Impact of re-refined engine oil bottoms on binder properties and mix performance on two pavements in Minnesota

Gerald Reinke[1, a], Andrew Hanz[1, b], R. Michael Anderson[2, c], Mary Ryan[1, d], Steven Engber[1, e], Douglas Herlitzka[1, f]

[1] *MTE Services, Inc., Onalaska, United States*
[2] *Asphalt Institute, Lexington, United States*

[a] gerald.reinke@mteservices.com
[b] andrew.hanz@mteservices.com
[c] manderson@asphaltinstitute.org
[d] mary.ryan@mteservices.com
[e] steve.engber@mteservices.com
[f] doug.herlitzka@mteservices.com

Digital Object Identifier (DOI): dx.doi.org/10.14311/EE.2016.284

ABSTRACT

The use of vacuum distilled residues from the re-refining of used motor oils as blend stock in paving grade bitumen has occurred in isolated markets in North America for more than 30 years. Recently in the United States the increasing need for low stiffness bitumen for use in high binder replacement mixtures, coupled with economic considerations, has led to an expanding market for these products. Little in-depth investigation into the long-term impact of these additives on mixture performance has taken place until recently. Conflicting studies from researchers in Canada and private organizations have been published. Here, we review the eight-year performance of four virgin mixture test sections placed on a county highway Minnesota. Three test sections used PG 58-28 bitumens from different crude sources, one of which contained approximately 8% of the re-refined engine oil residuum. The fourth utilized PG 58-34 polymer-modified bitumen. Pavement distress surveys were conducted over the eight year period. Tests of the original bitumen following extended laboratory aging were conducted. Properties of the recovered bitumen from eight-year-old field cores were correlated to pavement distress. Double Edge Notched Tension (DENT) tests were conducted on 20- and 40-hour PAV residues of the original bitumen. Iatroscan and extensive rheological testing were conducted on laboratory aged bitumen and on bitumen recovered from laboratory aged mixtures and field cores to determine the colloidal index, rheological index and the extent to which laboratory aged and recovered bitumen materials were m-controlled. Also evaluated were three test sections constructed at the Minnesota DOT MnROAD test site. A comparative study of PG 58-28, PG 58-34 (PMA) and PG 58-40 (PMA +re-refined engine oil bottoms) were evaluated over eight years. Bitumen used on the projects was tested as described above. The PG 58-40 exhibited the worst performance. The results of binder evaluations correlated to the fatigue cracking observed in the field with the test sections containing the re-refined engine oil residuum exhibiting the most extensive fatigue cracking. Based on this investigation we provide recommendations for test criteria to identify bitumens that could increase an asphalt mixture's likelihood to exhibit poor durability and fatigue cracking performance.

Keywords: Complex Modulus, Fatigue Cracking, Low-Temperature, Reclaimed asphalt pavement (RAP) Recycling, Rheology

1. INTRODUCTION

The use of vacuum distilled residues from the re-refining of used motor oils as a blend stock in paving grade bitumen has occurred in isolated markets in North America for more than 30 years. Published information discussing the potential benefits of the product are more recent (Herrington, 1992) (Hayner, 1999) (Villanueva A., 2008). Within the last three years, the increasing need for low stiffness bitumen for use in bituminous mixtures with ever increasing amounts of RAP and RAS (Reclaimed Asphalt Shingles) has created a growing market for this low cost residual by-product. In North America the availability of relatively inexpensive energy in the form of natural gas has resulted in bituminous paving companies switching from burning used motor oils in their production facilities and thus more used motor oil is being re-refined creating more of the residual by-product. Re-refined used oil bottoms have gone by many names in the bitumen supply industry, which has had the effect of creating uncertainty as to when the product had been added to bitumen. The current nomenclature agreed upon within the last year by suppliers in North America is Vacuum Tower Asphalt Extender (VTAE), but the common name used in many studies is Re-refined Engine Oil Bottoms (REOB) and is the term used in this paper. In Europe the same material is being marketed under several different product descriptions, among them flux oil, vacuum residue, and asphalt. Within the last five years some researchers have raised concerns that the use of REOB in bitumen has led to early pavement cracking. Tests have been developed by Hesp and his co-workers to identify bitumen properties believed to be related to the presence of REOB and indicative of poor pavement performance (Hesp S. &., 2010) (Rubab, 2011). Within the last 18 months, MTE Services became aware of two research projects constructed in Minnesota, USA in 1999 and 2006 that utilized REOB in one test section of each project. The research objective of neither project was an evaluation of REOB. Extensive records detailing laboratory tests of the original mixtures and the in service performance of the test sections on each project were available. Original bitumen samples were available from each research project as well as original mixture samples collected from the 2006 project. MTE's investigation of historical field performance data and new data generated from the available materials form the basis of the current research.

2. RESEARCH BACKGROUND

MTE began studying the impact of REOB on bitumen properties in May of 2014 as a result of discussions at the April 2014 meeting of the Asphalt Institute. Suppliers of REOB and Canadian Asphalt Institute members requested that AI form a task force to develop a state of the knowledge document on REOB and its impact on pavement performance. This request was initiated because the Ontario Ministry of Transportation (MTO) had implemented bitumen specifications aimed at limiting the amount of REOB that could be present in bitumens supplied to MTO. MTO based their limit on tests and data provided by Hesp in a report to MTO covering his research (Hesp S. (., 2006). Having relatively little first hand knowledge of the impact of REOB on bitumen properties, especially aged bitumen properties, MTE began to investigate bitumens blended with REOB and other additives capable of reducing the low temperature stiffness properties of bitumens. The elements of MTE's initial study of REOB are shown in Table 1. Two samples produced from PG 64-22 bitumen blended with 5% REOB and 5% of a bio derived oil were compared unaged, after RTFO, 20 hour and 40 hour PAV aging. The inclusion of a bio derived oil in the initial investigation was to provide a basis of comparison for the performance behavior of bitumens produced with these two different additives used to soften bitumen. The impact of adding 20% bitumen recovered from post-consumer waste shingles to the REOB and bio derived oil blends was also evaluated, as one of the target applications of softening bitumen with oil additives is to enable use of higher levels of recycled bitumen products.

Testing consisted of determining the high temperature bitumen grade using the dynamic shear rheometer (DSR) according to ASTM D7175 and the use of a 4 mm DSR geometry test methodology developed by Western Research Institute (Sui C. Farrar, 2010) consisting of a frequency sweep from 0.2 to 100 radians/sec at temperatures ranging from -40°C to 50°C. All 4 mm data was generated on TAInstruments DHR3 Dynamic Shear Rheometers equipped with air chillers capable of achieving -50°C. The 4 mm data was used to determine the low temperature properties using an analysis procedure reported at TRB in 2011 (Sui C., 2011) and revised by Farrar, et al at the 2012 E&ECongress in Istanbul (Farrar, 2012). Based on the determination of the low temperature stiffness critical grade (S-critical) and the low temperature relaxation critical grade (m-critical) a parameter (designated ΔTc) was calculated by subtracting the m-critical low temperature grade from the S-critical low temperature grade. The impetus for generating the ΔTc parameter was research reported by Anderson, et al at the 2011 AAPT meeting (Anderson, 2011), which identified ΔTc as a parameter that captures bitumen relaxation properties and correlated ΔTc to non-load associated cracking . Anderson's work had generated the ΔTc parameter as m-critical – S-critical which resulted in positive value for ΔTc as the bitumen aged. The choice to reverse the calculation was predicated on having the ΔTc parameter becoming more negative as the bitumen aged thus indicating greater potential for pavement distress as the bitumen in the mix became more m-controlled. From the 4 mm DSR data the Rheological Index (R-Value) (Christensen, 1992) at a reference temperature of 25°C was also calculated as another measure of bitumen relaxation. R-value was identified in the SHRP program as a shape parameter for the complex modulus master-curve, and is established as sensitive to aging. At this stage of the investigation data was being developed to quantify the impact of REOB and other softening agents on bitumen durability properties by evaluating ΔTc and R-value at multiple aging conditions..

Figure 1 is a composite plot exemplifying many of the concepts relevant to the current study that utilizes data for samples discussed in more detail later. The relaxation modulus plots (data traces from upper left to lower right) generated at a reference temperature of -25°C show that depending on the bitumen the rate of relaxation varies with increasing relaxation time. A reduced time of 60 seconds is indicated because utilizing updated work of Farrar, et al (Farrar, 2012) the relaxation modulus (G(t)) at 60 seconds and the slope of G(t) at 60 seconds is used to calculate the 4 mm DSR equivalent of BBR stiffness and m-value. Determining G(t) and slope of the relaxation modulus mastercurves derived at higher and lower reference temperatures allows one to interpolate results that allow calculation of the limiting S-critical and m-critical values equivalent to those produced via the BBR. MTE utilizes a 4 mm DSR limiting G(t) relaxation modulus value of 143 MPa (equivalent to 300 MPa from the BBR) and a m-critical (slope) value of -0.275 (equivalent to 0.300 BBR) rather than the value of -0.28 proposed in (Farrar, 2012) because of better correlation to BBR m-critical results. The second reduced time reference point is at 1E7 seconds. Examining the rate of change of the relaxation moduli for these bitumens between 60 seconds and 1E7 seconds demonstrates that the bitumen with highest modulus at 60 seconds relaxes at a faster rate than the two other bitumen materials. The bitumen with the lowest relaxation modulus at short relaxation times ultimately has a relaxation modulus value equal to the bitumen with the second highest relaxation modulus at low relaxation times. This inability to relax stiffness with increasing time or decreasing frequency is the basis for understanding the causation of the pavement distress discussed later. The two complex shear modulus (G*) plots (data traces from upper right to lower left) are shown at a reference temperature of +25°C. The MN1-5 bitumen is stiffer at the higher frequencies, but the MN1-4 bitumen stiffness does not relax with decreasing frequency as rapidly as MN1-5. Also graphically shown is the determination of the Rheological Index (R-Value), defined as the difference between the glassy modulus and cross-over modulus (where G'=G"). Because of the reduced ability to relax stiffness, the MN1-4 bitumen's crossover modulus occurs at a substantially lower frequency than that of the MN1-5 bitumen resulting in a greater difference between the glassy modulus (1E9 Pa) and the crossover modulus and hence a greater R-Value.

After removal of asphaltenes as n-heptane insoluble (ASTM D3279), Iatroscan (NTS Iatroscan® MK-6) evaluation of the remaining maltene fraction of all bitumen blends at the four aging conditions was performed. Using the procedure described by Loeber, et, al (L. Loeber, 1998) the Colloidal Index (CI), $\frac{Resins+Cyclics}{Asphaltenes+Saturates}$, was calculated from the Iatroscan data. As the bitumen ages the asphaltenes increase, saturates change (increase) slightly, cyclics decrease and resins increase. Addition of REOB causes the saturate content to start higher than for the unaged base bitumen. Use of bio derived oil, because it contains little or no saturated chemistry, results in lower saturates relative to the unaged base bitumen. The Colloidal Index column in Table 1 shows that regardless of the bitumen source or blends as the bitumen ages the Colloidal Index decreases.

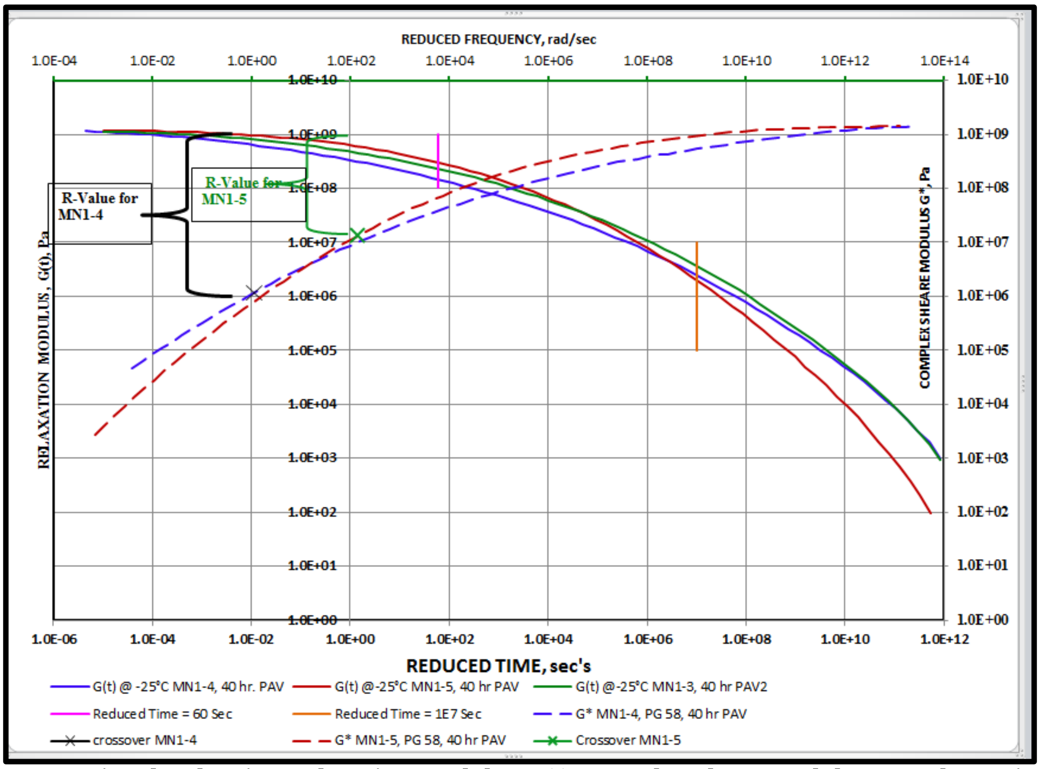

Figure 1: Composite plot showing Relaxation Modulus G(t), Complex Shear Modulus G*, Change in slope of G(t) with time & G* with Frequency

Examination of the high temperature DSR data, the S-critical, m-critical and ΔTc data in Table 1 shows that 5% REOB does not change the high temperature grade of PG 64-22 and reduces the low temperature PG grade after 20 hour PAV aging by 2°C which is not sufficient to change the low temperature grade. Addition of 5% bio derived oil reduces the high temperature grade by 8.8°C and the low temperature grade by 6°C. Regardless of the oil additive type, all of these materials are m-controlled. The REOB and bio derived oil blends containing shingle bitumen do not show much difference in the low temperature PG grade, relative to the blends without shingle bitumen, based on the standard 20 hour PAV. After an additional 20 hours of PAV aging the ΔTc values for all samples decrease, primarily because the m-critical temperatures increase at a faster rate than the S-critical temperatures. As a summarization of the initial investigation of REOB and other softening materials, it was found that 5% REOB in this particular PG 64-22 can neither change the high nor the low temperature grade of the bitumen. After 40 hour PAV aging the use of REOB causes the bitumen blend to become more m-controlled than a blend produced with an equivalent amount of a bio derived oil and also more m-controlled than the original base bitumen. Neither additive can prevent a continuing decrease in the ΔTc parameter resulting from addition of 20% shingle bitumen to the blends.

TABLE—1: Summary of Test Results for REOB, Bio Oil and Shingle Bitumen Blends

MATERIAL	AGING	DSR Grade, °C	S-critical, °C	m-critical, °C	Δ Tc, °C	R-value	CI
64-22 base	Unaged	67.1	-30.3	-32.2	1.8	1.582	3.808
64-22 base	RTFO	68.3	-29.4	-30.7	1.3	1.782	3.132
64-22 base	20 hr PAV		-27.1	-26.2	-0.86	2.174	2.436
64-22 base	40 hr PAV		-25.8	-23.2	-2.6	2.506	2.185
64-22 + 5% REOB	unaged	64.3	-34.6	-35.6	1.3	1.669	3.673
64-22 + 5% REOB	RTFO	65.5	-32.9	-33.4	0.6	1.937	3.065
64-22 + 5% REOB	20 hr PAV		-30.9	-28.2	-2.7	2.439	2.390
64-22 + 5% REOB	40 hr PAV		-29.8	-24	-5.8	2.826	2.115
64-22 + 5% BIO OIL	unaged	58.3	-37.1	-39.3	2.2	1.549	4.051
64-22 + 5% BIO OIL	RTFO	59.9	-35.8	-37.1	1.2	1.783	3.525
64-22 + 5% BIO OIL	20 hr PAV		-33.3	-32.2	-1.1	2.208	2.559
64-22 + 5% BIO OIL	40 hr PAV		-31.5	-29.6	-1.9	2.501	2.219
64-22 +20% SB+ 5% REOB	unaged	69.5	-34.5	-35.4	0.9	2.014	2.861
64-22 +20% SB+ 5% REOB	RTFO	72.1	-33	-32.4	-0.6	2.355	2.344
64-22 +20% SB+ 5% REOB	20 hr PAV		-31.9	-28.6	-3.3	2.812	2.058
64-22 +20% SB+ 5% REOB	40 hr PAV		29.8	-22.7	-7.1	3.307	1.833
64-22 + 20% SB + 5% BIO OIL	unaged	63.5	-38.2	-39.8	1.6	1.881	3.054
64-22 + 20% SB + 5% BIO OIL	RTFO	65.7	-35.9	-37.7	1.8	2.099	2.661
64-22 + 20% SB + 5% BIO OIL	20 hr PAV		-33.9	-32.4	-1.5	2.632	2.208
64-22 + 20% SB + 5% BIO OIL	40 hr PAV		-31.5	-27.7	-3.8	2.948	1.933

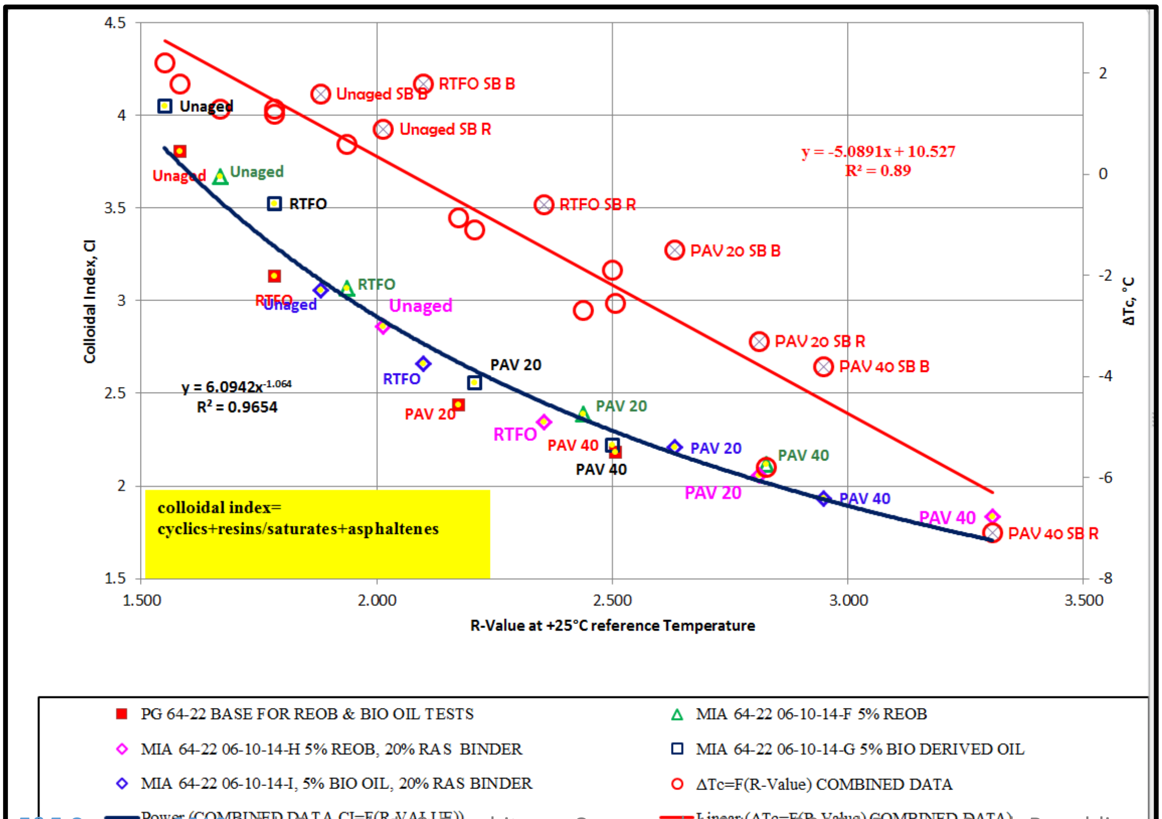

Figure 2: Colloidal Index as a Function of R-Value & ΔTc as a Function of R-Value for REOB, Bio Oil and Shingle Bitumen Blends

Figure 2 is a composite plot of Colloidal Index and ΔTc as a function of R-Value for the five bitumen samples and all aging conditions shown in Table 1. The Colloidal Index and R-Value parameters derived from two completely different tests exhibit a very strong correlation showing that as the R-Value increases the Colloidal Index decreases. Aging factors that cause a decrease in Colloidal Index such as an increase in asphaltenes and a greater decrease in cyclics relative to increase in resins are the same factors resulting in a reduced ability of the bitumen to relax stresses resulting in a reduced cross over frequency and therefore an increased R-Value. The R-Value and ΔTc parameters were derived from the same 4 mm DSR tests and should be correlated if the same mechanism that results in decreasing values of ΔTc, namely an decreasing ability of the bitumen to relax stresses at low temperature as it ages, also results in decreasing stiffness values at the crossover frequency and therefore increased R-Values. While the correlation for R-Value versus ΔTc is quite good at 0.89, it is not as robust as the R^2 for the Colloidal Index correlation with R-Value. It is worth noting that with one exception all of the data for the oil blends containing shingle bitumen plot above the regression plot of ΔTc versus R-Value, while the other data is on or below the fitted data plot. The shingle bitumen blends are labeled on Figure 2 using "SB" to denote shingle bitumen and "R" and "B" to denote REOB or Bio oil. This discrepancy between the 20% shingle blends and blends without shingle bitumen is most likely a result of the different chemistry of the oxidized shingle bitumen compared to the paving bitumen and the impact that has on the rheological behavior between the two bitumen materials. Similar issues with R-value were observed for polymer modified binders, as the presence of polymer has a significant impact on the relaxation behavior of modified asphalts (Reinke, 2015). Independent correlations between ΔTc versus R-Value for blends with and without shingle bitumen resulted in R^2 of 0.95. It is also worth noting that the relationship between R-Value and Colloidal Index does not exhibit any bias between the blends with and without shingle bitumen. The Colloidal Index is capable of capturing the compatibility variation resulting from the inclusion of shingle bitumen in the blends especially the changes in compatibility after extended aging.

In another study MTE investigated the impact of blending virgin paraffinic base oils into bitumen. It was found that 5% of a 130 cps viscosity virgin base blended into a PG 64-22 bitumen resulted in ΔTc after a 20 hour PAV aging period of – 3.1° C and a ΔTc after 40 hours in the PAV of -8.7°C (Reinke G. H., 2014). These ΔTc levels exceed the values for similar amount of REOB and indicate that the paraffinic oil component present in the REOB is the main reason for the low ΔTc values measured.

The basics learned from the testing summarized in Table 1 and Figure 2 and the concepts exemplified by Figure 1 carry over to testing performed for the two field projects detailed in this paper. Anderson,20 et al reached the conclusions in their 2011 paper based on recovered bitumen from aged field mixtures that a ΔTc equivalent to -2.5°C represented a cracking warning limit and a ΔTc equivalent to -5°C represented a bitumen at or beyond its cracking limit. Based on that interpretation the 5% REOB blend and the REOB blend plus shingle bitumen were well beyond their cracking limits after 40 hours of PAV aging. The bio derived oil plus shingle bitumen at 40 hours and the 20-hour PAV REOB plus shingle bitumen were also beyond the warming limit.

3. STUDY BACKGROUND

3.1 Olmsted County Highway 112 Comparative Crude Source Research, 2006 - Continuing

In August 2006 Rochester Sand and Gravel, a subsidiary of Mathy Construction Co, which is also the parent of MTE Services, participated in a comparative crude source research study in cooperation with Western Research Institute (WRI) (WRI Asphalt Research), Minnesota DOT and the Olmsted County, Minnesota highway department. The objective of the study was to evaluate the relative performance of bitumen meeting the same PG grade but derived from different crude sources. MTE Services chose the bitumen sources detailed in Table 2. The original study design included only three PG 58-28 bitumen products being used to produce virgin mixes. WRI requested inclusion of a virgin PG 58-34 PMA test section and that mix was then included in the study. The majority of the project was constructed using PG 58-34 bitumen with 20% RAP, which was the original project design. All mixtures were placed in 3 lifts on newly prepared aggregate base.

TABLE--2 Comparative Crude Source Study Information

COMPARATIVE CRUDE SOURCE STUDY OLMSTED COUNTY HIGHWAY 112			
BITUMEN GRADE	TEST SECTION	CRUDE SOURCE	Zinc, ppm
PG 58-34 PMA	MN1-2	Canadian Blend, Elvaloy® + PPA	Note 1
PG 58-28	MN1-3	Canadian Blend	12

PG 58-28	MN1-4	Arab heavy/Arab medium/Kirkuk blend	321
PG 58-28	MN1-5	Venezuelan blend	11

Note 1-Formulated at Mathy bitumen terminal and no REOB was used in formulation

WRI conducted distress surveys on the test sections annually from 2007 through 2012. In spring of 2014 the sales representative for the MN1-4 bitumen casually informed the Mathy bitumen supply department that the PG 58-28 bitumen supplied in 2006 was produced using REOB. Until that point, no one was aware of this fact. MTE had retained samples of the four bitumen products and an X-Ray fluorescence check of the three PG 58-28 bitumen products showed high zinc in the MN1-4 sample (Table 2), thus corroborating the use of REOB in the MN1-4 bitumen. The distress surveys through 2012 showed that section MN1-4 was had the greatest distress, but with the revelation that REOB was present in the MN1-4 bitumen MTE launched a full-scale investigation of the properties of the four virgin bitumen materials and the relationship with field cracking distress.

2.2 MnROAD Comparative Bitumen Study, 1999-2007

In 1999, a bitumen supplier in Minnesota sponsored a comparative performance study at the Minnesota test site known as MnROAD (www.dot.state.mn.us/mnroad/index.html). The study was designed to evaluate the cold weather and fatigue performance of three bitumen grades; a PG 58-28 straight run bitumen, a PG 58-34 PMA bitumen produced from a PG 52-34 base bitumen and a PG 58-40 PMA bitumen produced from the PG 52-34 to which a softening oil had been added to achieve the -40°C PG grade. Over the eight years of the comparative study it was found that the PG 58-40 had ultimately exhibited the worst performance of all the mixtures. In the spring of 2015 amidst the concerns being raised by several state DOT's regarding the use of REOB the question was posed by the head of the MN DOT bituminous laboratory as to whether the PG 58-40 placed on MnROAD had contained REOB. MTE had conducted two separate investigations of the three mixtures placed on this MnROAD project in 2000 and again in 2004 and retained original samples of the three bitumen materials. Table 3 shows the X-Ray fluorescence test results of the three bitumen samples. Further verbal confirmation of the use of REOB in the PG 58-40 bitumen were made by representatives of the supplier.

TABLE—3: X-Ray Fluorescence Results for MnROAD Bitumen Samples

X-Ray Fluorescence Test Results for Three MnROAD Bitumen Samples				
Bitumen grade	sulfur, %	phosphorus, %	molybdenum, ppm	zinc, ppm
58-28	4.896	0.001	9	19
58-34	4.374	0.001	8	10
58-40	3.969	0.059	18	925

The Olmsted county 112 comparative crude source study and the MnROAD comparative bitumen performance study each contained one test section constructed with a bitumen grade produced using REOB. For both projects, the test sections paved with bitumen containing REOB exhibited the highest level of distress after four plus years of service. MTE conducted a suite of tests similar to those discussed in Section 2 on the bitumen materials used to produce the MnROAD mixtures. The test properties of the bitumen materials were then correlated to the pavement distress performance.

4. TESTING AND RESULTS

4.1 Olmsed County Highway 112 Comparative Crude Source Study-Phase 1 Results

Two phases of investigation were conducted for this project. The first phase consisted of aging the retained bitumen samples used on the four virgin test sections through 40 hours of PAV conditioning. These results were then correlated against the distress survey data from 2012. Table 4 summarizes the results from 4 mm DSR testing, the Colloidal Index and asphaltene content determined prior to the Iatroscan evaluation of the maltene fraction.

TABLE-- 1: Summary of Results for Four Bitumen Materials Used on Olmsted County 112

Bitumen Source	Aging	S-Critical	m-critical	ΔTc	Asphaltenes, %	Colloidal Index	R Value
MN1-2	unaged	-38.7	-41.5	2.8	14.5	3.742	1.933
MN1-3	unaged	-35.3	-38.5	3.2	14.7	3.739	1.626
MN1-4	unaged	-38.5	-37	-1.5	18.4	2.806	2.037
MN1-5	unaged	-34.2	-37.7	3.5	12.7	4.291	1.370
MN1-2	RTFO	-37.7	-39.6	1.9	19.1	2.953	2.255
MN1-3	RTFO	-34.4	-36.7	2.3	17.9	3.000	1.909
MN1-4	RTFO	-38.3	-36.4	-1.9	19.8	2.581	2.465
MN1-5	RTFO	-32	-34.8	2.8	15.2	3.587	1.606

MN1-2	PAV-20	-35.5	-36	0.8	23.3	2.254	2.687
MN1-3	PAV-20	-31.9	-31.8	-0.2	22.2	2.493	2.416
MN1-4	PAV-20	-35.5	-30.7	-4.2	23.9	2.344	2.967
MN1-5	PAV-20	-30.5	-32.2	1.7	19.8	2.788	1.877
MN1-2	PAV-40	-34.6	-34.3	-0.3	25.1	2.015	3.061
MN1-3	PAV-40	-31.8	-28.1	-4.2	25.4	2.115	2.872
MN1-4	PAV-40	-35.2	-27.6	-7.6	25.5	1.889	3.281
MN1-5	PAV-40	-29.3	-30.1	0.8	21.8	2.497	2.162

TABLE--5: Summary of Distress Data from 2012 Survey, Length Data in Meters, Fatigue area in Meters^2

Bitumen Source	Transverse Cracks, m	Fatigue Cracking, area, m^2	Net Longitudinal Cracks, (WP, NWP- CL), m	Center Line, CL, m	Total Distress (Net Long + CL, m) + (Fatigue area, m^2)	Total Longitudinal (Total Cracks - Fatigue area), m
MN1-2	4.9	0	24.7	32.3	61.9	61.9
MN1-3	14.2	6	58.1	52.2	130.5	124.5
MN1-4	44.1	12.4	157.5	4.8	218.8	206.4
MN1-5	18	0	0	0	18	18

Table 5 summarizes the distress data from the 2012 survey. In figures below the centerline cracking is included as part of the plotted distress data. This decision seems reasonable considering that some test sections had no (MN1-5) or very little centerline cracking (MN1-4). The decision was also made to plot a Total Distress value, which summarizes all linear cracking and the area of fatigue cracking for each test section into a single distress value.

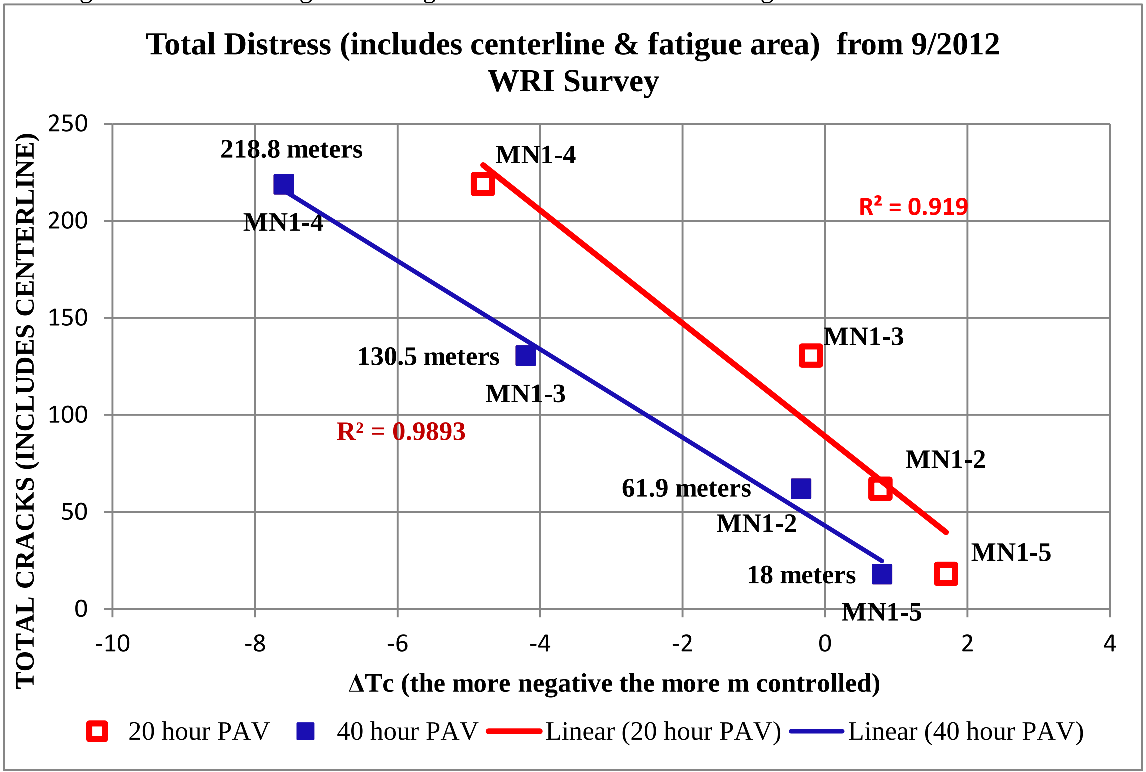

Figure 3: Total Pavement Distress as Function of ΔTc of 20 and 40 Hour PAV Residues

The correlation of total distress as a function of ΔTc is very good; it is slightly weaker for the 20-hour PAV data compared to the 40 hour PAV data. Plotting total distress as a function of S-critical temperature or m-critical temperature produced nonsense results (data plots not shown) (Reinke G. H., 2014). The data is provided for those wishing to engage in examining other relationships. Although ΔTc is a parameter derived from low temperature limiting stiffness properties, the fact that ΔTc is strongly correlated to the R-value (Figure 2), which is a direct indicator of aging and increases as the bitumen relaxation decreases implies that ΔTc should be a good predictor of fatigue or non-load associated cracking. . Figure 4 is a composite plot showing only longitudinal cracks but no transverse cracks (blue solid diamonds) and only transverse cracks (red solid squares) as a function of ΔTc. The relationship for transverse cracks is not as robust as is the relationship for longitudinal minus transverse cracking. In addition, the test section with bitumen exhibiting the best ΔTc value had 18 meters of transverse cracking.

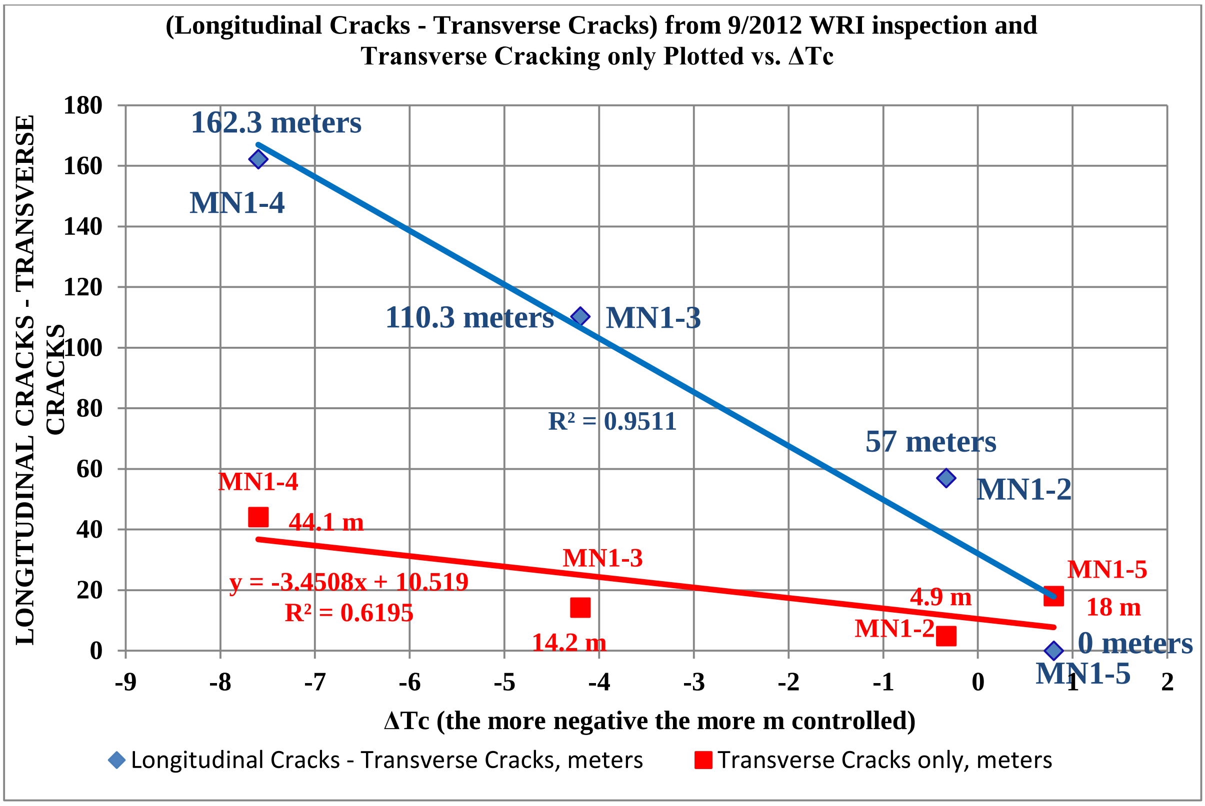

Figure 4: (Longitudinal Cracks - Transverse Cracks) and Transverse Cracks only Plotted as Function of ΔTc

4.2 Olmsted County Highway 112 Comparative Crude Source Study-Phase 2 Results

After performing the initial analysis of evaluating the relationship between ΔTc for 20 and 40 hours of PAV aging and obtaining a very good predictive correlation between ΔTc and the observed pavement distress a phase 2 investigation was launched. MTE commissioned the person who had performed the prior distress surveys to perform another survey in October of 2014. At the same time cores were cut from the different test sections for torsion bar modulus testing and bitumen recovery and testing. A second round of PAV aging of the four test bitumen materials was conducted. Sufficient 20 and 40 hour PAV aged material was generated so that Double Edge Notch Tension tests (DENT) could be conducted on the PAV samples. The DENT test for bitumen was developed by Hesp (Hesp S. G., 2009) to determine the Crack Tip Opening Displacement (CTOD) of the bitumen which he then correlated to field performance. PAV residue samples were submitted to the Asphalt Institute for DENT testing. Additionally, the top 12.5 mm of cores from each test section were extracted, recovered and subjected to 4 mm DSR testing and Iatroscan compositional testing. The results are reported in Table 6. Table 7 is a summary of the pavement distress data from the 2014 survey.

TABLE-- 2: Properties of Bitumen Recovered from Top 12.5 mm of 2014 Field Cores (NOTE: CTOD data was obtained from 20 and 40 hour PAV Residue)

Bitumen Source	Asphaltenes, %	CI	S_Critical, °C	m-critical, °C	ΔTc, °C	R-Value	CTOD_20 hr. PAV, mm	CTOD_40 hr. PAV, mm
MN1-2	23.3	2.300	-31.1	-30.2	-0.9	2.577	9.2	4.9
MN1-3	25.3	2.106	-28.7	-25.7	-3.0	2.365	6.2	4.3
MN1-4	26.7	1.863	-31.2	-24.8	-6.4	2.861	5.4	4.3
MN1-5	22.2	2.497	-27.8	-29.3	1.5	1.968	10.7	7.1

Results using the CTOD test generally have a lower limit of 3-4 mm; higher CTOD values should be indicative of better performance. The data presented in Table 6 show that after 40 hours of PAV aging three of the four bitumen materials tested are trending towards the lower limit. It was impossible to obtain sufficient recovered bitumen from the top 12.5 mm field core samples and therefore the best option for examining the relationship between CTOD and field performance was to utilize results on PAV residues.

Bitumen Source	Total Distress	Transverse cracks	Total Fatigue (Total distress-Transverse)	Center line	Non-Centerline_ fatigue	ΔTc 20 Hr. PAV	ΔTc 40 Hr. PAV
MN1-2	205.9	13.5	192.4	78.8	113.6	0.8	-2.6
MN1-3	363.4	19.5	343.9	73.3	270.6	-0.5	-4.2
MN1-4	472.6	51.2	421.4	82.2	339.2	-4.8	-7.6
MN1-5	44.1	19.5	24.6	12.3	12.3	1.7	0.8

TABLE-- 3: Summary of Distress Data from 2014 Survey

The only agency that currently requires DENT testing and utilizes CTOD data as a specification is the Ontario Ministry of Transportation. The current MTO requirements are listed in Table 8.

TABLE-- 4: Ontario Ministry of Transportation CTOD Specification

Low Temperature Performance Grade (LTPG) (°C)	CTOD (mm)		
	Acceptance, min	Major Borderline	Rejectable
XX-28	10	6	4
XX-34	14	10	8
XX-40	18	14	12

The MTO acceptance criteria are based on results from the 20-hour PAV test. Figure 5 is plot of the 20-hour CTOD data versus total pavement distress and also versus total transverse cracking in meters. As with the previous plots of total distress this data includes total linear cracking and block cracking in m². When the total distress metric is adjusted by removal of the transverse crack data the R^2 remains unchanged. Figure 6 is plot of the same distress values as a function of the ΔTc results determined by 4 mm DSR for the bitumen recovered from the top 12.5 mm field core layers.

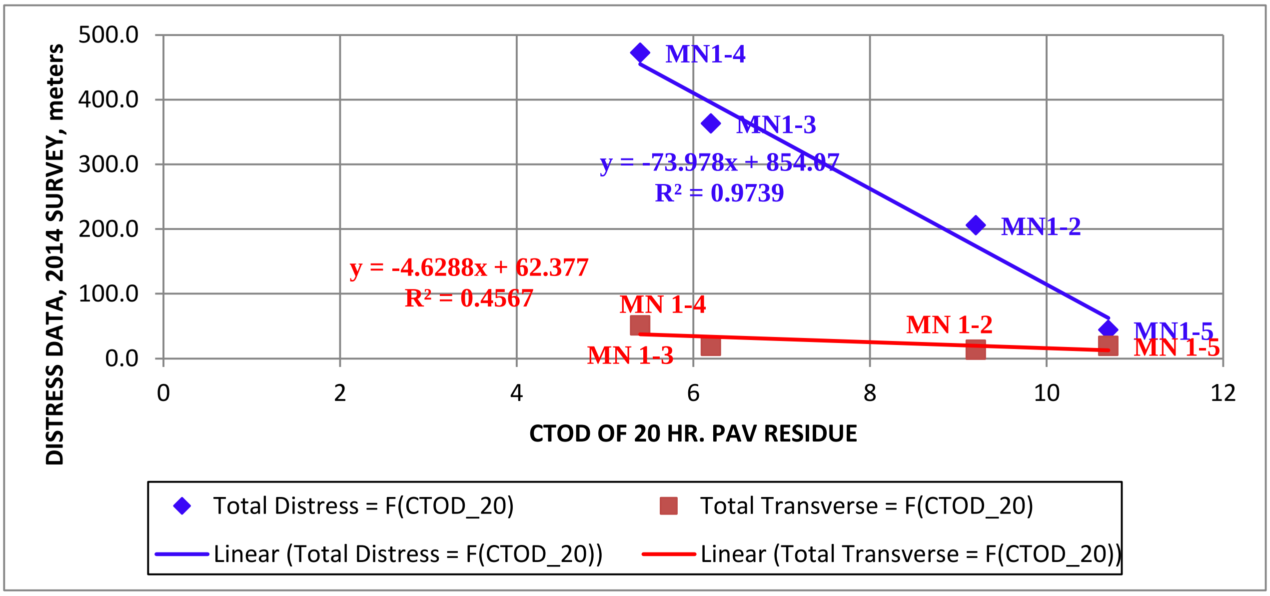

Figure 5: Total Distress and Transverse Distress plotted as Functions of CTOD for 20 hour PAV

The results shown in Figures 5 and 6 suggest that neither CTOD nor ΔTc is capturing the complete cause of single event thermal cracking as represented by transverse cracking. Both parameters are well correlated to total pavement distress, most of which for the Olmsted County 112 test sections is fatigue related. The 20 hour PAV CTOD data would have flagged MN1-4, MN1-3 and MN1-2 as borderline according to MTO criteria, which would have enabled them to be placed although MN1-4 and MN1-2 would have been penalized for being under the Borderline limit but still above the Rejectable limit. The 40-hour PAV ΔTc data for these four bitumens would have, according to the Anderson et. al criteria, flagged MN1-3 (ΔTc= -4.2) as beyond the warning limit and MN1-4 (ΔTc= -7.6) as well beyond the cracking limit. The ΔTc data for the bitumen recovered from the eight year old field cores show that both MN1-4 and MN1-3 are approaching the values obtained from the 40 hour PAV residue, however MN1-3 is at the warning limit and MN1-4 is beyond the cracking limit.

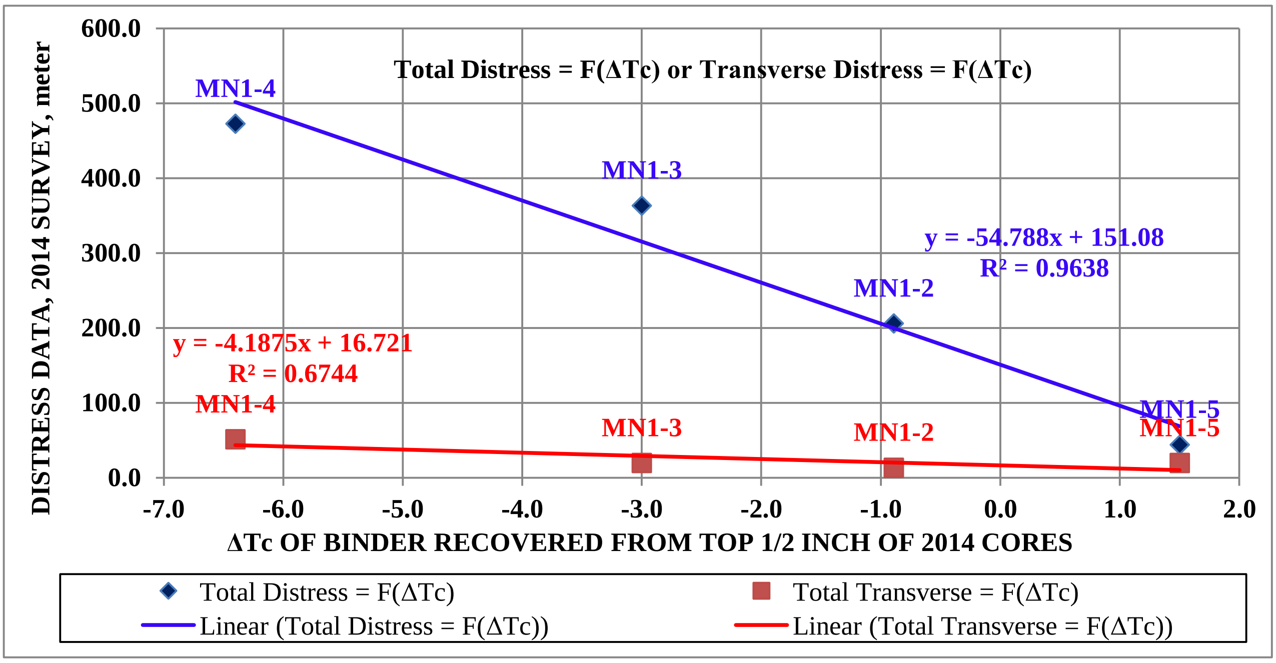

Figure 6: Total Distress and Transverse Distress plotted as Functions of ΔTc of 12.5 mm Recovered Bitumen

Figure 7 is a composite plot of ΔTc for the 20 and 40 hour PAV residues as a predictor of the ΔTc of the bitumen recovered from the top 12.5 mm of the eight year old field cores. The data shows that the 20 hour PAV residue under predicts the ΔTc of the eight year old pavement bitumen and the 40 hour PAV residue over predicts the eight year ΔTc by 1.5°C or less. For the Minnesota climate, which is temperate, the ΔTc of 40 hour PAV residue is a reasonable, if somewhat conservative; predictor of in service bitumen aging at eight years; for hotter climates the time frame could be reduced considerably.

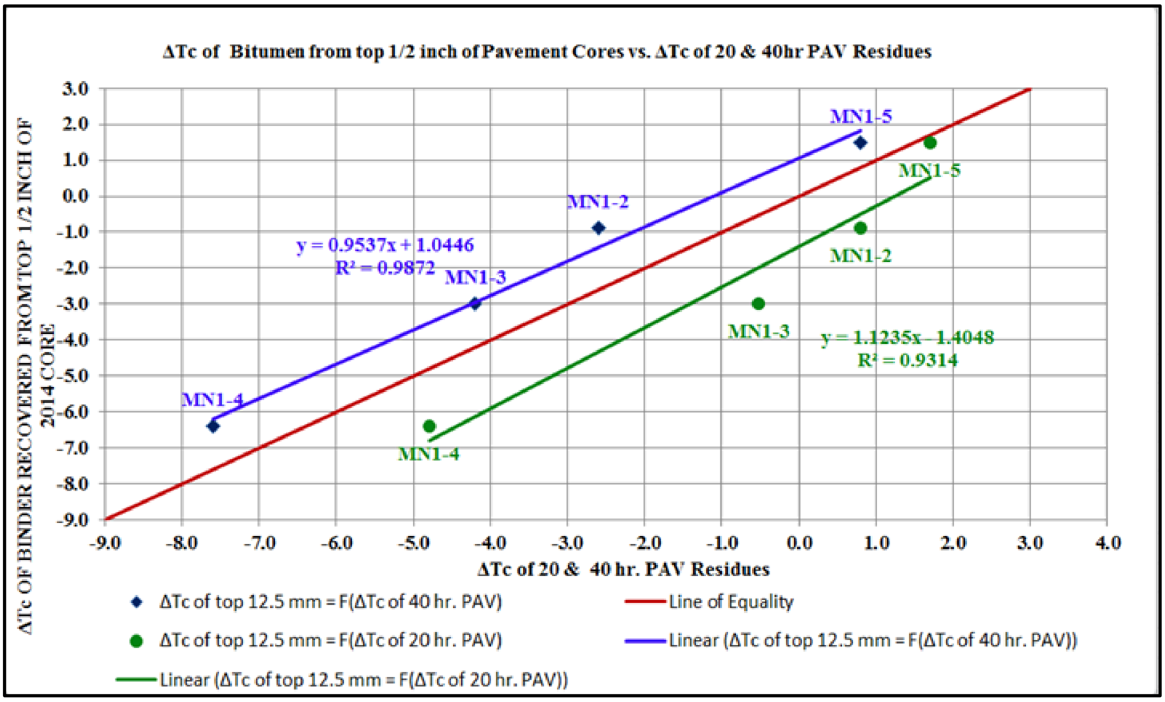

Figure 7: ΔTc of Bitumen from Top 12.5 mm of Field Cores vs. ΔTc of 20 & 40 hour PAV Residues

The data in Figure 5 shows the CTOD result for the 20 hour PAV residue correlates well with the eight year distress data and it should not be surprising given the relationship shown in Figure 7 that the ΔTc result of the 40 hour PAV residue correlates well with the eight year distress as well ($R^2 = 0.97$, data not plotted). A plot of CTOD results for 20 and 40 hour PAV residues also correlated well with ΔTc for those same samples (Reinke G. H., 2015). The relationship between R-value for MN1-2 through MN1-5 and total distress was investigated in a separate study but not included in this paper because strong relationships between performance and R-value were not observed (Reinke, 2015). Due to the influence of compositional factors and evaluation of the parameter at one reference temperature, R-value varies by asphalt binder type (i.e. unmodified, polymer modified, blended with RAP/RAS) and therefore is not a good performance indicator when comparing multiple binder formulations.

4.3 MnROAD Comparative Bitumen Study Results

Once MTE determined the presence of high levels of zinc and the presence of molybdenum in the PG 58-40 bitumen used on the MnROAD project, an investigation commenced to evaluate the pavement performance over time of the three test sections in relationship to the aging properties of the retained bitumen samples. The staff at MnROAD was very helpful in providing distress survey data for every year of the comparative study; unfortunately there were no retained mixture samples available to supplement the bitumen study. The investigation was quite straight forward; having performed two investigations of pavement performance versus bitumen properties for the Olmsted County 112 project, a similar methodology was employed. The three bitumen materials used on the MnROAD construction were tested through 20, 40 and 60 hours of PAV aging, 4 mm DSR testing was performed on the unaged, RTFO, 20, 40 and 60 hour samples and Iatroscan evaluation was performed. MTE had performed standard PG grading on the 3 bitumen samples in 2000 and that 20-hour PAV data was compared to the new 20 hour results using the 4 mm DSR. The test results are summarized in Table 9. Results for S-critical, m-critical and ΔTc between the 2000 BBR tests and the 2015 4 mm DSR tests are within 1°C for all data. Detailed distress data for MnROAD are available from the corresponding author.

TABLE-- 5: S-critical, m-critical, ΔTc for 20, 40 & 60 hour PAV of MnROAD Bitumen

	20 hr. PAV, 2000			20 hr. PAV, 2015			40 hr. PAV			60 hr. PAV		
Bitumen Grade	BBR, S, 2000	BBR, m, 2000	**ΔTc, 2000**	4 mm, S, 2015	4 mm, m, 2015	**ΔTc, 2015**	4 mm, S, 2015	4 mm, m, 2015	**ΔTc, 2015**	4 mm, S, 2015	4 mm, m, 2015	**ΔTc, 2015**
58-28	-30.9	-30.3	**-0.5**	-31.3	-30.5	**-0.8**	-29.5	-26.7	**-2.8**	-28.5	-22.7	**-5.8**
58-34	-34.8	-35.4	**0.6**	-35.6	-35.4	**-0.2**	-34.9	-32.4	**-2.5**	-33.1	-27.6	**-5.5**
58-40	-44.2	-42.9	**-1.3**	-44.4	-42.0	**-2.4**	-42.9	-34.6	**-8.3**	-42.9	-30.5	**-12.4**

The distress disparity over time is so skewed towards the PG 58-40 bitumen mix that it is difficult to show perspective when comparing the test sections. Figure 8 is a comparison plot showing the total linear cracking (without the center line crack length) for the three bitumen materials at 4, 5.5 and 7.5 years of service as functions of ΔTc for the 40 hour PAV. The distress was not evident at year three and was not severe at year four, but increased substantially after year four. It is evident from Figure 9 that the PG 58-40 underwent an substantial increase in cracking between year 4 and year 5.5. A ratio of the slopes of the fitted data plots shows that between years 4 and 5.5 the rate of change of cracking was 12.6 and rate of change between years 5.5 and 7.5 was 1.9. Fitting a correlation coefficient to the three data points for each year is somewhat meaningless given the similarity of the data for the PG 58-28 and PG 58-34 sections, however the change in the slopes in the trend lines demonstrates the rate at which the severity of the distress in the PG 58-40 section increased with aging time.

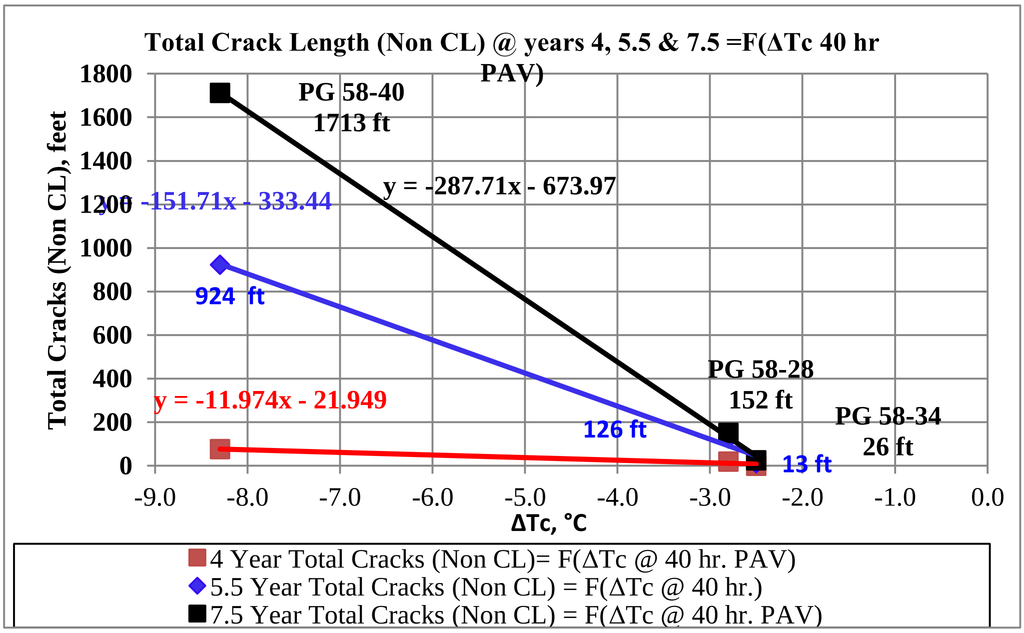

Figure 8: Total Crack Length (Non CL) at Years 4, 5.5 and 7.5 of Service as Function of ΔTc 40 hr PAV

Figure 9 is a comparison plot showing the change in ΔTc for each of the MnROAD bitumen materials over the 20, 40 and 60 hour PAV aging cycles. The PG 58-28 and PG 58-34 have nearly the same rate of decrease in ΔTc with PAV aging time as exemplified by the slopes of the fitted data. The PG 58-40 however exhibits double the rate of decrease in ΔTc with PAV aging time. Given that the PMA PG 58-40 was produced from the same PG 52-34 base used to produce the PMA PG 58-34 and the same polymer was used for those two bitumens, the only chemical factor contributing to this accelerated rate of change in ΔTc is the REOB additive.

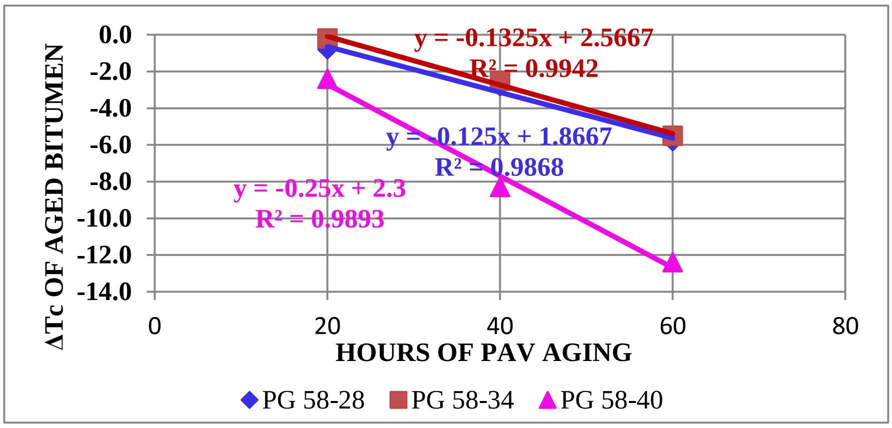

Figure 8: Variation in the Rate of Change of ΔTc with PAV Aging Time for MnROAD Bitumen

It is easy to look back with hindsight and identify causative relationships between test properties of bitumen and the development of pavement distress. In the case of this particular project, data that might have suggested a problem in the making was available, but no one bothered to perform the evaluation that might have foretold the problem. In 2000 MTE tested the three MnROAD mixtures for low temperature thermal cracking using the Low Temperature Indirect Tensile Test developed during SHRP. In the course of that study the compacted mixtures were aged for 5 and 10 days at 85°C prior to testing. As expected the PG 58-40 exhibited the best low temperature cracking results because even after 10 days of aging the recovered bitumen still maintained the lowest low temperature stiffness grade (Table 10). Bitumen had been extracted from the 5 and 10 day aged mixtures followed by BBR characterization and determination of Critical Cracking Temperature (CCT) using the direct tension test. The data missed and in fact never calculated were the ΔTc values. In 2000 would anyone have suspected the implications of a ΔTc value of -4.51°C for the recovered bitumen from the 10 day aged mix?

TABLE-- 6 : Summary of BBR and CCT Test Results on Bitumen Recovered from Aged Mixtures

Bitumen	Aging condition	BBR S critical, °C	BBR m critical, °C	Δ Tc, °C	CCT, °C
PG 58-28	PAV	-30.87	-30.34	-0.53	-27.7
PG 58-28	5 day aged IDT specimens	-31.72	-32.17	0.45	-29.4
PG 58-28	10 day aged IDT specimens	-30.85	-29.08	-1.77	-26.7
PG 58-34	PAV	-34.77	-35.36	0.59	-33.4
PG 58-34	5 day aged IDT specimens	-35.15	-36.2	1.05	-34.4
PG 58-34	10 day aged IDT specimens	-35.45	-35.98	0.53	-35.9
PG 58-40	PAV	-44.18	-42.92	-1.26	-46
PG 58-40	5 day aged IDT specimens	-44.93	-43.27	-1.66	-42.6
PG 58-40	10 day aged IDT specimens	-43.73	-39.22	-4.51	-42.6

5.0 CONCLUSIONS

1. The main distress factor caused by the use of REOB is the reduction in ability of the bitumen to relax stress as it ages.
2. Results discussed in this paper and available at www.asphaltetgs.org showed that blends of virgin paraffinic lube base oils in a bitumen samples produce ΔTc values after 40 hours of PAV aging equal to or more negative than blends produced with the same amount of REOB in the same bitumen. The implication of this comparison is that the paraffinic or saturated chemistry of the additives is the main reason for the reduction in bitumen relaxation properties.
3. Determination of the parameter ΔTc (S critical low temperature grade – m critical low temperature grade) after 40 hours of PAV aging was strongly correlated to pavement performance on the two Minnesota test projects reported in this study
4. ΔTc levels of -5°C and lower were associated with high levels of pavement distress at 5 years of service and longer.
5. The common factor associated with the greatest level of pavement distress on the Minnesota projects was the presence of REOB in the bitumen

6. Testing reported here showed that for a PG 64-22 bitumen the addition of 5% REOB resulted in more than a doubling of the ΔTc value after 40 hours of PAV aging compared to the base bitumen. The addition of an equal amount of a bio derived oil to the PG 64-22 resulted in a 25% decrease in the ΔTc value after 40 hours of PAV aging.
7. PAV aging of 20 hours is not sufficient to stress the bitumen to result in ΔTc properties equivalent to the recovered bitumen properties after 8 years in service in south central Minnesota. PAV aging of 40 hours is a closer approximation to the 8 year properties. It is unknown what the relationship would be for warmer climates.
8. Of the bitumen parameters evaluated in this study ΔTc is most blind to bitumen formulation, composition and aging. ΔTc is based on determination of specific stiffness values regardless of temperature to determine the S and m critical values. CTOD is performed at 15°C and therefore the result is impacted by bitumen stiffness and elastic properties at that temperature. Rheological index (R-value) is impacted by cross over frequency which at any given determination temperature will be impacted by bitumen composition such as high levels of RAP and/or RAS and also polymers.

Works Cited

Anderson, R. M. (2011). Evaluation of the Relationship between Asphalt Binder Properties and Non-Load Related Cracking. Association of Asphalt Paving Technologists, 2011 Volume 80, pp 615-663.

Christensen, J. D. (1992). INTERPRETATION OF DYNAMIC MECHANICAL TEST DATA FOR PAVING GRADE ASPHALT CEMENTS. *Association of Asphalt Paving Technologists, Vol 61*, (pp. 68-116).

Farrar, M. G. (2012). Thin Film Oxidative Aging and Low Temperature Performance Grading Using Small Plate Dynamic Shear Rheometry: An Alternative to Standard RTFO, PAV and BBr. *Eurasphalt & Eurobitume 5th E&E Congress-2012 Istanbul* (pp. Paper O5ee-467). Istanbul: Foundation Euraspalt.

Hayner, R. E. (1999, June 15). *Patent No. 5911817.* USA.

Herrington, P. (1992). Use of rerefined oil distillation bottoms as extenders for road bitumens. *Journal of Materials Science 27(1992)*, 6615-6626.

Hesp, S. &. (2010). X-Ray fluorescence detection of waste engine oil residue in asphalt and its effect on cracking in service. *International Journal of Pavement Engineering 11(6)*, 541-553.

Hesp, S. G. (2009). Five year performance review of a northern Ontario pavement trial. *Proceedings, Canadian Technical Asphalt Association*, (pp. 99-126). Moncton, NB.

L. Loeber, G. M. (1998). Bitumen in colloid science: a chemical, structural and rheological approach. *Fuel, Vol 77, No 13*, 1443-1450.

NTS Iatroscan® MK-6. (n.d.). Retrieved from www.ntsinstruments.com: http://www.ntsinstruments.com/sara-method.html

Reinke, G. H. (2014, September 17). A DISCUSSION OF SOME FACTORS IMPACTING PERFORMANCE OF BINDERS BLENDED WITH ADDITIVES FOR REDUCING LOW TEMPERATURE PROPERTIES OF ASPHALT BINDERS & THEIR IMPACT ON MIX PERFORMANCE. *FHWA Mixture and Binder Expert Task Group Meeting*. Baton Rouge, LA, USA. www.asphaltetgs.org

Reinke, G. H. (2015). Further Investigations into the Impact of REOB & Parafinnic Oils on the Performance of Bituminous Mixtures. *FHWA Binder ETG Meeting.* Fall River, MA. www.asphaltetgs.org

Rubab, S. H. (2011). Effects of engine oil residues on asphalt cement quality. *Proceedings, Canadian Technical Asphalt Association*, (pp. 1-12). Quebec City, Quebec.

Sui C. Farrar, M. T. (2010). *"New Technique for Measuring Low-Temperature Properties of Asphalt Binders with Small Amounts of Material".* Transportation Research Record, Journal of the Transportation Research Board, Vol. 2179.

Sui C., F. M. (2011). *"New Low Temperature Performance Grading Method Using 4mm Parallel Plates on a Dynamic Shear Rheometer".* Transportation Research Record, Journal of the Transportation Research Board," Vol. 2207,.

Villanueva A., H. S. (2008, February). Asphalt modification with used lubricating oils. *Canadian Journal of Civil Engineering, vol 35*, 148-157.

WRI Asphalt Research. (n.d.). Retrieved from Western Research Institute: http://www.westernresearch.org/transportation.aspx?id=168

www.dot.state.mn.us/mnroad/index.html. (n.d.). Retrieved from MnRoad Minnesota's Cold Weather Testing Facility: http://www.dot.state.mn.us/mnroad/index.html