in that it likely provides the most cost effective way to increase the Superpave grade span (Rubab et al. (2011)). Waste oil residue sells for a significant discount to regular asphalt cement and hence profits increase when more is added. Our research has shown that when the amount of waste engine oil goes up binders high in asphaltenes become increasingly sensitive to both physical and chemical hardening (Hesp and Shurvell (2010), Rubab et al. (2011)). Materials stored for three days at low temperatures can easily lose anywhere from one to two full grades (6-12°C). It has also been shown that slightly modified pressure aging vessel treatments can depreciate the low temperature grades by similar amounts (Erskine et al. (2012). Hence, there is a need to look at how the low temperature Superpave protocol can be improved.

1.4 *Improved low temperature specifications*

A number of simple changes can be implemented to vastly improve current specification tests. First, physical hardening phenomena can be accounted for by conditioning the bending beam rheometer (BBR) specimens for various lengths of time prior to testing. Ontario's recently developed extended BBR protocol, *LS-308 Determination of Performance Grade of Physically Aged Asphalt Cement Using Extended Bending Beam Rheometer (BBR) Method* (MTO (2012)), specifies that materials are conditioned for one, 24 and 72 hours prior to testing at pass and fail temperatures. The absolute grade after 72 hours is required to meet the contract requirements and the grade loss from the one hour results is limited to 6°C. This simple modification can prevent a significant number of premature and excessive failures.

A second improvement relates to the inclusion of a failure strain criterion. Ontario's double-edge-notched tension (DENT) test method, *LS-299 Asphalt Cement's Resistance to Fatigue Fracture Using Double-Edge-Notched Tension Test (DENT)* (MTO (2012)), determines an approximate critical crack tip opening displacement (CTOD). The CTOD provides a measure of strain tolerance in the ductile state under severe constraint (e.g., at the tip of a propagating crack or in between two large aggregate particles). The CTOD has a lower limit that depends on the low temperature grade of the material.

A final improvement that can be made to the specification protocol is the way in which the asphalt cement is chemically aged prior to grading. The current pressure aging vessel (PAV) protocol ages the material in 3.2 mm thick films. Such films are not saturated uniformly and hence binders of the gel-type are at an advantage due to the formation of a skin that slows oxygen entering the film. Aging in thinner films of 0.8 mm and in the presence of moisture, according to Ontario's method *LS-228 Modified Pressure Aging Vessel Protocol* (MTO (2012), has proven to produce a much improved grading with poor performing materials losing as much as 6-12°C from their regular low temperature grade (Erskine et al. (2012)).

The first objective of this research project was to investigate and document the drawbacks of using regular Superpave specifications that promote the use of waste engine oil residues and similarly cheap modifiers in asphalt cement. A second objective is to show how improved test methods can be used to better control cracking distress due to accelerated thermal and chemical hardening. The extended BBR and DENT test methods LS-308 and LS-299 are used in this study to explain the premature failure in a northern Ontario paving contract.

2 MATERIALS AND EXPERIMENTAL METHODS

2.1 *Materials*

The materials investigated in this study were extracted from core samples taken from two large paving contracts on Highway 11 north of North Bay, Ontario and west of Cochrane, Ontario (Hesp et al. (2010)). The North Bay material provides an example of where Superpave has failed whereas the previously reported results from Cochrane (Hesp et al. (2010), provide an example of how a judicious choice of asphalt cement properties can prevent cold temperature cracking under extreme conditions for extended periods of time.

Approximately 4 kg of core sample for each site was cut into smaller pieces and soaked in just enough tetrahydrofuran (THF) to cover the material. After several hours the THF solution was removed from the aggregate. A graduated cylinder was used for sedimentation of fine particulate matter before evaporation of the solvent. This procedure was repeated several times with a total of approximately 4-6 L of solvent until no residual asphalt cement remained on the aggregate. The solvent was carefully removed by using a rotary evaporator to apply heat and vacuum. A final temperature of 150ºC and aspirator pressure of 20 mm Hg were maintained for 1.5 hours to ensure complete removal of all solvent with minimal hardening or oxidation of the binders. Approximately 200 g of asphalt cement was recovered for each pavement location, which was enough for testing in both LS-299 and LS-308.

2.2 *Experimental methods*

X-ray fluorescence (XRF) spectra for recovered and virgin asphalt cements and aggregates were collected using a hand-held Bruker Instruments Tracer III XRF analyzer. The instrument irradiates the surface of the material with high energy X-rays, causing the ejection of inner K-shell electrons from heavy elements. The vacancies so produced are reoccupied by electrons from the outer L- and M-shells. The descent of electrons from these outer shells is accompanied by the emission of a lower energy X-ray with a characteristic energy for the element being irradiated. The XRF analyzer detects the emitted radiation, and a plot of intensity versus the X-ray energy provides qualitative as well as quantitative information on the presence of a range of heavy elements. Peak heights in the spectrum provide a quantitative measure of the presence of the metal, but calibrations for each metal are required to provide absolute comparisons between metals (fluorescence yields vary between elements).

Ductile failure properties were determined according to Ontario's double-edge-notched tension method *LS-299 Asphalt Cement's Resistance to Fatigue Fracture Using Double-Edge-Notched Tension Test (DENT)* (MTO (2011)). In brief, samples were poured into brass double-edge-notched tension moulds and kept overnight at the test temperature of 15ºC. The specimens were tested at a constant rate of 50 mm/min, and the total failure energy was determined by integration of the force-displacement curve. The total specific failure energy, w_f, was plotted versus the ligament length (distance between the notches), L, and the specific essential work of fracture, w_e, was determined from the intercept of the curve (Andriescu (2006)). The specific essential work of fracture was divided by the net section stress, $\sigma_{n,\ 5mm}$, in the smallest ligament sample to obtain an approximate critical crack tip opening displacement, CTOD. The CTOD provides a measure of strain tolerance in the ductile state and has shown a strong correlation with thermal and fatigue cracking distress (Andriescu (2006), Hesp et al. (2009)).

The tendency for the binders to harden during cold conditioning was assessed through the extended BBR protocol. The test as embodied in *LS-308 Determination of Performance Grade of Physically Aged Asphalt Cement Using Extended Bending Beam Rheometer (BBR) Method*, is a simple elaboration on the regular BBR protocol (MTO (2012)). Specimens are conditioned at -10ºC and -20ºC for one, 24 and 72 hours prior to testing. The continuous low temperature grade is determined from the warmest temperature where the creep stiffness, S(t), reached 300 MPa or the slope of the creep stiffness master curve, m(t), reached 0.3 after 60 seconds of loading. Both pass and fail tests are conducted to determine continuous grades by interpolation. Besides the absolute grade, LS-308 also determines the grade loss after 24 and 72 hours compared to American Association of State Highway and Transportation Officials (AASHTO) standard M320 (AASHTO (2002)), which stipulates only one hour of conditioning.

3 RESULTS

3.1 *Field distress surveys*

Both contract locations were visited and inspected for thermal and traffic induced cracking distress. The 30 km long contract west of Cochrane is located about 800 km north from Toronto and was constructed in 1999. Today, it remains largely free of any distress over its entire length. The centerline joint is in very good shape as are the shoulders. Representative photographs are provided in Figure 1.

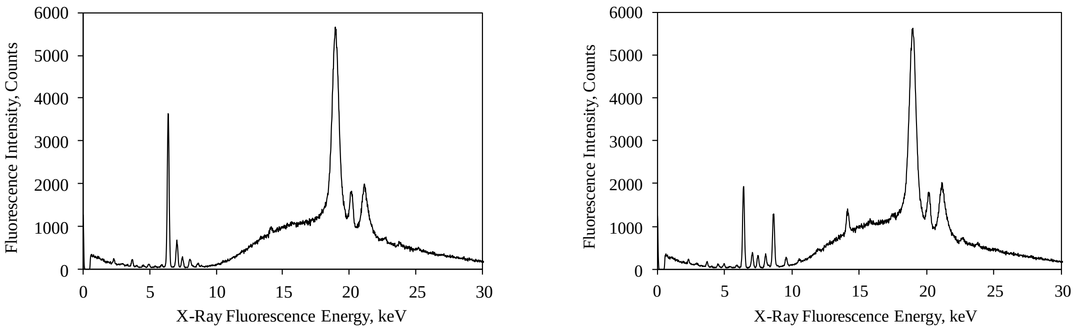
Figure 1. Representative images for Highway 11 condition west of Cochrane.

The 10 km long North Bay contract is located about 500 km north from Toronto and has presented opposite performance with regular cracking from beginning to end. Joints have opened up to significant degrees and transverse cracks are spaced anywhere from 30 to 50 cm apart over the entire length of the contract. Representative images for this pavement are provided in Figure 2. While the distress started as longitudinal cracks in early life, a large number of transverse cracks have now sprouted out from the original cracks.

3.2 *X-ray fluorescence*

The X-ray fluorescence (XRF) finding for the Cochrane and North Bay materials are provided in Figure 3. The spectra show a number of distinct peaks that indicate the presence of several metals of interests. Aggregate samples were also scanned and these were found to be free of zinc peaks at 8.6 and 9.6 keV and molybdenum peaks at 17.4 keV. It is clear from the result that there was a lot of zinc and a small amount of molybdenum in the North Bay sample while neither were found in the Cochrane material. For comparison, Figure 4 provides an X-ray fluorescence spectrum for 10W30 graded engine oil obtained from a local store.

Figure 2. Representative images for Highway 11 condition north of North Bay.



(a) (b)

Figure 3. X-ray fluorescence spectra for the recovered binders from (a) Highway 11 Cochrane and (b) Highway 11 North Bay. Note: Zinc (8.6 and 9.6 keV) and molybdenum (17.4 keV) peaks suggest waste engine oil residue is present in the North Bay contract. Other peaks for sulfur, calcium, vanadium, nickel, iron and strontium originate either from aggregate remnants or the asphalt cement.

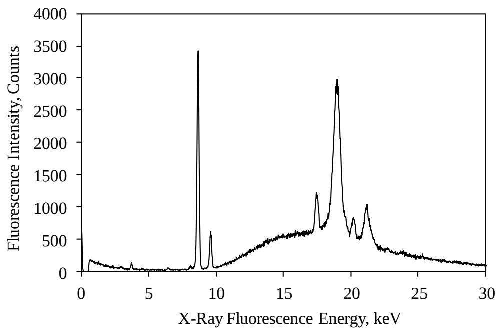
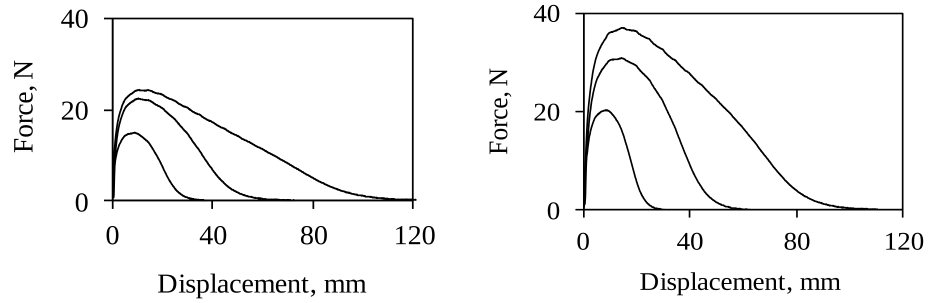


Figure 4. X-ray fluorescence spectrum for straight 10W30 graded engine oil. Note: Zinc peaks at 8.6 and 9.6 keV and the molybdenum peak at 17.4 keV originate from anti-wear additives in engine oils. Peaks at 19, 20.2 and 21.2 keV originate from the XRF instrument.

3.3 *Double-edge-notched tension testing*

The double-edge-notched tension results for the Cochrane and North Bay sample are provided in Figures 4 and 5. Repeat tests for the force-displacement traces in Figure 4 have shown that the test method is generally highly reproducible. Figure 4 shows that the force-displacement traces for different ligament lengths are self-similar (i.e., are similarly shaped). Figure 5 shows that the data follows a straight line with a high degree of accuracy. Hence, the essential work of failure analysis is applicable for this type of material. A comparison of the findings for the LS-299 analysis of both the Cochrane and North Bay materials is provided in Table 1.



(a) (b)

Figure 4. Raw force-displacement data from the double-edge-notched tension analysis for (a) Highway 11 Cochrane material and (b) Highway 11 North Bay material.

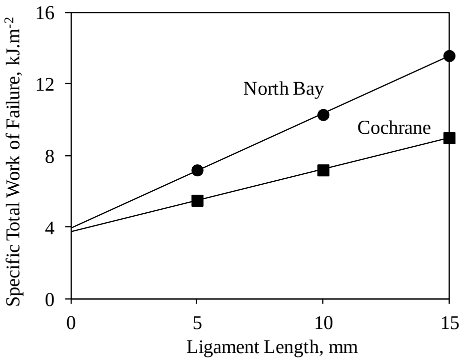


Figure 5. Essential work of failure analysis for Highway 11 Cochrane and Highway 11 North Bay materials. Note: The straight line relationship relates the specific total work of failure (w_f) to the essential work (w_e) and plastic work term (βw_p) as follows: $w_f = w_e + \beta w_p L$, where β is the geometry factor that describes the shape of the ductile failure zone and L is the ligament length between two notches (Andriescu (2006)).

Table 1. Essential Work of Failure Analysis on Recovered Materials

Sample	Essential Work of Failure, kJ.m^{-2}	Critical Crack Tip Opening Displacement (CTOD), mm
Cochrane	3.8	12.7
North Bay	3.9	6.0

3.4 *Extended bending beam rheometer testing*

The extended bending beam rheometer results for the North Bay and Cochrane materials are provided in Table 2. The stiffness and m-value after one hour of conditioning at the respective pass and fail test temperatures provides the AASHTO M320 grade whereas the warmest limiting temperature after 72 hours of conditioning at both -10°C and -20°C provides the LS-308 grade. In addition to the LS-308 grade, the worst grade loss from one hour to three days is provided for each binder.

Table 2. Regular and Extended Bending Beam Rheometer Analysis on Recovered Materials

Sample	AAHTO M320 Low Grade, °C	LS-308 Low Grade, °C	LS-308 Three Day Grade Loss, °C
Cochrane	-38.0	-38.6	1.8
North Bay	-27.4	-21.4	6.5

4 DISCUSSION

The construction quality assurance test results for both contracts were governed by Ontario's end result specification (ERS) system. Under the ERS the contractor either receives a bonus for superior construction quality (low voids, high compaction, good asphalt cement content, etc.) or is penalized or forced to replace the pavement for borderline and gross failures in meeting the specifications. There were no irregularities in quality that can explain the premature and excessive cracking in the North Bay contract. Both roads were newly constructed on thick layers of fresh granular material and the underlying sub-grade in North Bay was largely rock based while in Cochrane this was sedimentary clay. Both the Cochrane and North Bay contracts specified PG 52-34 asphalt cement grades.

The Cochrane area reached low surface temperatures of close to -34°C during a cold spell between January 9 and 15, 2004 (Hesp et al. (2009)). The North Bay contract would have reached low temperatures a few degrees higher during the same period of early 2004.

The Cochrane contract has remained largely free of cracks over its entire 30 km length except for a few occurrences over culverts. Cracking in North Bay has been consistently severe over the entire 10 km contract length. The distress started as wheel path longitudinal cracking in early life but has since progressed to include transverse and block type cracking.

The presence of waste engine oil residue in the North Bay contract has made the binder more susceptible to chemical and physical hardening (Hesp and Shurvell (2010), Hesp et al. (2011)). The typically large amounts of paraffin in waste oil residue make the asphaltene fraction less soluble in the maltene phase and this in turn predisposes the binder to accelerated physical and chemical hardening.

The high degree of chemical hardening is clearly evident from the differences in failure properties (Table 1) and low temperature grades (Table 2). The higher CTOD for the Cochrane material shows that this pavement will likely be able to withstand many more years of low temperature exposures. The PAV residue of good quality western Canadian asphalt cement has a CTOD of around 14-16 mm. A lower limit of 14 mm is set in Ontario as an acceptance criterion for PG -34 grades. The regular AASHTO M320 grade of the recovered material from Cochrane shows that this pavement has hardly aged and the LS-308 grade shows that it also suffers little from the effects of cold conditioning. In contrast, the North Bay material has chemically aged considerably and is also significantly more susceptible to physical hardening.

The fact that the recovered grade in North Bay is around -27.4°C or slightly more than a full grade above the required -34°C indicates that the confidence that the surface is not exposed to damage has been reduced from the intended 98% to around 50%. If the true grade is better represented by the -21.4°C value as obtained according to the LS-308 protocol (which we believe to be correct) then the confidence is further reduced to around 10%. This analysis reveals some of the deficiencies of the current AASHTO M320 specification. Superpave clearly rewards the wrong modification technologies. Canada is awash in superior quality asphalt cement but in Ontario this has not been used to construct superior performing pavements. The presented case study from North Bay is just one of numerous contracts that we have investigated for premature and excessive cracking due to the presence of waste engine oil sludge and other detrimental additives (Hesp et al. (2009), Hesp and Shurvell (2010), others).

It is possible that other factors have contributed to the excessive and premature cracking. Waxes and polyphosphoric acid are known to have profoundly negative effects and these were not tested for in this study (Hesp et al. (2007), Kodrat et al. (2007)).

5 CONCLUSIONS

Given the findings reported in this paper, the following conclusions are offered:

- The current AASHTO M320 specification as implemented in much of North America fails to adequately prevent thermal and traffic induced cracking and promotes the use of undesirable modification technology.
- Waste engine oil sludge that is widely used to modify asphalt cement is easily detected with X-ray fluorescence through the presence of zinc and molybdenum, elements that originate from anti-wear additives in all lubricating oils.
- Paraffins in engine oil sludge precipitate asphaltenes from the base asphalt cement or those formed through chemical oxidation. This premature precipitation leads to increased hardening.
- Parrafins in waste engine oil sludge promote additional physical hardening during cold storage.

The problem that we have identified is of a significant enough magnitude that it deserves further discussion between users and producers of asphalt cement.

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