asphalt institute

STATE-OF-THE-KNOWLEDGE

THE USE OF REOB/VTAE IN ASPHALT

IS-235

FOREWORD

The Technical Advisory Committee of the Asphalt Institute (AI) formed a task force (TF) in 2014 on re-refined engine oil bottoms (REOB), also known as vacuum tower asphalt extender (VTAE). The charge of the TF was to learn more about REOB /VTAE and its effects when blended into asphalt, and to recommend education, engineering and research efforts that may be needed. Members of the REOB/VTAE TF were from AI's Technical Advisory Committee and AI's Health, Safety & Environment Committee. A liaison from FHWA also served on the TF.

This document was produced by AI's REOB/VTAE TF and approved for publication by AI's Technical Advisory and Health, Safety & Environment Committees on April 12-13, 2016. It is provided as a free download on AI's website, www.asphaltinstitute.org.

Observations, conclusions and recommendations provided herein are based on the review of information as of the end of 2015.

INFORMATION SERIES NO. 235 (IS-235)

The Asphalt Institute can accept no responsibility for inappropriate use of this publication. All reasonable care has been taken in the preparation of this publication; however the Asphalt Institute can accept no responsibility for the consequence of any inaccuracies it may contain.

Printed in USA
First Edition
ISBN 978-1-934154-74-8
Library of Congress Control Number: 2016947584

asphaltinstitute.org

FHWA ACKNOWLEDGEMENT and DISCLAIMER

The development of Section III of this document was partially funded through the Federal Highway Administration (FHWA) Cooperative Agreement DTFH61-11-H-00033. The information presented does not reflect the official view of FHWA, nor does the U.S. Government assume any liability for the accuracy or use of the information in that section.

EXECUTIVE SUMMARY

This document provides a comprehensive review of available information regarding the modification of paving grade asphalt binders with material commonly referred to as Re-refined Engine Oil Bottoms (REOB) or Vacuum Tower Asphalt Extender (VTAE). Producers of REOB/VTAE report that their material has been used to modify asphalts since the 1980s. However, a heightened level of interest in this topic has recently developed. The intent of this document is to help facilitate informed decisions regarding the use of REOB/VTAE.

This document is careful to define REOB/VTAE as the non-distillable residuum from a vacuum tower in a used oil re-refinery. Other re-refined products derived from used oil not meeting this definition are not addressed herein, and the extent of their use in asphalt is unknown. REOB/VTAE has been used as a blending agent to soften binders. The demand for softer binder grades has grown due to higher levels of RAP and RAS being used in mixtures. The use of higher concentrations of hard oxidized binder from RAP and RAS require a softer virgin binder to meet the combined blend requirements. This factor has led to a heightened use of softening agents, which include non-asphalt blending stocks such as REOB/VTAE.

Section III of this paper is a review of 26 published papers pertaining to REOB/VTAE which can be effectively summarized as inconclusive regarding the use of REOB/VTAE with asphalt binders. Published research has provided conflicting conclusions regarding pavement performance impacts. There is a scarcity of information on comparative field performance of mixtures produced using binders with and without REOB/VTAE. Such head-to-head, real world comparative performance data is desirable when attempting to qualify the impact of REOB/VTAE on ultimate mixture performance.

Some research indicates REOB/VTAE has an adverse effect on the aging characteristics of the asphalt binder and in turn the cracking resistance of in-service pavements. Other research indicates REOB/VTAE blended asphalt has equivalent or better asphalt mixture performance relative to mixtures with neat asphalts of similar stiffness. The literature is largely inconsistent; with various authors suggesting that REOB/VTAE may be innocuous while others suggest its usage may be detrimental to performance. Further, there is data indicating that the performance of binders and mixtures containing REOB/VTAE is dependent on the REOB/VTAE dosage, the REOB/VTAE source and the binder source.

Many papers suggest improvements are needed to current binder specifications, such as extended PAV aging to better characterize all types of modified binders. X-Ray Fluorescence (XRF) technology was shown to be a qualitative tool to identify the presence of REOB/VTAE in asphalt; however XRF is not a reliable quantitative measurement of REOB/VTAE without having the individual blend components available for evaluation.

The Asphalt Institute's website has an open repository of REOB/VTAE information, which includes a listing of the published literature and recent presentations at industry meetings (http://www.asphaltinstitute.org/re-refined-engine-oil-bottom-residue/).

Section IV of this paper details the Health, Safety & Environmental aspects related to the use of REOB/VTAE in paving asphalt. Data is provided demonstrating the re-refining manufacturing process removes carcinogens present in the unrefined used oils. This was done by testing for the Polycyclic Aromatic Compounds (PACs) and mutagenicity index (MI) of the raw materials and products throughout the re-refining process. The MI is a measure of carcinogenic potential, with values less than 1 considered non-carcinogenic. Vacuum distillation concentrates the carcinogens in the distillate product, resulting in the REOB/VTAE having a MI less than 1. The carcinogenic molecules in the vacuum distillate are removed by hydrotreating before being typically used as lubricating base oils. In addition, the MI values for four different REOB/VTAE sources were shown to be less than 1. These results are supported by the PAC analysis of REOB/VTAE modified asphalts. Data shows no increase in PACs when asphalt is modified with REOB/VTAE.

Also covered in Section IV is the potential of REOB/VTAE modified asphalts to leach metals. The metal leaching concern has arisen because REOB/VTAE has a high concentration of metals, coming from lubricant additives or wear metals. Leaching studies show no differences between REOB/VTAE modified asphalts and unmodified asphalts. A final aspect of Section IV provides the chemical analysis of laboratory generated fumes of unmodified asphalt and REOB/VTAE modified asphalts. The data shows similar fume composition for each.

Section V of this paper provides a review of asphalt testing approaches beyond Superpave specifications to improve material characterization for enhanced binder performance prediction and formulation. The focus is on binder durability and ductility tests that are linked to field performance evaluations. Past studies that link pavement performance to binder cracking and fatigue properties such as ductility and brittleness are covered in detail. A general theme is the significant effect aging has on the relaxation properties of mixtures and binders. Extended PAV aging (> 20 hrs.) may be required to differentiate performance in binders that age differently. The difference between the limiting temperature for BBR m-value and limiting temperature for BBR Stiffness is known as ΔT_c, and it is an indicator of the binder's ability to relax stresses. A negative ΔT_c value means the binder is m-controlled. All binders undergo a trend towards being more m-controlled as they age, becoming more brittle and less able to relax stresses.

The addition of RAP and RAS binder also tend to make ΔT_c more negative as these materials are extensively field aged. Additives containing paraffinic base oils, along with REOB/VTAE, have been shown to increase the rate and extent to which a binder becomes m-controlled (more negative ΔT_c). The presence of REOB/VTAE in binders can negatively impact binder relaxation properties as the binder progressively ages.

Based on the information presented, it is recommended that the binder parameter ΔT_c after 40 hours of PAV be explored further as an improved method for characterizing long-term (5+ years) binder durability and performance. However, ΔT_c after 40 hours of PAV is not recommended as a purchase specification at this time due in part to the extended length of time required to age binders and the need for validating research.

The last section of this paper provides answers to 21 frequently asked questions submitted by state highway agencies and serves to summarize the highlights of this document.

TABLE of CONTENTS

I. INTRODUCTION

Purpose of Document

The use of Re-refined Engine Oil Bottoms (REOB) / Vacuum Tower Asphalt Extender (VTAE) to modify paving grade binders has become a topic of significant focus in the asphalt paving community, with many questions regarding its impact on binder and pavement performance. There is a need for an objective and thorough review of available information. This publication is intended to provide that review. While there is much on-going research on this topic at the time of writing, this document is intended to synthesize the current "state-of-the-knowledge."

This document is not intended to promote nor dissuade the use of REOB/VTAE. Rather, the goal is to clarify issues regarding REOB/VTAE modification in order to help user agencies, consultants, contractors and suppliers make informed decisions regarding its use. Observations, conclusions and recommendations provided herein are based on the review of information as of the end of 2015.

While REOB/VTAE is used in roofing asphalts, this document focuses on its use in paving asphalts. Roofing asphalts have a different set of performance conditions and critical properties compared to paving asphalts, and the performance questions raised regarding REOB/VTAE have generally come from the paving industry.

Scope and Terminology

The term REOB/VTAE as used in this document refers strictly to the residual distillation product from a vacuum tower in a re-refinery of used lubricating oil. REOB/VTAE is neither cleaned up waste engine oil, nor is it the residue from only an atmospheric tower of a more simple used oil re-refinery. It is equally important to understand that this product is compositionally different than asphalt that comes from a vacuum tower of a crude oil refinery. The production of REOB/VTAE as defined above is covered in the next section.

The terminology used over the years for additives derived from recycled engine oil has not been consistent, as many names are found in the published literature describing material that may or may not have come from a vacuum tower of a used oil re-refinery. Many of those names are listed in Table 1, and a review of that literature is provided later in this document. Fortunately, as more attention has recently been placed on this material, the asphalt industry has settled on using either the term REOB or VTAE. The two terms are currently being used interchangeably.

“Re-refined Engine Oil Bottoms” (REOB) is the prevailing name used by state highway agencies and FHWA, while “Vacuum Tower Asphalt Extender” (VTAE) is the preferred name used by the manufacturers of the product. Since each name provides an important descriptor of production elements (“re-refined” and “vacuum tower”) for the specific product this document addresses, this document uses the term “REOB/VTAE.”

TABLE 1. Various Names Associated with REOB/VTAE

Acronym	*Name*
	Asphalt Flux
	Asphalt Blowdown
EOR	Engine Oil Residue
RHVDB	Re-refined Heavy Vacuum Distillation Bottoms
RHVDO	Re-refined Heavy Vacuum Distillation Oil
REOB	Re-Refined Engine Oil Bottoms
RVTB	Re-refined Vacuum Tower Bottoms
VTB	Vacuum Tower Bottom
VTAB	Vacuum Tower Asphalt Binder
VTAE	Vacuum Tower Asphalt Extender
WEOR	Waste Engine Oil Residue
WODB	Waste Oil Distillation Bottoms

Background and Use

Asphalt binder can be produced directly from the crude oil refining process or by blending a variety of binders with different properties. Higher viscosity “hard” asphalts and lower viscosity “soft” asphalts can be used to meet specification requirements. Asphalt can also be modified with non-asphalt components such as adhesion agents, crumb rubber, emulsifiers, polymers, polyphosphoric acid (PPA), warm mix additives, vegetable or paraffinic base oils and REOB/VTAE. For an in-depth discussion of asphalt production, see IS-230 “The Bitumen Industry – A Global Perspective: Production, chemistry, use, specification and occupational exposure”.

REOB/VTAE is typically used as a blending agent to soften asphalt binders, reducing both the high and low continuous temperature performance grades. Producers of REOB/VTAE report their material has been used to modify asphalts since the 1980s (multiple papers and presentations).

There has been a heightened level of interest and use in REOB/VTAE over the past few years. Softer binder grades in the North American cold climates are in greater demand to reduce low temperature cracking. In addition, the trend to use higher levels of recycled asphalt pavement (RAP) and recycled asphalt shingles (RAS) has also driven up the demand for softer grades. The binder from RAP is hard and oxidized, and the binder from RAS is even more so. These higher concentrations of hard oxidized recycled binder require softer virgin binder to meet the combined blend requirements. Regional availability of softer grades of asphalt is generally limited. These factors have led to the heightened interest and use in softening agents which include non-asphaltic blending stocks such as REOB/VTAE.

In an FHWA study, approximately 20 percent of the 1000 samples submitted from States across the U.S. were reported to contain some level of REOB/VTAE [Arnold, TRB, 2015]. Some State DOTs have expressed concerns based on recently published research that suggests the addition of REOB/VTAE may have an adverse effect on pavement performance. Approximately 10 States have prohibited REOB/VTAE at the time of this writing, with many waiting to reassess once more research becomes available. Section III of this document provides a review of the literature and timeline of these concerns in more detail.

Manufacturers of REOB/VTAE report typical dosage rates to be 4-8%, dropping both the high and low PG grade as much as a full grade (from a 64-22 to a 58-28, or from a 58-28 to a 52-34). The rate of grade drop for both the high and low temperature grade is dependent on the REOB/VTAE material itself (available in a wide range of viscosities), as well as the interaction with the base asphalt.

There are approximately 14 REOB/VTAE manufacturing sites in the U.S. and approximately 17 in North America. The REOB/VTAE manufacturers are represented by NORA, an Association of Responsible Recyclers. This association represents the broader spectrum of approximately 400 companies in the oil recycling industry. It has been reported that approximately 160,000 tons of REOB/VTAE is produced in North America annually, which represents approximately 0.5% of all asphalt used in North America (Bouldin, Safety-Kleen, Aug 2014 presentation to AI's TAC). This amount may not represent all the products from used oil that may be used in the market place. As stated earlier, this document only addresses vacuum tower residue from re-refined engine oil, and not the full spectrum of products from used oil.

Asphalt Institute's Position

The Asphalt Institute supports the responsible modification of asphalt materials for improved performance and better life cycle costs, but does not endorse any specific or proprietary form of modification. Furthermore, the Asphalt Institute encourages the continued development of performance-related specifications to replace recipe-type binder specifications whenever feasible.

REOB/VTAE is just one of many products used to soften asphalt. Testing on the final binder should be performed after the addition of all additives to ensure that the final product specification is met.

Document Overview

The document covers key aspects of the manufacture and properties of REOB/VTAE, its current use as an additive/modifier to asphalt, its effect on binder and mix properties and ultimately field performance as reported in peer-reviewed literature and industry meetings. In addition, this document addresses HS&E aspects of REOB/VTAE and its use as a modifier in asphalt. A discussion is also provided on new binder parameters and aging protocols currently being considered and their relationship to field performance that may provide insight on effects of REOB/VTAE. The last section provides frequently asked questions from states highway agencies and the best available answers provided by the contributors of this document.

A major component of this document is the synthesis of available, relevant published literature on REOB/VTAE through the end of 2015. Paper summaries are provided on the effects of REOB/VTAE on binder and mix performance. Recognizing the dynamic nature of available science, the task force has developed an open repository of published papers and presentations given on the topic of REOB/VTAE available at: http://www.asphaltinstitute.org/re-refined-engine-oil-bottom-residue/. The presentations can be downloaded directly while the papers are linked to the published journal.

II. PRODUCTION of REOB/VTAE

REOB/VTAE is the non-distillable residuum from the recycling of used oil via atmospheric distillation followed by vacuum distillation.

Used oils are collected and screened for re-refining suitability, which includes testing for contaminants such as polychlorinated biphenyls (PCBs).

The used oil feedstock comes into the re-refinery and is temporarily stored in guard tanks where it is tested again for re-refinery suitability. In the first processing step the used oil is processed through a dehydration unit to remove water and then through an atmospheric distillation tower to recover any light volatile products. Stripped of water and fuels, the material then undergoes vacuum distillation which provides a low pressure environment to enable higher boiling point material to be recovered. In this process the distillates, called the lube cuts, are separated from the non-distillable fraction that is REOB/VTAE. The fractionated lube cuts are further processed to make different grades of lube oils, sometimes using hydrotreating. The non-distillable residue, or REOB /VTAE, is used in the asphalt industry as a modifier. Re-refining of used oil produces roughly 12-15% REOB/VTAE, with the remaining products being fuel oil and lube oil.

FIGURE 1 provides a diagram of the re-refined used oil process that includes REOB/VTAE production.

FIGURE 1. Re-refined Oil Process and REOB/VTAE Production

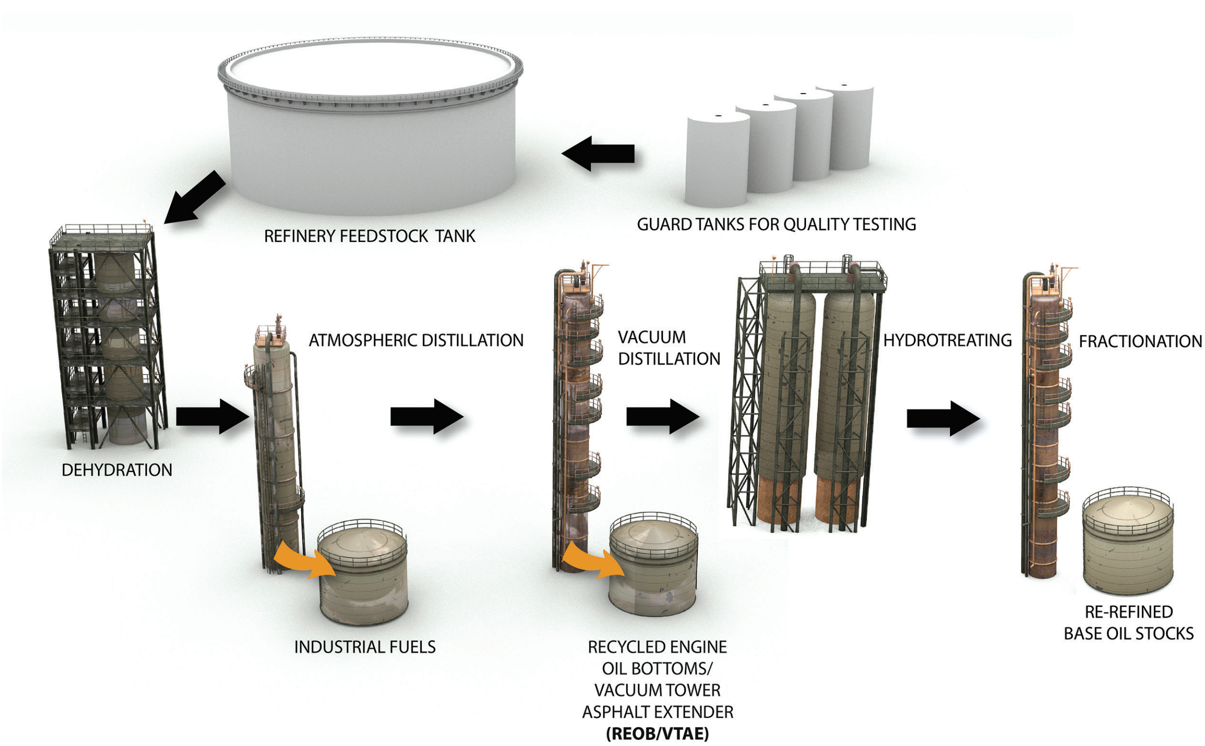


IMAGE COURTESY OF KLEEN PERFORMANCE PRODUCTS - A CLEAN HARBORS COMPANY

III. LITERATURE REVIEW PERTAINING to REOB/VTAE

Background

Re-refined engine oil bottoms (REOB/VTAE) have been blended with paving grade binders to achieve the low temperature properties in parts of the Midwestern U.S. and in Canada since the 1980s. The demand for softer binder grades required by the use of high RAP/RAS mixtures has expanded the use of REOB/VTAE throughout the country. The Federal Highway Administration (FHWA) Turner Fairbank laboratory has recently tested more than 1000 asphalt samples from various parts of the U.S. and found approximately 20 percent of the samples contained some level of REOB/VTAE [24]. The many State Departments of Transportation (DOTs) participating in that study "…were not aware that binders they were purchasing had been modified in this way."

In recent years, there has been increased interest in the properties of asphalt binders containing REOB/ VTAE and the effect on asphalt mixture performance. Recent research has been conducted related to the chemical properties and binder performance of REOB/VTAE-modified asphalt binders, and, in turn, their impact on asphalt mixture performance. Published research has provided conflicting conclusions regarding performance impacts. Some research states that REOB/VTAE has an adverse effect on the aging characteristics of the asphalt binder and in turn the cracking resistance of in-service asphalt mixture pavements. Other research states that REOB/VTAE blended asphalt has equivalent or better asphalt mixture performance than mixtures with neat asphalts of similar stiffness. These conflicting research conclusions, coupled with the lack of common knowledge regarding the use of REOB/ VTAE and anecdotal reports about premature pavement failures, have prompted concerns about the use of REOB/ VTAE-modified binders.

A number of published studies dating back to 1992 have been reviewed and are listed in TABLE 2 below. All of these studies involved the modification of asphalt binders with either REOB/VTAE or used oils (waste engine oil). This review includes research papers showing both favorable and unfavorable conclusions on the use of REOB/VTAE. The authors' findings from each paper are briefly summarized in the paragraphs that follow. Additional information and commentary on each paper may be found in the Appendix. The information provided herein should not be considered all inclusive – every attempt was made to provide the best information available through the end of 2015. Research on this topic is posted on the AI website:

http://www.asphaltinstitute.org/re-refined-engine-oil-bottom-residue/

Commentary

This commentary is based on a review of the literature that is summarized in the subsequent section. The papers that follow suggest that blending REOB/VTAE in asphalt binder at low dosages seems to be innocuous while at high dosages it may be detrimental to performance. Binders and mixtures containing moderate dosages of REOB/VTAE may perform well but are dependent on the source of REOB/VTAE and binder used. It has also been suggested that improvements may be needed to current binder specifications as standard PAV aging may not properly characterize REOB/VTAE blended binders. X-Ray Fluorescence (XRF) technology was shown to be a qualitative tool to identify the presence of REOB/VTAE in asphalt binders but in the absence of virgin materials, it does not provide a quantitative measurement.

Field evaluation should continue using XRF to determine the presence of REOB/VTAE blended binders in asphalt pavements exhibiting good and poor field performance. New field studies designed specifically around the use of REOB/VTAE with tight controls are needed as much of the available information is determined from forensic work. Development of a practice to determine the allowable dosage of REOB/VTAE when blended with an asphalt binder is also needed. The practice should provide a better understanding and insight into the interactions or consequences of blending REOB/VTAE with other binder components such WMA additives, anti-strips, PPA, emulsifiers, etc.

Summary of Papers

The studies involve the modification of asphalt with REOB/VTAE and/or used lubricating oils.

1. "Use of Re-refined Oil Distillation Bottoms As Extenders For Roading Bitumens"
   P.R. Herrington
   Journal of Materials Science – Vol. 27 No. 24 – December 1992

In 1992, Herrington investigated the potential for use of vacuum distillation bottoms produced during the re-refining of waste lubricating oils, as extenders for asphalt binders. The simple rheological properties of waste oil distillation bottoms (WODB) and both simple and air-blown blends with asphalt binders were investigated. Results were compared with unmodified binders. Additionally, the oxidative properties of the WODB blended binders were also studied. The consistency of WODB was evaluated as measurements were made on a number of batches over a 13 month period indicating considerable variation in viscosity from batch to batch. The WODB had a lower temperature susceptibility -based on the viscosity temperature susceptibility (VTS) (Puzinauskas 1979) - than the 80/100 pen base binder. WODB flash points were found to be greater than 320°C. Many different WODB's were blended with the base 80/100 pen binder at dosage rates from 2 – 20%. Observed effects on binder properties were approximately linear with increasing WODB concentration and the effect at any given concentration was greater at low temperatures than at high temperatures. Storage stability tests were run and it was determined that there was no significant separation of the two components. Testing showed that at temperatures up to around 200°C the WODB may slightly improve the oxidative stability of the base binder. At higher temperatures, the WODB blends oxidize at a faster rate.

2. "Waste Oil Distillation Bottoms As Bitumen Extenders"
   P.R. Herrington, V.K. Dravitzki, C.W.B. Wood, and J.E. Patrick
   Road & Transport Research – Vol. 2 No. 4 – December 1993

In 1993, Herrington et al. looked at two blended binders manufactured by air blowing a 180/200 penetration grade bitumen plus 9 and 20% waste oil distillation bottoms (WODB) to an 80/100 and compared to a conventional 80/100 pen bitumen. The air blown/WODB blended binders were less temperature sensitive - based on viscosity temperature susceptibility (VTS) (Puzinauskas 1979)- than the conventional 80/100 bitumen at low temperatures while exhibiting similar temperature susceptibility at temperatures of 70 -135°C. The leaching potential of the metals found in REOB was found to be negligible; in many cases this was below the detection limit. Field performance after 14 months in service was similar between the conventional bitumen sections when compared to the sections containing bitumen/WODB blends.

3. "Recycling of Waste Oil Distillation Bottoms in Asphalt"
   P.R. Herrington and P.G. Hamilton
   Transfund New Zealand Research Report No. 102, 1998

In 1998, Herrington et al. followed up on their earlier work by looking at a field trial of hot mix asphalt using waste oil distillation bottoms (WODB) produced during the re-refining of waste lubricating oils for use as an asphalt extender. Three sections of hot mix asphalt were laid in May 1991 on a roundabout in New Zealand. The trial consisted of hot mix asphalt manufactured using 80/100 grade asphalt on a control section, and using blends of 180/200 grade asphalt with 9% and 20% of WODB respectively, air-blown to a nominal 80/100 grade asphalt on the remaining two sections. The physical and age-hardening properties of these binders were examined and found to be comparable to those of the control 80/100 asphalt. Marshall stability and flow were satisfactory for the WODB asphalt mixtures and were placed without any noticeable differences between the control and WODB-blended mixes. The three sections were compared after 14 months and again after 57 months and all appeared to be performing well with no evidence of surface deterioration, cracking, or rutting. Binder was extracted from cores taken from each of the three sections. Viscosity was measured and compared to that of the initial binders. All of the binders used showed an expected hardening but the hardening appeared to be somewhat greater for the WODB-blended binders than that for the control. However, the variability introduced by the recovery procedure casts doubt on this finding. The WODB were known to contain lead and metals. After 57 months, analysis of lead, chromium, and zinc contents of the hot mix asphalt in the trial sections showed that up to 25% of the zinc had been lost while lead and chromium were similar to those initially present in the asphalt.

4. “Asphalt Modification with Used Lubricating Oil”
Aaron Villanueva, Susanna Ho, and Ludo Zanzotto
Can. J. Civ. Eng. Vol. 35, 2008

In 2008, Villanueva et al. investigated several asphalt materials modified with increasing amounts of used lubricating oil using standard Superpave tests – both MP-1 and MP-1a. Two base asphalts were prepared by air blowing 300/400 Husky asphalt at 240°C for 3 and 3.5 hours. The two binders were then blended with different percentages of used lubricating oil. All samples became softer as oil was added, as the softening point decreased and penetration increased at 25°C with increasing oil level. The high temperature grade also decreased as more oil was added. Using the AASHTO MP-1 specification, there was a decrease in the low temperature grade with an increasing level of used lubricating oil. The high-low temperature spread also decreased with increasing oil levels. Using the MP-1a test specification, it was observed that the low temperature grade ($T_{critical}$) was decidedly lower than the resulting low-temperature grade determined using the MP-1 test. This grade difference between the two tests increased with rising oil levels suggesting that the MP-1 specification underestimated the low temperature improvements with the addition of used lubricating oil to the air blown asphalts.

5. “Five Year Performance Review of a Northern Ontario Pavement Trial: Validation of Ontario’s Double-Edge-Notched Tension Test (DENT) and Extended Bending Beam Rheometer (BBR) Test Methods”
S.A.M Hesp, S.N. Genin, D. Scafe, H.F. Shurvell and S. Subramani
Proceedings of the Canadian Technical Asphalt Association, 2009

In 2009, Hesp et al. discuss a need for improvement in the Superpave (M 320) protocols to better predict cracking performance through the use of the Double Edged Notched Tension Test (DENT) and extended Bending Beam Rheometer (BBR) methods. Seven pavement sections placed in 2003 with seven different asphalt binders – all meeting the current M 320 low temperature requirement of -34°C – were investigated. The sections had varying low temperature cracking performance so chemical analysis results from recovered asphalt binders was used to confirm the validity of the proposed DENT and extended BBR test methods. X-ray fluorescence (XRF) spectrum detected the presence of zinc in three of the failed sections. Zinc may be a fingerprint element for waste engine oil residues, since it is typically present in large quantities after the refining of waste engine oil and never occurs in either straight asphalt cement or the aggregate. However zinc-carbonate or zinc-sulphate is sometimes used to scavenge hydrogen sulfide in asphalt binders.

6. “Asphalt Cement Loss Tangent as Surrogate Performance Indicator for Control of Thermal Cracking”
Abdolrasoul Soleimani, Shanan Walsh, and Simon A. M. Hesp
Transportation Research Record: Journal of the Transportation Research Board, No. 2126; 2009

In 2009, Soleimani et al. and Hesp discuss the field validation of a simple performance indicator for specification grading of asphalt binders for thermal cracking. Asphalt binders from 20 contract sites in Ontario, Canada were tested in torsion bar geometry to determine their viscoelastic properties. Tan(δ), as defined by the ratio of viscous over elastic modulus, G”/G’, was able to distinguish good from poor performers from these sites with 95% accuracy which is an improvement over current bending beam rheometer protocol. In addition to the use of tan(δ), it is suggested that a measure of critical strain tolerance in the ductile state could be included to provide a significantly improved performance grading method. Using x-ray fluorescence (XRF), most of the worst performing contracts were found to contain zinc suggesting the use of waste engine oils. The paper states that waste oils likely bring about premature hardening in the binder and this hardening will limit the strain tolerance as reflected in the double edge notched tension (DENT) data. These same asphalts would be penalized on a limit of tan(δ) since waste engine oils effectively reduce the loss tangent.

7. “Characteristics of Rejuvenated Bitumen With Used Lubricating Oil As Rejuvenating Agent”
Kemas A. Zamhari, Madi Hermadi, Choy Wai Fun
International Conference On Sustainable Infrastructure and Built Environment in Developing Countries – November 2009 – ISBN 978-979-98278-2-1

In 2009, Zamhari et al. conducted a study to assess the technical viability of using used lubricating oil as a rejuvenator for aged asphalt binders. The binder used in this study was an 80/100 Pen grade which was artificially aged using a PAV so that the binder characteristics before and after aging could be identified and compared. Five different sources of used lubricating oils were collected as well as a sample of unused motor oil and their characteristics were compared to the ASTM RA-2 specification for asphalt rejuvenating materials. The flash point was found to be too low for safe handling during the asphalt mixing operation. It was determined that by preheating the used lubricating oil for a period of three hours increased the flash point to a safe level, fulfilling the requirements of ASTM RA-2. The preheated used oil was added to the PAV aged 80/100 pen binder at rates of 5 and 10%. At the right proportion, used lubricating oil may restore the characteristics of an aged binder in terms of viscosity and penetration values as well as binder rheology. However, the addition of used oil cannot restore the chemical composition of the aged binder to pre-aged levels.

8. “X-ray Fluorescence Detection of Waste Engine Oil Residue In Asphalt and Its Effect on Cracking In Service”
Simon A. M. Hesp and Herbert F. Shurvell
International Journal of Pavement Engineering, Vol. 11, No. 6, Dec. 2010

In 2010, Hesp and Shurvell discuss the discovery of waste engine oil residues in pavements across Ontario, Canada, finding that recovered asphalts from a large majority of poorly performing contracts test positive for zinc through X-ray fluorescence (XRF) analysis. The XRF spectra were collected using a handheld Innovative X-Ray Technologies (Innov-X) model XT-440L analyzer. Since zinc dialkyldithiophosphates are universal additives in engine oils, the authors inferred that the use of waste oil residues in asphalt must be widespread with typical modification levels in the 5–20% range. However, zinc carbonate or zinc sulphate is sometimes added to scavenge hydrogen sulfide (H_2S) produced not only during the routine production of asphalt but also during the production of polymer-modified asphalt cements where a sulphur grafting reaction occurs. The authors found that 12 of the 15 poorly performing contracts tested positive for the presence of significant quantities of zinc and hence were likely contaminated with waste engine oil residues. An additional 2 of the 15 poorly performing contracts showed traces of zinc and manganese in the asphalt cement while none of the 11 superior performing contracts showed any sign of zinc. The sum of all cracking was included in the determination of performance and the type of cracking was not deciphered.

9. “Oxidative Aging Of Asphalt Cements From an Ontario Pavement Trial”
Logan Wright, Amit Kanabar, Eric Moult, Syed Rubab, and Simon Hesp
International Journal of Pavement Research and Technology 4.5 (2011)

In 2011, Wright et al. and Hesp discuss and document a rheological and spectroscopic investigation of the oxidative aging in asphalt cements from a 2003 northern Ontario pavement trial. Seven asphalt cements – two of which contained waste engine oil residue- were aged according to standard methodology (rolling thin film oven test (RTFOT) and pressure aging vessel test (PAV)), as thin films at temperatures of 45, 65, and 85 °C and standard pressure (thin film oven test (TFOT)), and under modified PAV conditions (extended time of 40 hours). Of the seven sections, only two performed as expected with two sections identified by the authors as containing waste engine oil residue performing poorly. It was found that the current RTFOT/PAV protocol provided insufficient aging for the poor performing asphalt cements. Infrared analysis of the TFOT-aged samples for the formation of carbonyl groups could largely explain the performance ranking in service.

10. “Effects of Engine Oil Residues on Asphalt Cement Quality”
    Syed Rubab, Kezia Burke, Logan Wright, Simon A. M. Hesp, Pamela Marks, and Chris Raymond
    Proceedings of the Canadian Technical Asphalt Association, 2011

In 2011, Rubab et al. and Hesp discuss and document an investigation of chemical aging in asphalt cement modified with Engine Oil Residue (REOB/VTAE). Five blends were investigated – Cold Lake 80/100 pen, Cold Lake 80/100 + 10% EOR, +20% EOR, +30% EOR, and Cold Lake 200/300 pen . The 80/100 Cold Lake modified with 20 percent EOR provides a low temperature grade that is equal to that of the straight 200/300 Cold Lake (-34°C), but with a high temperature grade that is 5°C higher (61°C versus 56°C). Changes in carbonyl formation rates were investigated with the introduction of EOR in straight Cold Lake base asphalt. Carbonyl formation is closely linked to increases in viscosity and hence it is generally accepted as a convenient indicator to monitor oxidative hardening. It was found that engine oil residues significantly increase both the spurt and steady rate oxidation which could cause premature and excessive fatigue and thermal cracking.

11. “Penetration Testing of Waste Engine Oil Residue Modified Asphalt Cements”
    Burke, K., and S. A. Hesp
    Proc., 1st Conference of Transportation Research Group of India (CTRG), Bangalore, India, Dec. 2011

In 2011, Burke and Hesp believe that the current AASHTO M-320 protocols for chemical aging in the rolling thin film oven (RTFO) and the pressure aging vessel (PAV) and physical hardening in the bending beam rheometer (BBR) are inadequate and useful insights can be obtained from empirical tests methods and acceptance criteria such as penetration and penetration index (PI). Two binders were investigated in this study – a Cold Lake 200/300 pen binder and a Cold Lake 80/100 pen binder blended with 20% waste engine oil residue (WEOR). The true grade of the 200/300 pen material was a PG 56-34 while the WEOR blended binder was a PG 61-34. Different conditioning and aging protocols were considered for each of the binders. The WEOR-blended material was consistently less temperature susceptible as indicated by higher PI values. As binders age in the field, PI typically increases making them more prone to cracking. Based on some field studies done in the 1960’s the authors suggest that higher PI values lead to premature cracking and that the WEOR-blended binders are already prematurely aged.

12. “Waste Engine Oil Residue in Asphalt Cement”
    S.A.M. Hesp and H.F. Shurvell
    MAIREPAV Paper – 2012

In 2012, Hesp and Shurvell discuss and document a case study of two paving projects in Ontario Canada. The first experienced widespread cracking in just 5-7 years of service. The second project, paved around the same time, had experienced no cracking at the time of their report. Recovered asphalt binders from each of the projects revealed the presence of zinc and molybdenum in the failed pavement but were not present in the good performing trial sections. The low temperature M320 grades of the extracted binders for the good and the poor performing sections were -38°C and -27.4°C respectively. The extended BBR low grades were -38.6°C and -21.4°C with a three day grade loss of 1.8°C and 6.5°C respectively – which the authors attributed to the differences in cracking performance. The presence of waste engine oil residue in the poor performing pavement has made the binder more susceptible to chemical and physical hardening. It is possible that other factors have contributed to the excessive and premature cracking.

13. “Asphalt Binder Modification with Re-Refined Heavy Vacuum Distillation Oil (RHVDO)”
John D’Angelo, Ken Grzybowski, and Steve Lewis
Proceedings of the Canadian Technical Asphalt Association, 2012

In 2012, D’Angelo et al. discuss and document an evaluation performed to determine the performance characteristics of asphalt binders modified with Re-refined Heavy Vacuum Distillation Oil (RHVDO) at several different levels. Chemical analysis of the RHVDO and the asphalt binders was performed to determine what changes are taking place in the modified binder. Rheological testing was also conducted on the binders to evaluate the physical properties of the binders under several different aging conditions. This study looked at two mid-continent asphalts - Marathon-Detroit, MI and BP-Calumet, IL [sic. BP-Whiting, IN]- blended with RHVDO at different percentages (0, 2, 4, 6, 8, and 20%) using two different sources (Safety-Kleen-Chicago, IL and Safety-Kleen-Breslau, Ontario) – RHVDO is typically used at less than 10%. Extensive testing was carried out on the individual RHVDO, the asphalt binders, and the various blends. This included full AASHTO M320 Table 1 and 2 testing, AASHTO MP-19 MSCR testing, modified PAV (extended time of 35 hours) aging, separation tests on the blends, aggregate adhesion screening ASTM 3625, and chemical analysis ASTM 4124. The authors suggest that the addition of RHVDO does not increase the aging rates – aging rates are controlled by the base asphalt and not the RHVDO.

14. “Evaluation of Rejuvenator’s Effectiveness With Conventional Mix Testing For 100% RAP Mixtures”
Martins Zaumanis, Rajib B. Mallick, and Robert Frank
Transportation Research Board Compendium of Papers 2013 – Paper 13-1447

15. “Evaluation of Rejuvenator’s Effectiveness With Conventional Mix Testing For 100% RAP Mixtures”
Martins Zaumanis, Rajib B. Mallick, and Robert Frank
Transportation Research Record: Journal of the Transportation Research Board, No. 2370 – 2013, pp. 17-25

In 2013, Zaumanis et al. evaluated the effectiveness of rejuvenators for production of 100% RAP mixtures. Nine different softening agents were tested which included waste engine oil (WEO) and waste engine oil bottoms (WEOB). Based on the previous work by Burke and Hesp (10), penetration index (PI) and penetration-viscosity number (PVN) was considered along with their assumption that while high PI or high PVN values indicate low thermal susceptibility, these same high values increase the likelihood for premature cracking. WEO and WEOB were each blended with extracted RAP binder at a dosage rate of 18.26% - addition of WEO lowered both the PI and PVN when compared to the RAP binder. Addition of WEOB increased the PI but had no effect on PVN. It was later decided to eliminate the PVN approach as all of the data was non-linear and outside the typical PI range for linearity. Low temperature mixture testing was also completed including IDT creep compliance and indirect tensile strength testing. Mixtures using both WEO and WEOB showed increases in both creep compliance and fracture energy when compared to the reference RAP mixture – however both WEO and WEOB lowered the tensile strength of the mixtures.

16. “Evaluation of the Performance Properties of Asphalt Mixes Produced with Re-refined Heavy Vacuum Distillate Bottoms”
John A. D’Angelo, Ken Grzybowski, Steve Lewis, and Rodney Walker
Proceedings of the Canadian Technical Asphalt Association, 2013

In 2013, D’Angelo et al. discuss and document an evaluation to determine the performance characteristics of asphalt mixtures produced with binders modified with Re-refined Heavy Vacuum Distillation Bottoms (RHVDB) at several different levels. The rutting, moisture damage, fatigue and low temperature properties of the mixtures were evaluated against control mixes produced with unmodified binders. Multiple tests were run to compare the mixtures with RHVDB against control mixes. In this study, two coarse-graded Superpave mix designs were produced with asphalt binders blended with RHVDB at dosage rates of 2, 4, 6, and 10%. The Illinois mixes and aggregates were selected because they were reported to have a higher potential for moisture damage. Rutting was evaluated using both the HWT test and Flow Number test. Moisture damage potential was evaluated using AASHTO T 283 and the HWT test. Four-point bending beam fatigue testing was used to evaluate the fatigue properties of mixes. Overall in rut resistance, moisture damage potential, fatigue response and cracking potential the RHVDB modified binders provided equal or better performance than the control neat binders. The authors suggest that binders modified with RHVDB may provide good performance in the field.

17. “Pushing the Asphalt Recycling Technology to the Limit”
Joel R. M. Oliveira, Hugo M.R.D. Silva, Carlos M.G. Jesus, Liliana P.F. Abreu, and Sara R.M. Fernandes
International Journal of Pavement Research and Technology - 6.2 (2013)

In 2013, Oliveira et al. looked at the use of non-rerefined used motor oil as a rejuvenator in 100% RAP mixtures and found that the incorporation of 100% RAP and used motor oil (as a binder rejuvenator) in the production of asphalt mixtures may be a paving solution with performance as good as conventional asphalt mixtures.

18. “Investigation of the Effect of Oil Modification on Critical Characteristics of Asphalt Binders”
Amir Golalipour
A dissertation at the University Of Wisconsin – Madison 2013

In 2013, Golalipour investigated the effect of oil modification, including REOB/VTAE on the rheological properties of asphalt binders. To examine the effect of oil modification on binder characteristics, low temperature properties as well as high temperature performance of oil-modified binders were evaluated. It is found that oils vary in their effects on asphalt binder performance. 10 different oils were used – 1 petroleum aromatic oil, 3 petroleum paraffinic oils, 1 petroleum naphthenic oil, 3 bio-oils, and 2 REOBs at dosage rates of 4-5%. Both REOB blends tested provided the least aging susceptibility (lowest aging indices) of all of the oil blends used. The REOBs also reduced the intermediate temperature stiffness of the binder which may be beneficial to fatigue life. One REOB did well and the other not so well using the AASHTO TP 101 Linear Amplitude Sweep (LAS) test while both REOB blends provided the greatest decrease in low temperature stiffness when compared to the neat binder – they also provided the highest increase in m-value at the lowest temperature. Based on the single edge notched beam (SENB) test, REOB improved the low temperature performance of the binder and was the most effective oil in improving low temperature properties as determined by glass transition temperature (Tg) testing.

19. “Effect of Waste Engine Oil Residue on Quality and Durability of SHRP Materials Reference Library Binders”
Kelli-Anne N. Johnson and Simon A. M. Hesp
Transportation Research Record: Journal of the Transportation Research Board. 2014

In 2014, Johnson and Hesp discuss the effects of widely used waste engine oil (WEO) residue on asphalt binder properties. Five SHRP Materials Reference Library binders and one commercial material from Ontario, Canada, were modified with 15% WEO. Adding WEO to binders with moderate to high asphaltene contents result in effect analogous to regular aging – loss in phase angle and increase in stiffness. Binders that retain a high phase angle at both high and low temperatures are known to do well in resisting both thermal and fatigue cracking. The authors suggest WEO should only be used with great caution for modification of low asphaltene binders.

20. “The Performance of Aged Asphalt Materials Rejuvenated with Waste Engine Oil”
Christopher D. DeDene and Zhanping You
International Journal of Pavement Research and Technology – March 2014

In 2014, DeDene and You blended a PG 58-28 neat, virgin binder with reclaimed asphalt binder (RAB) and waste engine oil. The blends were then tested to study the interactions between RAB and waste engine oil using Fourier-Transform Infrared Spectroscopy (FTIR). Asphalt mixture testing was then performed with mixtures of virgin asphalt, virgin binder, RAP and waste engine oil, in quantities similar to the binder testing. FTIR testing showed reductions in carbonyl and sulfoxide structural indices as more waste engine oil is added to the blend. That reduction translates to an increase in the relative amount of maltenes. The mixture results indicated increased rutting with the addition of WEO. The addition of WEO did not adversely affect TSR values but did reduce the indirect tensile strengths of the mixture.

21. “Evaluation of Oil Modification Effect on Asphalt Binder Thermal Cracking and Aging Properties”
Amir Golalipour, Ph.D. and Hussain Bahia, Ph.D.
Proceedings of the Canadian Technical Asphalt Association, 2014

In 2014, Golalipour and Bahia investigated the effect of oil modification on a binder’s low temperature cracking resistance and in terms of its effect on stiffness, stress relaxation rate, and fracture properties. The study compared a series of asphalt blends modified with different oils, including a petroleum-based aromatic oil, a petroleum-based paraffinic oil, a bio-based oil, and a re-refined used oil (REOB). The oils were blended at 5% by weight with a PG 64-22 and then tested. The study found that the REOB blend had the lowest Tg and the highest coefficient of thermal contraction which suggests REOB is the most effective oil regarding low temperature properties. The REOB blend had the highest m-value and had the same increase in low temperature stiffness (physical hardening) as the virgin binder. The REOB blend exhibited the best properties of all of the oil-modified binders.

22. “The Impact of Asphalt Blended with Re-refined Vacuum Tower Bottoms (RTVB) and Its Effect on HMA Mixture Performance”
Jason C. Wielinski, Anthony J. Kriech, Gerald A. Huber, Andreas Horton, Linda V. Osborn, Heritage Research Group
Proceedings of the Canadian Technical Asphalt Association, 2014

23. “Analysis of Vacuum Tower Asphalt Extender and Effect on Bitumen and Asphalt Properties”
Jason C. Wielinski, Anthony J. Kriech, Gerald A. Huber, Andreas Horton, Linda V. Osborn, Heritage Research Group
Taylor and Francis – Road Materials and Pavement Design, 2015

In 2014, Wielinski et al. provides information regarding the viability, compatibility and integrity of re-refined vacuum tower bottoms (RVTB) in asphalt binders and asphalt pavement mixtures. One source of RVTB was blended with PG 64-22 to achieve binder grade of PG 58-28. A second PG 58- 28 was formulated without RVTB using a soft asphalt blended with PG 64-22. The asphalt binders were analyzed for Superpave PG Binder Testing, X-Ray Fluorescence, Polycyclic Aromatic Compound (PAC) content, Gel Permeation Chromatography (GPC) and ThermoGravimetric Analysis (TGA). The study also compares the performance of asphalt mixtures prepared with neat PG 58-28 and PG 58-28 containing RVTBs. Analysis includes volumetric analysis, resistance to moisture damage, resistance to rutting, mixture stiffness, fatigue resistance and environmental assessment using leachate testing. This study concluded that introducing RVTBs into an asphalt binder at a rate of 9%, sufficient to convert a PG 64-22 to a PG 58-28, does not compromise mixture stiffness or aging. In this case it enhanced resistance to moisture damage and resistance to fatigue damage. The RVTB additive did not significantly affect PAC levels and were similar to other asphalt reported in the literature.

24. “The Analysis of Asphalt Binders for Recycled Engine Oil Bottoms by X-Ray Fluorescence Spectroscopy”
Terence S. Arnold CChem. and Anant Shastry Ph.D.
Transportation Research Board Compendium of Papers 2015

In 2015, Arnold and Shastry presents a methodology to detect and measure the presence of REOB in asphalt binders using X-ray Fluorescence Spectroscopy. Engine oils contain additives that enhance their performance. The additives are typically zinc dithiodialkyl phosphates, calcium phenate and molybdenum disulfide. These metals survive the re-refining process and persist in the REOB. The additives and engine wear metals, mainly iron and copper, can readily be detected and measured using X-ray fluorescence spectroscopy (XRF). The analysis is not straightforward as some of these metals also occur in asphalt binders, several occur in ground tire rubber (GTR), and some occur in scavengers added to control plant emissions of hydrogen sulfide. The composition of REOB varies, not only between different producers but also within samples taken from the same producer at different times. Unless the analysis of the actual REOB used in the blend being tested is available, the XRF test method can only provide an approximate analysis. X-Ray Fluorescence Spectroscopy can readily determine the presence of these residues in asphalt binders. The authors suggest the presence of calcium, zinc, copper and molybdenum can be used as a fingerprint to show that a binder contains REOB.

25. “Cold Winter and Early Asphalt Pavement Cracking Observed in Ontario”
Ludomir Uzarowski, Ph.D., P. Eng., Gary MacDonald, P. Eng., John Rizzo, CET, Gary Moore, P.Eng., and Vimy Henderson, Ph.D., P. Eng.
Proceedings of the Canadian Technical Asphalt Association, 2015

In 2015, Uzarowski et al. discuss and document a number of recent premature pavement failures in Ontario due to cracking after 2 to 3 years and occasionally in as little as 6 months. The standard for this area is a PG 58-28 with a PG 64-28 being used in areas of heavier or slower moving traffic. This premature cracking has mainly occurred in pavements constructed with PG 64-28 causing the agencies to focus their attention on the quality of the asphalt binders being used and the method of modification used to bump the asphalt grade from PG 58-28 to PG 64-28. After completing a pavement failure investigation, it was concluded that the distressed pavements were constructed of adequate thickness and quality to provide sufficient structural support and that the materials used were in specification. During the investigation, it was determined that cracking was only found in the asphalt wearing course. Binder was extracted from the mixtures and tested using standard PG testing as well as the Multiple Stress Creep Recovery Test (MSCR), the ash test, Double Edge Notched Tension (DENT) test, and the Extended Bending Beam Rheometer (ex BBR) test. The recovered binders were also tested for the presence of zinc, molybdenum, and carbonyls to check for the potential that the PG 64-28 was modified by blowing and the addition of REOB. Only two of the binder sample results were reported and both showed a presence of zinc and molybdenum. Other sample results were not reported.

26. “Laboratory Crack Testing of Hot Mix Asphalts Containing Vacuum Tower Asphalt Extender”
Jason C. Wielinski, Andreas Horton, Anthony J. Kriech and Gerald A. Huber
Proceedings of the Canadian Technical Asphalt Association, 2015

In 2015, Wielinski et al. discuss and document the aging of binders formulated with different percentages of REOB/VTAE to generate a final PG 58-28 grade. One source of REOB/VTAE was mixed with neat binder at 0, 3, 6, 9, and 27.5 percent by weight. The binders were subjected to the standard long-term aging protocol of 20 hours in the pressure aging vessel (PAV) as well as extended aging of 40 and 60 hours. Results indicate that increasing the amount of REOB/VTAE in the asphalt binder decreased low temperature stiffness of the asphalt binder. The stiffness decreased in direct proportion to the amount of REOB/VTAE added. Critical temperature difference (ΔT_c) is defined as the limiting temperature for BBR stiffness minus limiting temperature for BBR m-value. It was determined that as the percentage of REOB/VTAE increased, the ΔT_c increased. It was also determined that stiffness of all of the binders increased with increased aging but were not considered highly sensitive to the PAV aging time. Changes in BBR stiffness with increased PAV aging did not depend strongly on the percentage of REOB/VTAE present in the binder. However it was determined that BBR m-value was sensitive to the amount of PAV aging as well as ΔT_c, which was significantly affected by the amount of PAV aging. This study also evaluated the crack resistance of an asphalt mixture containing three of the resulting PG 58-28 binders. The mixtures also contained 25 percent asphalt binder replacement from reclaimed asphalt pavement (RAP) and were subjected to both short term loose mix aging as well as an extended long-term aging procedure of 480 hours at 85°C. Mix specimens were evaluated for cracking potential using the Semi-Circular Bend Test as used by the University of Illinois (SCB-IL). Values of Flexibility Index of the short-term aged specimens ranged from 7.9 to 13.5, however no trend with REOB/VTAE content was observed. Results showed that the work of fracture decreases with increased percentages of REOB/VTAE. The work of fracture and Flexibility Index were found to be lower for all mixes after extended long term aging.

Presentations

In addition to the papers cited above, many of the authors and other industry experts have made presentations at conferences, user producer group meetings and symposiums. A partial listing of some of these presentations on the modification of asphalt with REOB/VTAE follows.

1. Simon Hesp, Detection and analysis of waste engine oil (WEO) residues in asphalt cements using X-Ray Fluorescence (XRF) spectroscopy, 2012

2. John D'Angelo, Asphalt Binder Modification With Re-refined Heavy Vacuum Distillate Bottoms, 2013

3. Mark Buncher, Ph.D., P.E., Asphalt Institute's Re-refined Engine Oil Bottoms (REOB) Residue Task Force, FHWA Mix ETG Meeting, Sept. 2014

4. Gerald Reinke, 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 Mix ETG Meeting, Sept. 2014

5. Ken Grzybowski, VTAE Oils In Asphalt, Rocky Mountain Asphalt User Producer Group, Oct. 2014

6. Jack Youtcheff, Session 831 – Use of Re-refined Engine Oil Bottoms As Asphalt Binder Modifier, 94th Annual TRB Meeting, Jan. 2015

7. Tony Kriech, REOB – History and Use, 94th Annual TRB Meeting, Jan. 2015

8. Terry Arnold/Nelson Gibson, Laboratory Evaluation of REOB Modified Binders & Mixtures, 94th Annual TRB Meeting, Jan. 2015

9. Jean-Pascale Planche, Lab and Field Performance of Mixtures Containing REOB Modified Asphalt, 94th Annual TRB Meeting, Jan. 2015

10. Louay N. Mohammad, Intermediate Temperature Fracture Properties of Asphalt Mixtures Containing RAS: Impact of Recycling Agents, FHWA Binder ETG Meeting, April 2015

11. William Ahearn, P.E, Why Is There An AASHTO REOB Task Force?, FHWA Binder ETG Meeting, April 2015

12. William Ahearn, P.E., Where's My Pavement Today?, FHWA Binder ETG Meeting, April 2015

13. Mark Buncher, Ph.D., P.E., AI's REOB Task Force, FHWA Binder ETG Meeting, April 2015

14. Gerald Reinke, Further Investigations Into The Impact of REOB & Paraffinic Oils On The Performance of Bituminous Mixtures, FHWA Binder ETG Meeting, April 2015

15. Walaa S. Mogawer, P.E., F.ASCE, Evaluating The Influence of Aging on The Chemical and Performance Characteristics of REOB Modified Asphalt Binders and Mixtures, FHWA Binder ETG Meeting, April 2015

16. Nelson Gibson, Recycled Engine Oil Bottoms as Asphalt Binder Additive, FHWA Binder ETG Meeting, April 2015

17. Thomas Bennert, Ph.D., Asphalt Binder and Mixture Properties Produced With REOB Modified Asphalt Binders, FHWA Binder ETG Meeting, April 2015

18. Jean-Paul Planche, Blended REOB Binder Advanced Chemical & Physical Characterization, FHWA Binder ETG Meeting, April 2015

19. Al Palmer, Introduction To Re-refined Vacuum Tower Asphalt Extenders, Ontario Hot Mix Producers Association Road Show, April 2015

20. William Ahearn, P.E., AASHTO Subcommittee on Materials Recycled Engine Oil Task Force Report To SOM, AASHTO Subcommittee on Materials Meeting, August 2015

21. Mark Buncher, Ph.D., P.E., AI's REOB Task Force, FHWA Binder ETG Meeting, Sept. 2015

22. Gerald Reinke, Some Properties of Binders With and Without REOB With RAP & RAP+RAS NHDOT & Data From Vermont Cores, FHWA Binder ETG Meeting, Sept. 2015

23. Nelson Gibson, REOB Update From FHWA TFHRC, FHWA Binder ETG Meeting, Sept. 2015

24. Geoff Rowe, ETG REOB Task Force Update, FHWA Binder ETG Meeting, Sept. 2015

25. John D'Angelo, Review of Several Binder and VTAE Blends, FHWA Binder ETG Meeting, Sept. 2015

26. Nelson Gibson, Examining The Use of ΔT_c To Screen Presence of High REOB, FHWA Binder ETG Meeting, Sept. 2015

27. Louay Mohammad, Asphalt Mixtures Containing RAS: Effect of REOB on Laboratory Performance, FHWA Binder ETG Meeting, Sept. 2015

28. Thomas Bennert, Ph.D., Asphalt Binder and Mixture Properties Produced With REOB Modified Asphalt Binders, Northeast Asphalt User Producer Group Meeting, Oct. 2015

29. Ryan Barborak, P.E., Quantifying Re-Refined Engine Oil Bottoms (REOB) In Asphalt – TxDOT's Approach, Southeast Asphalt User Producer Group Meeting, Nov. 2015

The presentations above were made through the end of 2015 and should not be considered all-inclusive. Presentations are available on the Asphalt Institute's webpage: http://www.asphaltinstitute.org/re-refined-engine-oil-bottom-residue/ or on the FHWA Binder ETG website: http://www.asphaltetgs.org/.

IV. HEALTH, SAFETY and ENVIRONMENTAL CONSIDERATIONS

There is little published HSE data on asphalt binders modified with REOB/VTAE. Much of the knowledge is derived from studies on asphalt/bitumen. Information in this section is limited to studies conducted to aid in the understanding of the health and environmental effects of using REOB/VTAE in asphalt binders. Unless stated otherwise, testing was conducted by Heritage Research Group (HRG) (Indianapolis, IN) in response to questions surrounding the use of REOB/VTAE in asphalt. This information is relevant to worker exposure concerns while using REOB/VTAE asphalt binders, although laboratory-generated emissions provide a worst case scenario. Data gaps include a need for additional testing on a greater number of samples including roofing materials; and field occupational hygiene assessments of actual worker exposure during use.

CAS Number

CAS number 129893-17-0 has been typically used by manufacturers for REOB/VTAE. REOB/VTAE is only a subset since other materials not from a vacuum tower also fall under this category. The National Institute of Health (NIH) ToxNet, ChemID description is: "Lubricating oils, used, residues".

Toxic Substances Control Act (TSCA) Definition 2008: A very complex combination of high molecular weight hydrocarbons consisting mostly of spent polymers and organometallic based additives which have been removed as a non-volatile residue from waste lubricating oils. This material consists primarily of hydrocarbons with a carbon number greater than 25, and with high carbon to hydrogen ratios. This material will contain metals such as zinc, calcium, sodium and magnesium [1].

Carcinogenicity

Used oils can contain Polycyclic Aromatic Compounds (PACs) such as benzo[a]pyrene. A subset of this group of chemicals is considered carcinogenic. In processing used oil in a vacuum tower refining the PACs are generally removed out of the residuum (bottoms) and end up in the heavy fuels and lubes, not the REOB/VTAE. REOB/VTAE materials show only trace levels of PACs.

It is unlikely that processes that do not include vacuum distillation, such as filtration, centrifugation, and atmospheric distillation, will remove PACs from the engine oil bottoms. At present, REOB/VTAE is not known to be listed as a carcinogen.

There have been concerns about PAC content of REOB/VTAE due to potentially carcinogenic levels in used oil. To address this concern, seven samples that follow the re-refining process were taken at different stages of the re-refining process and tested for mutagenicity (MI) and PACs. The point at which these were taken is shown in FIGURE 2. Points 1, 2, and 3 reflect the unprocessed used oil, the dehydrated used oil, and the REOB/VTAE respectively. Points 4-A, 4-B, 4-C, and 4-D are the lube cuts: 4-A = vacuum oil, 4-B = hydrotreated 80 Base Oil, 4-C = hydrotreated 150 base oil and 4-D = hydrotreated Low Sulfur Fuel.

FIGURE 2. Source Location of Seven Samples Taken from a Re-refinery. The numbers in the red boxes refer to REOB/VTAE processed samples. Identifiers in the blue boxes refer to the lube oil processes, two of which involve hydrotreating to convert the PACs to naphthenic compounds

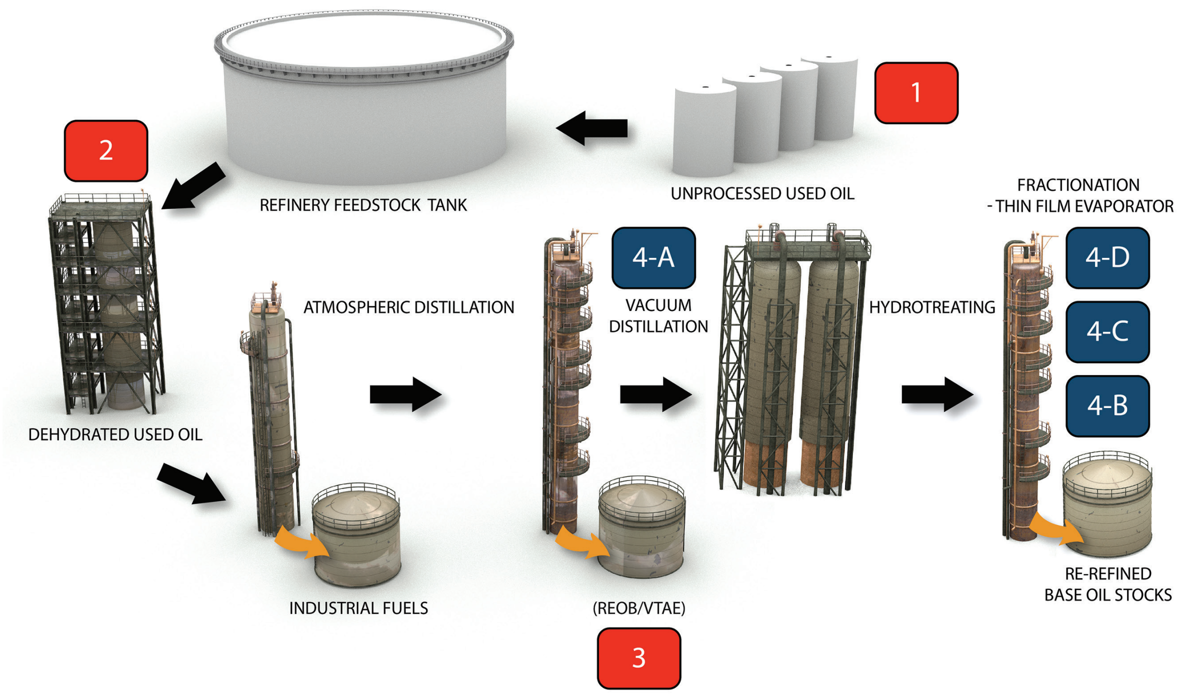


IMAGE COURTESY OF KLEEN PERFORMANCE PRODUCTS - A CLEAN HARBORS COMPANY

Modified Ames testing was performed by PetroLabs (Ivyland, PA) in accordance with the procedures described in ASTM Standard Method E 1687-95 [2]. This test has been shown to have a high correlation with carcinogenic potential of petroleum-derived materials in animal skin painting studies. Skin painting bioassays have shown that most oils with Mutagenicity Indices (MI) greater than 1.0 are carcinogenic, while most with MIs less than 1.0 are not. MI results for these seven samples are shown in TABLE 3.

TABLE 3. Mutagenicity Indices for Seven Samples at Various Stages in a Re Refinery

Figure Label	Description	MI
1	Unprocessed Used Oil	2.5
2	Dehydrated Used Oil	5.2
3	REOB/ VTAE	0.57
4-A	Vacuum Oil	1.2
4-B	HT 80 Base Oil	0.30
4-C	HT 150 Base Oil	0.21
4-D	HT Low Sulfur Fuel	0.18

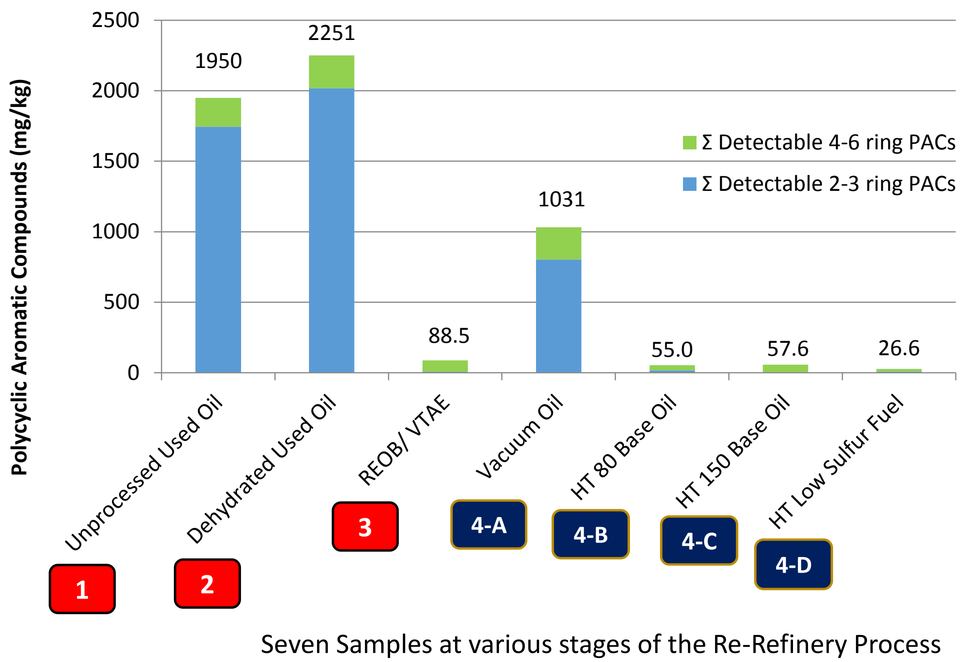
Analysis of PACs using Gas Chromatography/Mass Spectrometry (GC/MS) was also performed on these seven samples. Forty PACs were investigated using GC/MS (Agilent Q Time of Flight) following a modified SW-846 8270 method [3]. With an Agilent Select PAH 30m x 0.25mm column, 0.15µm film (Agilent p/n CP 7462); the transfer line temperature is 300°C and source temperature is 275°C. Acquisition includes 35-500 masses at 5 spectra/second and 200 ms/spectra. These PAC results are shown in TABLE 4 and are graphically displayed in FIGURE 3.

Although the dehydrated used oil (Figure 2, Label 2) contains significant PACs with an MI >1.0, after atmospheric distillation PACs are substantially concentrated in the lube cut (Figure 2, Label 4-A) by the vacuum tower distillation. Hydrotreating of the lube cut converts PACs to naphthenic oils in the lube cut (Figure 2, Labels 4-B through 4-D) reducing the MI to below 1.0.

TABLE 4. Detectable Concentrations of PACs of Seven Samples at Various Stages in the Re-Refinery Process

	No. of Benzene Rings	Reference No.	1	2	3	4-A	4-B	4-C	4-D
		Sample Concentration (mg/kg)	Unprocessed Used Oil	Dehydrated Used Oil	REOB/ VTAE	Vacuum Oil	HT 80 Base Oil	HT 150 Base Oil	HT Low Sulfur Fuel
1	2	Naphthalene	1542	1770	2.41	600	7.65	nd	nd
2	2+	Fluorene	36.6	47.2	nd	33.3	nd	nd	nd
3	3	Anthracene	25.4	27.8	0.46	26.7	nd	nd	nd
4	3	Phenanthrene	121	151	2.34	120	nd	nd	nd
5	3+	Fluoranthene	20.2	22.1	1.40	22.9	8.85	1.92	8.70
6	4	Benz[a]anthracene	39.4	42.5	7.46	44.5	nd	nd	nd
7	4	Chrysene	16.0	18.3	2.34	19.3	nd	nd	nd
8	4	Pyrene	40.4	46.1	3.86	50.8	23.0	6.10	17.3
9	4	Triphenylene	2.14	13.0	nd	2.46	nd	nd	nd
10	4+	Benzo[b]fluoranthene	10.3	10.7	3.69	11.2	nd	nd	nd
11	4+	Benzo[j]fluoranthene	4.64	5.19	1.69	4.82	nd	nd	nd
12	4+	Benzo[k]fluoranthene	4.40	4.59	1.62	4.63	nd	nd	nd
13	5	Benzo[a]pyrene	27.5	29.8	11.5	30.1	nd	nd	nd
14	5	Benzo[e]pyrene	21.1	21.9	8.76	22.9	nd	3.06	nd
15	6	Benzo[ghi]perylene	23.5	23.8	22.6	24.2	10.1	35.7	0.59
16	6	Benzo[rst]pentaphene	5.44	6.11	10.4	4.51	nd	nd	nd
17	5	Dibenz[a,h]anthracene	nd	nd	nd	nd	1.88	6.27	nd
18	5+	Indeno[1,2,3-cd]pyrene	9.82	10.39	7.96	9.21	1.44	4.50	nd
nd = non-detected. Detection Limit = 0.5 mg/kg. HT = Hydrotreated									
Summary Data		∑ 40 PACs (mg/kg)	1950	2251	88.5	1031	55.0	57.6	26.6
		∑ 4-6 ring PACs (mg/kg)	205	233	81.9	229	38.5	55.7	17.9

FIGURE 3. PACs Detected in Seven Samples Taken at Various Stages of the Re-refinery Process as Shown in FIGURE 2.



REOB/VTAE Sources

REOB/VTAE samples from the four largest global REOB/VTAE producers (labeled Source A, Source B, Source C, and Source D) were tested for MI, PACs and elemental composition using XRF. XRF testing provides a wide scope of different elements (up to 70) ranging from fluorine to uranium in one single measurement run.

MI results were all below 1.0 (see TABLE 5). Source C is from a European producer whose feedstock consists primarily of diesel engine oil.

TABLE 5. Mutagenicity Index for Four 100% REOB/VTAE Samples Representing Largest Suppliers

REOB/VTAE	Mutagenicity Index (MI)
Source A	0.27
Source B	0.57
Source C*	0.88
Source D	0.25

* European source containing primarily diesel engine oil feedstock

From these same four sources, PAC results were all below 20 mg/kg for the sum of the 40 detectable compounds (see TABLE 6).

TABLE 6. PAC Content of Four 100% REOB/VTAE Samples

	No. of Benzene Rings	REOB/VTAE Neat Material Units = mg/kg	Source A	Source B	Source C	Source D
1	1+	Benzothiophene	<0.04	<0.04	<0.04	<0.04
2	2	Naphthalene	<0.04	<0.04	0.09	<0.04
3	2+	Acenaphthene	<0.04	<0.04	<0.04	<0.04
4	2+	Acenaphthylene	<0.04	<0.04	<0.04	<0.04
5	2+	Benz[a]acridine	<0.04	<0.04	<0.04	<0.04
6	2+	Benz[c]acridine	<0.04	<0.04	<0.04	1.46
7	2+	Carbazole	<0.04	<0.04	<0.04	0.56
8	2+	Dibenzothiophene	<0.04	<0.04	<0.04	0.58
9	2+	Fluorene	<0.04	<0.04	0.10	<0.04
10	3	Anthracene	<0.04	<0.04	<0.04	<0.04
11	3	Phenanthrene	0.65	<0.04	<0.04	0.36
12	3+	Benzo[b]naphtho[2,1-d]thiophene	<0.04	<0.04	<0.04	6.81
13	3+	Benzo[b]naphtho[2,3-d]thiophene	<0.04	<0.04	<0.04	<0.04
14	3+	Fluoranthene	0.60	<0.04	0.04	0.17
15	4	Benz[a]anthracene	0.90	<0.04	<0.04	0.32
16	4	Chrysene	0.30	<0.04	<0.04	<0.04
17	4	7,12-Dimethylbenz[a]anthracene	<0.04	<0.04	<0.04	<0.04
18	4	5-Methylchrysene	<0.04	<0.04	<0.04	<0.04
19	4	1-Nitropyrene	<0.04	<0.04	<0.04	<0.04
20	4	Pyrene	1.60	<0.04	0.05	0.44
21	4	Triphenylene	<0.04	<0.04	<0.04	<0.04
22	4+	Benzo[b]fluoranthene	0.65	<0.04	0.09	0.29
23	4+	Benzo[j]fluoranthene	0.25	<0.04	<0.04	<0.04
24	4+	Benzo[k]fluoranthene	0.25	<0.04	<0.04	<0.04
25	4+	Cyclopenta[cd]pyrene	<0.04	<0.04	<0.04	<0.04
26	4+	7H-Dibenzo[c,g]carbazole	<0.04	<0.04	<0.04	<0.04
27	4+	3-Methylcholanthrene	<0.04	<0.04	<0.04	<0.04
28	5	Benzo[a]pyrene	1.65	0.29	0.32	0.46
29	5	Benzo[e]pyrene	1.40	0.24	0.21	0.34
30	6	Benzo[ghi]perylene	4.19	2.43	1.81	1.19
31	6	Benzo[rst]pentaphene	1.20	1.80	0.84	0.63
32	5	Dibenz[a,h]acridine	<0.04	<0.04	<0.04	1.19
33	5	Dibenz[a,j]acridine	<0.04	<0.04	<0.04	1.75
34	5	Dibenz[c,h]acridine	<0.04	<0.04	<0.04	<0.04
35	5	Dibenz[a,h]anthracene	<0.04	<0.04	<0.04	<0.04
36	5+	Dibenzo[a,e]fluoranthene	<0.04	<0.04	<0.04	<0.04
37	5+	Indeno[1,2,3-cd]pyrene	1.05	0.58	0.44	0.29
38	6	Dibenzo[a,e]pyrene	<0.04	<0.04	<0.04	<0.04
39	6	Dibenzo[a,h]pyrene	<0.04	<0.04	<0.04	<0.04
40	6	Dibenzo[a,l]pyrene	<0.04	<0.04	<0.04	<0.04
Summary		∑ 40 PACs (mg/kg)	14.7	5.34	3.41	16.9
		∑ 4-6 ring PACs (mg/kg)	13.4	5.34	3.20	6.91

From these same four sources, X-Ray Fluorescence (XRF) results are shown in TABLE 7. XRF testing provides a wide scope of different elements (up to 70) ranging from fluorine to uranium. Samples were run on a Thermo Advant'X WDXRF. Data were processed through Thermo Uniquant™ using the integrated fundamental parameters' calculations.

TABLE 7. Elemental composition of Four Sources as determined by XRF Analysis

Element	Source A	Source B	Source C	Source D
mg/kg - XRF				
Ca	0.81	1.19	1.17	1.16
P	0.40	0.47	0.51	0.44
Zn	0.39	0.63	0.54	0.45
S	0.51	0.21	0.24	1.26
Fe	0.11	0.13	0.28	0.14
Mg	0.08	0.09	0.10	0.07
Al	0.03	0.02	0.08	0.05
Mo	0.02	0.04	0.04	0.01
Na	0.25	0.19	0.21	0.19
Si	0.04	0.04	0.10	0.09
Cu	0.02	0.02	0.03	0.02
Pb	<0.01	<0.01	<0.01	<0.01
Ba	<0.01	<0.01	<0.01	<0.01
Ti	<0.01	<0.01	<0.01	<0.01
Mn	<0.01	<0.01	<0.01	<0.01
Cr	<0.01	<0.01	<0.01	<0.01

Testing on the samples from four different sources found the MI's to all be below 1 and low levels of PACs revealing that these oils are similar to each other. This is in part based on similar processing methods for these sources. Metals varied slightly but were, in general, similar.

Laboratory-Generated Emissions

To simulate worst case workplace exposures to heated asphalt that may contain REOB/VTAE, the following laboratory study was conducted. Asphalt emissions (fumes) were generated in a laboratory to compare 0, 3, 6 and 9% REOB/VTAE in PG 58-28 asphalt. Source A (from TABLE 5) was used to dose the asphalt at these percentages. Analytical testing of these emissions included Total Organic Matter (TOM), simulated distillation, fluorescence, 40 polycyclic aromatic compounds (PACs), and extracted ion chromatogram comparisons. Results are summarized below with details provided at the end of this section.

A modified Brandt [4] laboratory fume generation apparatus was used for this study, which has been used routinely by HRG to study asphalt emissions [5]. Bitumen fumes generated in the laboratory using this protocol represent a worst case scenario and may not be representative of worker exposures in the field.

Laboratory-generated asphalt emissions, generated at 150°C, were consistent between asphalts prepared with and without REOB/VTAE. The differences seen amongst all samples tested – at 10.8% Relative Standard Deviation (RSD) - are within the error of the fume generation and analytical methods (samples = 10.8% Relative Standard Deviation (RSD), method = 27.9% RSD). Summary data are shown in TABLE 8. Duplicate fume generations were conducted for the asphalts containing REOB/VTAE. The average data for Total Organic Matter (TOM), 4-6 ring PACs by fluorescence at 385nmex/415nm em (FL-PACs) and simulated distillation at 10, 50 and 90 % are presented.

TABLE 8. Summary Data for Laboratory-Generated Asphalt Emissions

	% REOB/VTAE	TOM (mg/m^3)	FL-PAC EU/g ADJ	FL-PAC µg/m^3	10% Distilled °C	50% Distilled °C	90% Distilled °C
	0	121	52.5	0.81	136	241	378
	3	168	31.7	1.31	142	232	368
	6	89.0	53.0	1.06	128	236	376
	9	127	31.9	1.04	130	220	366
Without REOB/VTAE		121	52.5	0.81	136	241	378
Average With REOB/VTAE		108	47.7	0.57	127	230	374
%RSD with vs without		*7.99*	*6.83*	*25.5*	*4.84*	*3.20*	*0.82*
%RSD (All samples)		*25.7*	*28.6*	*19.2*	*4.6*	*3.9*	*1.7*

TOM=Total Organic Matter FL-PAC=Fluorescence Polycyclic Aromatic Compounds RSD=relative standard deviation

PACs detected in both the asphalt and the asphalt emissions are presented here based on gas chromatography/mass spectrometry (GC/MS) analysis using an Agilent Q Time of Flight as previously described. TABLE 9 shows results for the asphalts prepared at 0, 3, 6 and 9 percent REOB/VTAE. Of 40 PACs examined, only 11 were detected in the asphalts above a detection limit of 0.2 mg/kg.

TABLE 9. PACs in Asphalts Containing 0, 3, 6, and 9% REOB/VTA

	No. Benzene Rings	Sample Concentration (mg/kg)	0% REOB/VTAE	3% REOB/VTAE	6% REOB/VTAE	9% REOB/VTAE
1	1+	Benzothiophene	22.9	37.2	15.8	37.5
2	2	Naphthalene	503	730	291	635
3	2+	Dibenzothiophene	74.6	56.4	45.0	40.0
4	2+	Fluorene	18.3	14.9	6.9	8.9
5	3	Anthracene	4.96	4.67	2.33	2.64
6	3	Phenanthrene	29.9	24.3	14.1	14.6
7	3+	Benzo[b]naphtho[2,1-d] thiophene	1.58	1.27	0.53	0.68
8	3+	Fluoranthene	1.58	1.27	0.53	0.68
9	4	Chrysene	0.42	0.32	0.32	0.29
10	4	Pyrene	4.65	4.25	1.91	2.15
11	4	Triphenylene	0.63	0.53	0.21	0.29
Summary		∑ 40 PACs (mg/kg)	657	870	377	740
		∑ 4-6 ring PACs (mg/kg)	5.70	5.10	2.44	2.73

TABLE 10 shows PAC results for the laboratory-generated asphalt emissions in asphalts prepared at 0, 3, 6 and 9 percent REOB/VTAE. There is no indication upon examination of these data that the addition of REOB/VTAE causes an increase in PAC content in either the asphalt or its emissions.

TABLE 10. PACs in Laboratory-Generated Asphalt Emissions with 0, 3, 6, and 9% REOB/VTAE

	No. of Benzene Rings	Sample Concentration (μg/m³ air)	0% REOB/VTAE	3% REOB/VTAE	6% REOB/VTAE	9% REOB/VTAE
1	1+	Benzothiophene	22.2	40.2	37.0	47.9
2	2	Naphthalene	289	181	253	262
3	2+	Acenaphthene	18.3	19.6	14.6	10.4
4	2+	Acenaphthylene	2.50	1.99	1.65	1.14
5	2+	Dibenzothiophene	41.8	37.8	26.1	24.0
6	2+	Fluorene	39.1	39.0	27.1	24.8
7	3	Anthracene	2.15	1.86	1.39	1.24
8	3	Phenanthrene	31.5	26.5	19.2	16.8
9	3+	Benzo[b]naphtho[2,1-d]thiophene	1.37	1.11	0.99	0.78
10	3+	Benzo[b]naphtho[2,3-d]thiophene	2.29	1.99	2.05	1.93
11	3+	Fluoranthene	1.37	1.21	0.99	0.91
12	4	Benz[a]anthracene	0.21	0.16	0.17	0.16
13	4	Chrysene	0.42	0.39	0.33	0.36
14	4	7,12-Dimethylbenz[a]anthracene	0.67	0.49	0.36	0.39
15	4	Pyrene	4.29	3.43	2.64	2.45
16	4	Triphenylene	0.46	0.39	0.30	0.33
17	4+	Benzo[b]fluoranthene	0.11	0.10	0.10	0.07
18	5	Benzo[e]pyrene	0.21	0.13	0.13	0.13
Summary		∑ 40 PACs (μg/m³)	451	353	384	392
		∑ 4-6 ring PACs (μg/m³)	6.37	5.09	4.03	3.89

Leachate Studies

A leachate study was conducted on asphalt containing REOB/VTAE at 9% using the same REOB/VTAE as listed in TABLE 5 as Source B [6]. The purpose of the leachate testing was to determine if REOB/VTAE in the binder impacted the leachate potential from asphalt pavements. The Toxicity Characteristic Leaching Procedure (TCLP) was performed according to EPA Method SW846-1311. In this procedure, asphalt binder is mixed with aggregate to increase the surface area and facilitate leaching. Extensive testing included metals analysis for the regulated 8 elements, volatile organic analysis, semi-volatile organic analysis, and analysis of 40 PACs using GC/QTOFMS with detection limits significantly lower than that of traditional testing (between 0.1 and 5 ppb versus 50 ppb using the traditional methods). TCLP herbicides (SW 846-8151), TCLP pesticides (SW 846-8081), TCLP mercury by CVAA (SW 846-7470A), TCLP metals analysis by ICP-MS (SW 846-6020A), TCLP semi-volatile organics (SW 846-8270), and TCLP volatile organics (SW 846-8260) were analyzed using a National Environmental Laboratory Accreditation Program certified laboratory.

Results were non-detected for 39 of 40 of the traditional TCLP analytes in both studies. Barium was detected in the control sample without REOB/VTAE at 0.13 mg/L and 0.11 mg/l with the binder that contained REOB/VTAE at 9%. The regulatory level for barium is 100 mg/L. These trace levels of barium typically come from limestone in the aggregate not the binders. Results are shown in TABLE 11 (metals) and TABLE 12 (organics).

TABLE 11. Results for Leachable Elements in Asphalt Mix Samples

Element	CAS No.	PG 58-28 Neat mg/L	PG 58-28 with REOB/VTAE mg/L	Detection Limit mg/L	Regulatory Limit mg/L
Arsenic	7440-38-2	*BDL*	*BDL*	0.05	5
Barium	7440-39-3	0.13	0.11	0.05	100
Cadmium	7440-43-9	*BDL*	*BDL*	0.025	1
Chromium	7440-47-3	*BDL*	*BDL*	0.05	5
Lead	7439-92-1	*BDL*	*BDL*	0.05	5
Mercury	7439-97-6	*BDL*	*BDL*	0.002	0.2
Selenium	7782-49-2	*BDL*	*BDL*	0.05	1
Silver	7440-22-4	*BDL*	*BDL*	0.05	5

BDL = Below Detection Limit

TABLE 12. Results for Leachable Semi-volatiles in Asphalt Mix Samples

Compound	PG 58-28 Neat	PG 58-28 REOB/VTAE	Detection Limit µg/L
1,4-Dichlorobenzene	BDL	BDL	50
2,4-Dinitrotoluene	BDL	BDL	50
Hexachlorobenzene	BDL	BDL	50
Hexaachlorotutadiene	BDL	BDL	50
Hexachloroethane	BDL	BDL	50
Nitrobenzene	BDL	BDL	50
Pyridine	BDL	BDL	250
2-Methylpehnol (O-Cresol)	BDL	BDL	50
3-Methylphenol (M-Crosol)	BDL	BDL	50
4-Methylphenol (P-Cresol)	BDL	BDL	50
Pentachlorophenol	BDL	BDL	250
2,4,5-trichlorophenol	BDL	BDL	50
2,4,6-Trichlorophenol	BDL	BDL	50
Acenaphthene	BDL	BDL	50
Acenaphthylene	BDL	BDL	50
Anthracene	BDL	BDL	50
Benzo[a]anthracene	BDL	BDL	50
Benzo[a]pyrene	BDL	BDL	50
Benzo[b]fluoranthene	BDL	BDL	50
Benzo[g,h,i]perylene	BDL	BDL	50
Benzo[k]fluoranthene	BDL	BDL	50
Chrysene	BDL	BDL	50
Dibenz[a,h]anthracene	BDL	BDL	50
Fluoranthene	BDL	BDL	50
Fluorene	BDL	BDL	50
Indeno[1,2,3-cd]pyrene	BDL	BDL	50
Naphthalene	BDL	BDL	50
Phenanthrene	BDL	BDL	50
Pyrene	BDL	BDL	50

continued >>

Surrogate Recoveries	%	%	
2-Fluorophenol	46	53	
Phenol-D5	34	44	
Nitrobenzene-D5	76	79	
2-Fluorobiphenyl	84	81	
2,4,6-Tribromophenol	101	96	
Terphenyl-D14	101	93	

The QTOF GC/MS described previously provides detection limits a factor of >100 lower than achieved using typical environmental laboratory instrumentation. Analysis of 40 PACs showed detectable levels of 9 PACs in both the control and the REOB/VTAE samples (see TABLE 13); all results were below 3 μg/L (ppb). The sum of the 9 detectable PACs was 12.3 ppb versus 13.2 ppb for the control and REOB/VTAE samples respectively.

TABLE 13. Results for Leachable PACs in Asphalt Mix Samples using GC-TOFMS

Compound Sample Concentration (μg/L)	NEAT PG 58-28 CONTROL	PG 58-28 with 9% REOB/VTAE	Detection Limit μg/L
Naphthalene	1.98	2.70	0.33
Acenaphthylene	BDL	BDL	0.45
Acenaphthene	BDL	BDL	0.49
Fluorene	BDL	BDL	0.79
Phenanthrene	1.08	1.16	0.76
Anthracene	1.41	1.32	0.86
Fluoranthene	1.13	1.01	1.54
Pyrene	1.30	1.30	1.13
Benzo[b]naphtho[2,3-d]thiophene	1.70	1.77	2.87
Benz[a]anthracene	BDL	BDL	0.58
Cyclopenta[cd]pyrene	BDL	BDL	3.18
Triphenylene	BDL	BDL	3.34
Chrysene	BDL	BDL	0.30
5-Methylchrysene	BDL	BDL	0.26
1-Nitropyrene	BDL	BDL	1.75
Benzo[b]fluoranthene	BDL	BDL	0.22
7,12-Dimethylbenz[a]anthracene	BDL	BDL	0.26
Benzo[k]fluoranthene	BDL	BDL	0.21
Benzo[j]fluoranthene	BDL	BDL	0.22
Benzo[e]pyrene	BDL	BDL	0.11
Benzo[a]pyrene	BDL	BDL	0.25
3-Methylcholanthrene	BDL	BDL	0.11
Dibenz[a,j]acridine	BDL	BDL	0.74
Dibenz[a,h]acridine	BDL	BDL	1.36
Indeno[1,2,3-cd]pyrene	BDL	BDL	1.92
Dibenz[a,h]anthracene	BDL	BDL	4.76
Benzo[ghi]perylene	BDL	BDL	0.19
7H-Dibenzo[c,g]carbazole	BDL	BDL	0.20
Dibenzo[a,e]fluoranthene	BDL	BDL	0.35

Dibenzo[a,e]pyrene	BDL	BDL	0.26
Benzo[rst]pentaphene	BDL	BDL	1.22
Dibenzo[a,h]pyrene	BDL	BDL	0.30
Dibenzo[a,l]pyrene	BDL	BDL	0.31
Benzo[b]thiophene	BDL	BDL	0.18
Dibenzothiophene	1.95	2.10	0.19
Carbazole	0.63	0.66	0.21
Benzo[b]naphtho[2,3-d]thiophene	1.08	1.17	0.27
Benzo[c]acridine	BDL	BDL	0.42
Benzo[a]acridine	BDL	BDL	1.09
Dibenz[c,h]acridine	BDL	BDL	1.34
7H-Dibenzo[c,g]carbazole	BDL	BDL	0.56

Metals

The toxicity of metals present in REOB/VTAE is related to chromium, zinc and copper. Some wear metals are removed before vacuum tower, through the processing of the used engine oil. Additive metals in the REOB/VTAE are also present in the blended asphalt binder but were not found to be leachable in this study [6]. The levels found in this study are also not significantly different than other materials found in construction such as asphalt and concrete pavements.

Physical and Chemical Characteristics

REOB/VTAE is composed of the higher boiling components of used oil [7]. It generally contains paraffinic lube (neutral base oil), additives for oxidation protection, viscosity index improvers (polymer – i.e. poly butylene, poly iso-butylene), and anti-wear additives such as Zinc Dialkyl Dithiophosphate (ZDDP) and molybdenum sulfide. These additives are designed to protect the engine from damage, oxidation, and achieve cold and hot weather performance properties. REOB/VTAE can also contain low levels of wear metals from the engine. Because these metals are non-distillable, they will remain in the REOB/VTAE if not removed in the initial refining steps. REOB/VTAE is the highest boiling paraffinic lubricants and represents about ~12% of the used oil. It can contain about 4% polymers and about 50-60% of lower viscosity dewaxed lubricating oil. Engine oil additives, which can include metals such as zinc and molybdenum, are in a form such that they are soluble in REOB/VTAE and represent about 5% of the REOB/VTAE.

Although chemical and physical properties of REOB/VTAE vary, they are all viscous liquids at room temperature, the flash point (ASTM D 92) is greater than 232 °C, the RTFOT (ASTM D 2872) is generally <1% mass loss, and the solubility (ASTM D 2042) is greater than 98%. REOB/VTAE's Brookfield Viscosities (ASTM D 4402) vary widely between producers. For example, Producer A = 257.3 cps @ 135°C and Producer B = 28.2 cps @ 135°C. At 60 °C, REOB/VTAE is generally <50 P.

The full physical and chemical variability of REOB/VTAE samples is not known. Various physical parameters of REOB/VTAE measure in a subset of samples are reported in TABLE 14.

TABLE 14. Physical Parameters of REOB/VTAE Sources

	Source A	Source B	Source D	Average	%RSD
API@60°F	18.7	17.9	17.8	18.1	2.72
Specific Gravity	0.9415	0.9469	0.948	0.945	0.37
lb./gal	7.84	7.89	7.9	7.9	0.41
Ash %	4.81	4.54	4.96	4.77	4.46
Pour Point °F	-20	-18		-19	-7.44

REOB/VTAE Chemical Composition

Understanding the composition of REOB/VTAE requires separating the major components within the material. Using a fractionation procedure developed by Strieter [8], HRG isolated the Non-Polars, Mid-Polars and Polar fractions of REOB/VTAE products with results shown in TABLE 15. Based on GPC analysis, the Non Polar and Mid Polar fractions are of similar apparent molecular size. The REOB/VTAE was approximately 56.3% Non-Polars, 33.3% Mid-Polars, and 10.4% Polars.

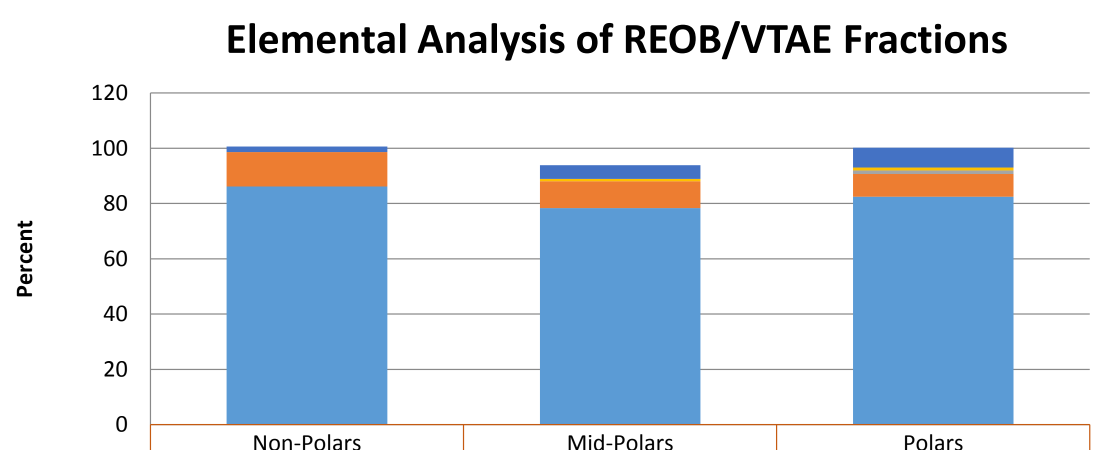
TABLE 15. Strieter Fractionation of REOB/VTAE into Non-Polars, Mid-Polars, and Polars

	Source A	Source B	Average
Polars %	10.8	10.1	10.4
Mid-Polars %	35.2	31.4	33.3
Non-Polars%	54.0	58.5	56.3

Polars are based on insolubility in pentane.

Elemental composition was determined using a combustion analyzer on each of the fractions. Results are shown in FIGURE 4, showing 98.6% of the Strieter Non-Polar fraction is comprised of C and H, 87% of the Mid-Polars are comprised of C and H, and 90.8% of the Polar fraction is C and H. Oxygen is detected in the Mid-Polar and Polar fractions, while nitrogen in only detected in the Polar fraction. Sulfur is detected in all three fractions.

FIGURE 4. Elemental Composition of REOB/VTAE Strieter Fractions Based on Combustion Analysis. The Zeros Represent < 0.5%. Recovery for the Mid-Polar fraction was ~94%.



	Non-Polars	Mid-Polars	Polars
Sulfur	2	4.92	7.15
Oxygen	0	0.94	1.1
Nitrogen	0	0	1.12
Hydrogen	12.5	9.7	8.3
Carbon	86.1	78.3	82.5

The Mid Polar does not add to 100% because not all of the material could be fully extracted from the media for analysis. These three fractions were further analyzed by WD-XRF. Estimated results are displayed in TABLE 16.

TABLE 16. Elements Detected Using XRF in each of the Strieter Method Fractions

	Source A			Source B		
m/m%	Polars	Mid-Polars	Non-Polars	Polars	Mid-Polars	Non-Polars
Element						
Ca	1.760	0.001		0.127		
Zn	1.380			0.114		
V	0.494	0.003		0.553		
Px	0.296					
Fe	0.344			0.119		
Ni	0.206	0.002		0.238		
K	0.148					
Si	0.068	0.011	0.002		0.040	0.713
Mg	0.076	0.024	0.013		0.360	0.028
Al	0.042	0.002	0.001			0.007
Mo	0.048					
Cu	0.051					
Ti	0.021					
Pb	0.020					
Sn	0.009	0.001				
Cr	0.007					
Na		0.080	0.040		0.080	0.038

Strieter Non-Polars

The Non-Polar fraction was 55% of the REOB/VTAE product and was paraffinic base oil with a molecular weight as determined by GPC of 925 daltons. The FTIR data shows this fraction to be primarily aliphatic hydrocarbons.

Strieter Mid-Polars

The molecular weight of this fraction was determined by GPC to be 1125 daltons. Aliphatic hydrocarbons, aldehydes, alcohols, and organic acids were the primary components detected in the volatile and semi-volatile analyses. Quantitative results for the PAC analysis detected ∑40PAC at 123 mg/kg. The RI @ 25°C is approximately 1.52. FTIR shows esters as being the main functional group in the resin fraction. Acids and amides are weak if present. Based on a total acid number of 4.4 mg KOH/g, calculating the acid as stearic acid, there would be about 2% long chain fatty acids in the Mid Polar fraction. Some of the esters are likely from synthetic oils.

Strieter Polars

Based on GPC analysis, the Polars are in the 7500 Dalton range. V, Ni, Ca, Fe, and Zn reside in this fraction. Most of the Polar fraction are metal complexes from additive packages and wear metals.

References

1. **TOXNET** Toxicology Data Network Substance Name: Lubricating oils, used, residues FN: 129893-17-0 [11/20/2015]. Available from: http://chem.sis.nlm.nih.gov/chemidplus/rn/129893-17-0

2. **ASTM: Method E 1687-95, in Volume 11.03**, Occupational Health and Safety, ASTM, W. Conshohocken, PA.

3. **U.S. Environmental Protection Agency (EPA)**: Method SW846-8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). In U.S. EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Method SW846, 3rd rev. ed. Washington, D.C.: EPA, 1996.

4. **Brandt HCA, Watson WP**. Monitoring human occupational and environmental exposures to polycyclic aromatic compounds. Ann of Occup Hyg. 2003;47(5):349-78.

5. **Bolliet C, Kriech AJ, Juery C, Vaissiere M, Brinton MA, Osborn LV**. Effect of Temperature and Process on Quantity and Composition of Laboratory-Generated Bitumen Emissions. *J Occup Environ Hyg.* 2015; 12(7):438-49.

6. **Wielinski, JC, AJ Kriech, GA Huber, A Horton, LV Osborn**. Chemical Analysis of Asphalt Blended with Re-Refined Vacuum Tower Bottoms (RVTB) and their Effect on Asphalt Mixture Performance, *Proceedings, Canadian Technical Asphalt Association, 59: 401 – 426 (November 2014)*.

7. **Wielinski, J., Kriech, A., Horton, A., Reece, T., and Huber, G.** Laboratory Evaluation of Vacuum Tower Asphalt Extenders Impact on HMA Fatigue Resistance and Asphalt Binder Aging, Transportation Research Board Annual Meeting, (January 2015).

8. **Strieter, OG**. Method for Determining the Components of Asphalt and Crude Oils. *J Res Natl Bur Stand, 26, 415-418 (1941)*.

V. A DISCUSSION of ALTERNATE TESTS and AGING PROTOCOLS and THEIR APPLICABILITY to the IDENTIFICATION of LONG RANGE MIX PERFORMANCE

Introduction

Within this section currently available research on REOB/VTAE containing binders and the aging procedures and tests performed on those binders that appear to be related to long term mixture performance is discussed. The focus is on evaluations that have been related to the performance of mixtures in the field. Previous sections of this document make clear that there is no shortage of reports and papers dealing with the subject of REOB/VTAE and its use in bitumen. There is however a scarcity of information on field performance of mixtures where there is documented evidence of the presence of REOB/VTAE being used in the binder. In addition there is limited data where comparative performance results on the same project are available where mixtures were produced using binders with and without REOB/VTAE. Such head to head, real world comparative performance data is desirable when attempting to qualify the impact of REOB/VTAE on ultimate mixture performance. Further discussion within this section attempts to contextualize the impact of REOB/VTAE within the larger framework of research performed on binders and the results of that research as it relates to long range mixture performance.

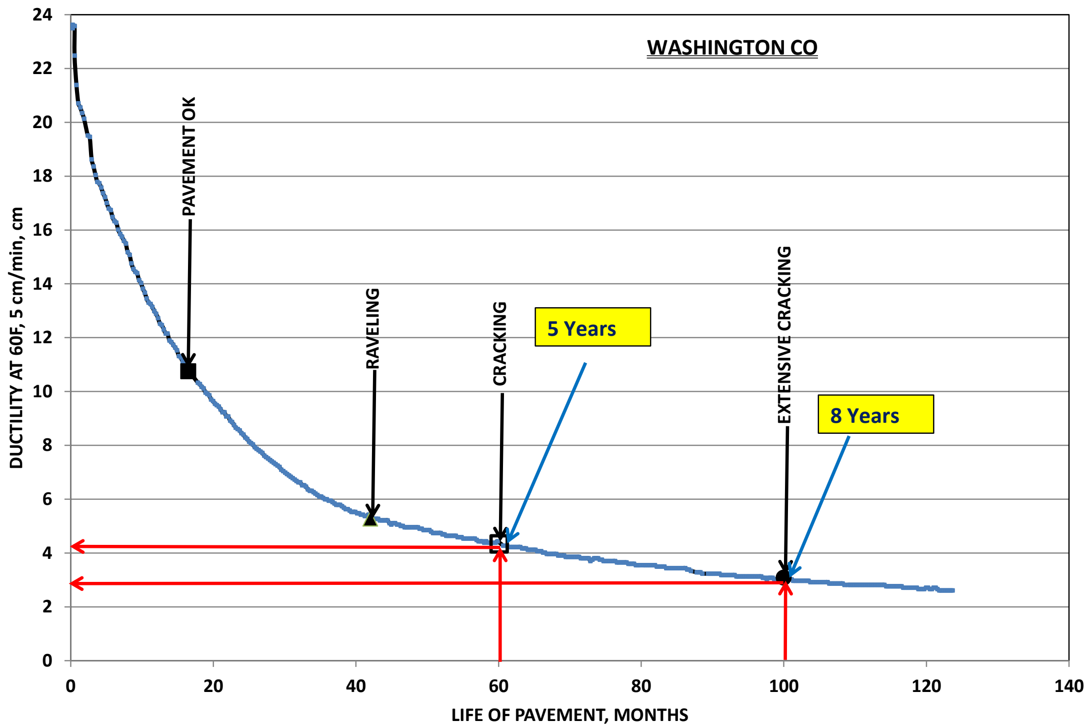
Synopsis

Research to understand factors affecting the durability of bituminous mixtures covers a span of more than fifty years. In this document only a few studies are discussed that inform the efforts to understand how REOB/VTAE can impact binder properties related to long term aging and the relationship of those properties to mixture field performance. One such study is early work by Kandhal and his use of ductility as an indicator of pavement distress. More recently, work by Glover has shown the interrelationship between ductility and binder rheology to predict long term pavement performance. Anderson utilized Glover's methods to develop correlations to air field pavement performance and in so doing developed the parameter of ΔT_c to relate to pavement distress. Some of the investigative work specifically related to REOB/VTAE conducted by Dr. Simon Hesp in Canada is discussed as are REOB/VTAE related presentations at 2014 and 2015 FHWA Expert Task Group meetings. A general theme throughout this section will be the impact that mixture and binder aging exerts on the relaxation properties of mixtures and binders and the extent to which the presence of REOB/VTAE in binders can negatively impact binder relaxation properties with aging.

Kandhal's Research: Investigate Relationship between Ductility of Binder from Field Aged Mixtures and Extent of Distress in Those Pavements

Research investigating the loss of binder ductility at 60°F (15.5°C) and its relationship to mixture cracking performance dates to the early 1960's and work conducted by Kandhal at the Pennsylvania DOT. A summary of Kandhal's work was presented by him as part of an ASTM Symposium in 1977 (Kandhal, 1977). An example of Kandhal's findings is shown in FIGURE 5.

FIGURE 5. Kandhal, Low Temperature Ductility in Relation to Pavement Performance, ASTM STP 628, Marek, Ed., 1977



The base plot is taken from the original publication; colored markups have been added for emphasis to this publication. Specifically, the indication that at 5 years cracking has been identified and the ductility has reached a value of 4 cm and at 8.3 years extensive cracking has been identified and the ductility has reached 3 cm is of importance. Subsequent information in this section will return to the impact of in service aging at the time period between 4 and 8 years and resultant pavement distress. Also note that for a test such as ductility performed on aged bitumen at cooler temperatures the ductility value must approach an asymptote as the ductility cannot ever drop below zero.

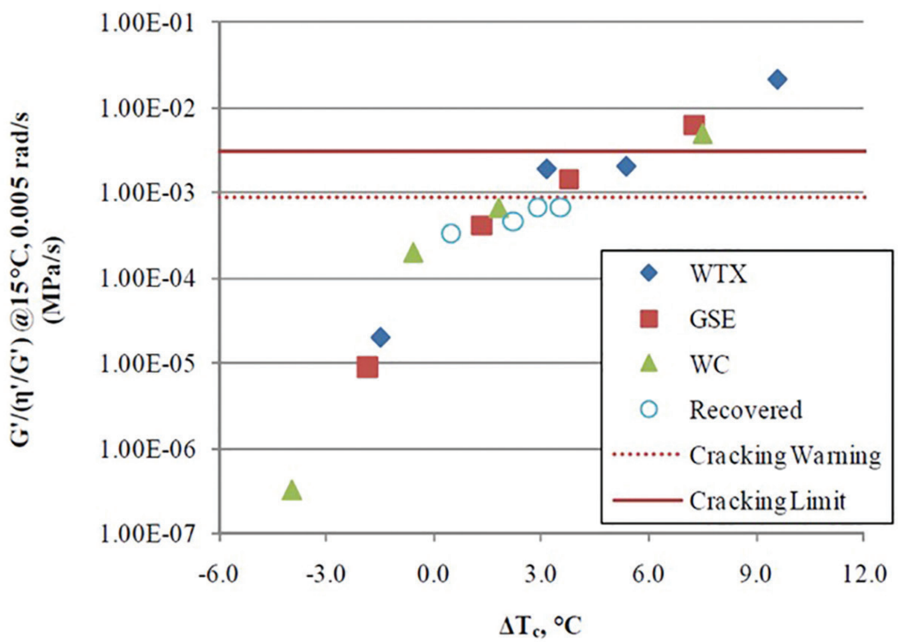
Glover's Research: Identify Properties Affecting Asphalt Performance and Develop Test Methods to Measure Thos°e Properties

In 2005 Glover (Glover, 2005) published the results of research his team had conducted for FHWA and TxDOT into the development of a new test method for evaluating asphalt binder durability which was tied to field validation. The result of Glover's work was to show that a ductility at 15°C and a pull rate of 1 cm/min correlated to long term pavement distress. Further he showed that the ductility result could be correlated to a binder rheological test from which the parameter G'/(η'/G') was determined at 15°C and a test frequency of 0.005 radians/second. As a result of his research Glover established a critical ductility value of 3 cm at 15°C at a pull rate of 1 cm/min and an associated rheological critical value of 0.003 MPa/second at 15C° at a test frequency of 0.005 radians/second. Note that the Glover determination coincides with the 8.3 year ductility Kandhal had labeled as "extensive cracking" in FIGURE 5 and while the test temperatures were nearly identical the pull rates were different. Glover also suggested a 32 hour, 90°C PAV aging step on a thin layer (0.857 mm) of bitumen as means of obtaining a sample that had been aged sufficiently to approximate long term aging of the bitumen in field mixtures.

Anderson's Research: Evaluation between Field Mixture Performance and Recovered Binder Properties and Introduction of ΔT_c Parameter

In research published in 2011 Anderson, et al (Anderson, 2011) investigated the rheological and ductility characteristics of PAV aged binders and binders recovered from aged air field cores to understand the relationship of those rheological properties to the level of non-load associated pavement distress. Key findings from that research suggested a cracking warning limit based on the difference between the BBR m-value critical temperature and the BBR S value critical temperature (defined as ΔT_c) at a ΔT_c value of +2.5°C and a cracking limit at a ΔT_c of +5°C. This concept is shown in FIGURE 6, taken from the Anderson, et. al paper. FIGURE 6 shows the relationship between the Glover parameter of (G'/(η'/G') and ΔT_c. Based on this work the authors concluded that a ΔT_c value of 5°C corresponded to a value beyond which the binder ductility would have decreased to a point where durability had been lost.

FIGURE 6. Relationship Between (G'/(η'/G') and ΔT_c Taken from (Anderson, 2011)



Note that the parameter ΔT_c reported by Anderson et al in 2011 is expressed as a positive value ($T_{critical}$ for BBR m-value – $T_{critical}$ for BBR Stiffness value). Recently the decision within the asphalt technology community has reversed the calculation so that ΔT_c is now expressed as the difference between $T_{critical}$ for BBR Stiffness – $T_{critical}$ for the BBR m-value. Since most binders become increasingly m-controlled due to aging, this approach to calculating ΔT_c results in the ΔT_c becoming more negative as a consequence of binder aging. This convention is followed in the remainder of this section.

Summary of Kandhal, Glover and Anderson

The findings of the Kandhal, Glover and Anderson research are particularly important because the pavement cracking results are not associated with REOB/VTAE or any other specific type of binder treatment. Although no investigations of binder additives were undertaken for any of the materials used in these three studies it is unlikely given the scope of Kandhal's and Glover's work that even if REOB/VTAE had been used in some of the binders evaluated it would have been included in all the samples tested. Given the nature (airfield pavements) and the age of the airfield pavements studied by Anderson it is also unlikely that REOB/VTAE would have been utilized in those asphalt materials. Three independent sets of research - conducted over a period of more than 50 years and focused on significantly different climatological regions and with different crude source binders - has identified relationships related to long term pavement durability. Therefore, the extension of these parameters to examine pavement performance of mixtures containing REOB/VTAE is appropriate. Moreover, as the review herein has shown, the relationships identified by those researchers, while arrived at independently, are clearly interrelated.

Hesp's Research: Relationship between Extended BBR and DENT Tests and Field Performance

Dr. Simon Hesp and his students at Queens University in Kingston, Ontario performed the earliest work attributing the use of REOB/VTAE to early pavement distress in Ontario, Canada (Hesp S. G., 2009). The referenced study utilized test methods that had been developed prior to 2009 by Hesp to investigate early pavement distresses that at the time had been attributed to the use of oxidized binders, gelled asphalt, and/or binders containing polyphosphoric acid (Hesp S. , 2006). The specific tests were the Double Edge Notched Tension test (DENT) (MTO test LS299) resulting in the determination of a Crack Tip Opening Displacement (CTOD) value and the Extended BBR test (MTO test LS308) which determines low temperature grade loss due to physical hardening of the binder as a consequence of isothermal conditioning at low temperatures over a 72-hour time period. In the 2009 paper a test project placed on Ontario Highway 655 in 2003 was reviewed. That project consisted of seven test sections paved with seven different binders each of which met a PG -34 grade based on conventional BBR testing. Sections 3, 4, 6, and 7 were identified as containing zinc and three of those sections contained phosphorus; both of which are marker elements present in REOB/VTAE. However, zinc is also used in some hydrogen sulfide scavengers and phosphorus is present in various forms of phosphoric acid, which is also used in some asphalt modifications not related to REOB/VTAE. No corroborating, independent information is provided to substantiate the assumption that REOB/VTAE was utilized in some of the binders. In the paper results of a 2008 pavement distress survey are reported as is data for CTOD and Extended BBR, for binders recovered from the top 50 mm of cores taken from each test section. TABLE 17 summarizes some data from the 2009 paper.

TABLE 17. Summary of Selected Data collected in 2008 from (Hesp S. G., 2009) **and collected in 2010 from** (Wright, 2011)

Test Section	Low Temp Limiting Grade after 3 days at -12°C & -24°C,2008	Grade loss after 3 days at -12°C & -24°C, °C, 2008	TOTAL CRACK Length in m/500m (TRANS + LONG) 2008	LOG TOTAL CRACK Length	CTOD 2008 mm	ΔLTLG 20 Hr. PAV - 40 Hr. PAV, Wright 2010	Total Crack Length in 2009, m/150 m Trans + Long, Wright 2010
1	-37	0.6	32	1.51	26	-3.2	10
2	-27	-3	275	2.44	10	-10.1	209
3	-26	-0.6	261	2.42	10	-6	113
4	-19	-2.4	628	2.80	9	-6.6	315
5	-30	2	43	1.63	52	-5.1	34
6	-21	-4	390	2.59	26	-7.8	68
7	-27	0.9	305	2.48	23	-7.3	93

For incorporation into this document correlations were evaluated using Grade Loss after 3 Days and Difference in Limiting Grade after 3 days correlated to Total Crack length in a 500 m monitoring section. The correlations are generally reasonable with R^2 values ranging from 0.71 to 0.85. Correlations between CTOD and the field cracking distress data were also evaluated with the best R^2 result being 0.48 for Log of Total Crack Length in the 500 m monitoring section and CTOD. An inspection of TABLE 17 shows that Sections 1, 6 and 7 have similar CTOD results and yet significantly different distress results, while Sections 1 and 5 (both of which are polymer modified and show no presence of zinc) have similar distress results but Section 5 has double the CTOD value of Section 1. These anomalous results for the CTOD data are probably explainable on the basis of binder formulation differences, but they do call into question the overall utility of CTOD test results as a predictor of mixture performance for a wide range of binder formulations.

In 2011 Wright, et.al. (Wright, 2011) revisited some of the data for these same test sections on Highway 655 and reported the results of the low temperature limiting grades (LTLG) after 20- and 40-hour PAV aging periods. The authors showed a strong correlation (R^2=0.92) for the difference between the 20-hour PAV and 40-hour PAV low temperature limiting grade results (ΔLTLG) and the three day grade loss at -10°C in the Extended BBR for the PAV residues. However, the correlation between the difference in the low temperature limiting grade between the 20-hour and 40-hour PAV and the log of total crack length measured in 2008 or in 2010 only exhibited R^2 values of 0.66 and 0.72 respectively. The implication of this level of correlation with respect to the utility of the difference between the 20- and 40-hour PAV low temperature limiting grade results will be revisited later in this section. Among his conclusions Wright characterizes the current 20-hour PAV aging protocol as not providing "sufficient oxidative hardening for poor performing asphalt cements to replicate properties from five to six year old pavement trial sections" and further states "A simple doubling of the PAV aging time to 40 hours appears to provide a significantly improved match between laboratory and recovered grades."

The Hesp et. al. research presented here shows a strong directional relationship between pavement distress and degraded low temperature properties as determined by the extended BBR test procedure. Wright's results for the difference in low temperature limiting grade between 20 hours of PAV aging versus 40 hours of PAV aging compared to extended BBR conditioning of PAV residues implies an interrelationship between PAV oxidative aging and laboratory physical hardening of PAV residues (Wright, 2011). However, the correlation between the difference in low temperature limiting grade between 20 hours and 40 hours of PAV aging and the grade loss of recovered binders from field cores after extended BBR aging was only $R^2 = 0.42$.

Expert Task Group Presentations Focused on REOB/VTAE

Much of the subsequent discussion in this section was conducted and reported since 2014 and has been presented at the FHWA sponsored Binder and Mixture Expert Task Group meetings. This work, while exhibiting a cause for concern regarding the long-term performance of binders containing REOB/VTAE, should not be construed as definitive. The current studies are limited in scope and climatic location and further research is needed to broaden the reach of targeted field studies on REOB/VTAE.

Nearly a full day of the April 2015 Binder ETG meeting held in Fall River, MA was devoted to information related to properties and performance of binders and mixtures containing REOB/VTAE. Several speakers suggested that the current 20 hour PAV aging of binders is not sufficient to capture the impact of REOB/VTAE on the binder's ability to relax stress and consequently capture the impact on mixture fatigue performance. Data was shown by these researchers demonstrating that a double PAV aging cycle, that is two sequential 20 hour PAV aging steps, is more likely to capture the impact of REOB/VTAE on binder properties related to fatigue. The response variables suggested for consideration were the Glover-Rowe parameter (Rowe, 2011), cross over frequency, Rheological Index or R-Value (Christensen, 1992), and the Delta Tc (ΔT_c) (Anderson, 2011), (Bennert, 2015), (G. Reinke, 2015). All of these test parameters show an increase in the level of m-control exhibited by the binders after 40 hours of PAV aging. The rate and extent at which a binder becomes m-controlled is a function of the binder chemistry. An excessive degree of m-control is not exhibited by all binders when blended with a given amount of REOB/VTAE and data presented by Bennert (Bennert, 2015) showed a difference in response with a given binder using REOB/VTAE from two different sources (FIGURES 7 and 8). FIGURE 7 shows that blends of 10% REOB/VTAE from two different sources have a decidedly different impact on ΔT_c after 40 hours of PAV aging. FIGURE 8 shows a similar differentiation for the addition of 6% and 20% REOB from two different sources when added to binders.

FIGURE 7. Impact of 10% REOB from Two Sources Blended with PG 70-22 to Make PG 64-22 (Bennert, 2015)

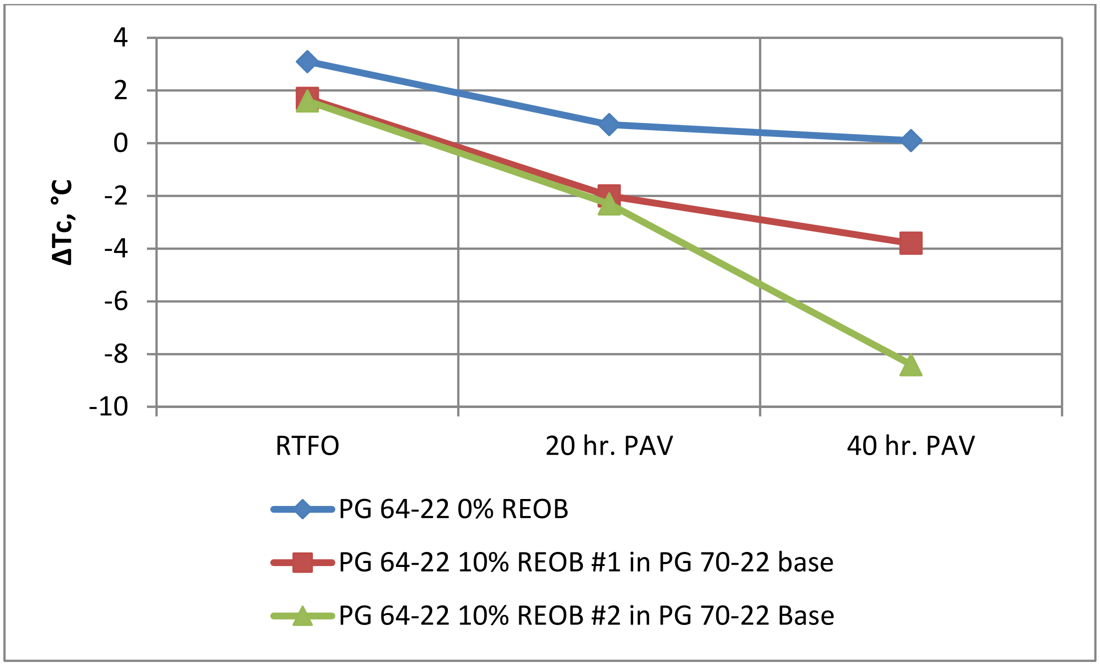
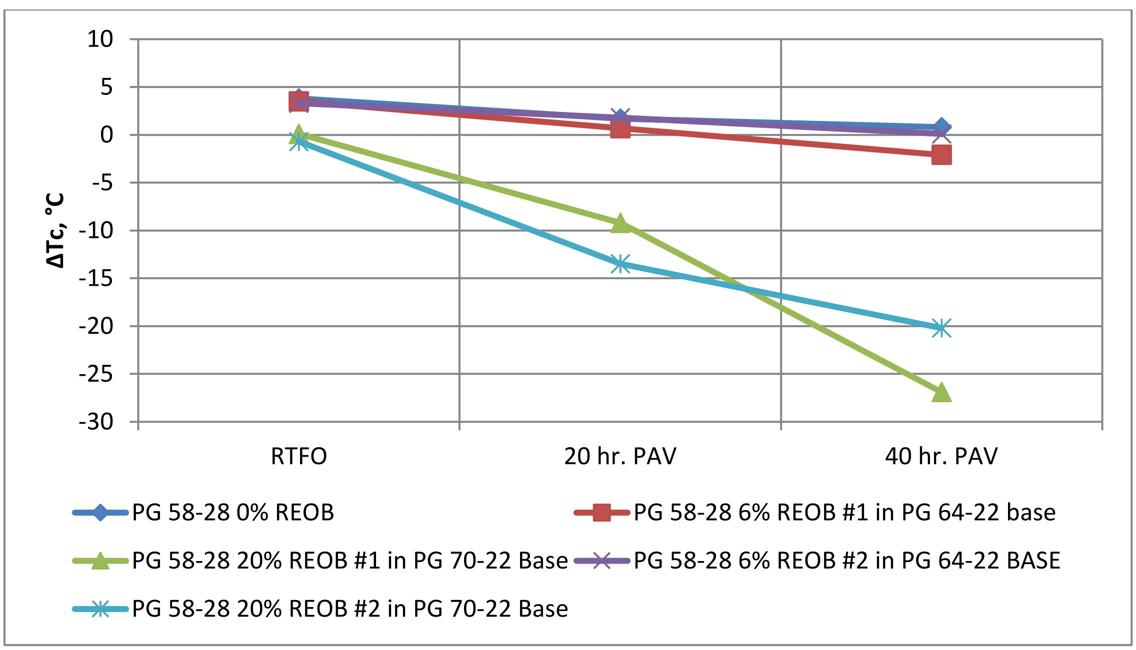


FIGURE 8. Impact of REOB from Two sources Blended at 6% in PG 64-22 and at 20% Into PG 70-22 Base to Produce Final Grade of PG 58-28, (Bennert, 2015)



Work presented by Reinke, Gibson and Bennert (G. Reinke, 2015) (Gibson, 2015) (Bennert, 2015) showed a correlation between ΔT_c and the Crack Tip Opening Displacement (CTOD) results obtained from Double Edge Notched Tension (DENT) test (Hesp S. , 2006). FIGURE 9 presents an example of the how ΔT_c varies with CTOD. CTOD results tend to reach a limiting value similar to earlier work using ductility as the test parameter. There is a strong relationship between the ΔT_c for the binders in FIGURE 9 and the CTOD results (MTO LS299). The binders MN1-2 through MN1-5 refer to a road trial study that will be discussed in more detail below and include polymer modified binder MN1-2. In the earlier discussion with respect to Hesp's work it was observed that CTOD results for polymer modified binders were not necessarily predictive of field performance. The results in FIGURE 9 show that the correlation between CTOD and ΔT_c is slightly reduced when the data for polymer modified binder MN1-2 is included.

FIGURE 9. Relationship between CTOD and ΔT_c for Olmsted CTH 112 PAV Residues (G. Reinke, 2015)

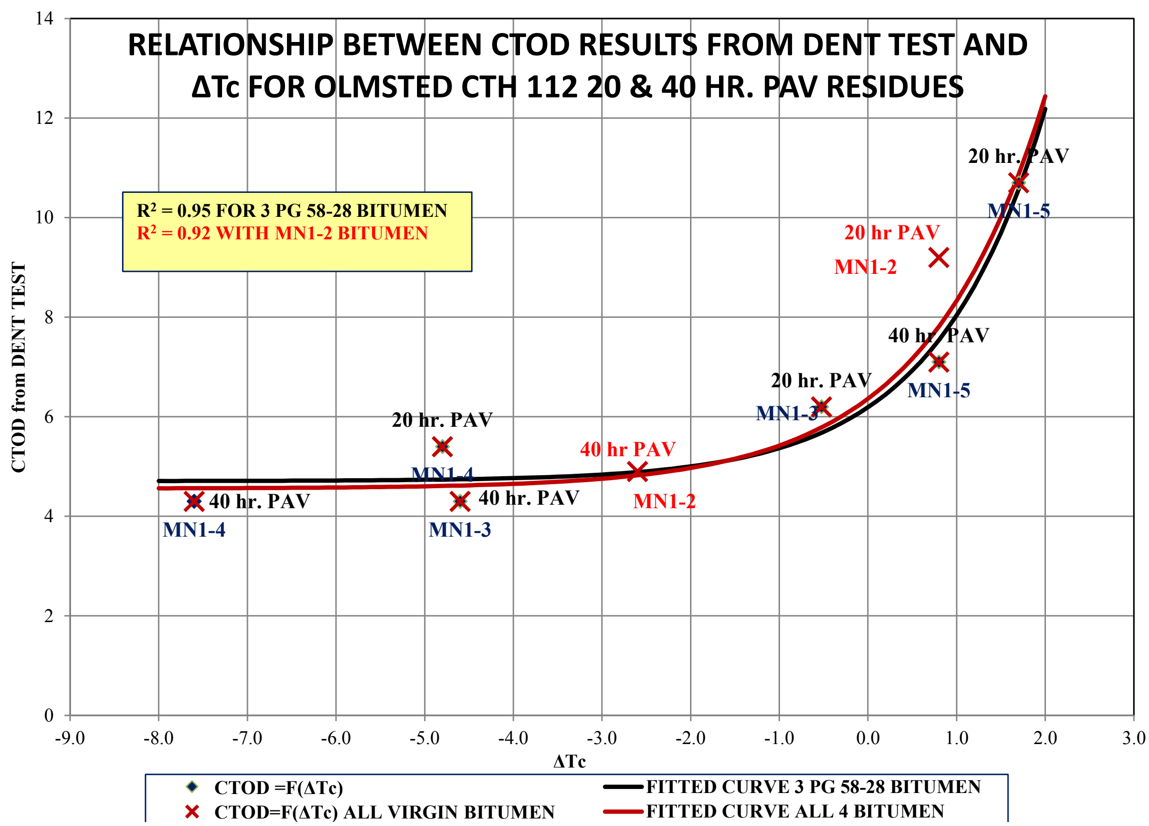
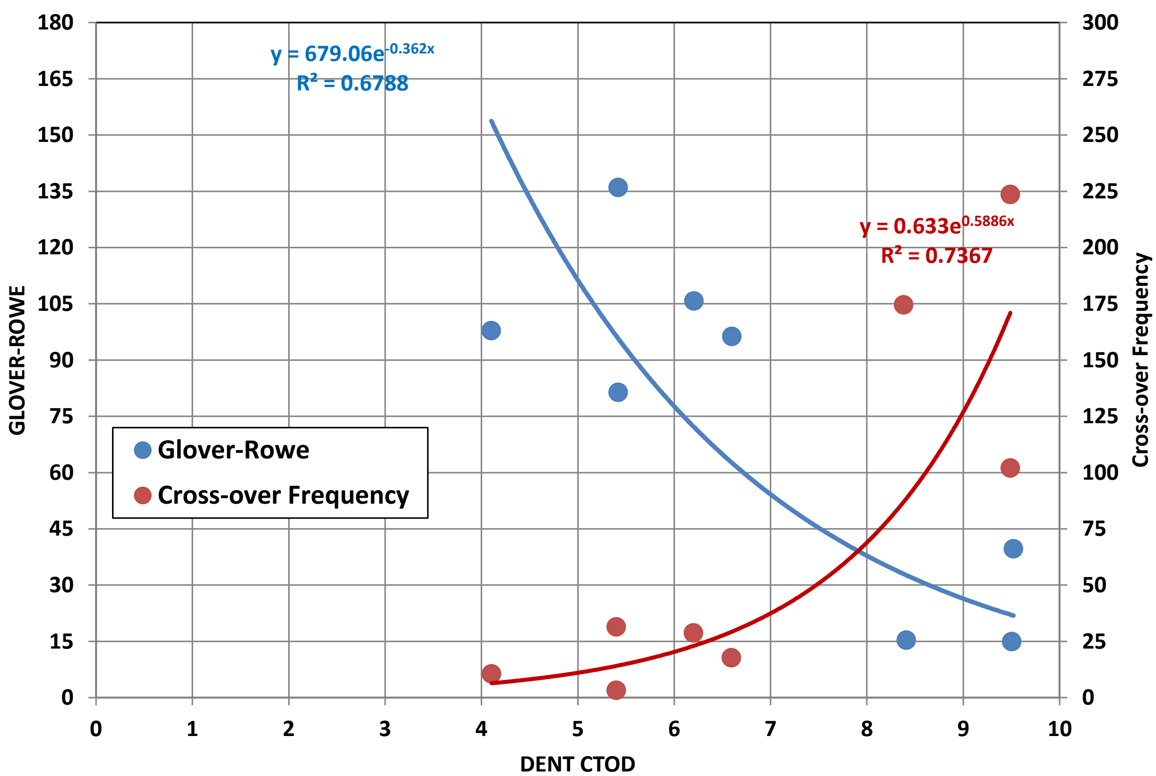


FIGURE 10 shows the manner in which the Glover-Rowe and Cross-over frequency vary as a function of CTOD. The cross-over frequency, Glover-Rowe parameter and ΔT_c are all interrelated as they are all derived from binder rheological data. Based on FIGURES 9 and 10 it appears that the DENT test CTOD results asymptote to a limiting value somewhere in the range of 3 or 4. While the CTOD parameter from the DENT test can segregate binders that are clearly well-performing from those that are clearly poor-performing, it is not as discriminating as rheologically-based parameters, generally, and the ΔT_c parameter, specifically. In contrast to the CTOD parameter, rheologically-based parameters do not have a lower limiting value, which represents an improved approach. In addition, the ΔT_c parameter has a lower coefficient of variation than the CTOD parameter, which represents a significant benefit of correlating performance and managing production control.

FIGURE 10. Relationship between DENT CTOD Results and Cross-Over Frequency and Glover-Rowe Parameter (Bennert, 2015)



Performance of Test Projects Containing REOB/VTAE

Reinke also presented data showing a correlation between ΔT_c of less than -5°C and long-term field performance for two test projects in Minnesota (G. Reinke, 2015). The work cited by Bennert and Gibson also supported the ΔT_c limiting value no lower than -5°C. One of the field projects was constructed on County Trunk Highway (CTH) 112 in Olmsted County, MN. The samples investigated have been designated MN1-2 through MN1-5. MN1-2 was a polymer-modified PG 58-34 and did not contain REOB/VTAE. MN1-3, MN1-4 and MN1-5 were PG 58-28 binders. Only MN1-4 contained REOB/VTAE. The other field project was constructed at the Minnesota DOT's test track, MnROAD, and the samples investigated have been designated as PG 58-28, PG 58-34 (PMA) and PG 58-40 (PMA + REOB/VTAE). The use of REOB/VTAE in the two Minnesota projects discussed in this document was confirmed verbally by the suppliers of those binders. Separate X-Ray Fluorescence testing confirmed the presence of the zinc marker element. FIGURE 11 shows a plot of pavement distress for an eight year old project in Olmsted County, Minnesota as a function of ΔT_c of the binder recovered from the top ½ inch (12.5 mm) of field cores from each test section using binders MN1-2 to MN1-5 as previously discussed. The relationship between ΔT_c and just transverse cracking, usually associated with low temperature fracture, is not as robust as the relationship between ΔT_c and overall pavement distress, which includes wheel path cracking, fatigue area, and non-load associated cracking in the non-wheel path areas as well as transverse cracking. The binder for each test section was derived from a different crude source. All pavement sections were built by the same contractor using the same aggregate structure on the same day. All test sections were constructed with virgin binder over newly prepared aggregate base. Only the MN1-4 section contained REOB/VTAE, estimated at ~8% based upon Zn levels. The modest correlation of transverse cracking with ΔT_c suggests that ΔT_c is not strongly tied to single event thermal cracking. However, when total pavement distress data such as wheel path cracking and non-load associated cracking plus transverse cracking are correlated to ΔT_c the predictive ability of ΔT_c increases substantially.

FIGURE 11. Relationship between ΔT_c of Binder Recovered from 8 year Old Field Cores and Total Pavement Distress after 8 Years in Service. Olmsted County Trunk Highway 112. MN1-3, MN1-4 and MN1-5 were PG 58-28 and MN1-2 was PG 58-34 PMA. MN1-4 Contained REOB (G. Reinke, 2015)

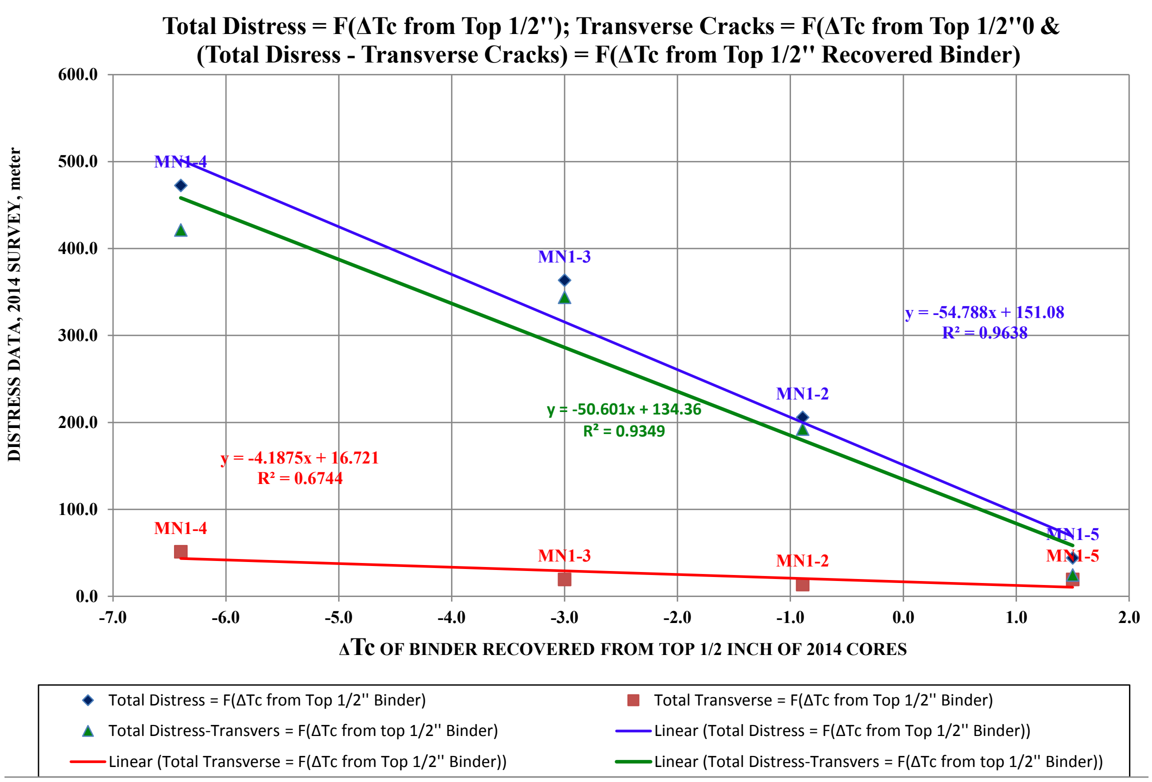
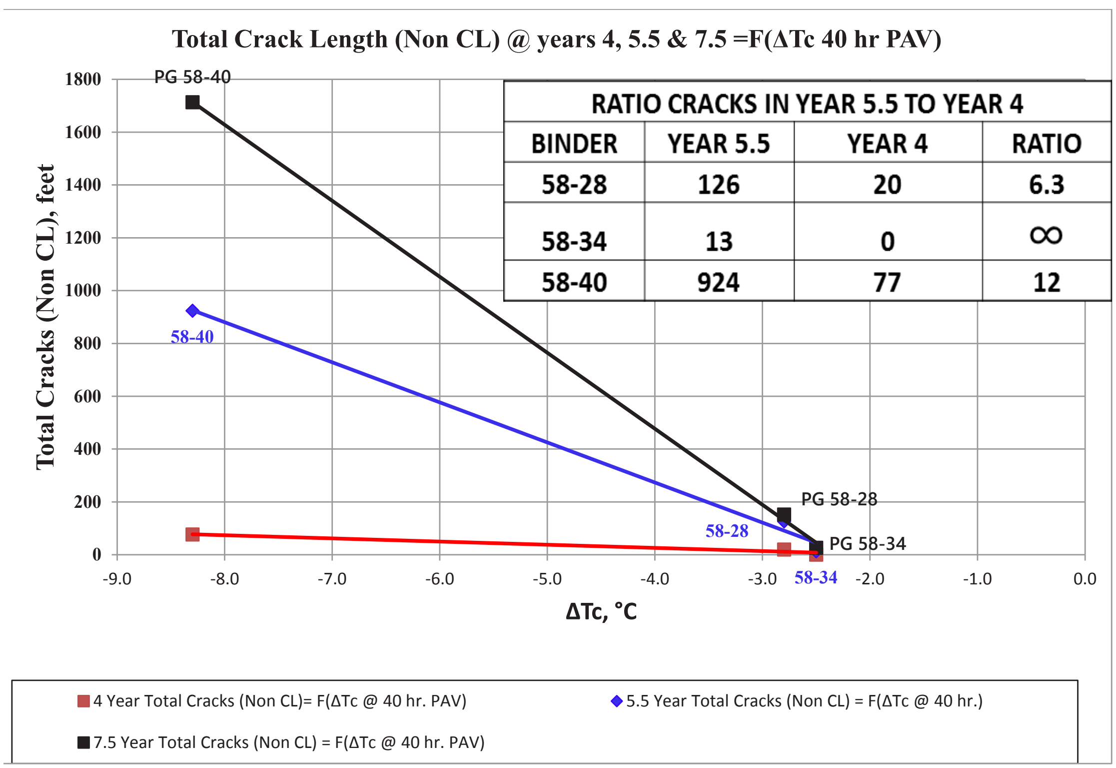


FIGURE 12 is a plot of field performance for a test project at the MnROAD test site (www.dot.state.mn.us/mnroad/index.html). Three binders from the same refinery were used in the construction of the test project in 1999 and the PG 58-34 and PG 58-40 were polymer modified. In addition, the PG 58-40 contained REOB/VTAE to obtain the requisite low temperature grade. As noted in Kandhal's work, the onset of substantial distress seems to occur at the fifth year and following on many pavements. The inset table in FIGURE 12 shows the ratio of cracking between the distress surveys taken at Year 4 and Year 5.5 and also shows plots for the cracking of the three test sections at 4, 5.5 and 7 years.

While the PG 58-40 exhibits the greatest increase in cracking over the time period plotted, it is also obvious that the better performing test sections also show noticeable changes in cracking at Year 5 and later in keeping with the findings of Kandhal.

FIGURE 12—Total Crack Length after 4, 5.5 & 7 YEARS for MnROAD Comparative Binder Study



Paraffinic Base Oils

It should be emphasized that all binders undergo a trend towards being m-controlled as they age. Furthermore REOB/VTAE is not the only binder additive or mixture component that has been shown to alter the rate and extent to which a binder becomes m-controlled. Data presented at the April 2015 ETG meeting (G. Reinke, 2015) showed PMA binders produced from a base binder containing virgin paraffinic base oil exhibited ΔT_c levels lower (more negative) than those resulting from the use of equivalent levels of REOB/VTAE (FIGURE 13). Furthermore, binders recovered from a four-year old field project in Wisconsin that utilized paraffinic oils exhibited a more negative value for ΔT_c as the dosage level of the paraffinic oil increased (TABLE 18).

FIGURE 13. Impact of Equal Amounts of REOB/VTAE & Virgin Paraffinic Base Oil on Binder ΔT_c after Aging

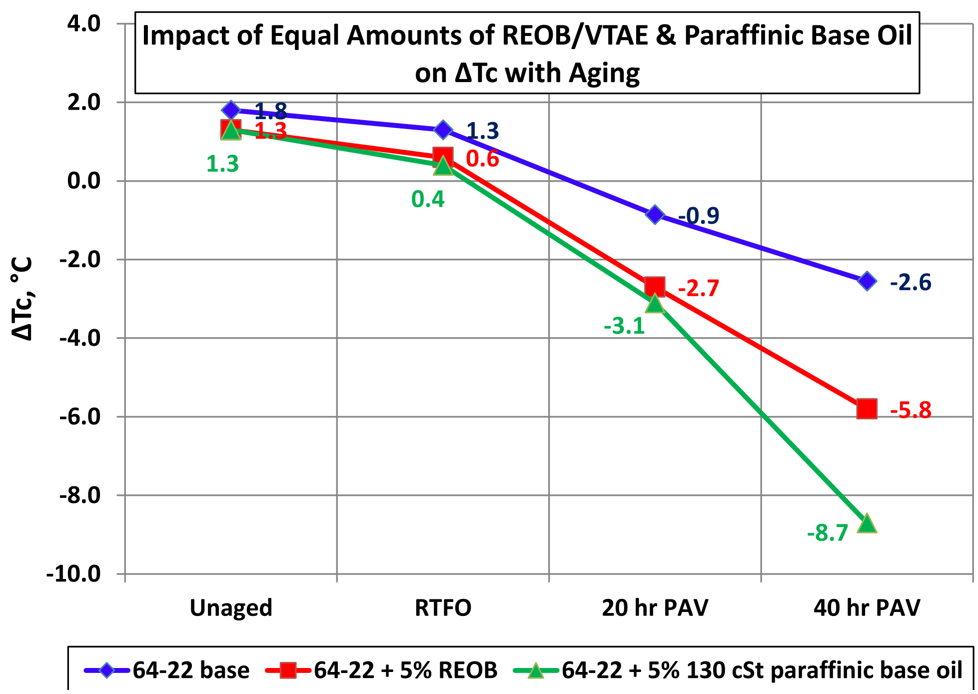


TABLE 18. Impact of Paraffinic Base Oil on ΔT_c of Binders Recovered from 4 Year Old Field Mixture

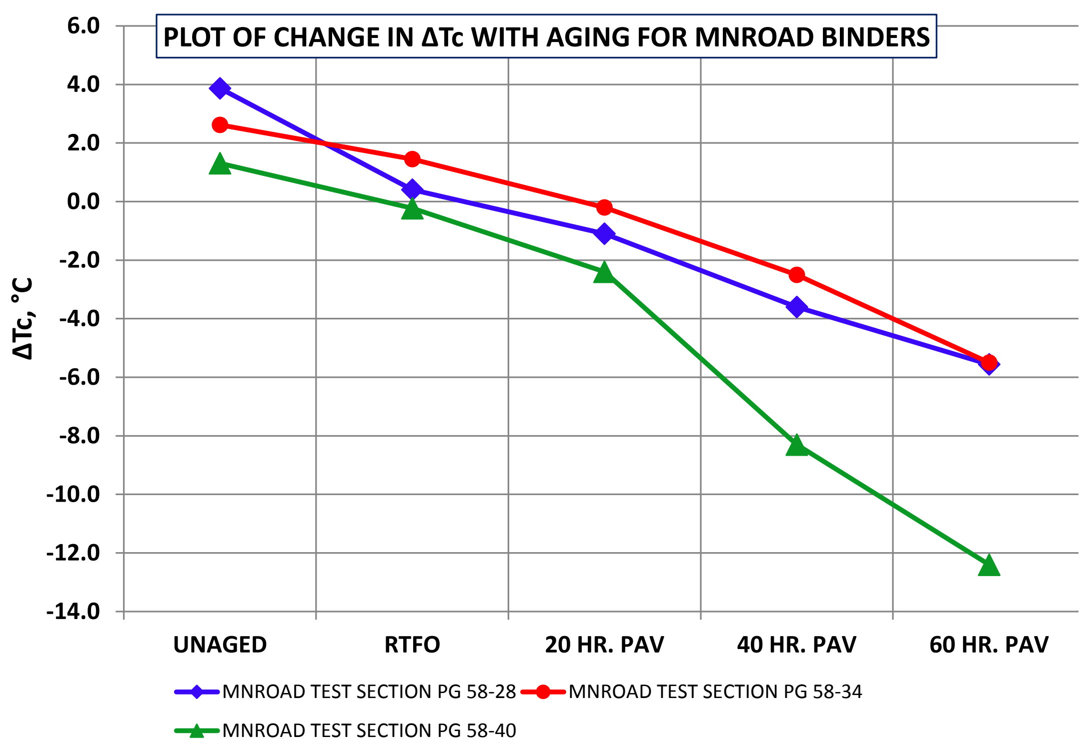
Data from Binder Recovered from WI Highway 53 after 4 years in Service, Constructed in 1994			
Binder Grade	BBR, S Critical Temp	BBR, m Critical Temp	ΔT_c, °C
PG 58-28	-34.5	-35.4	0.9
PG 58-34 (PG 58-28 + 3% 300 cSt paraffinic base oil) Polymer modified	-38.1	-33	-5.1
PG 58-40 (PG 58-28 + 5% 300 cSt paraffinic base oil) Polymer modified	-43.1	-34.9	-8.2

The influence of virgin paraffinic base oils shown in these two examples strongly suggests the reduction of ΔT_c seen in REOB/VTAE modified binders is the result of the paraffinic oil component of the REOB/VTAE and not the other constituents (i.e., metals, additives, etc.).

Variation in ΔT_c with Aging Time and Binder Source

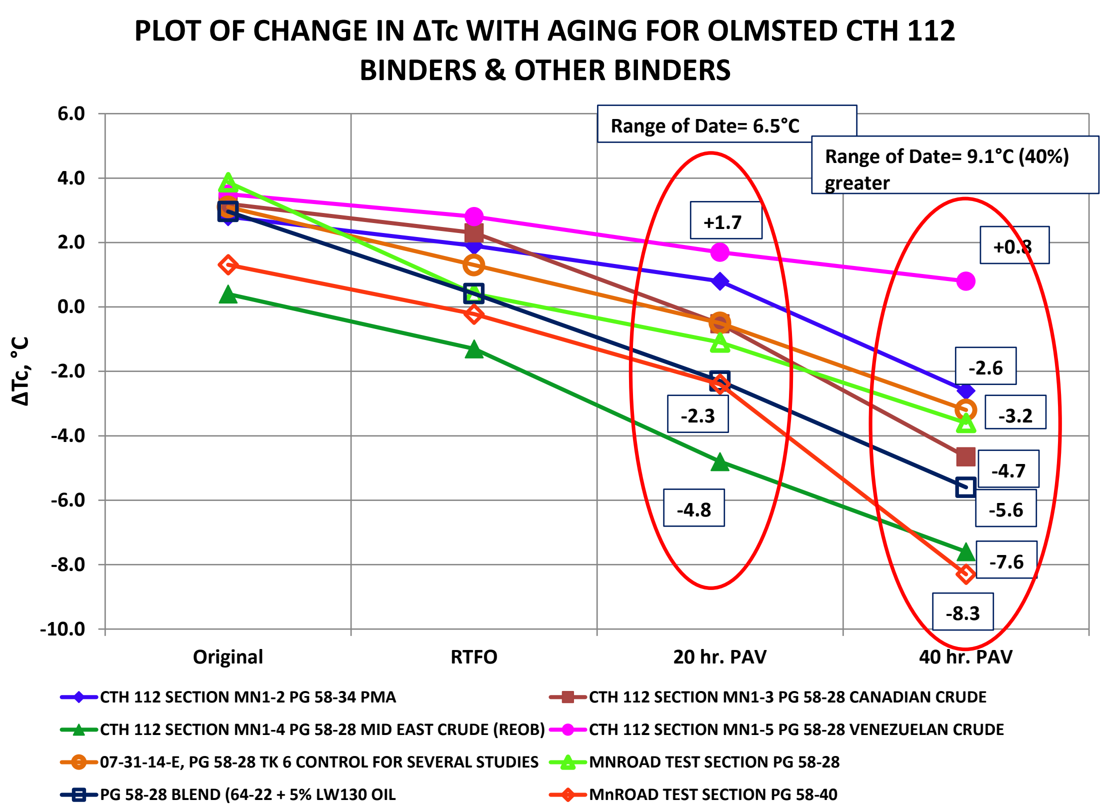
It has been suggested that the ΔT_c results or other rheologically-derived parameters obtained from the 20-hour PAV residue sufficiently rank order binders and there is no need to perform extended binder aging beyond the current 20-hour procedure. The data that has been so far correlated to field performance is based on long term aged (five or more years) field mixtures (Anderson, 2011), (G. Reinke, 2015) and at least for the Reinke, 2015 data the binder recovered from aged field mixtures was better correlated to 40-hour PAV residue properties than 20-hour PAV residue properties. Additionally for some binders there is a different rate of decrease in the value of ΔT_c as the binder is aged from 20 to 40 hours. This implies that the ΔT_c value at PAV40 cannot be reliably estimated from the PAV20 condition. For example, FIGURE 14 shows that at 20 hours the PAV residues ΔT_c results were rank ordered, but the results for the poor performing PG 58-40 binder would not have been flagged because the ΔT_c value was only -2.3°C, whereas after 40 hours of PAV aging the result for the PG 58-40 was -8.3°C. Additional PAV aging past 40 hours did not provide any further performance discrimination.

FIGURE 14. Change in ΔT_c with PAV Aging Time for MNROAD Binders (G. Reinke, 2015)



For a broader view of the importance of extending the PAV aging beyond 20 hours FIGURE 14 plots 20- and 40-hour data for the Olmsted CTH 112 binders, some of the MnROAD binders and some other binders as noted in the legend. The 20-hour PAV results do rank order most of the binders as they rank after 40 hours of aging, but not in all cases. Additionally after the additional aging period the overall range covered by the ΔT_c results is approximately 40% greater, providing greater insight into discerning potentially problematic materials. The rate of decrease in the value of ΔT_c as the binders are aged from 20 to 40 hours is also informative. The best performing binders on Olmsted CTH 112 (MN1-2 and MN1-5) exhibit slight change in the rate at which their ΔT_c values decrease, whereas there is a marked change in the rate of decrease for the MN1-3 binder from a non-concerning value of -1.1°C at 20 hours to a marginal value of -4.7°C at 40 hours. Examination of the performance of the MN1-3 test section (which does not contain REOB/VTAE) shows that this binder is not performing well compared to MN1-2 and MN1-5. It is also worth noting that the PG 58-28 binder used on the MnROAD test project exhibits a lower rate of change as it ages from 20 to 40 hours and its 40-hour ΔT_c value of -3.6 is consistent with its good level of performance after seven years on the MnROAD test site.

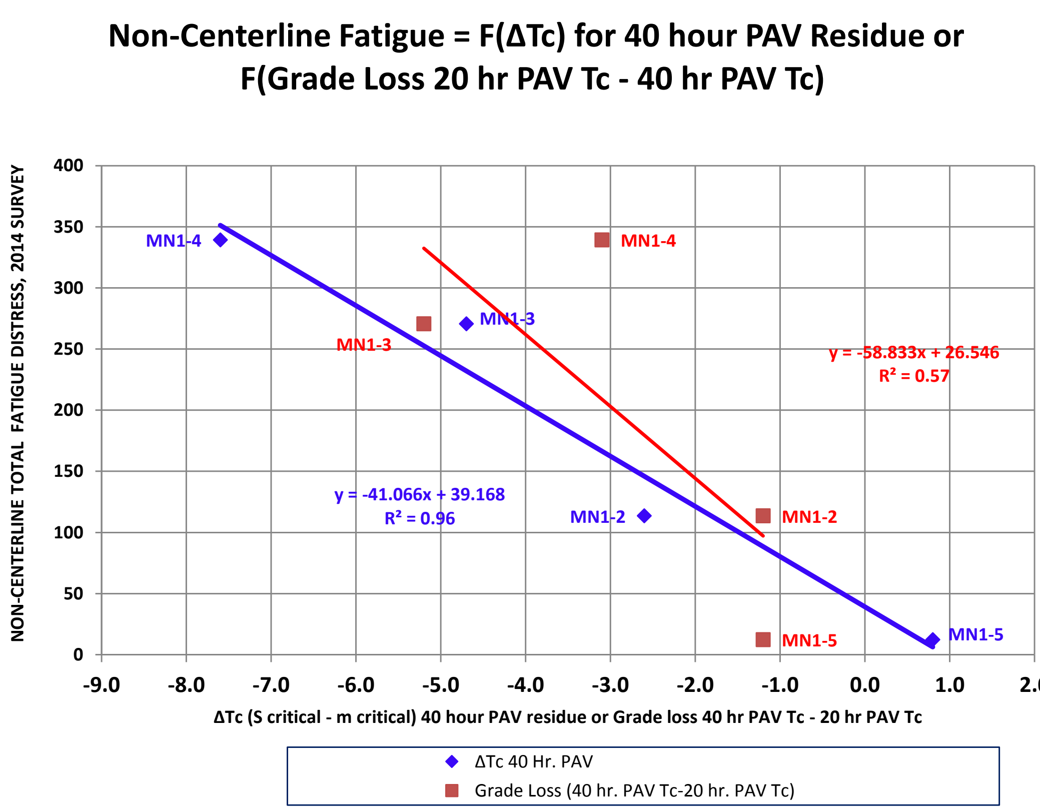
FIGURE 15. Plot of Variation of ΔT_c for Various Binders for 20- and 40-Hour PAV Aging



Comparative Evaluation of Two Parameters Based on 40 Hour PAV Properties

In their 2011 paper Wright et al (Wright, 2011) presented CTOD data on binders recovered from cores taken in 2009 on Highway 655. The authors also determined the low temperature grade loss between the 20-hour PAV and the 40-hour PAV residues. Correlations between the Grade loss data and CTOD data from 2009 result in a R2 value of 0.24. The correlation between the low temperature grade loss and log of crack length in the 150-m monitoring section had an R2 value 0.72. Neither of these results based on Low Temperature Grade Loss are comparable to the correlations of CTOD versus ΔT_c or to pavement distress of Olmsted County 112 mixes with ΔT_c. This is most likely due to the fact that the low temperature grade loss between the 40- and 20-hour PAV results only takes into account the actual low temperature grade which is usually determined by the BBR m-value. The determination of ΔT_c for either the 20-hour or 40-hour PAV takes into account the extent to which the BBR m-value failure critical temperature increases (becomes warmer) compared to the BBR Stiffness critical temperature. For binders that are particularly susceptible to aging, regardless of the causative factors, the m-value critical temperature increases much more rapidly than the Stiffness value critical temperature and therefore ΔT_c is not measuring the same binder characteristic as the Grade Loss value determined by Wright. The information presented in this section has put forth the argument that ΔT_c is a better predictor of fatigue related distress as opposed to single event thermal cracking. It therefore should not be surprising that Low Temperature Grade Loss does not predict the measured pavement distress that has been measured on Olmsted County 112. ΔT_c is a more reliable indicator of when binders are rapidly losing the ability to relax stresses and therefore will exhibit reduced CTOD values. FIGURE 16 is a plot of Non-Centerline Fatigue for Olmsted County 112 plotted as a function of either Low Temperature Grade loss between the 20- and 40-hour PAV residues or the 40-hour PAV ΔT_c results. For these materials, the worst performing binder only had a Low Temperature Grade loss of 3°C. The second worst performing binder exhibited little difference between the Low Temperature Grade Loss and the ΔT_c value. The chemistry of the binder controls these results, and while both Low Temperature Grade Loss and ΔT_c show a similar directional trend they do not provide the same information.

FIGURE 16. Non-Centerline Fatigue Plotted as a Function of Either ΔT_c of 40 hour PAV or as a Function of Low Temperature Grade Loss between the 20- and 40-hour PAV Residues



Summary

Currently only the Province of Ontario requires tests intended to identify binders with a diminished ability to relax stress with conditioning. The specific tests are the DENT test (MTO test LS299), which allows determination of the Crack Tip Opening Displacement (CTOD) value and the extended BBR test (MTO test LS308), which determines the loss in the binder low temperature grade after 72 hours of isothermal conditioning at low BBR testing temperature. Agencies within the United States are considering some of these options and the FHWA Expert Task Groups continue to evaluate the utility of implementing the ΔT_c parameter and moving to a 40-hour PAV requirement.

Kandhal's research showed that in the 5 to 8 year time frame bitumen had lost sufficient ductility to be associated with extensive pavement cracking. Glover's work pointed to similar behavior. The results of Kandhal's and Glover's field related studies suggest that if we are to understand the performance limits of a given bitumen it must be aged more aggressively than present Superpave test methods require if we are to model the aging that takes place at five plus years in service. Anderson's work certainly supports the need to more aggressively age mixtures and binders. The authors of the Wright et al (Wright, 2011) paper also support the need for additional aging of the binder to better predict field performance. The more current research studies discussed and referenced in this document suggest that binder test results from the 40-hour PAV conditioning of binders is a means of gauging field performance after 5 plus years in service. The studies discussed herein are not exclusively focused on binders containing REOB/VTAE. The work conducted by Kandhal and Glover is blind to whatever might have been present in the binder. The work by Anderson et al and the results of Olmsted CTH 112 test section MN1-3 do not contain REOB/VTAE and yet exhibited concerning levels of distress. Other work not discussed in this report, but available in the referenced documents, point to additives capable of causing ΔT_c values below -5°C with extended aging. Work is currently under way to ascertain whether it is possible to perform alternative analysis on 20-hour PAV test data and obtain a prediction of 40-hour test results. Work has been performed independently by Hesp and Reinke looking at reducing the film thickness while maintaining a 20-hour PAV aging process with the objective of producing results comparable to the 40-hour PAV procedure using the standard 50 g sample. It is too soon to predict whether those efforts will be successful, but is obviously in the best interest of the industry to reduce testing time as much as possible while still producing results that help to predict long term field performance. Additional testing time represents real challenges to asphalt binder production.

Based on the information presented, it is recommended that the binder parameter ΔT_c after 40 hours of PAV be explored further as an improved method for characterizing long-term (5+ years) binder durability and performance. However, ΔT_c after 40 hours of PAV is not recommended as a purchase specification at this time due in part to the extended length of time required to age binders and the need for validating research.

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.

Bennert, T. (2015). *www.asphaltetgs.org*. Retrieved from Asphalt Binder ETG, April 9-10, 2015.

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).

G. Reinke, e. a. (2015). *www.asphaltetgs.org*. Retrieved from Asphalt Binder ETG April 9-10, 2015.

Gibson, N. H. (2015). *www.asphaltetgs.org*. Retrieved from Asphalt Binder ETG, April 9-10, Fall River, MA.

Glover, C. J. (2005). *Development of a New Method for Assessing Asphalt Binder Durability with Field Validation*. FHWA/TX-05/1872-2.

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. (. (2006). *Development of an Improved Asphalt Binder Specification Testing Approach HIIFP-034*. Ontario Ministry of Transportation Provincial Highways Mangement Division Report.

Hesp, S. (2006). *Development of an Improved Asphalt Binder Specification Testing Approach HIIFP-034*. Ontario Ministry of Transportation Provincial Highways Mangement Division Report.

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

Kandhal, P. (1977). Kandahl, Low Temperature Ductility in Relation to Pavement Performance, ASTM STP 628, Marek, Ed., 1977. *Low Temperature Properties of Bituminous Materials and Compacted Bituminmouis Paving Mixtures, ASTM STP 628*. ASTM.

Presenters, V. (2015, September 16). *www.asphaltetgs.org*. Retrieved Sept 16, 2015, from www.asphaltetgs.org: www.asphaltetgs.org

Reinke, G., Anderson, R.M., Hanz, A., Impact of re-refined engine oil bottoms on binder properties and mix performance on two pavements in Minnesota, 6th Eurasphalt & Eurobitume Congress, 1-3 June 2016, Prague, Czech Republic, DOI: dx.doi.org/10.14311/EE.2016.284

Rowe, G. (2011). Prepared Discussion for Anderson, et al AAPT 2011 paper "Evaluation of the Relationship between Asphalt Binder Properties and Non-Load Related Cracking". *Proceedings of Association of Asphalt Paving Technologists, (pp. 649-663).*

Wright, L. K. (2011). Oxidative Aging of Asphalt Cements from an Ontario Pavement. *International Journal of Pavment Research and Technology, Vol. 4 No. 5* , 259-267.

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

VI. FREQUENTLY ASKED QUESTIONS (FAQ) ABOUT REOB/VTAE

This chapter is a result of numerous industry forums; including regional user/producer groups, AASHTO Subcommittee on Materials, and FHWA Asphalt Expert Task Groups; where the REOB/VTAE topic was highlighted and discussed. These are actual questions posed by suppliers, users, owner agencies, and academia resulting from an increased highway industry awareness of REOB/VTAE products and the scarcity of documented product usage guidance. This condensed question and answer format is based on the information provided in each detailed chapter associated with REOB/VTAE production, literature search, health, safety and environment, and material conditioning and testing. The reader is encouraged to review the specific chapters and referenced literature for more detailed information and gain further insight into the background and rationale for each response.

1. Are there used oil (not re-refined) products that are being used in asphalt?

Response: Unrefined used oil is not recommended for blending with asphalt products because of health, safety, environment, quality, and performance concerns of their use in heated asphalt binders. Many of these oils can contain free water causing foaming, solubility issues, flash point issues, or other hazardous conditions. When these products are used for asphalt mixture production they can cause blue smoke, opacity, and emission issues. In asphalt emulsions applications there are additional concerns about unrefined used oil products' effect on the emulsion stability and are not recommended. Used oil generally has a Mutagenicity Index (MI) greater than 1.0, which is an indication of carcinogenic potential. Refer to the Safety Data Sheets for further information from the supplier.

2. Are used oil processing methods used to manufacture products intended as asphalt additives/ modifiers?

Response: This document is only intended to address REOB/VTAE products which are re-refined using vacuum tower distillation. Oil re-refiners use various processes to produce base lube products using vacuum tower distillation. There are additional ways to process used oils which do not use vacuum tower distillation; they only use filtration, centrifugation, or atmospheric distillation. These simplified processes are generally intended for fuel or vacuum gas oil uses and not intended for use in pavement asphalts. Misuse of these products can potentially create a hazardous situation, may have detrimental impacts to health, safety, and environment, and should be avoided.

3. Is REOB/VTAE a single substance; are there different re-refining processes that might lead to it being physical or chemically different?

Response: Again, this document is only intended to address REOB/VTAE products which are re-refined using vacuum tower distillation. When blended into asphalt in more than trace amounts (less than 1%), products not derived from vacuum tower distillation may have a detrimental effect on the asphalt binder's physical and chemical properties. Like asphalt, REOB/VTAE is not a single substance. It is a mixture of thousands of hydrocarbons with a more narrow range of paraffinic molecules than those found in asphalt. REOB/VTAE products can be manufactured by various process conditions and from various used oil sources such as diesel engines, gasoline engines, and non-engine lubricant sources. The chemistry is likely influenced by the manufacturing method and the source of the used oils.

4. Are there asphalt tests or other tests that can differentiate REOB/VTAE products (re-refined using vacuum tower distillation) from those products that are not re-refined using a vacuum tower?

Response: There is not an asphalt test that can that can differentiate re-refined versus non re-refined oil products. Flash point, moisture, solubility, weight loss, viscosity, and ash content may help indicate differences but these tests are not conclusive. Typically, products not re-refined using a vacuum tower have a lower flash point, a higher weight loss, and a lower viscosity than REOB/VTAE. Mutagenicity Index (MI) and Polycyclic Aromatic Compounds (PACs) testing may also help to differentiate between REOB/VTAE and other used oil products. Supplier or producer certification of vacuum tower distillation may be the best way to ensure the material is vacuum tower derived.

5. What CAS Number(s) are used for re-refined oil products?

Response: The CAS number typically encountered to designate REOB/VTAE products is CAS# 129893-17-0. The National Institute of Health (NIH) ToxNet, ChemID description for this CAS number is: *Lubricating oils, used, residues*. REOB/VTAE is only one of many products that may fall under this CAS Number designation. Re-refined vacuum tower distillation products are also typically designated with CAS# 129893-17-0. Other products which are not re-refined using vacuum tower distillation may be designated with this same CAS Number. Non re-refined products are not the focus of this document because those products should not be used to modify asphalt for pavement construction. This CAS number designation does not assure the product is re-refined using vacuum tower distillation processes. Supplier or producer certification may be the best way to ensure the product is re-refined using vacuum tower distillation.

6. How uniform and consistent are REOB/VTAE products across used oil sources, re-refiners, and regions?

Response: The physical and chemical variability of REOB/VTAE products is not fully characterized at this time. This document only contains information about the physical and chemical characterization of four REOB/VTAE sources. REOB/VTAE products can be manufactured by various process conditions and from various used oil sources. The uniformity and consistency of the product is influenced by these factors.

7. Are there standards/specifications that address the consistency of REOB/VTAE?

Response: Draft specifications for REOB/VTAE use in both roofing and asphalt paving have been proposed to the American Society for Testing and Materials (ASTM) by the REOB/VTAE suppliers. The specifications were proposed to measure and control the consistency of REOB/VTAE products by the re-refiner. The specifications are currently under review by their respective ASTM roofing and asphalt pavement specification committees.

8. What are the tests and criteria for the proposed standards/specifications?

Response: The proposed American Society for Testing and Materials (ASTM) specification tests include flash point, weight loss, solubility, and viscosity. Additionally, an ash content criterion is also being investigated to control product usage. These and other test criteria are being developed and vetted by experts within the asphalt pavement industry.

9. The ash test has been suggested as a potential test for controlling and monitoring REOB/VTAE usage. Is the ash test an effective test to control and monitor REOB/VTAE?

Response: The Ash Test does not address the performance of the binder or the mixture. It is a relatively simple procedure that relies on the difference between the residue after the ash test of the particular REOB/VTAE product and the asphalt binder to which it is added. It only addresses the mass of material added and can only be used if the properties of the base materials are known. The ash test will not guarantee performance.

10. How is REOB/VTAE detected and quantified in the final asphalt product?

Response: It is difficult to quantify the amount of REOB/VTAE after asphalt blending without data on the individual components due to compositional variability.

The presence of REOB/VTAE in asphalt binders can be determined using X-Ray Fluorescence (XRF) spectroscopy. The detection of trace metals such as calcium, zinc, copper, and molybdenum have been used to determine the presence of REOB/VTAE. The simultaneous presence of all four metals (calcium, zinc, copper, and molybdenum) indicates REOB/VTAE. The interpretation of the analysis results is not simple because both REOB/

VTAE and asphalt binder composition varies. Refer to ASTM D6481-14 *Standard Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by Energy Dispersive X-ray Fluorescence Spectroscopy and ASTM 6443-14 Standard Test Method for Determination of Calcium, Chlorine, Copper, Magnesium, Phosphorus, Sulfur, and Zinc in Unused Lubricating Oils and Additives by Wavelength Dispersive X-ray Fluorescence Spectrometry (Mathematical Correction Procedure)*

Proton Nuclear Magnetic Resonance (^{1}H NMR) spectroscopy is a method used in the determination and structure of unknown organic compounds. Asphalt binder has few aromatic protons and numerous aliphatic protons. REOB/VTAE contains much less aromatic protons and is mostly saturate alkanes (aliphatics); some of which are very different from those found in asphalt.

Inductively Coupled Plasma (ICP) techniques are powerful tools for detecting and analyzing trace elements. ICP mass spectroscopy (ICP-MS) is used in analytical laboratories to provide accurate and precise measurements and lower limits of detection. The intensity of a specific peak in the mass spectrum is proportional to the amount of the elemental isotope from the original sample. Refer to ASTM D5185-13e1 *Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES).*

Although there are standards associated with conducting these tests, there is currently no published guidance for using these three analytical methods to specifically detect and quantify REOB/VTAE.

11. Describe confounders which affect trace metal detection and quantification methods?

Response: There are a number of ways to modify asphalts including ground tire crumb rubber and polyphosphoric acid (PPA). They contain either zinc or phosphorus which can be detected in the blended asphalt. REOB/VTAE also contains both phosphorous and zinc compounds from oil additives. Phosphorus is not a useful indicator since the levels are too low to be useful.

Scavengers are compounds/molecules that bind to other molecules/compounds. REOB/VTAE products contain compounds also found in scavengers added to asphalt in the refinery to control hydrogen sulfide emissions. Hydrogen sulfide scavengers are used in asphalt binders even when there has been no modification with REOB/VTAE, rubber, or PPA. The presence of these hydrogen sulfide scavengers can complicate the analysis.

Rubber contains zinc as do the hydrogen sulfide scavengers. The levels of zinc in rubber are higher than those typically found in binders containing REOB/VTAE. The levels of zinc from hydrogen sulfide scavengers are much lower. High zinc levels indicate rubber is present, low levels of zinc, particularly if calcium, molybdenum, and copper are absent would suggest a hydrogen sulfide scavenger is present. Lubricating oil additives which contain zinc dialkyl dithiophosphate and molybdenum sulphide are not typically found in asphalt.

Additional confounding factors can result during pavement forensic investigations when analyzing asphalt from pavement cores. Investigations on the detection of REOB/VTAE in extracted and recovered asphalt binders are in their early stages. Pavement cores may contain compounds from outside sources and other compounds from leaking automobiles or other sources. Careful investigation is required to understand which product(s) and source(s) are providing these compounds.

12. Are there any known interactions between common asphalt additives such as amine based liquid antistrips, polyphosphoric acid (PPA), polymers, emulsifiers, or others?

Response: There may be interactions between REOB/VTAE and other additives such as PPA, styrene-butadiene-styrene (SBS), styrene-butadiene rubber (SBR), ground tire crumb rubber, liquid antistrips, and emulsifiers. When used in combination, the final rheological and performance properties must be assessed to determine their combined impacts and compatibility. REOB/VTAE blended asphalt can cause stability problems with certain emulsifiers and are not recommended for these applications.

13. Are there interaction and compatibility effects between the base asphalt and REOB/VTAE that impact performance?

Response: It is important to understand the base binder asphalt source properties and REOB/VTAE properties, both separately and in combination, to understand their impact on binder and mixture performance. Asphalts with similar performance grades from different crude sources are impacted differently by a given amount and source of REOB/VTAE. There are several studies underway that are looking at binder and mixture cracking and aging characteristics of REOB/VTAE blended asphalts. A number of researchers are investigating extended pressure aging vessel (PAV) conditioning durations to observe the REOB/VTAE impact on binder properties and determine its susceptibility to accelerated aging. This document describes additional aging and rheological testing that may address performance effects.

14. What are the aging characteristics of REOB/VTAE modified asphalt binders in comparison to non-REOB/VTAE modified asphalt binders?

Response: From a binder formulation and life cycle cost standpoint it is valuable for specifiers, suppliers and purchasers to understand long term binder aging characteristics and its impact on pavement performance. The importance is highlighted within this document to understand the relationship between laboratory oven conditioning to in-place pavement field aging; and also recognize the impact modifiers have on asphalt binder aging rates that influence this relationship and ultimately affect pavement performance. For binders which are particularly susceptible to increased rates of aging, the BBR creep rate (m-value) critical temperature [$T_{c(m\text{-}value)}$] increases more rapidly than the BBR creep stiffness (S) critical temperature [$T_{c(S)}$] and therefore the ΔT_c [difference between $T_{c(S)}$ and $T_{c(m\text{-}value)}$] is a reliable indicator of when binders are losing the ability to relax stresses and therefore losing the ability to resist crack formation.

There are several studies underway that are looking at the aging characteristics of REOB/VTAE modified binders. The magnitude of aging has a significant impact on the performance of REOB/VTAE modified asphalt binders and mixtures. Asphalts with similar performance grades from different crude sources are impacted differently by a given amount and source of REOB/VTAE. When compared to neat asphalt binders of equivalent PG grade, asphalt binders modified with REOB/VTAE typically exhibit higher rates of aging, with small changes observed after RTFO aging and larger changes observed after 40 hours of PAV aging. Higher percentages of REOB/VTAE have a larger impact on aging. As the REOB/VTAE dosage rate increases, the blended binders age faster than lower dosage and neat binders.

Current test data correlated to field performance is based on long term aged (five or more years) field mixtures. The binder recovered from aged field mixtures was better correlated to 40-hour PAV residue properties. The rate of decrease in the value of ΔT_c as the binders are aged from 20 to 40 hours is also informative. A higher rate of decreasing ΔT_c values between 20 to 40 hours PAV aging indicates potentially problematic materials as the binder loses its ability to resist crack formation.

15. Will a *mixture* using REOB/VTAE blended asphalts "age" the same, faster, or slower than a typical *mixture* using non REOB/VTAE blended asphalt?

Response: Mixture aging is influenced by many factors including production, construction, location, climate, volumetric properties, binder film thickness, permeability, asphalt binder properties and aggregate properties. Although initial REOB/VTAE studies focused on the aging characteristics of binders, there are several studies underway looking at mixture aging and performance. Similar to the asphalt binder studies, the magnitude of mixture aging has a significant impact on its laboratory performance. Laboratory conditioning of mixtures to simulate field aging of at least five years in service is an important variable when assessing performance. Aging during a pavements service life is more difficult to simulate by laboratory tests because it takes place at lower temperatures and a much longer time duration. Those considering the use of REOB/VTAE in their binders and mixtures should investigate their materials utilizing one or more of the testing procedures referenced in this document and incorporate additional long term laboratory conditioning protocols.

16. Is the REOB/VTAE product the same quality and consistency as traditional asphalt binders?

Response: REOB/VTAE is compositionally different from asphalt. Asphalt is a complex mixture containing hundreds of individual chemical species comprised primarily of carbon, hydrogen, nitrogen, oxygen and sulfur. Asphalt does not have a specific chemical formula; individual molecules vary and the composition is highly dependent on the crude oil source.

The components of asphalt are typically classified according to solubility into four classes of compounds by the well-known SARA analysis:

1. Saturates (S) – saturated alkanes or aliphatic hydrocarbons,
2. Aromatics (A) – partially hydrogenated polycyclic aromatic compounds or naphthene aromatics,
3. Resins (R) – polar aromatics consisting of functionalized aromatic compounds such as high molecular weight phenols and carboxylic acids, and
4. Asphaltenes (A) – high molecular weight heterocyclic compounds insoluble in n-heptane and soluble in toluene.

Asphalt is a blend of aliphatic and aromatic molecules of varying polarity. The majority of asphalt components are typically naphthene aromatics and polar aromatics; followed by asphaltenes and saturates. Asphalt contains few aromatic protons and numerous aliphatic protons.

REOB/VTAE is also a complex mixture of hydrocarbons primarily composed of aliphatic molecules with very little aromatic character; and is lower in polarity than asphalt. REOB/VTAE contains less aromatic protons than asphalt and is mostly saturated alkanes (aliphatics); some of which are very different from those found in asphalt. Unlike asphalt, although REOB/VTAE may contain components that are insoluble in n-heptane or other aliphatic solvents, these insoluble components are not identifiable as asphaltenes.

When REOB/VTAE is blended with asphalt, it shifts the ratio of aliphatic to aromatic compounds found in the resulting blended product. These ratio differences change the rheological characteristics to “soften” the asphalt. These changes are displayed in lower Bending Beam Rheometer (BBR) flexural creep stiffness (S) test results in relation to the BBR creep rate (m-value). This alters the relationship between the (S) critical temperature [$T_{c(S)}$] grade and the (m-value) critical temperature [$T_{c(m\text{-}value)}$] grade and the difference between the two (ΔT_c) becomes greater as aging increases. The decrease in ΔT_c is correlated with an associated decrease in the binder’s colloidal index (CI) which is the ratio of aromatics plus resins to asphaltenes plus saturates. As a binder ages, its CI decreases primarily due to a loss of aromatics and an increase in asphaltenes. This demonstrates the impact that REOB/VTAE, an aromatic deficient and saturate rich material, can have on the asphalt composition; ultimately resulting in decreases in both colloidal index and ΔT_c as the binder is aged.

High performance liquid chromatography (HPLC) based saturate, aromatic, resin, asphaltene determinator (SAR-AD) analysis splits asphalts and oils into distinct chromatographic and solubility subfractions, and separates the saturates, aromatics, resins, and asphaltenes. Unlike asphalt, REOB/VTAE analysis reveals mostly paraffinic saturates (maltenes) and resins; but very low naphthene saturates and virtually no asphaltenes. Investigations into these compositional differences, their compatibility, and effects on performance are unknown at this time.

17. Are there asphalt binders that will not benefit from the addition of REOB/VTAE?

Response: Benefits are dependent on both the asphalt binder source and the REOB/VTAE source. Bending beam rheometer (BBR) creep rate (m-value) controlled asphalt binders are more susceptible to the addition of REOB/VTAE modification. REOB/VTAE products are also highly m-value controlled and tend to increase a binder’s level of m-value control further with increasing amounts of REOB/VTAE. This characteristic, results in a larger difference between the BBR creep stiffness (S) and BBR creep rate (m-value) critical temperatures (ΔT_c); especially at longer conditioning times, such as a 40 hour pressure aging vessel (PAV) conditioning time used to simulate pavement field aging. Pavements built with binder ΔT_c values approaching -5°C and colder have shown to exhibit significant pavement cracking distress in the field.

The rate and extent at which a binder becomes m-value controlled is a function of the binder's chemistry. All binders undergo a trend towards being m-value controlled as they age. Aged binder sources from reclaimed asphalt pavement (RAP) and reclaimed asphalt shingles (RAS) can also provide highly m-value controlled binders impacted by REOB/VTAE modification. An excessive degree of m-value control is not exhibited by all binders when blended with a given amount of REOB/VTAE. The m-value control and ΔT_c progress with aging should be investigated to observe the REOB/VTAE impact on binder properties and determine its susceptibility to accelerated aging.

18. Since REOB/VTAE is derived from lubricants, what are its effects on the adhesive and cohesive properties of asphalt binders?

Response: Limited studies using surrogate tests for evaluating adhesion and cohesion have been conducted using Hamburg wheel track and tensile strength ratio testing. These studies, referenced in Chapter 3, have not found evidence of adhesion and cohesion issues in the laboratory.

19. Are there guidelines for the use of REOB/VTAE residue, such as a max limit either by weight of binder or by the drop of PG grade?

Response: This document is intended to provide the state of the knowledge on the usage of REOB/VTAE to modify asphalt binders. The REOB/VTAE source, dosage, compatibility with the base asphalt binder, and the base asphalt binder properties influences performance. It is the authors' opinion that the current body of research is inconclusive in regards to recommending a limit. Some state agencies are considering weight (percentage) limits. Some REOB/VTAE producers are also recommending an acceptable usage level based on their experience. REOB/VTAE can also be limited by considering a ΔT_c (difference between BBR critical temperature for creep stiffness (S) and creep rate (m value). Those considering the use of REOB/VTAE in their binders and mixtures should investigate their materials utilizing one or more of the testing procedures referenced in this document and incorporating additional long term laboratory conditioning protocols.

20. Is there a practical limit for the percentage of REOB/VTAE that can be blended with asphalt binders to ensure long term pavement performance?

Response: The REOB/VTAE source, dosage, compatibility with the base asphalt binder, and the base asphalt binder properties influences performance. Due to these variables, a practical limit may be established based on specific materials sources available and supplied to a specific region. Those considering the use of REOB/VTAE in their binders and mixtures should investigate their materials utilizing one or more of the testing procedures referenced in this document and incorporate additional long term laboratory conditioning protocols for their specific materials to establish practical limits.

21. What performance tests can be used to determine the appropriate REOB/VTAE percentage limit to ensure long term pavement performance?

Response: There is not a single test to ensure long term pavement performance. Additional asphalt binder performance properties, such as ΔT_c with extended PAV conditioning, are being considered to properly characterize the long term performance of REOB/VTAE and other materials used to modify binders. Additional mixture evaluation protocols are also being developed to evaluate pavement performance. Those considering the use of REOB/VTAE in their binders and mixtures should investigate their materials utilizing one or more of the testing procedures referenced in this document to assess binder performance; and incorporate additional long term laboratory conditioning protocols for both binders and mixture evaluations.

APPENDIX A – Supplemental Information On Fume Generation and Collection Methods Reported In Section IV

Asphalt fumes were generated following a standard protocol used by HRG since 2006 [1, 2]. After the blended REOB/VTAE/bitumen was pre-heated in an off-line oven to 150 °C for 60 minutes, 200 ± 1 grams of the heated bitumen were transferred to a vessel within the generation system, re-equilibrated to that temperature, and stirred at a controlled rate (125+10 rev/min). This temperature was selected because it represents the typical hot-mix asphalt paving application temperature used today. Emissions released were captured on a series of three sorbent tubes (FIGURE 2) using a sampling train linking two 600 mg XAD-2 sorbent tubes (Cat. No. SKC 226-30-06 SKC, Inc.) and a XAD-2/charcoal sorbent tube (150 mg XAD-2 followed by 50 mg activated charcoal; Custom Order Cat. No. CPM032509-001; SKC, Inc.). Using this collection system, fumes emitted from the generator were drawn through the sampling train using a Thomas Vacuum Pump MN 107 CAB18-310C sampling pump calibrated to a flow rate of 2.0 liters per minute (lpm). The fume generation was performed at 150°C with a stir rate of 100 rpm. Emissions were collected for 15 minutes. To recover the emissions from the XAD-2/charcoal sorbent tubes, dichloromethane was used as the elution solvent. The final elution volume was 10 mL for each individual sorbent tube.

- **Eurobitume (European Bitumen Association)**, Heritage Research Group, Kriech AJ. Comparison of straight run vacuum distilled bitumen and semi-blown bitumen. *Eurobitume (European Bitumen Association)*, 2006 Report.

- **Eurobitume (European Bitumen Association)**, Heritage Research Group, Kriech AJ. Bitumen Fume Composition as A Function of Binder Temperature - Physical and Chemical Analysis of Binder Fumes At Various Relevant Temperatures Using Two Different Laboratory Fume Generation Techniques. *Eurobitume (European Bitumen Association)*, 2006 Report.

APPENDIX B – REOB/VTAE Literature Review Summary

Use of Re-refined Oil Distillation Bottoms As Extenders For Roading Bitumens
P.R. Herrington
Journal of Materials Science – Vol. 27 No. 24 – December 1992 – pg. 6615-6626

Vacuum distillation bottoms from the re-refining of waste automotive oils (WODB) can be successfully blended with standard petroleum bitumen to produce a stable binder with acceptable physical properties for roading applications. The increase in viscosity and shear susceptibility after accelerated aging of thin films at 163°C is slightly less for bitumen extended with WODB than for bitumen itself; but at temperatures above ~200° the WODB appears to accelerate the rate of bitumen oxidation. Air blowing at 240°C can be used to produce an acceptable binder from blends of WODB and a softer grade of bitumen. The rate of hardening during air blowing depends on both the batch and concentration of WODB.

- The purpose of this study was to investigate the potential use of vacuum distillation bottoms produced during the re-refining of waste lubricating oils, as extenders for roading bitumens.
- This work was a preliminary study to investigate more fully the simple rheological properties of waste oil distillation bottoms (WODB) and both simple and air-blown blends with bitumen.
- Results are compared with those for a standard unmodified roading bitumen.
- The oxidative properties of the extended bitumen were also studied.
- The WODB used were obtained from vacuum distillation of waste automobile lubricating oils at 370°C at 3-6 mbar.
- The standard bitumen was 80/100 penetration grade, from a Safaniya Arabian crude. The 80/100 grade is produced by blending back a straight-run 180/200 and air-blown 45/55 grades.
- To evaluate the consistency of the various WODB materials used, measurements were made on a number of batches collected over a period of 13 months – it was clear there was considerable variation in viscosity from batch to batch, which reflects variations in vacuum efficiency and feedstock.
- Flash points of a number of WODB batches were found to be greater than 320°C.
- Experiments were carried out with blends stored at 135°C for three days showing no apparent problems with compatibility.
- Accelerated oxidative hardening properties indicate that the blends are hardening at a similar but somewhat slower rate to the base bitumen.
- Test results suggest that at 163°C the WODB may actually slightly improve the oxidative stability of the base bitumen; at the very least the WODB have no deleterious effect. However, at higher temperatures there is some evidence to the contrary.
- Results of the air-blowing experiments show that all of the WODB blends harden at a faster rate than the 180/200 bitumen. The rate varies with WODB batch and increases with concentration of WODB.
- To compare the blown products with the standard 80/100 bitumen, penetration curves were interpolated to determine the time required to reach a penetration of 90.
- It is evident that the different rates of oxidative hardening amongst the blends means that the blowing does not remove the initial variation in their consistency.
- This study suggests that although the effects of WODB on the physical properties of roading bitumen are complex, they are not so great as to preclude the use of viable practical concentrations (≈10%) as bitumen extenders
- The variation in bitumen-WODB blend properties with WODB batch may pose difficulties meeting specifications.
- The oxidative hardening properties of the blends are similar to one another and to those of a standard bitumen at least up to 200°C – at higher temperatures, the blends oxidize at a higher rate likely due to the high levels of wear metals present in the WODB.

Comments:

- *Many different batches and sources of WODB were used*
- *Dosage rates as high as 50% - authors suggest 10% is reasonable*

Waste Oil Distillation Bottoms As Bitumen Extenders
P.R. Herrington, V.K. Dravitzki, C.W.B. Wood, and J.E. Patrick
Road & Transport Research – Vol. 2 No. 4 – December 1993 – pg. 56-68

This paper discusses a field trial in which binders produced from bitumen and waste oil distillation bottoms (WODB) were used to manufacture asphalt concrete. Two blended binders were manufactured by air blowing a 180/200 penetration grade bitumen plus 9 and 20% WODB, to an 80/100 and compared to a conventional 80/100 pen bitumen.

- The air blown/WODB blended binders were less temperature sensitive than the conventional 80/100 bitumen at low temperatures
- The air blown/WODB blended binders exhibited similar temperature susceptibility compared to the conventional 80/100 bitumen at temperatures of 70 -135°C.
- After RTFO, the blended binder residues were similar to that of conventional bitumen except for a lower ductility
- Measurements made on a number of batches of WODB produced over 13 months showed considerable variation in physical properties – this reflects variations in plant vacuum efficiency and feedstock oil
- The air blowing process did not eliminate problems associated with variability in the WODB – the rate of oxidative aging of the blends differed considerably as a result of the variability of initial viscosities of different batches of WODB
- Contaminant levels found in WODB will vary depending on feedstock oil but the level of polyaromatic hydrocarbons are low compared to conventional binder
- The Marshall properties of the mixtures incorporating the blended binders were found to be similar to those using the conventional binder
- The use of WODB in road construction is environmentally more acceptable than dumping in a landfill
- The laying of asphalt using bitumen/WODB blends is perfectly feasible and requires no specialized techniques or equipment
- Field performance after 14 months in service was similar between the conventional bitumen sections when compared to the sections containing bitumen/WODB blends
- The leaching potential of the metals found in WODB was studied by means of accelerated photo-oxidation and found to be negligible in many cases below the detection limit
- The 2000 hours of exposure of accelerated aging procedures used in this study correlates to approximately 3 years of natural exposure in New Zealand

Comments:
- *Long term pavement performance comparisons were not included*

Recycling of Waste Oil Distillation Bottoms in Asphalt Institute
P. R. Herrington and P.G. Hamilton
Transfund New Zealand Research Report No. 102, 1998

This paper discusses and documents a field trial of hot mix asphalt using waste oil distillation bottoms (WODB) produced during the re-refining of waste lubricating oils as an asphalt extender. Three sections of hot mix asphalt pavement were laid at a site on a roundabout in Hamilton, New Zealand, in May 1991. The trial consisted of hot mix asphalt manufactured using an 80/100 PEN grade asphalt on the control section, and using blends of 180/200 PEN grade asphalt with 9% and 20% of WODB respectively, air-blown to a nominal 80/100 PEN grade on the other two sections. A full laboratory study had already been done these binders by Herrington et al. [2].

The physical properties of the mixtures were monitored at 14 months and after 57 months. The WODB-blended mixtures did not show greater levels of surface deterioration, cracking, or rutting compared to the control section.

The WODB were known to contain lead (from additives) and metals (from engine wear). After 57 months, analysis of lead, chromium, and zinc contents of the hot mix asphalt in the trial sections showed that up to 25% of the zinc had been lost, while lead and chromium levels were similar to those initially present in the asphalt.

- Conventional techniques and equipment were used during production and placement of the three mixtures. The mixtures were produced at 135°C.
- 31 tonnes of control mix, 34 tonnes of mixture containing 80/100 asphalt - 9% WODB, and 85 tonnes of mixture containing 80/100 asphalt – 20% WODB were placed at 25mm thick
- No differences were observed with the exception that the 20% WODB blend was considered “lively” during compaction

- The WODB blended binders have a slightly lower temperature susceptibility than the control 80/100 binder in the low temperature region but similar susceptibility in the 70° - 135°C region.
- Measurements were made on the rolling thin film oven (RTFO) residues from each of the three trial binders – when compared to the control section binder, the WODB blended binders had higher retained penetration values but significantly lower ductilities. The ratio of before and after viscosities show that the relative increase in viscosity of the blended binders is apparently less than that of the 80/100 control. Due to errors inherent in the measurement, it is perhaps safer to say that the increases in blend viscosity are, at least, not greater than the increase in viscosity of the control.
- However, findings by Herrington et al. 1997 on non-volatile fluxes for chip sealing are consistent with lower hardening rates observed in this trial with WODB blends. In that project, 3mm films of simple blends of 10% WODB and 180/200 asphalt showed significantly reduced hardening rates compared to control samples after 3.3 years of oven aging at 43°C.
- The low ductility measures contradict the viscosity and retained penetration results. Ductilities can be affected by the presence of particulate matter which caused premature breaking of the asphalt thread. Particulate matter is found in WODB.
- Other evidence casting a doubt on the validity of the ductility results comes from a relationship established by Heukelom (1966) who found ductility to be inversely proportional to stiffness modulus. Using penetration index and van der Poel's nomograph, ductilities are estimated 0.6 m and 0.9 m for 9% WODB blend and 20% WODB blend respectively – much higher that the measured values.
- Marshall tests and sieve analysis were run on the three mixtures during the time of construction and all three were comparable – the only difference being a trend to slightly higher stabilities for the mixtures with the blended binders.
- A walkover inspection of the site in January 1996 showed the asphalt in all three sections to be in generally good condition with the control section to be slightly lighter in color
- All surfaces were smooth in appearance but showed a constant matrix of aggregate
- Rutting depths had not increased since last measured in 1994 with average rut depths of 2.3, 1.75, and 0.8 mm for the control, 9% WODB, and 20% WODB blends respectively
- Cores were taken from each of the sections with the binder extracted and recovered
- The viscosity data from the recovered binders after 57 months in service indicates that the WODB-modified binders are hardening significantly faster than the control 80/100 binder. However, the rates of hardening are inconsistent with results measured at 14 months. This inconsistency may be due to the relatively small change in viscosity which had taken place in that period.
- The 57-month results are also inconsistent with the RTFO measurements made on the initial binders and as well, they are not supported by the results of the non-volatile flux for chip sealing project (Herrington et al. 1997)
- An explanation for these apparent contradictions may lie in the differences in the air voids which is apparent between the three sites – higher levels of air voids allow greater ingress of oxygen and hence faster oxidation rates. The 9% site had the highest percentage of air voids while the 20% section, while having an average similar to the control, had a much greater range in air voids.
- WODB contains various levels of lead, chromium, zinc, and other metals arising from engine wear, and from fuel and oil additives.
- Of particular concern in early work were the high levels of lead
- The possibility of lead and other metals being leached from asphalt by rainwater was investigated in the laboratory (Herrington et al. [2]) – WODB blends were prepared as thin films and photo-oxidized under UV lamps. Periodic washing and gentle brushing of the surface showed that no significant leaching occurred.
- High levels of lead are no longer of concern since the removal of leaded gasoline from the market
- Potential leaching of metals was investigated from the trials sections by comparing the metals contents of the initial retained binder samples with those recovered after 57 months
- In all cases except one the metals content measured from the cores was not significantly different from the initial binder samples. A reduction in zinc for both of the WODB sections was determined to be around 25%
- This result contradicts previous work – a possible explanation is that the rate of the leaching was too slow to be observed in the laboratory study. Alternatively, other factors not allowed for in the laboratory study may be influencing the rate of zinc loss from the field trial. The leaching process from the field trial has been assumed to be principally mechanical abrasion of the binder surface.

Comments:

- *Would the leaching effect of zinc increase with increased in-place air voids?*
- *What impact does aggregate properties have on the leaching?*
- *Is leaching of zinc of environmental concern?*
- *Why does zinc leave and not chromium or lead? What about other metals?*

Asphalt Modification With Used Lubricating Oil
Aaron Villanueva, Susanna Ho, and Ludo Zanzotto
Can. J. Civ. Eng. Vol. 35, 2008

This paper looks at the viability of used lubricating oil as an asphalt modifier, with the enhancement of the low- temperature grade as the specific goal. Used oil modification was found to improve the Superpave low-temperature performance grade (PG), but at the expense of the high-temperature PG grade. When evaluated according to the Superpave MP1 specification, the low-temperature grade of the modified asphalt was not significantly improved due to failure of the bending beam rheometer (BBR) test's m value. When evaluated according to the Superpave MP1a specification, the modified asphalt overall PG grade temperature spread remained essentially constant, varying only by approximately two degrees. The asphalt took as much as 12% of oil and still had less than the maximum limit of 1.0% rolling thin film oven test (RTFOT) mass loss (emissions). However, the oil may possibly have a detrimental effect on the asphalt quality, such as reduced adhesiveness to the aggregates, leading to stripping and raveling. Field performance should be checked before considering lubricating oil as a modifier.

- Two different base asphalt binders were prepared by air blowing a 300/400 Husky asphalt at 240 °C for 3 and 3.5 h. The two samples were then mixed with different percentages of used lubricating oil in a low shear mixer at 500 rpm for 1 h at 160 °C.
- The high- temperature results of the samples were obtained from the dynamic shear rheometer (DSR) test, whereas the low temperature grades were determined from two separate tests, the bending beam rheometer (BBR) and the direct tension test (DTT).
- The DTT along with the BBR results were used to determine $T_{critical}$.
- The samples became softer as more used oil was added, as the softening point decreased and penetration at 25 °C increased with increasing oil level.
- The high-temperature grade also decreased as more used oil was added.
- Using the MP1 (AASHTO 2004b) test specification, there was a decrease in the low-temperature grade with an increasing level of used lubricating oil in the modified asphalt. The high–low temperature spread also decreased with increasing oil levels.
- It can be observed that the MP1a low-temperature grade ($T_{critical}$) was lower than the resulting low-temperature grade determined using the MP1 (BBR) test. The grade difference between the two tests increased with rising oil levels. This improvement in the MP1a low-temperature grade, in turn, improved the overall temperature spread of the samples, in comparison with the results from the MP1 test specification.
- In this study of oil-modified air- blown asphalts, the MP1a low-temperature grades were decidedly lower than the MP1 low-temperature grades, whereas the DTT test results (i.e., DTT secant modulus, failure energy) and the BBR thermal stress curve all pointed to low-temperature improvements.
- PG64 binders in this study were compared against an air-blown PG64 and a straight-run PG64 asphalt binder. Comparing the two PG64 binders in this study with the straight-run sample, it is quite clear that the low-temperature grade for the used oil-modified asphalt is better, enhancing the overall temperature spread by 6 °C.
- The RTFOT mass loss for the two sets of samples in this study were all below the 0.90% mark, which is well below the accepted maximum mass loss of 1.0% - the oil percentage in the sample that would go over the 1% loss was estimated to be 19% in the 3-hour oxidized base asphalt and 12% in the 3.5-hour oxidized base asphalt.
- The Superpave MP1 test specification using the BBR was found to underestimate the low-temperature improvements with the addition of used lubricating oil to the air-blown asphalts.
- The Superpave MP1a test specification, combining the BBR test results with DTT results, was found to be a better determinant of the low-temperature grade.
- The oil- modified samples, however, lowered the high-temperature grade and did not improve the overall quality of the asphalt.
- At the same PG64 grade, oil-modified air-blown asphalts had a better high–low overall temperature spread than straight-run asphalt and a slightly better overall temperature spread than air-blown asphalt without oil modification.

Comments:

- *Used lubricating oil – not REOB/VTAE – was used in this study*
- *No extended aging beyond the standard 20 hour PAV aging was considered*
- *The author's suggest that the use of this oil may possibly have a detrimental effect on the asphalt quality such as reduced adhesiveness to the aggregates which could lead to stripping and raveling.*

Five Year Performance Review of a Northern Ontario Pavement Trial: Validation of Ontario's Double-Edge-Notched Tension Test (DENT) and Extended Bending Beam Rheometer (BBR) Test Methods

S.A.M Hesp, S.N. Genin, D. Scafe, H.F. Shurvell and S. Subramani - Department of Chemistry – Queen's University, Kingston, Ontario

CTAA - 2009

This paper discusses the physical and chemical differences that may be responsible for the variation in cold temperature performance of some pavement test sections placed on Hwy 655 north of Timmins in Ontario, Canada. Test results on laboratory residues and recovered asphalt binders using two proposed test methods – Double-Edge-Notched-Tension test (DENT) and the extended Bending Beam Rheometer (extended BBR) – may explain why two sections have performed according to expectation while the others have failed. Chemical analysis suggests that the likely presence of waste engine oil residues, air-blown residues, and/or acids in some of the asphalt binders may have contributed to the excess cracking.

- Seven pavement sections were placed on Highway 655 in 2003 with seven different asphalt binders – all meeting the current M 320 low temperature requirement of -34°C
- In early 2004 air temperatures reached a record low of -48°C – the pavement surface temperature on this project reached -34°C on two different occasions
- At the time of this report, sections 1 and 5 had performed as expected – sections 2, 3, 6, and 7 were cracked significantly more than desired – section 4 was cracked excessively
- The authors feel that there is a need for improvement in the Superpave protocols to better predict cracking performance through the use of the DENT and extended BBR methods
- Discussion of both physical and chemical analysis results from recovered asphalt binders is used to confirm the validity of the proposed DENT and extended BBR test methods
- As it has proven challenging to convince others of the merit of both methods, a chemical explanation of the findings was sought to provide an added validation of this new asphalt cement grading approach.
- X-ray fluorescence spectroscopy provides a qualitative analysis of the presence of heavy elements from sulfur (S), calcium (Ca), vanadium (V), iron (Fe), nickel (Ni), zinc (Zn), and a host of other metals.
- Zinc may be a fingerprint element for waste engine oil residues, since it is typically present in large quantities after the refining of waste engine oil and never occurs in either straight asphalt cement or the aggregate. However zinc-carbonate or zinc-sulphate is sometimes used to scavenge hydrogen sulfide in asphalt binders
- The authors refer to waste engine oil residues, low-cost waste engine oil flux, and waste engine oil as an asphalt binder modifier
- NMR and XRF data reveal that the binders used in section 4, 6, and 7 tested positive for zinc and phosphorous, suggesting the presence of waste engine oil residue

Comments:

- *This section of Hwy 655 is located at 48.5585N and 81.2782W. Using LTPPBind v3.1 with 98% reliability and rut depth specified as 12.5 mm, the recommended grade is PG 52-40. Using a PG xx-34 is based on common Ontario practice in 2003.*
- *It is unclear if REOB is used in these sections or waste engine oil*

Asphalt Cement Loss Tangent as Surrogate Performance Indicator for Control of Thermal Cracking

Abdolrasoul Soleimani, Shanan Walsh, and Simon A. M. Hesp

Transportation Research Record: Journal of the Transportation Research Board, No. 2126; 2009 pp. 39 – 46

This paper discusses the field validation of a simple performance indicator for specification grading of asphalt binders for thermal cracking. Recovered asphalt binders from 20 contract sites varying from 8 to 20 years in age in Ontario, Canada were tested in torsion bar geometry to determine their viscoelastic properties. Tan(δ), as defined by the ratio of viscous over elastic modulus, G"/G', was able to distinguish good from poor performers from these sites with 95% accuracy which is an improvement over current bending beam rheometer protocol. In addition to the use of tan(δ), it is suggested that a measure of critical strain tolerance in the ductile state could be included to provide a significantly improved performance grading method.

- Regular BBR testing (AASHTO M320) predicted performance with an accuracy of 55% - 0% of the time able to predict poor performance and 100% of the time predicted good performance; extended BBR testing, an Ontario method LS-308 proposed by the authors, predicted performance with an accuracy of 90% - 100% of the time predicting poor performance and 82% of the time predicting good performance; a limiting value of

Tan(δ)=0.3 was considered at both 0 and 72 hours of conditioning – predicted performance was 75 and 90%, respectively – with the poor performance prediction being 66% and 78%, while good performance predictions were 82% and 100% respectively
- Most of the worst performing contracts were found to contain zinc. The presence of this element suggests that the use of waste engine oils in asphalt production is likely widespread, since zinc is a universal additive in engine oil and is never found in straight asphalt cement
- Using x-ray fluorescence (XRF), samples obtained from sites A, J, K, L, P, and S tested positive for phosphorous and zinc while none of the aggregates tested positive for zinc. This suggests that the binders were likely modified with waste engine oils that are known to contain both phosphorous and zinc (a universal anti-wear additive in engine oil is zinc dialkyldithiophosphate) possibly together with polyphosphoric acid or some other chemical gelling agent to improve the high temperature grade and limit the volatile loss in the RTFO.
- The authors also provide XRF spectra for waste engine oil sludge (the residue that is left after waste engine oils are re-refined)
- Waste oils likely bring about premature hardening through gel formation, volatilization, exudation (sweating of oils), and oxidation, and this will also limit the strain tolerance as reflected in the double edge notched tension (DENT) data. These same asphalts would be penalized on a limit of Tan(δ) since gelling agents effectively reduce the loss tangent.
- It has been reported that asphalt mixtures modified with waste oils may possibly lack interfacial adhesion between binder and aggregate which would appear as reduced strain tolerance in the mixture.
- Using the DENT test, those binders modified by the likely addition of waste lubricating oils or excessive gelling were easily identified by their low strain tolerance in the ductile state
- Regarding implementation, the authors suggest that it is up to the supplier to make sure the asphalt they sell is not high in wax, unstable in a colloidal sense, or made with problematic upgrading technologies such as waste engine oil sludge or polyphosphoric acid

Comments:
- Some of the contracts were originally made with penetration graded asphalt cement. The remainder were likely specified with a PG 52-34 as was common practice in Ontario at the time. Many of the locations tested should have used a PG xx-40 according to LTPPBind v 3.1 using 98% reliability and a specified rut depth of 12.5 mm.

Characteristics of Rejuvenated Bitumen With Used Lubricating Oil As Rejuvenating Agent

Kemas A. Zamhari, Madi Hermadi, Choy Wai Fun

International Conference on Sustainable Infrastructure and Built Environment in Developing Countries – November 2009 – ISBN 978-979-98278-2-1

World-wide motor vehicles require millions of gallons of lubricating oil. The used lubricating oil generates a waste disposal problem because improperly disposed of used oil may contaminate the environment. On the other hand, each year billions of dollars are spent on road maintenance and rehabilitation. Reclaimed asphalt pavement is among the best option for road maintenance in terms of construction sustainability. This technique requires the use of an asphalt rejuvenator to revive the aging bitumen. A study was carried out to assess technical viability of using used lubricating oil as rejuvenator. The study aimed (i) to identify the properties of typically available used lubricating oil and the requirement for standard bitumen rejuvenating agent; (ii) to determine appropriate methods to improve the characteristics of used oil in fulfilling the rejuvenator standard and (iii) to assess the laboratory performance of reclaiming asphalt with used lubricating oil as rejuvenator. Only the first two are discussed in this paper. The bitumen used in this study is penetration grade 80/100. The bitumen was artificially aged so that the characteristics of bitumen before and after aging could be identified and compared. A series of chemical, physical and rheological tests related to standard for rejuvenator were carried out to recognize the characteristics of used oil. The results indicate that, technically, used lubricating oil can be used as rejuvenating agent in reclaiming asphalt pavement. However, for a safe handling during asphalt mixing operation, the flash point of the used oil should be increased.

- The bitumen used in this study was a 80/100 penetration grade – it was put through the RTFO and PAV to simulate long term aging of the binder
- Used lubricating oil was collected from five locations and tested and eventually blended together to be used as a rejuvenator
- The used lubricating oil exhibited a low flash point so the authors heated it at 135°C for three hours prior to blending with the aged bitumen in order to increase its flash point
- The heated used lubricating oil was blended at 5 and 10% by weight with the bitumen

- Used lubricating oil contains some volatile light fraction that not only affects its viscosity but also the safety during transportation, storage and application process due to its low flash point temperature. However, by preheating, the flash point can be increased to an acceptable level as required by technical specification of rejuvenating agent.
- The preheated used lubricating oil fulfils the requirements for rejuvenating agent similar to ASTM RA-2.
- At a right proportion, used lubricating oil may restore the characteristics of an aged bitumen, especially in terms of viscosity and standard penetration value, even so, the change of chemical composition cannot fully recover.
- Although the investigation on the performance based rheology is yet to conclude, the initial finding indicates that used lubricating oil as rejuvenator can produce a rejuvenated bitumen that satisfies the criteria of Performance Grade (PG) Binder that suitable for tropical climate.

Comments:

- *Used lubricating oil was used in this study – not REOB/VTAE*
- *Low flash points are a great concern requiring additional processing of the used lubricating oil*
- *Actual RAP or RAP binder was not used in this study*

X-ray Fluorescence Detection of Waste Engine Oil Residue In Asphalt and Its Effect on Cracking In Service

Simon A. M. Hesp and Herbert F. Shurvell

International Journal of Pavement Engineering, Vol. 11, No. 6, December 2010, pp. 541-553

This paper documents the discovery of waste engine oil residues in pavements across Ontario, Canada. The authors found that recovered asphalts from a large majority of poorly performing contracts test positive for zinc through X-ray fluorescence (XRF) analysis. In contrast, neither the aggregates nor any of the well-performing asphalts showed any signs of the metal. Since zinc dialkyldithiophosphates are universal additives in engine oils, they inferred that the use of waste oil residues in asphalt must be widespread. Further analysis of 2008 quality assurance samples taken for the Ontario Ministry of Transportation substantiated this, with most samples testing positive for zinc. XRF analysis of straight waste oil residues suggests that typical modification levels are in the 5–20% range. The damaging effect of this additive through increased physical and chemical hardening is briefly discussed with reference to previous studies on unexplained, premature and excessive thermal cracking.

- Authors spent a great deal of time discussing the benefits of their proposed test methods LS-299 – Determining asphalt cement's resistance to ductile failure using double-edge-notched tension (DENT) test and LS-308 – Determination of performance grade of physically aged asphalt cement using extended bending beam rheometer (BBR) method.
- Low temperature grade loss after 3-days of conditioning at -10°C showed that good performing sites lost less than 3°C while the poor performers lost more than 3°C. the suggestion that colloidal instability, as reflected in the higher grade losses due to physical hardening, leads to a reduced ability to relax thermal stress, and this in turn leads to higher thermal cracking distress.
- Other testing such as the loss tangent, tan(δ), the viscous creep compliance, Jv(t), and the elastic recovery (ER) in the BBR could all provide better accuracy compared to the regular BBR test (AASHTO M320) and equal or better accuracy to the extended BBR test.
- It has proven rather difficult to convince those with an interest in the current state of affairs of the benefits offered by the new specifications. Hence, our focus has switched to the chemical analysis of asphalt cements used in Ontario.
- While the physical properties of the asphalt cement determine the low-temperature performance, it is of course the chemical composition that affects these physical properties in positive or negative ways. Paraffins play an important role in thermal cracking since this class of compounds is known to promote asphaltene precipitation, in addition to having a tendency to crystallize at low temperatures. Hence, they can contribute to physical hardening and thermal cracking distress.
- This study also investigated the presence of waste engine oil residues in all of the 2008 quality assurance (QA) samples from the Ministry of Transportation contracts in the northeastern and eastern regions of Ontario.
- Additional experiments were done to show how waste engine oil residue affects a superior quality Cold Lake asphalt cement obtained from the Imperial Oil refinery in Edmonton, Alberta. Waste engine oil residues for these experiments were obtained from two local suppliers.
- XRF spectra for recovered and virgin asphalt cements and aggregates were collected using a handheld Innovative X-Ray Technologies (Innov-X) model XT-440L analyzer. The XRF analyzer detects the emitted radiation, and a plot of intensity vs. 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.

- Two different waste engine oil residues from local suppliers were scanned and analyzed in order to compare the XRF results from straight and recovered asphalt cements with samples of known composition.
- Both residues produced similarly strong zinc signals, so the element was selected to gauge the amount of waste engine oil residue in recovered asphalt cements.
- Zinc dialkyldithiophosphate (ZDDTP) is a universal and deliberate antioxidant and anti-wear additive in engine oils, and zinc is therefore expected to be present in these residues in relatively constant amounts. However, this is only an assumption since this study merely obtained XRF spectra for two single samples obtained from two Canadian sources. Furthermore, zinc carbonate or zinc sulphate is sometimes added during the production of polymer-modified asphalt cements to scavenge hydrogen sulfide (H_2S) produced during the sulphur grafting reaction. However, most producers of polymer-modified asphalt use scrubbers to remove H_2S and do not rely on scavengers.
- Twelve of the 15 poorly performing contracts tested positive for the presence of significant quantities of zinc and hence were likely contaminated with waste engine oil residues. An additional 2 of the 15 poorly performing contracts showed traces of zinc and manganese in the asphalt cement, which could have caused premature oxidation and gel formation.
- None of the 11 superior performing contracts showed any sign of zinc.
- All 2008 QA samples were tested for the presence of zinc in order to obtain more quantitative information regarding the amounts of waste engine oil residue likely added to the asphalt supply. All samples from the northeastern region test positive for approximately 11 – 15% waste engine oil residue and 70% of the samples from the eastern region test positive for approximately 4–12%.
- The experimental results for these model systems show that LS-299 is able to distinguish the Cold Lake asphalt cements that were modified with waste engine oil residue from the unmodified materials of similar low-temperature grades.
- Sites A – T found that phosphorous could be detected in most of the poorly performing contracts - his could be a sign for the presence of phosphoric acid (H3PO4) or polyphosphoric acid (PPA), added by the asphalt supplier, or it could have been a remnant of ZDDTP (i.e. ZDDTP from the waste engine oil residue).
- Previous DENT testing on PPA-modified asphalt cements has shown that these can reduce the strain tolerance in the ductile state due to excessive gelling.
- It is striking that the AASHTO M320 grade goes down with increasing amounts of waste engine oil residue, whereas the LS-308 grade appears to first remain constant and go up at high levels of modification.
- XRF spectroscopy is able to detect zinc and other heavy elements in asphalt cements sampled during construction and recovered from the field.
- The presence of zinc and a host of other heavy elements indicates that the use of waste engine oil residues for asphalt cement modification is wide- spread in Ontario.
- Physical hardening and losses in strain tolerance due to the presence of waste engine oil residues are largely to blame for the observed premature and excessive failures in the Ontario road network.
- It is up to the government to exercise its role as guardian of the public interest. Given the findings of this research, an early ban on waste engine oil residues and other detrimental additives in asphalt cement appears justified. In the long term, these problems can only be avoided through the implementation of improved specification test methods and acceptance protocols. Both LS-299 and LS-308 provide real improvements to specification grading and deserve to be implemented at an early opportunity.

Comments:

- *Many of the binder samples from the failed pavement section contained high amounts of waste engine oil residues – often 11 – 15% by weight – this is higher than typically recommended by REOB manufacturers*

Oxidative Aging Of Asphalt Cements From an Ontario Pavement Trial
Logan Wright, Amit Kanabar, Eric Moult, Syed Rubab, and Simon Hesp
International Journal of Pavement Research and Technology 4.5 (2011)

This paper documents and discusses a rheological and spectroscopic investigation of the oxidative aging in asphalt cements from a 2003 northern Ontario pavement trial. Seven asphalt cements were aged according to standard methodology (rolling thin film oven test (RTFOT) and pressure aging vessel test (PAV)), as thin films at temperatures of 45, 65, and 85 °C and standard pressure (thin film oven test (TFOT)), and under modified PAV conditions (extended time of 40 hours). It was found that the current RTFOT/PAV protocol provided insufficient aging for the poor performing asphalt cements. Infrared analysis of the TFOT-aged samples for the formation of carbonyl groups could largely explain the performance ranking in service. The only significant outlier was modified with polymer and waste engine oil residue. Rheological investigations of the recovered and the TFOT-aged residues were able to show that such materials likely suffer from early gel formation. These findings illustrate the need for improved asphalt cement aging protocols. A preliminary example is provided that shows how simply doubling the PAV aging time to 40 hours can produce materials that more closely replicate those aged for 5-6 years in a northern Ontario climate. It was further confirmed that both chemical and physical hardening tendencies are closely correlated, likely because they involve similar asphalt constituents.

- Insufficiencies of the current M 320 standard promote the use of undesirable asphalt sources and modification technologies (air blowing, use of waxes, polyphosphoric acid, waste engine oils, gelling agents, blending of incompatible asphalts, etc.) often causing premature and excessive hardening and consequent thermal cracking
- An improved and practical aging method is needed so the authors developed a modified version of the current RTFOT/PAV protocol. This investigation began by looking at premature hardening that took place on several Ontario trial pavement sections.
- The objective was to obtain a better understanding of the oxidative aging processes that cause the performance to vary enormously between the seven test sections constructed with seven different binders all meeting the same M320 grade
- All seven binders were graded according to the current AASHTO M320 as well as using Ontario's extended BBR test method and the double-edged-notched tension (DENT) test.
- Additional aging protocols investigated were the thin film oven test (TFOT) at 45°C, 65°C, and 85°C and extending the PAV aging time to 40 hours
- TFOT-aged materials were investigated using infrared spectroscopy (IR) to monitor changes in carbonyl, sulfoxide, aromatic, and butadiene concentrations, and dynamic shear rheometer (DSR) to monitor changes in rheological performance
- Carbonyl content from the IR data ranks the sections in a reasonably accurate fashion – the two good performing sections had low carbonyl formation rates and low carbonyl indices – four of the poor performing sections had intermediate carbonyl formation rates and intermediate to high carbonyl indices
- The worst performing section was somewhat of an anomaly as it had low to intermediate carbonyl formation rates and a moderate carbonyl index – however low linear phase slopes as measured by the DSR on the TFOT samples aged at 45°C for 1000 hours indicate early gelation likely due to its modification with largely paraffinic waste engine oil residues
- Current M320 aging protocols does not provide sufficient oxidative hardening for poor performing binders to replicate properties from five- to six-year-old pavement sections
- TFOT aging tests show that carbonyl formation rate provides a reasonable prediction for performance
- Early formation of a gel is important in explaining the observed embrittlement of binders modified with waste engine oil residues of paraffinic nature
- Extended PAV aging appears to provide a significantly improved match between laboratory and recovered binder grades and potentially eradicate a significant amount of the widespread thermal cracking in Ontario

Comments:

- These are the same pavement sections referred to in Paper #4. All were specified as PG 64-34. According to LTPPBind v3.1 using 98% reliability and a rut depth of 12.5 mm, it would be been appropriate to specify a PG 64-40.

Effects of Engine Oil Residues on Asphalt Cement Quality
Syed Rubab, Kezia Burke, Logan Wright, and Simon A. M. Hesp, - Queen's University
Pamela Marks and Chris Raymond – Ontario Ministry of Transportation
CTAA Paper – 2011

This paper discusses and documents an investigation of chemical aging in asphalt cement modified with Engine Oil Residue (EOR). EOR is produced during the recycling of used motor oils collected from vehicles. It has been reported that a significant fraction of the asphalt cement used in parts of Canada is blended with EOR to meet certain low temperature Superpave grades. Such material can provide a low-cost method of increasing the grade span when blended with asphalt high in asphaltenes. However, concerns exist that such modifiers may negatively affect asphalt quality in several ways. First, the paraffinic nature of the EOR and possible presence of engine detergents can reduce the adhesion to the aggregate and thereby increase moisture damage. Second, metals such as iron, copper, and chromium, abundantly present in EOR, can act as catalysts in the oxidation of asphalt cement. Finally, paraffin can precipitate asphaltenes, which may accelerate further chemical and physical hardening. This paper presents results of aging tests on straight and EOR-modified asphalt cements. The study found that EOR significantly increased oxidation rates and the tendency for asphaltenes to precipitate, leading to a less ductile material with higher stress at low temperatures, which typically results in early and increased cracking.

- It is accepted by some that a mild degree of network (gel) formation is desirable in terms of overall asphalt cement quality
- The primary objective of this study was to investigate how EOR affects the oxidative hardening tendency of straight Cold Lake asphalt cement.
- A secondary objective was to show if and how the current Rolling Thin Film Oven/Pressure Aging Vessel (RTFO/PAV) protocol can be improved to better capture the effects of such additives on asphalt durability.
- The long-term objective of this research is to aid in the development of improved asphalt cement aging and specification test methods allowing user agencies to better ensure performance.
- Cold Lake crude is largely naphthenic in nature, and thus produces good quality asphalt cement relatively high in asphaltenes and low in wax (paraffin or microcrystalline). It suffers little from chemical and physical hardening processes – providing good performance for long periods of time. Both 80/100 and 200/300 penetration grade asphalt cements were evaluated.
- The EOR was obtained from a local supplier. The material is produced as a leftover in the recycling process of used engine oil and constitutes approximately 20 percent of the total used engine oil stream. These EOR have high ash contents, reflective of the high concentrations of metals in the residue. They are also high in saturated paraffin oil and degraded polymeric dispersants typically found in engine lubricants.
- Five blends were investigated – Cold Lake 80/100 pen, Cold Lake 80/100 + 10% EOR, +20% EOR, +30% EOR, and Cold Lake 200/300 pen
- The 80/100 Cold Lake modified with 20 percent EOR provides a low temperature grade that is equal to that of the straight 200/300 Cold Lake (-34°C), but with a high temperature grade that is 5°C higher (61°C versus 56°C)
- It has been shown that the blending of incompatible asphalt cements can provide significant gains in the grade span, this benefit may subsequently be lost due to increases in both physical and chemical hardening
- To address concerns with the potential for accelerated oxidative hardening, they looked at changes in carbonyl formation rates with the introduction of EOR in straight Cold Lake base asphalt. Carbonyl formation is closely linked to increases in viscosity and hence it is generally accepted as a convenient indicator to monitor oxidative hardening.
- The data show that the addition of EOR increases dramatically the absolute carbonyl indices and the carbonyl formation rates during the spurt and steady rate periods. These changes are likely due to the presence of metals and oxidized components found in EOR.
- Black space diagrams were plotted for temperatures between 34°C and 70°C at 12°C intervals and at five frequencies for each test temperature. Black space diagrams provide a comprehensive picture of the rheological performance of asphalt cements. A plot of the phase angle, δ, versus the log of the complex modulus, log G^*, gives a direct overview of the elastic and viscous nature of a material.
- After thin film aging, it is clear that the EOR affects the material by lowering the phase angle at all stiffness levels. Asphaltenes formed during the oxidation process are less soluble when EOR is present and hence produce a more pronounced gel-type network. This is, of course, the same reason why the grade span increases under the current Superpave specification. While there may be some benefit in rutting resistance, this gain will likely be offset by a significant decrease in fatigue and low temperature performance.
- Modified pressure aging vessel protocols were looked at using two binders – a cold lake 200/300 and a cold lake 80/100 + 20% EOR. These binder both graded at -34 using current M320 specifications. Film thicknesses were reduced by 75% in the PAV pans and the binders experienced an increase in low temperature properties of 2°C and 6°C respectively. The authors also increased the PAV aging time to 40 hours and the increases in low temperature grades were 4°C and 9°C respectively.
- Materials with very low initial stiffness, as reflected by the RTFO limiting temperature of -31°C for Cold Lake 80/100 + 20 % EOR, may also result in problems with mix tenderness during construction. Oregon has reported observing this tenderness and that it can cause a loss in pavement durability directly linked to the amount of reclaimed oil contaminant
- Engine oil residues significantly increase both the spurt and steady rate oxidation. This detrimental effect is likely due to the presence of large amounts of metal catalysts (Fe, Cu, and Cr) and/or oxidized engine oil components. Higher oxidation rates lead to more rapid gelation which could significantly increase restraining stress during winter. This increase in restraining stress is likely causing premature and excessive fatigue and thermal cracking.

- The benefit of the increased Superpave grade span from EOR modification is likely offset by a loss in low temperature grade when these asphalt cements are aged in a manner that more closely reflects the in service oxidation process.
- In addition to an increased tendency for chemical aging, these EOR modified asphalt cements also suffer from excessive physical hardening, likely because both chemical and physical aging impact similar asphalt constituents.

Comments:

- *The authors were not clear as to the nature of the EOR used – was it a re-refined product or a distilled product or was it just a filtered waste oil? It is described as "leftover in the recycling process of used engine oil".*
- *The blend percentages used were typically higher than those recommended by suppliers of these components*
- *Much of the data presented was for a dosage of 20% - much higher than generally recommended amounts – what would the impact on the binders have been at 5-7%?*
- *The authors mentioned that EOR was found in a majority of cracked pavements in Ontario – however this assumption was based on the presence of zinc found in the binders – while zinc is found in EOR, there are zinc compounds sometimes used as a H_2S scavenger in asphalt binders*
- *While the addition of EOR did increase the formation rates of carbonyl and the carbonyl indices of the Cold Lake binder, how would these results compare to other binders (non Cold Lake) that have good performance?*

Penetration Testing of Waste Engine Oil Residue Modified Asphalt Cements

Burke, K., and S. A. Hesp

Proc., 1st Conference of Transportation Research Group of India (CTRG), Bangalore, India, Dec. 2011

Asphalt cements sold in most of North America are currently graded according to the Superpave™ specification. Superpave specifies the Rolling Thin Film Oven (RTFO) followed by the Pressure Aging Vessel (PAV) for accelerated laboratory aging. Unaged material and RTFO residue are tested in the Dynamic Shear Rheometer (DSR) to determine a high temperature grade (PG XX) while PAV residue is tested in the Bending Beam Rheometer (BBR) to determine a low temperature grade (PG -YY). The main weaknesses of the Superpave test methods and specifications relate to insufficient aging in the RTFO/PAV and insufficient conditioning in the BBR. These deficiencies promote the use of many undesirable additives that can provide low cost ways to increase the grade span (XX-YY) through the gelation of the colloidal asphaltene structure. Hence, Superpave often results in North American pavements under-designed for thermal and fatigue cracking due to the widespread use of poor quality asphalt cements.

This paper discusses the effects of one such additive, Waste Engine Oil (WEO) residue, on the relative degree of steric hardening (i.e., structure formation) in asphalt cement. Steric hardening was monitored by means of a simple penetration test to determine temperature sensitivity expressed by the Penetration Index (PI). The negative effects of WEO are twofold. First, the abundant presence of metals in WEO promotes accelerated oxidative hardening. Second, the presence of large amounts of paraffin in WEO causes the asphaltenes to precipitate, resulting in the formation of a gel-type structure that is able to retain higher thermal stress levels during periods of cool temperatures and unable to heal microcracks during summer. These higher thermal stresses persist long enough during winter and spring to promote the formation of early cracking distress. The formation of a gel-type structure is clearly illustrated by an increase in the PI. Detrimental effects of adding WEO are briefly illustrated with real world experiences in Ontario trial sections and regular contracts. The findings are compared with data from field trials in southern Ontario from nearly 40 years ago that indicated that high PI materials have a tendency to suffer from excessive thermal cracking.

- The two asphalt cements investigated in this study were obtained from the Imperial Oil of Canada refinery in Edmonton, Alberta.
- The crude source for this refinery comes from Cold Lake, Alberta
- Penetration grades included 200-300 pen and 80-100 pen materials. The 80-100 pen material was blended with 20 % WEO obtained from a local supplier. The blended material graded as a PG -34, which is identical to the straight Cold Lake 200-300 pen asphalt.
- Penetration tests were conducted on all of the asphalt blends – with a penetration index (PI) calculated
- The penetration tests were conducted on unaged and PAV-aged samples.
- Additional PAV aging was conducted with just 12.5 g of asphalt cement in each pan in order to study how film thickness in the PAV affects the penetration test results.
- Penetration values are provided for the unaged and PAV-aged materials after one hour at 25°C, one week at 25°C, and one additional week at 0°C.

- The penetration data show that the WEO-modified asphalt cement is consistently harder at 25°C while being equal to or softer at 0°C.
- The PI data shows that the WEO-modified material is consistently less temperature sensitive (higher PI). However in addition to a (potential) gain in rutting resistance (lower penetration at high temperatures), the thermal cracking performance could deteriorate significantly due to higher retained thermal stress in winter and spring and a reduced ability to heal microcracks during summer.
- The addition of WEO to Cold Lake asphalt cement increases the Superpave grade span compared to straight Cold Lake asphalt cement of equal low temperature grade.
- The addition of WEO decreases the temperature susceptibility as indicated by a reduced penetration at high temperatures, a slightly increased penetration at lower temperatures, and a significantly increased PI.
- Field experience with asphalt cements modified with WEO suggests that high PI materials are prone to early and excessive thermal cracking.
- Recent experience across Ontario agrees with early pavement trial findings of McLeod, published some 40 years ago, that showed high PI materials to be prone to thermal cracking.

Comments:

- *It would have been useful for the authors to provide the properties for the base 80-100 penetration binder used in the 20% WEO blend*

Waste Engine Oil Residue in Asphalt Cement
S.A.M. Hesp and H.F. Shurvell
MAIREPAV Paper – 2012

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.

- 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.
- 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. 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. Approximately 200 g of asphalt cement was recovered for each pavement location, which was enough for testing in both LS-299 and LS-308.
- 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.
- 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)*. 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, wf, was plotted versus the ligament length (distance between the notches), L, and the specific essential work of fracture, we, was determined from the intercept of the curve. The specific essential work of fracture was divided by the net section stress, σn, 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.

- The CTOD on the recovered materials from Cochrane and North Bay were 12.7mm and 6.0mm respectively.
- The tendency for the binders to harden during cold conditioning was assessed through the extended BBR protocol LS-308. Specimens are conditioned at - 10°C and -20°C 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.
- The low temperature M320 grades of the extracted binders for the Cochrane and North Bay were -38°C and -27.4°C respectively. The LS-308 low grades were -38.6°C and -21.4°C with a three day grade loss of 1.8°C and 6.5°C respectively.
- XRF findings show that zinc and molybdenum were found in the North Bay samples while neither were found in the Cochrane material. These elements suggest that waste engine oil residue was present in the North Bay material.
- 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 presence of waste engine oil residue in the North Bay contract has made the binder more susceptible to chemical and physical hardening. 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 and low temperature grades. 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 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 then the confidence is further reduced to around 10%. This analysis reveals some of the deficiencies of the current AASHTO M320 specification and that it may not be ideal at characterizing binders with this type of modification.
- 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.
- 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.
- Paraffins in waste engine oil sludge promote additional physical hardening during cold storage.

Comments:

- *The site North of North Bay on Highway 11 is at 46.8538N and 79.7864W and the site west of Cochrane is at 49.0610N and 811749W. Using LTPPBind v3.1 with a reliability of 98% and a rut depth of 12.5 mm, 3 of the 5 closest stations (all are over 50 km away) would require a PG 52-40 and all of the 5 closest weather stations to the Cochrane section would require a PG 52-40.*
- *The authors discuss that "waste engine oil residue" or "waste engine oil sludge" has been added to the North Bay binder as determined by XRF detection of zinc and molybdenum – however it is unclear what that additive specifically is? Waste engine oil? Distilled waste engine oil? Re-refined engine oil bottoms? It is also not known at what dosage REOB may have been added.*

- *The authors provide no information regarding the types of aggregates used, the % binder, volumetric properties, film thicknesses, etc. They only mention that "no irregularities in quality can explain the premature and excessive cracking in the North Bay contract".*

Asphalt Binder Modification with Re-Refined Heavy Vacuum Distillation Oil (RHVDO)
John D'Angelo, Ken Grzybowski, and Steve Lewis
CTAA Paper – 2012

Asphalt binders used in the paving industry are required to perform under extreme conditions. At high temperatures when the binder is softer, it must withstand heavy traffic loading to prevent rutting. At low temperatures, it must remain flexible to resist cracking from traffic and thermal stresses. Additives have been used to improve the performance of asphalt binders and extend the temperature range where they can be used. These additives include many different organic polymers and chemicals to change the morphology or chemistry of the binders. One of these additives is the residue from the re-refining of used engine oil.

In this study, an in-depth evaluation was performed to determine the performance characteristics of asphalt binders modified with Re-refined Heavy Vacuum Distillation Oil (RHVDO) at several different levels. Chemical analysis of the RHVDO and the asphalt binders was performed to determine what changes are taking place in the modified binder. Extensive rheological testing was also run on the binders to evaluate the physical properties of the binders under several different aging conditions. This study provides a full evaluation of the real nature of the effects of RHVDO modification of asphalt binders.

- RHVDO is the heavy distillation residue from refining used engine oil. Re-refining typically produces 75% base oil, 12% fuel oil, and 12% residue which is used for modifying asphalt. The used oil is dewatered and any solids removed prior to the re-refining process. The used oil is compositionally evaluated to screen out contaminates and undesirable compounds and any oil containing hazardous material is diverted for burning or processed to remove the material.
- The re-refining process is typically a 3-stage process – stage 1 is a steam stripping process to remove light volatiles – stage 2 is an atmospheric distillation at moderate temperatures to remove of the lighter fuels such as home heating oil – stage 3 is a vacuum distillation at higher temperatures to separate the lubricating oil from the flux or residue. During this process, some of the larger wear metals found in the used oil have been removed leaving lower concentrations than what is seen typically in motor oils.
- Villanueva et al (2008) published a study looking at blending RHVDO with air blown Husky asphalt at various percentages. The RHVDO blended binders improved the low temperature properties and slightly decreased the high temperature properties in all cases according to AASHTO M320 Table 2 testing.
- Hesp et al (2011) claimed that RHVDO increased the aging of the asphalt blends by looking at the carbonyl indices. However, they failed to correct for the baseline carbonyl content and had they factored that out, it would have shown that RHVDO blended binders have the same aging rate as the base asphalt.
- This study looked at two mid-continent asphalts (Marathon-Detroit, MI, and BP-Calumet, IL) blended with RHVDO at different percentages (0, 2, 4, 6, 8, and 20%) using two different sources (Safety-Kleen-Chicago, IL, and Safety-Kleen-Breslau, Ontario) – RHVDO is typically used at less than 10%
- Extensive testing was carried out on the individual RHVDO, the asphalt binders, and the various blends. This included full M320 Table 1 and 2 testing, MP-19 MSCR testing, extended PAV (35-hour) aging, separation tests on the blends, aggregate adhesion screening ASTM 3625, and chemical analysis ASTM 4124.
- Both RHVDO's are predominantly made up of polar aromatics and saturates with a small percentage of asphaltenes. Both have very low wax contents less than 0.35% and neither have any naphthene aromatics
- Paraffinic wax or aliphatic materials are known to cause cracking in asphalt and have been to be the primary cause of physical hardening – both RHVDO's contain little to no wax and engine oil will not contain wax (even though they are produced from paraffinic oil) as they would not be able to perform in cold climates
- Asphaltenes are the viscosity building components of asphalt. As asphalt ages or oxidizes, polar aromatics change to asphaltenes and naphthene aromatics convert to polar aromatics. Saturates act as a solvent to disperse the asphaltenes and prevent them from developing into large molecules that will embrittle the asphalt. With no naphthene aromatics, the RHVDO-treated asphalt has less material that will age to stiffen the binder and the increased saturates help disperse the asphaltenes and reduce embrittlement of the asphalt.
- M 320 continuous grading shows that RHVDO reduces both the high and low temperature grade of the binders. It also shows that RHVDO's react differently with different binders.

- M 320 Table 2 grading indicates that RHVDO has a greater reduction in low temperature grade than the high temperature – low temperature reductions were as much one to one and a half low PG grade
- Each binder blend was subjected to both 20-hour and 35-hour PAV aging – as the percentage of RHVDO increases, BBR m-values increase – m-values for the 35-hour conditioned samples were lower than those aged for 20 hours – however the rate of aging does not increase with increased percentage of RHVDO
- The loss of m-values with the extended conditioning is similar for the neat binder with RHVDO as it is for 20% blend – this shows that the aging rate is controlled by the base binder and not the RHVDO
- RHVDO has a much more significant effect on the intermediate DSR properties – a 10°C reduction in the intermediate continuous grade was observed with the 20% blend
- The relationship of percent RHVDO to reduction in intermediate continuous grade was linear for both the 20-hour and 35-hour aged samples and the data shows that the addition of RHVDO does not increase the aging rates – aging rates are controlled by the base asphalt and not the RHVDO
- The DSR was used to evaluate the separation of the RHVDO asphalt binder blends and it was found to be less than 2% - this shows that RHVDO is very compatible with the binders used and will not separate easily
- An increase in the amount RHVDO does not cause any additional increase in the asphaltenes but does increase the saturates. Increased saturates are likely the reason for the reduction in intermediate DSR values and the low temperature properties – clearly showing that the increase RHVDO does not increase the age hardening of the binder
- Extended PAV aging shows little to no changes to the component fractions – the improvements to the intermediate and low temperature properties of the binder from the increased saturates of the RHVDO is not degraded with additional aging
- Component fractions of the RHVDO blends were compared to a Lloydminster 58-28 (good performer) and a California valley 64-16 (poor aging properties). The RHVDO blends had a much closer resemblance to the Lloydminster 58-28.
- Aggregate adhesion of the RHVDO blends was measured using a boiling test (ASTM D 3625) – aggregates used were a Trap Rock (known to have good binder aggregate adhesion) and a Lithonia Granite (known to have poor binder aggregate adhesion) – all binder blends tested (0, 4, and 8% RHVDO) exhibited good adhesion to the trap rock and poor adhesion to the granite. However, in some cases at 4 and 8% RHVDO, adhesion was improved with the granite. There is no indication that RHVDO negatively affected the adhesion.

Comments:

- *Other sources of RHVDO i.e. not Safety-Kleen should be considered to see if they behave the same and how their component fractions may vary*
- *Do all re-refining processes consist of the three stages referenced?*
- *One of the RHVDO used in this study was produced in Ontario – was this the same material used on any of the projects Hesp describes that had premature cracking?*
- *The authors should have added all of the component fractions for the Marathon blends after the 20 and 35-hour PAV aging*

Evaluation of Rejuvenator's Effectiveness With Conventional Mix Testing For 100% RAP Mixtures

Martins Zaumanis, Rajib B. Mallick, and Robert Frank

Transportation Research Board Compendium of Papers 2013 – Paper 13-1447

This paper presents research evaluating effectiveness of rejuvenators for production of very high (40-100%) Reclaimed Asphalt pavement (RAP) content mixtures. Nine differently originated softening agents were tested, including plant oils, waste derived oils, engineered products, as well as traditional and non-traditional refinery base oils. Two different dosages of the agents were added to binder extracted from RAP to evaluate their softening potential through testing of kinematic viscosity and penetration at two different temperatures. At 25°C the softening efficiency varied by a factor of twelve between the most and least effective rejuvenators. Consistency results at different temperatures were used to express temperature susceptibility by means of Penetration Index (PI), Penetration-Viscosity Number (PVN) and Bitumen Test Data Chart (BTDC) of the softened binders. The PI results varied measurably depending on the rejuvenator and supported the low temperature mixture test results, showing that PI may be a good and simple measure of rejuvenation effectiveness. Low temperature mixture embrittlement was evaluated at -10°C through determination of the indirect tensile strength and creep compliance for rejuvenated 100% RAP mixture samples. It can be concluded that four of the nine tested rejuvenators reduced extracted binder consistency to the necessary level and reduced susceptibility of RAP mixtures to low temperature embrittlement. Of the four, two engineered products tested had notably different performance but neither was superior to similar generic oils.

- Waste engine oil (WEO), waste engine oil plus a FT wax (WEO+FT Wax), and waste engine oil bottoms (WEOB) were two of the softening agents considered in this study – both were blended with extracted binder from RAP at a dosage rate of 18.26% by weight
- Penetration Index (PI) describes the temperature susceptibility of an asphalt and ranges from around -3 for highly temperature susceptible asphalt to +7 for highly blown or low-temperature susceptible asphalt.
- When asphalt ages, its PI usually increases, indicating a more structured and brittle material that is less able to flow and thus more prone to cracking.
- A successful restoring of aged RAP binder properties should reverse this process and thus the decreasing of PI compared to the extracted asphalt may be a good indicator of the rejuvenation quality.
- Most of the rejuvenators used in this study decreased PI of the RAP binder – WEOB and WEO+FT Wax both increased the PI. However a drawback to this method is that the determination of penetration at low temperature (4°C) may have a large statistical deviation since the measured values are small: therefore small inaccuracies in measurement can significantly influence the calculated PI.
- Penetration-Viscosity Number (PVN) was considered as an alternative to PI.
- PVN is calculated from kinematic viscosity at 135°C and penetration at 25°C – high PVN indicates low thermal susceptibility while low PVN indicates high thermal susceptibility.
- All of the rejuvenators decreased PVN while WEOB increased PVN
- PVN for RAP binders may be beneficial for characterization of medium to high temperature properties but may not be appropriate for use of characterization of low temperature properties and cracking
- Ten different sets of 100% RAP mixture samples were prepared, including reference and nine rejuvenated mixes. The RAP material had a binder content of 5.1% and Superpave 9.5-mm aggregate gradation with high dust content (10%).
- The samples were mixed with the respective dosage of rejuvenator after 2 h of heating at 150°C and aged for 2 h before compaction at the same temperature.
- The mixtures were tested for low-temperature properties at −10°C by using the indirect tensile creep compliance test for 1,000 s according to AASHTO T 322 and indirect tensile strength test according to ASTM D6931 Procedure A.
- The asphalt mixture samples were prepared according to the following considerations: the penetration at 25°C for the rejuvenated samples must be similar, if possible without exceeding reasonable rejuvenator content, and all samples must have equal binder content and therefore film thickness.
- The target binder penetration at 25°C of the rejuvenated samples was defined as 90 1/10 mm, which is close to the NuStar PG 64-22 penetration.
- The required rejuvenator amount needed to attain the required penetration for the WEO, aromatic extract, and paraffinic base oil was close to the dosage used in the asphalt binder study; therefore, the verified amount (18.26% of asphalt binder mass) was chosen for the preparation of the respective asphalt mixture samples.
- The products that cannot ensure the required penetration, without exceeding reasonable dosage range, were added at a rate of 18.26% (from binder mass) to reach equal film thickness with the rest of samples.
- For the mixtures for which a lower dosage rate of rejuvenator was required to reach the target penetration, the difference between the binder contents was compensated for by the addition of virgin NuStar PG 64-22 asphalt.
- Creep compliance is a way of characterizing the stiffness of material. The less stiff the pavement is at low temperature, the lower is the possibility of cracks. The results show that in most cases creep compliance has been increased after addition of rejuvenating agent, therefore reducing cracking potential. Creep compliance increased in both of the mixes containing WEO and WEOB.
- Fracture energy is defined as the energy required to initiate fracture of the mixture and it is not dependent on loading rate (13). Therefore, it can be used as an indicator of crack resistance. WEO and WEOB both increased fracture energy while WEO+FT Wax showed a decrease in fracture energy.
- Products used in the mixtures (when compared to the reference mixture) that have maintained or increased the creep compliance, without reducing the tensile strength and fracture energy, can be considered to have reduced the embrittlement of the mixture. WEO and WEOB both increased the creep compliance and fracture energy but decreased tensile strength. WEO+FT Wax increased creep compliance but decreased both fracture energy and tensile strength.

Comments:

- *Virgin binder PG 64-22 was added to the reference (RAP) mixture at 0.93% to maintain film thickness - this may have skewed the results*
- *What would the mixture and binder results be if lower dosages of WEO and WEOB were used?*
- *What would mixture test results have been from a mixture using virgin binder and extracted aggregate from the RAP?*

Evaluation of Rejuvenator's Effectiveness With Conventional Mix Testing For 100% RAP Mixtures
Martins Zaumanis, Rajib B. Mallick, and Robert Frank
Transportation Research Record: Journal of the Transportation Research Board, No. 2370 – 2013, pp. 17-25

Comments:
- *This paper reports the same data and information as paper #14 by the same other authors – however in this paper the HMA mixture fatigue testing was not included*

Evaluation of the Performance Properties of Asphalt Mixes Produced with Re-refined Heavy Vacuum Distillate Bottoms
John A. D'Angelo, Ken Grzybowski, Steve Lewis, and Rodney Walker
CTAA Paper – 2013

Asphalt mixtures used for paving are required to perform under extreme conditions. At high temperatures, the mix must withstand heavy traffic loading to prevent rutting. At low temperatures, it must remain flexible to resist cracking from traffic and thermal stresses. The aggregate structure is a primary performance characteristic for high temperature rutting, but it is the asphalt binder that has a much more critical role when it comes to cracking. Re-refined Heavy Vacuum Distillation Bottoms (RHVDB) - the residue from re-refining of used engine oil - has been used to improve the low temperature properties of asphalt binders and improve its cracking response. Previous papers have shown the improved binder properties with the addition of RHVDB, but the question still remains - will these materials perform well in a mixture?

An in-depth evaluation was performed to determine the performance characteristics of asphalt mixtures produced with binders modified with RHVDB at several different levels. The rutting, moisture damage, fatigue and low temperature properties of the mixtures were evaluated against control mixes produced with unmodified binders. Multiple tests were run to compare the mixtures with RHVDB against control mixes. The study provides an extensive evaluation of the effects of RHVDB modification on mix performance.

- In this study, two coarse graded Superpave mix designs typically used by the Illinois Department of Transportation (DOT) were produced with asphalt binders blended with RHVDB (brand name EcoAddz) at various add rates. The Illinois mixes and aggregates were selected because they were reported to have a higher potential for moisture damage.
- RHVDBs were added at 2, 4, 6, and 10 percent levels to a BP PG 64-22. The PG 64-22 was used as the control for the 2 percent RHVDB addition. Separate control binders were developed for the four and six percent RHVDB additions by blending a soft flux (PG 46-34) produced by BP with the PG 64-22. The continuous grades show that control blends match the RHVDB blends within 0.5°C for both the high and low temperature grade. A liquid anti-strip was added to the RHVDB six percent blend.
- The aggregates for the mixes were a Dolomitic Limestone which are 100% crushed material. A coarse graded N70 (70 gyration) Superpave mix and a coarse graded N90 (90 gyration) Superpave mix were produced using 19 mm nominal maximum aggregate size.
- The mix design binder content was established for the PG 64-22 control in each mix and the optimum binder content from the control was maintained for each of the other mixes. There were only minor variations in air voids and VMA, from the control mixtures.
- The experimental design included two aggregate gradations and eight different asphalt binders. This provided 16 different mixes to test for rutting, dynamic modulus, fatigue, cracking and moisture damage properties. The mixes included binder blends with 2, 4, 6, and 10 percent RHVDBs and PG 64-22 control along with the controls for the 4 percent and 6 percent RHVDBs.
- Rutting was evaluated using both the HWT test and Flow Number test.
- All HWT tests specimens were compacted to 7 percent air voids and tested under water at 50°C. Rutting ranged from 4 to 8 mm for the various binders blends used in with the N70 mix and ranged from 5 to 10 mm for the binder blends in the N90 mix. Rutting basically followed the binder stiffness with the PG 64-22 control having the lowest rutting and the 10 percent RHVDB blend having the highest rutting. All the mixes were well below the Illinois DOT requirement that rutting be less than 12.5 mm at 7500 wheel passes for a PG 64-22 binder. This was the case even for the PG 58 binders. Comparison of the various RHVDB percentage blends with the specific control developed for the specific percent indicates that they are equivalent or better in rutting to the control.

- The Flow Number test was done at 47.5°C, which is the 50 percent reliability pavement temperature. A cyclic axial 600 kPa load is applied to the specimen for 10,000 repetitions or until two percent permanent strain is reached. The Flow Number data mirrored the HWT test data. The Flow Number the results followed the binder stiffness with the PG 64-22 having the highest Flow Number and the 10 percent RHVDB PG 58-28 having the lowest Flow Number. In both mix types, N70 and N90, the RHVDB mixes provided equivalent results to the individual controls for each percentage.
- Moisture damage potential was evaluated using AASHTO T 283 and the HWT test.
- Illinois modified AASHTO T 283 and the HWT test were both run as moisture damage tests on all mixes in the study. The Illinois modification to AASHTO T 283 is to not require a freeze thaw cycle. The HWT test was run at 50°C under water with a 705 Newton loading. All the samples were tested to 20,000 wheel passes.
- All mixes for both the N70 and N90 designs except the 10 percent RHVDB met the Illinois DOT requirement for Tensile Strength Ratio (TSR) of 85 percent - the 10 percent RHVDB mixes had TSR values greater than 80 percent, but were just under the 85 percent requirement. The PG 64-22 control mixes for both the N70 and N90 designs actually had the lowest passing TSRs of all the mixes.
- The HWT tests verified what was seen in the AASHTO T283 testing. Only the 10 percent RHVDB mix shows any sign of the stripping inflection point, which is typically defined as indicating moisture damage in the test. The addition of a liquid anti-strip or hydrated lime would eliminate any indication of moisture damage even in the 10 percent mixes.
- Four point bending beam fatigue testing was used to evaluate the fatigue properties of mixes
- Fatigue properties of the mixes were evaluated using ASTM 7460 4 – Point Flexural Fatigue. Samples for the test were made by compacting slabs to 7 percent air voids and then cutting 380x50x68 mm beams from the slabs. Two strain levels were used to evaluate the mix properties; a 300 micro-strain level to simulate low strain levels in a pavement and 700 micro-strains to simulate high strain levels. All testing was done at 20°C for fatigue evaluation. Cycles to failure for the testing were determined at the number of cycles to reach 50 percent loss of the initial beam modulus.
- The PG 64-22 control for both the N70 and N90 mixes had the lowest number of cycles to failure at both the high and low strain levels, indicating the poorest fatigue response. The 6 percent RHVDB with 0.5 percent liquid anti-strip had the highest cycles to failure at both strain levels for both mix types. All of the RHVDB mixes provided better fatigue response than the straight run PG 64-22.
- Low temperature cracking was evaluated using the ASTM D7313 "Determining Fracture Energy of Asphalt-Aggregate Mixtures Using the Disk-Shaped Compact Tension Geometry." Cylindrical shaped mix specimens 150 mm in diameter by 50 mm wide with a specified size notch are pulled in direct tension to determine the fracture energy of the mix.
- N70 mixes show a clear increase in fracture energy with the addition of the RHVDBs, indicating improved cracking properties. In each case, the RHVDB mix is equal or better than the corresponding control mix and better than the PG 64-22 control. The N90 mixes indicate that the 2, 4 and percent RHVDB mixes have similar fracture energy properties to the PG 62-22 control and the 4 and 6 percent EcoAddz equivalent. It is not until the 6 percent RHVDB with 0.5 percent AS and the 10 percent RHVDB mix that the improvement in fracture energy is seen. None of the RHVDB mixes provide reduced fracture energy.
- Overall in rut resistance, moisture damage potential, fatigue response and cracking potential the Re-refined Heavy Vacuum Distillate Bottoms provided equal or better performance that the control neat binders. This study indicates that binders modified with RHVDB should provide good performance in the field.

Comments:

- *It would be interesting to see how other RHVDB and/or other binder/aggregate sources would perform using the same arsenal of tests – the current combination of BP asphalt and EcoAddz appears to be very compatible*
- *A nice follow-up report would be to construct trial pavement sections using each of these blends and monitor actual field performance*

Pushing the Asphalt Recycling Technology to the Limit

Joel R. M. Oliveira, Hugo M.R.D. Silva, Carlos M.G. Jesus, Liliana P.F. Abreu, and Sara R.M. Fernandes
International Journal of Pavement Research and Technology - 6.2 (2013) pp. 109-116

The environmental and economic benefits of using Reclaimed Asphalt Pavement (RAP) material in hot mix asphalt (HMA) applications could be pushed up to the limit, by producing totally recycled HMAs (100% RAP), but the performance of this alternative must be satisfactory. In the present study, the utilization of a used motor oil as a rejuvenator was evaluated. This would allow the aged binder to restore some of its original properties, thus promoting an adequate performance of the mixture. After studying the RAP moisture content, the optimal amount of oil was determined by conventional bitumen tests, using the penetration grade as the selection criterion. Then, the binder was evaluated through rheological testing, and laboratory specimens were prepared and tested for water sensitivity, permanent deformation, stiffness and fatigue, in order to confirm that the totally recycled mixture will perform as good as a conventional mixture used for comparison purposes.

- Since the RAP binder is too hard for a conventional bituminous mixture, an aliphatic-aromatic used motor oil was also used to rejuvenate the binder and improve its properties.
- To determine the amount of additive that should be added to the mixture (to improve its properties), a new binder (10/20 pen grade), with properties similar to those of the RAP binder, was blended with the used motor oil rejuvenator to achieve a 20/30 penetration grade
- The minimum amount of rejuvenator necessary to modify the aged binder and to achieve a 20/30 penetration grade was 5% - this also allows reducing the mixing temperature of the recycled HMA mixture by approximately 10 to 15°C
- Master Curves (TREF = 30 °C) of Complex Modulus (G*), tan(δ), Elastic Modulus (G') and Viscous Modulus (G'') were developed for aged and rejuvenated binders.
- The complex modulus of the rejuvenated binder is nearly 10 times lower than that of the aged bitumen almost for all frequencies and temperatures, especially due to the reduction of the elastic modulus, although the viscous modulus has also decreased. Thus, the values of tan(δ) of the rejuvenated binder increase for all frequencies due to the higher decrease of its elastic modulus.
- The relaxation time corresponding to the inverse of the frequency where tan(δ) = 1 decreases after the addition of the rejuvenator. Since the relaxation time is proportional to the bitumen viscosity and the cube of the size of the asphaltenes/resins micelles, this suggests that the rejuvenator effectively reduced the size of the micelles of the binder, thus causing a change in the behavior of the binder at higher frequencies.
- Since the RAP materials do not meet the mixture gradation specifications, the performance of the 100% RAP mixture was compared to that of a conventional HMA mixture with a different composition.
- The conventional mixture (produced with a 35/50 pen grade bitumen) was slightly more sensitive to the presence of water. The recycled mixture with incorporation of a rejuvenator showed a better performance due to the lower volume of voids
- The rut resistance of asphalt mixtures was assessed using a wheel tracking test (WTT) - the wheel tracking slope in air (WTS_{air}) was measured between the 5000th and the 10000th cycles - the recycled asphalt mixture had a WTS_{air} value of 0.14 mm/10^3 cycles while the conventional mixture had a slightly worse slope of 0.16 mm/10^3 cycles.
- The stiffness modulus and phase angle were obtained using the four-point bending beam test - the recycled mixture is less susceptible to the loading frequency since the stiffness modulus variation is smaller and the phase angle is significantly lower than that of the conventional mixture
- The recycled mixture had a better fatigue life that the conventional mixture most likely due to its high content of fines
- The rejuvenator increase the fatigue life of recycled mixtures due to the combined effect of the reduction on the penetration grade of the binder and the slight increase on the binder content of such mixtures. Those factors associated with a higher content of fines greatly enhanced the flexibility of the recycled mixture, thus increasing its fatigue cracking resistance.
- The incorporation of 100% RAP and used motor oil (as a binder rejuvenator) in the production of asphalt mixtures can be a paving solution with a performance as good as conventional asphalt mixtures, as long as adequate storing (RAP moisture) and production (temperature) conditions are assured.

Comments:

- *Used motor was used in this study (not REOB)*
- *All binder testing in this study assumed 100% blending of the RAP binder and the used motor oil*
- *Mixture properties were compared using different gradations and different air voids*

Investigation of the Effect of Oil Modification on Critical Characteristics of Asphalt Binders
Amir Golalipour
A dissertation at the UNIVERSITY OF WISCONSIN – MADISON 2013

Thermally induced cracking of asphalt pavement continues to be a serious issue in cold climate regions as well as in areas which experience extreme daily temperature differentials. Low temperature cracking of asphalt pavements is attributed to thermal stresses and strains developed during cooling cycles. Improving asphalt binder low temperature fracture and stiffness properties continues to be a subject of particular concern. Therefore, significant amount of research has been focused on improving asphalt binder properties through modification.

In recent years, wide ranges of oil based modifications have been introduced to improve asphalt binder performance, especially at the low service temperatures. Although, significant use of these oils is seen in practice, knowledge of the fundamental mechanisms of oil modification and their properties for achieving optimum characteristics is limited. Hence, this study focuses on better understanding of the effect of oil modifiers which would help better material selection and achieve optimum performance in terms of increasing the life span of pavements.

In this study, the effect of oil modification on the rheological properties of the asphalt binder is investigated. To examine the effect of oil modification on binder characteristics, low temperature properties as well as high temperature performance of oil modified binders were evaluated. It is found that oils vary in their effects on asphalt binder performance. However, for all oils used in the study, adding an oil to binder can improve binder low temperature performance, and this result mainly attributed to the softening effect. In addition to that, a simple linear model is proposed to predict the performance grade of oil modified binder based on the properties of its constituents at high and low temperatures.

Another part of this study focuses on the oil modification effect on asphalt binder thermal strain and stresses. A viscoelastic analytical procedure is combined with experimentally derived failure stress and strain envelopes to determine the controlling failure mechanism, strain tolerance or critical stress, in thermal cracking of oil modified binders. The low temperature failure results depict that oil modification has a good potential of improving the cracking resistance of asphalt binders during thermal cycles.

- Two different PG 64-22 binders were used in this study – one relatively high in asphaltenes and one relatively low
- 10 different oils were used – 1 petroleum aromatic oil, 3 petroleum paraffinic oils, 1 petroleum naphthenic oil, 3 bio-oils, and 2 REOBs
- The dosage of REOB required to reduce the high temperature PG grade by one full grade was 4-5% - additional testing was done using 5%
- Type of modification processing effects the binder blend properties – high shear blending for 30 minutes was used as this gave the best results
- Testing included : full PG testing, Glass Transition (Tg) Test, MSCR Test, Frequency Sweep, Linear Amplitude Sweep (LAS) Test, Single Edge Notched Beam (SENB) Test, and Bitumen Bond Strength (BBS) Test
- Both REOB blends tested provided the least aging susceptibility (lowest aging indices) of all of the oils blends used
- Both REOBs reduced the intermediate temperature stiffness of the binder which is beneficial to fatigue life based on the Superpave assumptions
- One REOB did well and the other not so well using the LAS test
- Both REOB blends provided the greatest decrease in low temperature stiffness when compared to the neat binder – they also provided the highest increase in m-value at the lowest temperature
- Based on SENB test, REOB improved the low temperature performance of the binder
- REOB was the most effective oil in improving low temperature properties according to the Tg Test

Comments:

- *A 5% dosage rate was used in this study.*

Effect of Waste Engine Oil Residue on Quality and Durability of SHRP Materials Reference Library Binders

Kelli-Anne N. Johnson and Simon A. M. Hesp

Transportation Research Record: Journal of the Transportation Research Board. 2014(2444): pp 102–9

This paper discusses the effects of widely used waste engine oil (WEO) residue on asphalt binder properties. Five SHRP Materials Reference Library binders and one commercial material from Ontario, Canada, were modified with 15% WEO. Materials were aged according to a standard rolling thin film oven protocol followed by 20 and 40 h of pressure aging vessel (PAV). Binder quality was determined according to AASHTO specification M 320 as well as dynamic shear characterization. Durability was assessed after additional conditioning at high temperature in the PAV and at low temperature in the bending beam rheometer. All but one of the WEO-modified materials transitioned to a gel-type state more readily upon chemical and physical conditioning; this result explains the poor thermal cracking performance seen for such binders on Ontario roads.

- WEO modified binders that become more elastic and less viscous suggest that these materials are less able to heal microcracks at high temperatures during summer months.

- Adding WEO to binders with moderate to high asphaltene contents result in effect analogous to regular aging – loss in phase angle and increase in stiffness. Binders that retain a high phase angle at both high and low temperatures are known to do well in resisting both thermal and fatigue cracking.
- 5 of the 6 binders tested had an increase in PG grade span with 15% WEO was added. However, after 72 hours of BBR specimen conditioning at -14C, the slight increases in grade span resulted in slight decreases.
- WEO modification causes the formation of gel-type binders when the base asphalt is high in asphaltenes. AASHTO M 320 fails to account for excessive physical and chemical hardening of the materials.
- WEO should only be used with great caution for modification of low asphaltene binders.

Comments:
- *The authors use the term WEO or waste engine oil residue. This appears to be the same as REOB.*
- *While a variety of binders was used, only one REOB material was used – how would other REOB sources behave?*
- *15% was the only dosage looked at – while this may be representative in the minds of the author as to the typical dosage in Ontario, it is higher than what is generally recommended*

The Performance of Aged Asphalt Materials Rejuvenated with Waste Engine Oil
Christopher D. DeDene and Zhanping You
International Journal of Pavement Research and Technology – March 2014 – Vol. 7 No. 2 – pp 145-152

The ability to recycle large amounts of asphalt pavement hinges on the capability of restoring the properties of the aged asphalt binder contained within the old pavement to that of virgin binder. Common practice in asphalt pavement recycling is to blend reclaimed asphalt pavements (RAP) with a recycling agent to chemically restore the aged asphalt binder. Waste engine oil from automobiles has been shown to improve asphalt binder when applied in small quantities.

In this study, a PG 58-28 neat, virgin binder was blended with reclaimed asphalt binder (RAB) and waste engine oil. The blends were then tested to study the interactions between RAB and waste engine oil. Using Fourier-Transform Infrared Spectroscopy (FT-IR), the differences in the samples were compared using the structural indices associated with asphalt binder aging. This testing revealed a decrease in the two aging indices of the blended asphalt binder, indicating that waste engine oil has the ability to chemically restore aged asphalt binder.

Asphalt mixture testing was then performed with mixtures of virgin asphalt, virgin binder, RAP and waste engine oil, in quantities similar to the binder testing, to see if the rejuvenation shown in FT-IR led to an improvement in the performance of the pavement specimens. After specimens were created, testing for freeze thaw durability, and rutting susceptibility was conducted. The results of the mixture testing failed to show an improvement of the freeze thaw durability or rutting susceptibility of specimens created with RAP and waste engine oil when compared to mixtures containing only new materials.

- The purpose of recycling agents is to restore asphalt consistency and chemistry – two types are generally used – rejuvenating agents and softening agents. More than 10% recycling agent by weight of the binder proves to be detrimental
- FT-IR testing was done on samples of: virgin binder (PG 58-28); virgin binder + 25% RAB (binder from RAP); waste engine oil (WEO); unused engine oil; virgin binder +25% RAB + 4% WEO; and virgin binder + 25% RAB + 8% WEO.
- Sulfoxides and carbonyls were considered as they are indicative of the amount of aging a binder has undergone
- When comparing the different blends, an increase in either the sulfoxide or the carbonyl index is indicative of an increase in the amount of asphaltenes present (aging)
- FT-IR testing showed reductions in carbonyl and sulfoxide structural indices as more waste engine oil is added to the blend. That reduction translates to an increase in the relative amount of maltenes.
- Pavements with excessive amounts of maltenes in them are known to suffer from rutting and moisture sensitivity issues. This can been seen by the mixture results where rutting increased with the addition of WEO. The addition of WEO did not adversely affect TSR values but did reduce the indirect tensile strengths of the mixture.

Comments:
- *This research involved the use of waste engine oil – not REOB*
- *No consideration was given to long term aging of either the binder blends or the mixture – it would be interesting to see the impact of aging on these materials*

Evaluation of Oil Modification Effect on Asphalt Binder Thermal Cracking and Aging Properties
Amir Golalipour, Ph.D. and Hussain Bahia, Ph.D.
CTAA – 2014

This study investigates the effect of oil modification on binder's low temperature cracking resistance and in terms of effect on stiffness, stress relaxation rate, and fracture properties. The study compares a set of different oils commonly used and explores the relation to binder's cracking criteria is defined.

An analytical method is used to numerically predict thermal stresses due to cooling cycle for oil modified binders by incorporating BBR creep stiffness values and non-linear thermal contraction rate data from the Glass transition (Tg) test results. Furthermore, fracture properties and critical stress and strain values were measured at different temperatures using the Single Edge Notched Beam (SENB) test procedure. Overall, these results confirm that oil modifiers can potentially improve pavement cracking resistance in cold regions besides the fact some of these materials are refined waste material that has low cost and usage of them will benefit the green mission of the countries.

- Various oils were blended at 5% by weight with a PG 64-22 and then tested, These oils included a petroleum based aromatic oil (PA), a petroleum based paraffinic oil (PP-3), a bio-based oil (BO-1), and a refined used oil (RW-1) – RW-1 is REOB as its origin is waste motor oil.
- At only 5%, the RW-1 lowered the base asphalt grade from a PG 64-22 to a PG 58-28 – nearly meeting PG 58-34
- The addition of oils lowered the glass transition temperature (Tg) in all cases with RW-1 having the greatest drop in Tg
- The addition of oils also increased the coefficient of thermal contraction of the binder in all cases
- RW-1 had the lowest Tg and the highest coefficient of thermal contraction which suggests RW-1 is the most effective oil regarding low temperature properties
- Oil modification increases m-value of neat binder – RW-1 showed the best results and nearly passed the criteria for -24C
- RW-1 showed the least temperature susceptibility by having the smallest change in m-value when going from -18C to -24C
- Physical hardening was tested by storing the BBR specimen before testing at extended times up to 72 hours – temperatures used were -12C, -18C, and -24C
- BBR stiffness increased with extended conditioning at low temperatures for all binders – at 24 hrs. the amount of increase in stiffness for the neat binder and the RW-1 blended binder were virtually the same – even at 72 hours, the increase in stiffness between these two binders were very similar with the RW-1 slightly higher
- BBR m-value decreased with extended conditioning at low temperatures for all binders – RW-1 however had the lowest reduction in m-value at all temperatures and all times – RW-1 did the best at decreasing m-value loss due to the physical hardening effect
- Oil modified have better performance with regards to stress build up during cooling – the RW-1 modified binder had the lowest stress build-up making it ideal for use in a cold region
- The single edge notched beam test was used to measure resistance to cracking – higher maximum deflection indicates more ductile behavior and higher total fracture energy means the material would tolerate higher amounts of stress or strain before failure – the RW-1 modified binder had the highest strain and stress failure when compared to all other binders
- Stress and strain failure was considered in this study – the intersection of the failure stress line with the build-up stress line (or the intersection of the failure strain line with the build-up strain line) is the failure temperature which the binder will crack due to thermal cycles.
- The RW-1 modified binder improved the cracking properties significantly – at only 5% RW-1 resulted in almost a 5 degree improvement in cracking temperature
- The best performance of the oil modified binders tested in this study was the RW-1 modified binder

Comments:

- *The RW-1 used in this study is likely to be REOB*
- *The results found somewhat contradict some of Hesp's work regarding physical hardening as the addition of REOB at 5% does not increase the rate of physical hardening or loss of grade when compared to the virgin binder*
- *Maybe more is not better – Hesp used 15% and this study used 5%*
- *Maybe all REOBs are not the same?*

The Impact of Asphalt Blended with Re-refined Vacuum Tower Bottoms (RTVB) and Its Effect on HMA Mixture Performance

Jason C. Wielinski, Anthony J. Kreich, Gerald A. Huber, Andreas Horton, Linda V. Osborn, Heritage Research Group
CTAA 2014

In the past few years, there has been increased interest in the properties of asphalt binders containing RVTBs, the non-distillable fraction from the re-refining of used engine oils, on HMA performance. Recent published research has provided conflicting results. This study provides additional information regarding the viability, compatibility and integrity of RVTBs in asphalt binders and HMA pavement mixtures.

One source of RVTB was blended with PG 64-22 to achieve binder grade of PG 58-28. A second PG 58- 28 was formulated without RVTB using a soft asphalt blended with PG 64-22. The asphalt binders were analyzed for Superpave PG Binder Testing, X-Ray Fluorescence, Polycyclic Aromatic Compound (PAC) content, Gel Permeation Chromatography (GPC) and ThermoGravimetric Analysis (TGA). The binder testing provides a comparison of the effects of blending RVTBs on binder grading, chemical composition, environmental impact, and compatibility of RVTBs and asphalt.

The second portion of the study compares mixture performance of HMA prepared with neat PG 58-28 and PG 58-28 containing RVTBs. Analysis includes volumetric analysis, resistance to moisture damage, resistance to rutting, mixture stiffness, fatigue resistance and environmental assessment using leachate testing.

- Chemical analysis by XRF showed higher presence of some inorganic compounds (metals) in the PG 58-28 with RVTB's than in the neat PG 58-28 asphalt binder. Compounds with greater presence in RVTB binder included Phosphorous, Zinc, Magnesium, Iron and Calcium. Phosphorous and Zinc are the two most prominent elements. Zinc has been identified as a potential indicator element for the presence of RVTBs.
- Testing results for polycyclic aromatic compounds (PAC) showed no significant difference in carcinogenic known PACs between the RVTB blended binder and the neat asphalts. Various PACs have been shown to cause mutagenicity when at elevated levels. Levels tested in the analyzed asphalts were comparable to levels found in other studies and were much below levels of concern. Based on these results, adding RVTBs to the asphalt binder does not pose any additional environmental or health issues. This is in part because these compounds remain in the asphalt and are not bioavailable from dermal contact, fumes by inhalation, or in leachate.
- There was no significant difference in molecular weight of the original asphalts analyzed by GPC. After undergoing aging through RTFO and PAV, the molecular weights increased as expected. The average molecular weights of the neat and RVTB PG 58-28 binders after PAV aging were very similar. This finding would suggest the formation of asphaltenes, which occurs as asphalt binder ages, is occurring at similar rates in both the RVTB- containing asphalt binder and the asphalt binder made without RVTB. The results suggest that both asphalt binders age at similar rates.
- There was also no appreciable difference in thermal stability of the PG 58-28 Neat, PG 58-28 RVTB and the PG 64-22 neat as measured by TGA. TGA would identify the presence of materials that would thermal crack at low temperatures. Blending 9% RVTB with a neat PG 64- 22 produced an asphalt binder that meets PG 58-28 specification. The PG 58-28 RVTB and PG 58-28 had comparable DSR results after RTFO and PAV aging protocols. This finding corroborates the GPC analysis that suggested that RVTB does not cause accelerated aging of the binder.
- Blending 9% RVTB with a neat PG 64-22 produced an asphalt binder that meets PG 58-28 specification. The PG 58-28 RVTB and PG 58-28 had comparable DSR results after RTFO and PAV aging protocols. This finding corroborates the GPC analysis that suggested that RVTB does not cause accelerated aging of the binder. The mid-temperature stiffness of the PG 28 RVTBs had a lower stiffness than the PG 58-28, suggesting good fatigue properties.
- Asphalt mixtures (9.5mm NMAS) were designed with 70 gyrations (N70) to meet IDOT HMA requirements. HMA mixtures containing the PG 58-28 neat and PG 58-28 RVTB asphalt both met the specifications.
- Results for tensile strength ratio (TSR) for the mixes analyzed show better TSR values for the PG 58-28 RVTB than for the PG 58-28 neat asphalt mix. The mixture containing RVTB asphalt binder had a slightly lower (but acceptable) dry tensile strength. It is not known if this finding can be generalized for all mixtures containing RVTB asphalt binders. The mixture containing RVTB binder was less susceptible to the loss of strength from water.
- Rutting resistance measured by Hamburg Wheel Track (HWT) testing and by Flow Number was about equal for both neat and RVTB binder mixes. Both mixes passed the rutting requirement of 12.5 mm after 7500 passes in the HWT test. This finding suggests that the lubricating effect of viscosity enhancers used in motor oil (and detectable in RVTB) does not cause the asphalt mixture to be more susceptible to rutting.

- Stiffness of the HMA mixes was found to be similar based on dynamic modulus testing at low and intermediate temperatures across all testing frequencies. This finding agrees with the rutting performance as indicated in the Hamburg Wheel Test. Rutting is predominantly a stiffness related property and both mixtures had similar stiffness and similar rutting performance.
- Both asphalt mixtures were artificially aged prior to fatigue testing to simulate long term aging. The asphalt mixture made with RVTB containing asphalt binder had slightly higher stiffness and resisted fatigue damage better than mixture containing neat asphalt binder. Repeatability among the triplicate specimens was reasonable for this test method. The effect was repeatable within the two mixtures. Asphalt mixtures contain RVTB asphalt binders had slightly improved fatigue resistance.
- Leachate results show trace levels of barium for both the asphalt mix and asphalt mix with RVTB at similar concentrations. This has been seen in previous studies and is related to low levels of barium in the limestone aggregate. No other regulated metals were detected in either sample.
- Leachate and subsequent organic analysis showed that all compounds tested were below the limit of detection. Additional analysis using GC-TOF/MS to obtain lower detection limits revealed trace levels of eight PACs; the sum was 12.3 ppb vs 13.2 ppb for the asphalt mix control and the asphalt mix with RVTB, respectively.
- The results of this study indicate that introducing RVTBs into an asphalt binder at a rate of 9%, sufficient to convert a PG 64-22 to a PG 58-28, does not compromise mixture stiffness or aging. In this case it enhanced resistance to moisture damage and resistance to fatigue damage. The RVTB additive did not significantly affect PAC levels and were similar to other asphalt reported in the literature.

Comments:

- *The authors use the term Re-refined Vacuum Tower Bottoms (RVTB) which is the same material as REOB.*
- *Testing was done at 9% RVTB – it would have been nice to see PAC and GPC data for binders with higher dosages of RVTB as well as mixture to determine leachate potential*
- *By itself, is RVTB bad? Do any PACs leach from it?*

Analysis of Vacuum Tower Asphalt Extender and Effect on Bitumen and Asphalt Properties

Jason C. Wielinski, Anthony J. Kreich, Gerald A. Huber, Andreas Horton, Linda V. Osborn, Heritage Research Group
Taylor and Francis – Road Materials and Pavement Design, 2015 – http://dx.doi.org/10.1080/14680629.2015.1030832

Comments:

- *This paper reports the same data and information as the paper above by the same other authors*

The Analysis of Asphalt Binders for Recycled Engine Oil Bottoms by X-Ray Fluorescence Spectroscopy

Terence S. Arnold, CChem. FRSC and Anant Shastry PhD
Transportation Research Board - Compendium of Papers – 2015

This research, which started in 2010, resulted from discussions with Illinois Department of Transportation. They had received asphalt binders which appeared to have been modified with phosphoric acid. Subsequent testing showed that they contained a residue from recycling used engine oil. It is also part of the responsibility of the chemistry laboratory staff at the Turner Fairbank Highway Research Center (TFHRC) to develop and provide test methods for use by State agencies and industry for the analysis of pavement materials. Since 2010, the increased use of these residues in paving asphalt binders coupled with negative research findings associated with low temperature cracking published in Canada has generated great interest and controversy in their use, particularly in the New England States.

In the US there is a practice of recovering lubricating oils from waste engine oils. The processing generates a residue, recycled engine oil bottoms (REOB). Engine oils contain performance enhancing additives. The sulfur, zinc, calcium, phosphorous and molybdenum from these, and engine wear metals (mainly copper and iron), all end up in the REOB. Their presence can be readily measured using X-ray Fluorescence Spectroscopy (XRF).
This paper presents a methodology to detect and measure the presence of REOB in asphalt binders.

The analysis is not simple since some of these metals also occur in asphalt, many are found in ground tire rubber and some are found in scavengers sometimes added to asphalt to control emissions of hydrogen sulfide. The variable composition of REOBs imposes some accuracy limitations.

- The use of Recycled Engine Oil Bottoms in the USA is widespread. They were found in samples from 16 of the 31 agencies that responded to the study.
- Recycled engine oil bottoms, unlike asphalt binders, are liquids at room temperature. They vary widely in their viscosity, effect on Superpave Performance grade and react differently with asphalt binders from different sources.
- X-Ray Fluorescence Spectroscopy can readily determine the presence of these residues in asphalt binders. The presence of calcium, zinc, copper and molybdenum can be used as a fingerprint to show that a binder contains REOB.
- Quantitative estimation of REOB content is complicated by the varying compositions of these additives. Analysis of more REOB and asphalt blends might improve the accuracy of the method.
- The analysis is complicated by the presence of zinc containing hydrogen sulfide scavengers and ground tire rubber. The levels of zinc in GTR are much higher than those in REOB. High zinc levels in binders calculate to unrealistic amounts of REOB and indicated the presence of GTR or hydrogen sulfide scavengers or both but do not necessarily imply a lack of REOB being used.
- If a binder has been modified with REOB, GTR, and a hydrogen sulfide scavenger then a more sensitive wavelength dispersive XRF spectrometer might be better able to detect the low levels of silicon, calcium, and copper.

Comments:

- *This type of quantitative analysis may not work for field materials (recovered binders) in the absence of the original binder used.*

Cold 2014 Winter and Early Asphalt Pavement Cracking Observed in Southwestern Ontario

Ludomir Uzarowski and Vimy Henderson, Gary MacDonald, John Rizzo, Gary Moore

Properly designed and constructed asphalt pavements incorporating good quality materials are typically anticipated to require major rehabilitation after about 20 or more years of service. The life of pavement that went through major rehabilitation should be about 15 years without the need of any major intervention. It is common belief that this should be true if weather conditions are not extreme. In Southeastern Ontario the standard asphalt cement grade is PG 58-28; it is often bumped to PG 64-28 for heavier traffic and even to PG 70-28 for very heavy traffic. The asphalt mixes incorporating these asphalt cement grades should be able to withstand the winter temperature down to -28°C. The 2014 winter was considered to be extremely cold in Southwestern Ontario with the temperature sometimes dropping to -25°C or even lower, sometimes for extended period of time.

There was early pavement cracking observed recently on number of roads in Southwestern Ontario. It has been noted in the last few years and particularly after the 2014 winter. Pavements that were considered to be properly designed and had good quality control/quality assurance (QC/QA) results exhibited cracking after 2 to 3 years or sometimes even after less than a year. It has been also noticed in some municipalities that mainly the pavements that incorporated PG 64-28 grade have cracked. An investigation is carried out to determine if the cracking is related to very low winter temperature and particularly if it is related to asphalt cement and what testing should be able to screen poorly performing materials.

This paper describes the case studies of three municipal roads that incorporated PG 64-28 asphalt cement and exhibited very early cracking and also so called "good roads" that also incorporated PG 64-28 but did not exhibit any cracking. The investigation included pavement visual condition inspections, QC/QA results review and pavement coring and slab cutting investigation to evaluate the depth of cracking and obtain materials for laboratory testing. The recovered asphalt cement testing included conventional PG grade verification, Multiple Stress Recovery (MSCR} and Extended Bending Beam Rheometer (eBBR) tests and Double Edge Notch Test (DENT). In addition, the recovered asphalt cements were tested for the presence of zinc and molybdenum considered by some researchers to be the indicators of the presence of recycled engine oil in asphalt cement. This paper will present the results for cracked and uncracked pavements and provide initial recommendations for updates for asphalt cement specifications.

- PG 58-28 is the standard grade for southeastern Ontario; PG 64-28 is used for heavier or slower moving traffic
- Recently some pavements have exhibited premature cracking after 2 to 3 years – some in as little as 6 months – this cracking has occurred mainly in pavements using PG 64-28 which has focused the attention of the road agencies on the quality of the asphalt used in Ontario.

- Premature pavement cracking starting with a mid-lane crack was observed for the first time in the Region of Waterloo in 2010
- Extensive field and laboratory investigation was carried out and the conclusions were not clear. Falling Weight Deflectometer (FWD) testing was done and the structural condition of the pavement was considered good. The asphalt layers, the foamed asphalt, and the granular layers met the specifications. The cracking was observed only in the asphalt wearing course.
- In 2014, an 'avalanche' of pavement premature cracking was reported. An initial thought was that such common pavement premature cracking was related to a very cold 2013/2014 winter when the temperature dropped to -25°C or less with the periods of cold temperature being relatively long.
- The cracking was observed almost exclusively on asphalt pavements that incorporated PG 64-28 asphalt cement. The Region of Niagara and City of Hamilton reported that no premature cracking was observed on projects where the base grade PG 58-28 was used. Therefore, it was concluded that the method(s) of asphalt cement modification to bump it from 58-28 to 64-28 had a dramatic negative impact on asphalt pavement performance.
- Besides aspects such as the percentage of Reclaimed Asphalt Pavement (RAP) or asphalt cement content the quality of the asphalt cement should be carefully considered at the asphalt mix design stage.
- By 2015, the Region of Waterloo observed premature cracking of pavements that were 2 to 4 years old on a total of 33 km of roadway. The Region of Waterloo commonly uses PG 64-28 on its roads. Fewer failures were observed at the Region of Niagara, likely because the Region commonly uses PG 58-28 asphalt cement and bumps the grade to 64-28 on only a few roads. However, premature cracking on Portage Road only 6 months after full depth rehabilitation raised the alarm. The Region used PG 64-28 asphalt cement on this road.
- Extensive pavement failure investigations were carried out on Katherine Street and Trussler Road in the Region of Waterloo and Portage Road in the Region of Niagara. The available QC and QA documentation was carefully reviewed. It revealed that the mix designs, the field results, and laboratory results generally met the specified requirements.
- A program of field and laboratory investigation was then developed that included pavement visual condition inspection, checking the pavement for permanent deformation (rutting), slab cutting in order to determine the depth and type of cracking, and obtaining material samples for laboratory testing. The program also included checking of the depth and quality of the underlying granular layers.
- During the investigation of a pavement in Region of Waterloo, none of the failed pavement exhibited permanent deformation. Asphalt layers cuts were typically carried out across medium severity cracks. One cut was done at a good location (i.e., with no cracks) for comparison purposes. The premature cracks in the investigated pavements were mainly observed only in the surface course. The underlying binder course was intact - clearly indicating that the cracks were not of the fatigue bottom up type.
- On all investigated pavements the quality of the granular layers was good to very good. The granular material was dry and met the specified requirements.
- The obtained samples were sent to the laboratory for asphalt mix and asphalt cement testing.
- Because of concerns about the quality of the incorporated asphalt cement it was decided that in addition to the conventional asphalt cement testing, more advanced testing was needed. It was decided to include the ash test, Multiple Stress Creep Recovery (MSCR) test, Double Edge Notched Tension (DENT) test, and the Extended Bending Beam Rheometer (Ex BBR) test.
- All laboratory testing of asphalt cement was carried on the binder recovered from the cut or cored samples. No virgin asphalt cement was available for testing. In addition the recovered asphalt cement was also tested at Queens University in Kingston for the presence of zinc, molybdenum, and carbonyls in order to check the potential that the PG 64-26 was modified by blowing and addition of recycled engine oil.
- Only the test results for Trussler Road and Portage Road were reported and both contained zinc and molybdenum - indicating that the PG 64-28 grade was likely obtained by blowing the base PG 58-28 asphalt cement and then adding recycled engine oil.

Comments:

- *As a comparison, it would have been appropriate to see the same testing done on those sections that have performed well – mainly as the authors suggest those sections constructed with PG 58-28*
- *Can a PG 58-28 not be manufactured by blending a PG 64-22 with recycled engine oil?*
- *The author states that premature cracking has occurred recently after 2 to 3 years – have there been any other changes i.e. mix design changes, increased use of RAP/RAS, etc. that have taken place in the recent past (5 to 10 years) that may have contributed to the premature cracking? REOB has been produced and sold into the asphalt market in Canada for 30 years*
- *How much zinc was present in the PG 64-28 binders?*
- *The authors show distress photos from 6 roads but state that extensive failure investigations were carried out on three; however they only report recovered binder test results from two of the roads – why?*

Laboratory Crack Testing of Hot Mix Asphalts Containing Vacuum Tower Asphalt Extender

Jason C. Wielinski, Andreas Horton, Anthony J. Kriech and Gerald A. Huber
Heritage Research Group
CTAA 2015

Vacuum Tower Asphalt Extender (VTAE) is blended with paving asphalt binder to improve low temperature properties. Some highway agencies have expressed concern regarding the use of VTAE in asphalt related to accelerated aging of the asphalt binder and premature cracking of the asphalt mixture.

The objective of this research is to analyze aging of binders formulated with different percentages of VTAE. One source of VTAE was mixed with neat asphalt binder at 0, 3, 6, 9 and 27.5 percent to generate a final PG 58-28 grade. The binders were submitted to the standard long-term aging protocol of 20 hours of Pressure Air Vessel (PAV) conditioning, as well as extended aging of 40 and 60 hours.
An HMA mixture was designed using 25 percent asphalt binder replacement from Reclaimed Asphalt Pavement (RAP). Each mixture was tested for cracking potential using the semi-circular bend test as modified by the Illinois Department of Transportation. Mixtures were aged using the standard two-hour oven aging protocol, as well as a long-term protocol for 480 hours.

This paper presents results of the aging research and recommends modifications to AASHTO M-320 including specification tests and criteria to ensure proper use of VTAE in the formulation of asphalt binder.

- The first portion of this study was to compare the properties of asphalt binder manufactured with different amounts of VTAE. Two asphalt binders, PG 64-22 and PG 52-28, were sampled from an asphalt manufacturing facility in the United States Midwest.
- Neat PG 58-28 bitumen without any VTAE was identified as the Control. It was manufactured using a blend of 20 percent PG 64-22 and 80 percent PG 52-28.
- The asphalt binders containing VTAE used the same PG 64-22 and PG 52-28 binders plus 3, 6 or 9 percent VTAE. The PG 58-28 asphalt binder containing 27.5 percent VTAE was blended with a hard penetration asphalt binder.
- Properties of all five asphalt binder formulations were determined - For the original and RTFOT condition, the stiffness ($G^*/\sin\delta$) decreased slightly with increasing percentage of VTAE. The trend is clear, but the effect is relatively minor. Going from zero to nine percent, the asphalt binder stiffness decreased 0.09 for the original condition and 0.42 for the RTFOT condition.
- At the intermediate temperature of 19°C, after long-term aging in the PAV there was a more distinct reduction in stiffness as the percentage of VTAE increased. The stiffness decreased from 4,597 kPa for zero percent VTAE to 2,602 kPa for 27.5 percent VTAE.
- For low temperature grade, m-value was the limiting parameter in all cases. The m-value was approximately the same for all the asphalt binders.
- As the percentage of VTAE increased, low temperature stiffness decreased.
- The limiting (“true”) high temperature grade of all the asphalt binders was nearly identical, either 59 or 60°C. The limiting low temperature grade (controlled by m-value) had a range from -28.4 to -30.5°C.
- Critical temperature difference (ΔT_c) is defined as the limiting temperature from the BBR stiffness minus the limiting temperature from the BBR m-value. Critical temperature difference increased proportionally with increase in VTAE content.
- According to AASHTO M320, long-term binder aging is defined as 20 hours of PAV aging. Extended aging was done on three of the five asphalt binders at 40 and 60 hours. These binders contained 0, 9 and 27.5 percent VTAE.
- Stiffness increased with extended PAV aging for all three asphalt binders. The change in stiffness was 72, 73 and 36 MPa, respectively for the 0, 9 and 27.5 percent VTAE binders.
- The failure temperature for stiffness remained relatively unchanged by the extended aging. The increase was 1.9, 1.9 and 1.8°C, respectively for the 0, 9 and 27.5 percent VTAE binders.
- The failure temperature for m-value changed significantly for all three binders. The failure temperature warmed by 14.3, 17.3 and 13.2°C, respectively for 0, 9 and 27.5 percent VTAE.
- The change in critical temperature (ΔT_c) changed significantly with the m-value. The amount of change was similar for all three asphalt binders. The difference (ΔT_c) changed 12.4, 15.4 and 11.4°C, respectively for the 0, 9 and 27.5 percent VTAE binders.
- The second portion of this study focuses on the engineering properties of HMA made with the resulting PG 58-28 binders. HMA was designed to meet Illinois DOT specifications. The design contained RAP sufficient to require a change in grade from PG 64-22 to PG 58-28.

- The HMA design was done with 25 percent ABR using PG 58-28, one grade lower than the usual grade of PG 64-22. The initial design was done using PG 58-28 with 0 percent VTAE. The other two formulations of PG 58-28 containing 9 and 27.5 percent VTAE were substituted into the mixture for evaluation of cracking behavior.
- The Semi-Circular Bend Test – IL was developed at the University of Illinois as an indicator of low temperature cracking behavior. Test specimens are made from gyratory compacted specimens compacted to yield test specimens with seven percent air voids. Two disks, 50 mm thick are cut from a 150 mm tall gyratory specimen. Each disk is cut into two semi-circles to yield a total of four test specimens. Each specimen is cut with a 15 mm deep notch.
- The test, which uses time–temperature superposition to simulate low temperature performance, is performed at 25°C with an actuator rate of 50 mm/minute; a high rate of loading. Load and displacement are measured during the test.
- Energy to cause failure is measured as the area under the curve. Slope of the failure curve is a measure of brittleness.
- The test defines Flexibility Index (FI) as a parameter that considers both the amount of energy to cause failure and the rate of strain energy release (slope) during fracture.
- The loose mixture was short-term oven aged at 135°C as specified by AASHTO R30. A second set of specimens were prepared in the same fashion except these compacted specimens were long-term oven aged. The long-term aging protocol in AASHTO R30 requires compacted specimens to be aged in an oven at 85°C for 120 hours. An extended version of AASHTO R30 was used in which the specimens were aged at 85°C for 480 hours.
- For both short-term and long-term aged specimens, the Work of Fracture decreased as percentage of VTAE increased. For the short-term aged specimens, the Work of Fracture decreased from 2026 kN-mm (Joules) for 0 percent VTAE to 1683 and 1191 Joules, respectively for 9 and 27.5 percent VTAE. For the long-term aged specimens, the Work of Fracture was 1611, 1410 and 1123 Joules, respectively.
- Extended long-term aging reduced the work of fracture in all of the mixtures regardless of VTAE content. For example, the 0 percent VTAE mixture decreased from 2026 to 1611 Joules, a reduction of 20 percent. For the 9 percent VTAE mixture the percentage decrease was 16 percent and for the 27.5 percent VTAE the decrease was 6 percent. Hence mixture with an increased percentage of VTAE did not experience as large a decrease in energy as a mixture with lower VTAE.
- For the short-term aged specimens, the slope of the fracture curve is not related to the percent of VTAE. The 9 percent VTAE samples had a higher slope than the 0 percent samples, -2.26 compared to -1.64 but the 27.5 percent VTAE mixture had a slope of -1.00 indicating it to be a more ductile material. The higher the slope, the faster the release rate of energy meaning the more brittle the material is behaving.
- For the short-term specimens, Flexibility Index did not provide a consistent view of the effect of VTAE. The Flexibility Index for the 9 percent VTAE mixture was lower than that of the 0 percent VTAE mixture. However; Flexibility Index for the 27.5 percent VTAE mixture is approximately the same as the 0 percent mixture.
- All mixtures subjected to extended long-term aging became more brittle (i.e., the slope of the failure curve became steeper). The 0 percent VTAE mixture slope changed from -1.64 to -6.59. Likewise for the 9 percent VTAE mixture the slope changed from -2.26 to -7.17. For the 27.5 percent VTAE mixture there is only one data point to compare but it appears to have become much steeper, -14.81 compared to -1.00.
- PG 58-28 asphalt binder prepared with increasing VTAE content were purposely fixed to produce the high temperature grade of 58°C to determine how other rheological properties would be affected. Short-term aging did not significantly affect these high temperature properties as VTAE concentrations were increased.
- Increasing the amount of VTAE in asphalt binder decreased low temperature stiffness of asphalt binder. As the percentage increased, the low temperature stiffness decreased in direct proportion to the amount of VTAE added.
- Critical temperature difference (ΔT_c) is defined as the limiting temperature for BBR stiffness minus limiting temperature for BBR m-value. As the percent VTAE increased, the ΔT_c increased.
- Stiffness of all the asphalt binders increased slightly with increased PAV aging time. The stiffness increase was not large; hence, the increase of stiffness that occurred was not highly sensitive to the amount of PAV aging time.
- Change in BBR stiffness with increased PAV aging did not depend strongly on the percentage of VTAE present in the asphalt binder. Neat asphalt binder stiffness increased about 35 percent with three times the PAV aging. Nine percent VTAE binder increased 50 percent and 27.5 percent VTAE increased 40 percent.

- Relaxation (m-value) was sensitive to the amount of PAV aging time. As PAV time increased the m-value decreased significantly. All three asphalt binders (0, 9 and 27.5 percent VTAE) had a similar absolute decrease in the m-value, 0.069, 0.064, and 0.068 respectively.
- The critical temperature difference, ΔT_c, is significantly affected by the amount of extended PAV aging regardless of the percentage of VTAE present. Each of the three binders had significant increases in ΔT_c, of 12.4, 15.4 and 11.4°C respectively for 0, 9 and 27.5 percent VTAE.
- As the percentage of VTAE increases, the work of fracture decreases. Mixture with 27.5 percent VTAE required about 55 percent of the energy required to break mixtures with no VTAE.
- Brittleness of the mixtures as measured by slope of the failure curve is not consistent with the percentage of VTAE present in the asphalt binder. Mixture that had 27.5 percent VTAE had a lower slope than mixture with no VTAE. The mixture containing 9 percent VTAE had a steeper slope.
- The Work of Fracture (i.e., energy to cause fracture) was lower for all mixtures after extended long-term aging regardless of the percentage of VTAE present.
- After extended aging the work of fracture expressed as a percentage of the work of fracture for short-term aged mixture is related to the amount of VTAE present. For example, the 0 percent VTAE Work of Fracture for long-term aging was 80 percent of the Work of Fracture for the short- term aged specimens. For the 9 and 27.5 percent binders, respectively, the percentages were 84 and 94 percent. The higher the percentage of VTAE present, the less impact extended aging had on the work of fracture.
- All asphalt mixtures, regardless of percentage of VTAE, become more brittle after extended long- term aging. For mixtures containing no VTAE the slope increased to -6.59 from -1.64. For 9 and 27.5 percent VTAE, the respective numbers were -7.17 from -2.26 and -14.81 from -1.00. The increase in brittleness was in direct proportion to the percentage of VTAE present.
- Recent proposals have been made to control ΔT_c after 20 hours of PAV aging (M320), to a maximum of 5.0°C as a method of controlling the amount of VTAE that can be added. Based on the results of this study, there is no need for extended aging to control the amount of VTAE. For the materials used in this study, ΔT_c for 9 percent VTAE was 4.8°C, near the proposed maximum of 5°C.

Comments:

- *What would the ΔT_c be if the 25 percent of RAP binder was added to the blend?*

asphaltinstitute.org