

54 Petersen Asphalt Research Conference

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Hilton Garden Inn

Laramie, Wyoming

July 17-19, 2017







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Welcome



Howdy and Bonjour!

As the conference chairman, it is my immense pleasure and privilege to welcome you to Laramie, Wyoming for the 54th Petersen Asphalt Research Conference (PARC).

The petroleum world, and particularly the asphalt world, is constantly changing. Huge variation in oil prices, new crude oil sources and an increased rate of recycling have considerably impacted asphalt crude selection, production and quality. The development of models, analytical tools and test methods for asphalt materials has never been more important. The overall goal is to make ever more sustainable and cost effective

pavements. While these changes present us with many challenges, they also give us opportunities for discovery and progress in our field of study.

As THE widely recognized forum for sharing new ideas and fresh knowledge, the PARC is especially interested in the presentation and discussion of research projects in progress.

The call for abstracts was very successful! The 2017 Petersen Asphalt Research Conference is proud to host over 30 presentations from around the globe. This offers a unique opportunity to expand your perspectives in both fundamental and cutting edge asphalt research. All of us, as international colleagues, can share and discuss applicable solutions to our real world problems.

"Chemistry matters" has always been the primary focus for this conference. Presentations reflect this basic premise, leading us toward innovative answers for our most difficult pavement questions. This year, you are invited to participate in four sessions concerning these topics: Asphalt Analytical Characterization, Asphalt Modification, Binder Testing and Recycling.

As always in Wyoming, visitors enjoy our friendly western hospitality. Home to the University of Wyoming, Laramie enjoys an atmosphere of curiosity and a quest for increased knowledge. On high plains surrounded by majestic mountains at the eastern edge of the Rockies, you are in a rare and beautiful location. Nearby adventures await you in every direction.

Welcome to our friends from near and far! I hope you'll have a great time at the PARC, enjoying the west "Wyoming style".

Jean-Pascal (JP) Planche Vice President, Asphalt and Petroleum Technologies Western Research Institute Laramie, Wyoming



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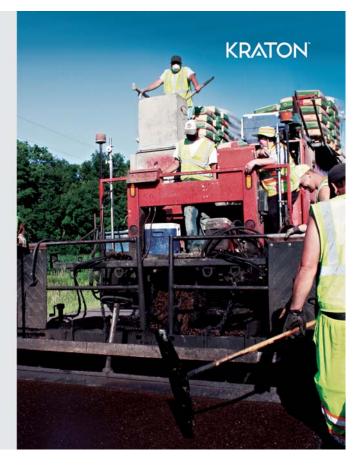
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54th Petersen Asphalt Research Conference

Presented by Western Research Institute

University of Wyoming Conference Center • July 17-19, 2017 • Laramie, Wyoming

Hilton Garden Inn and UW

Monday, July 17

Registration

7:30 a.m.

10:00-10:30

Break

7.50 a.m.	(Pick up conference materials)	Conference Center
8:00-8:30	Welcome and Opening Remarks	Jean-Pascal Planche and Joe Rovani Western Research Institute
Session 1	- Asphalt Analytical Characterization	
	Session Chair: Ashley Buss, Iowa State University	
8:30-9:00	Molecular Weight Distribution of Asphalt Binders from Laser Desorption Ionization (LDI) Technique and its Relationship to Relaxation Spectra	Akshay Gundla and B. Shane Underwood Arizona State University
9:00-9:30	MRI of Bitumen Emulsion Drying in Porous Medium	Marie Goavec, Pamela Faure, Stéphane Rodts, Emmanuel Keita and Philippe Coussot Université Paris-Est, Laboratoire Navier, Champs Sur Marne
		Vincent Gaudefroy IFSTTAR, MIT, Nantes
9:30-10:00	Analyzing a Boil Test to More Effectively Evaluate Moisture Damage	Andrew LaCroix InstroTek

Monday, July 17 (Continued)

10:30-11:00	Asphalt Base Variability: A Need and an Opportunity to Develop Innovative Characterization Tools	Jean-Pascal Planche, Joe Rovani and Fred Turner Western Research Institute Frederic Delfosse, Sabine Largeaud and Ivan Drouadaine Eurovia
11:00-11:30	Separation and Characterisation of Asphaltenes Based on Solubility Parameters and Their Influence on Basic Bitumen Properties	<u>Dawid D'Melo</u> , Rohit Gupta and Sathish Subramani Shell India Markets Richard Taylor Shell International Petroleum Co.
11:30-12:00	Use of Ion-Mobility-Mass Spectrometry for Understanding Asphalt Aging and Composition	Ashley Buss, Nacu Hernandez, R. Christopher Williams and Eric Cochran <i>Iowa State University</i> Andrew Hanz and Mary Ryan <i>Mathy Construction</i>
12:00-1:00	Lunch	
1:00-1:30	Hole Distribution in Asphalt Films After the Dewetting of Asphalt Films	Nibert Saltibus Sam Houston State University Nazimuddin Wasiuddin LA Tech University

Monday, July 17 (Continued)

Session 2 - Asphalt Modification

Session Chair: C.J. DuBois, DuPont Performance Materials

1:30-2:00	Chemical Effects from Epoxidized Plant Derived Oil Materials on Asphalt Binders Through Mass Spectrometry Utilizing Ion Mobility	Joseph Podolsky, Mohamed Elkashef, Nacu Hernandez, R. Christopher Williams and Eric Cochran Iowa State University
2:00-2:30	Determination of Recycled GTR Content on Rubberized Asphalt Mixtures Using an Automated Extraction Method	Ann Baranov InfraTest USA Joe Pitlik S.T.A.T.E. Testing, LLC
2:30-3:00	Hybrid Polymer Modifiers for Improved Blending in Asphalt	C.J. DuBois, James de Garavilla, George Prejean, Sara Reynoso and Felipe Sanchez DuPont Performance Materials
3:00-3:30	Break	
3:30-4:00	Experience from the Round Robin Testing on Selected Functional Bitumen Characteristics for the Second Generation of European Bitumen Standards	Guillaume Rousseau ValoChem SARL, France Jan Valentin, Ondrej Dasek, Vaclav Valentin, Vaclav Neuvirt, Radek Cerny and Petr Bures Czech Republic
4:00-4:30	Fuel Resistance Binder	Mohammed Memon Consultant Areeka Memon Virginia Tech. Medical Student
4:30-5:00	The Use of Gilsonite to Increase Performance Grade Properties of Asphalt Binders	Geoffrey Rowe Abatech Bill Britton American Gilsonite Company

Tuesday, July 18

Session 2 - Asphalt Modification (Continued)

Session Chair: C.J. DuBois, DuPont Performance Materials

8:00-8:30	High Performance Thin Bituminous Roofing Membranes	Erica Jellema and Sjaak Damen Kraton Polymers
8:30-9:00	The Circular Economy of Asphalt, New Additives from Old Resources	Domenic Di Mondo GreenMantra Technologies
9:00-9:30	Performance Modeling of a Highly Modified Asphalt Pavement	Robert Kluttz Kraton Polymers
		Richard Kim North Carolina State University
		Raj Dongre Dongre Lab Services Inc.
		Buzz Powell NCAT
		Richard Willis NAPA

Session 3 - Binder Testing

Session Chair: Jack Youtcheff, FHWA (TFHRC)

9:30-10:00	Evaluation of MSCR Test Specification Parameters and Their Relationship to Pavement Performance	Raj Dongre Dongre Laboratory Services Inc.
		Jack Youtcheff FHWA (TFHRC)
10:00-10:30	Break	
10:30-11:00	Testing of Ontario's Asphalt Binders to Improve Cracking Performance	<u>Heather Bell</u> and Pamela Marks Ontario Ministry of Transportation
11:00-11:30	Innovative Roofing Asphalt Characterization: A Simplified Approach to Routine Rheological Testing	John Casola Malvern Instruments

Tuesday, July 18 (Continued)

11:30-12:00	Effect and Significance of Ambient Pressure on the Aging of Asphalt Binders	Dave Anderson Consultant and Penn St. University Raymond Bonaquist Advanced Asphalt Technologies James Rosenberger Penn State University
12:00-1:00	Lunch	
1:00-1:30	Asphalt Binder Quality Assurance Test Method (ABQT)	Raj Dongre Dongre Laboratory Services Inc. Jack Youtcheff FHWA (TFHRC)
1:30-2:00	Study of Factors Affecting Low Temperature Strength and Performance of Asphalt Materials	Sang Soo Kim, Mohsen Radi and Moses Akentuna Ohio University
2:00-2:30	Measurement, Tolerance and Effect of PAV Pan Warpage and Film Thickness on Physical Properties of PAV Residue	<u>Dave Anderson</u> Consultant and Penn St. University
2:30-3:00	Inter-Laboratory Study of Performance Grade Testing of Crumb Rubber Modified Asphalt Binders	Sallie Houston & Gary Houston VSS Emultech Shakir Shatnawi Shatec Engineering Consultants Shauna Teclemariam US Oil Refining
3:00-3:30	Break	
3:30-4:00	Understanding Cracking Performance with Linear Visco-Elastic Parameters	Geoffrey Rowe Abatech

Tuesday, July 18 (Continued)

Session 4 - Recycling - Effects on Chemical, Physical Properties and Performance - Rejuvenation Mechanisms

Session Chair: Hassan Tabatabaee, Cargill Industrial Specialties

4:00-4:30	Materials for Extreme Rehabilitation Project	Frank Fee Frank Fee, Inc.
4:30-5:00	Mix Performance Improvement of HMA Incorporating High Amounts of RAP Through the Addition of Bio-additives	Zahra Sotoodeh-Nia, Nick Manke, Chris Williams and Eric Cochran Iowa State University
		Emmanuel Chailleux, Erik Bessmann, Pierre Hornych and Juliette Blanc IFSTTAR, France
		Davide Lo Presti and Ana Jimenez University of Nottingham, UK
		Laurent Porot Arizona Chemicals, Netherlands
		Jean-Pascal Planche and Ryan B. Boysen



Tuesday Evening Dinner

Oland

Western Research Institute
Simon Pouget and François

EIFFAGE Infrastructures, France

University of Wyoming Marian H. Rochelle Gateway Center

5:45 Social Hour (Cash Bar) 6:45 Dinner

Wednesday, July 19

Session 4 - Recycling - Effects on Chemical, Physical Properties and Performance - Rejuvenation Mechanisms (Continued)

Session Chair: Hassan Tabatabaee, Cargill Industrial Specialties

8:00-8:30	The Crossover Temperature: Significance and Application Towards Engineering Balanced Recycled Binder Blends	Lorena Garcia Cucalon, Fawaz Kaseer, Edith Arambula- Mercado Texas Transportation Institute Amy Epps Martin and Charles Glover Texas A&M University Nathaniel Morian, Sara Pournoman and Elie Hajj University of Nevada Reno
8:30-9:00	Determine the Correct Mixing and Compaction Temperatures for Using High RAP Content in HMA	<u>Huachun Zhai</u> , Alejandro Rosales and Erin Russell <i>Idaho Asphalt Supply</i>
9:00-9:30	Influence of RAP and RAS Addition on the Mixture and Binder Performance - Three Years of Loading History and Evaluation	Adrian Andriescu and X. Li SES Group and Associates Jack Youtcheff FHWA (TFHRC)
9:30-10:00	Chemical Characterization of a Rejuvenated Binder Using a Soybean-Derived Material Through Thermal Desorption / Gas Chromatography-Mass Spectrometry (TD/GC-MS)	Mohamed Elkashef, R. Christopher Williams and Eric Cochran Iowa State University
10:00-10:30	Break	
10:30-11:00	Thermal Analysis and Colloidal Stability of Asphalt Modified with Engineered Bio-Based Additives	Hassan Tabatabaee, Todd Kurth and Tony Sylvester Cargill Industrial Specialties

Wednesday, July 19 (Continued)

11:00-11:30 Compatibility of Recycled Binder Blends with Recycling Agents: Rheological and Physicochemical Evaluation of Rejuvenation and Aging Processes

Lorena Garcia Cucalon, Fawaz Kaseer and Edith Arambula-Mercado Texas Transportation Institute Gayle King GHK, Inc. Amy Epps Martin and Charles Glover Texas A&M University Fred Turner

Western Research Institute

11:30-11:45 *Closing Remarks*





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Session 1 - Asphalt Analytical Characterization

Molecular Weight Distribution of Asphalt Binders from Laser Desorption Ionization (LDI) Technique and its Relationship to Relaxation Spectra

Akshay Gundla and B. Shane Underwood Arizona State University

The SHRP studies of early 1990's used Size Exclusion Chromatography, and High Performance Liquid Chromatography to obtain molecular weight distributions (MWD) of asphalt cement and vapor phase osmometry to obtain number average molecular weight. In this study, an alternative approach for mass spectroscopy, laser desorption ionization (LDI) has been used to obtain MWD and molecular weights of asphalt cements. Five different neat and polymer modified asphalt cements including are used. For all materials, MWD is determined before and after laboratory aging and are compared using the polydispersity index, PDI. The aged asphalt cements have a lower PDI than the unaged samples, indicating less heterogeneity in the aged materials. Also, polymer modified asphalts have lower PDI than neat asphalts. Next, the linear viscoelastic parameters are characterized by temperature and frequency sweep tests. The results are interpreted using the rheological index, R, from the Christensen – Anderson model since this parameter is directly proportional to the width of the relaxation spectrum. The limited data in this study shows the efficacy of using LDI and finds that the PDI, or the MWD in general, had less impact on R of the neat asphalts in comparison to polymer modified binders.

MRI of Bitumen Emulsion Drying in Porous Medium

<u>Marie Goavec</u>, Pamela Faure, Stéphane Rodts, Emmanuel Keita, and Philippe Coussot (1); and Vincent Gaudefroy (2)

(1) Université Paris-Est, Laboratoire Navier, Champs sur Marne; (2) IFSTTAR, MIT, Nantes

The significant amount of water initially contained in cold mix asphalts, composed of aggregates and bitumen emulsion, delays the strengthening of the material; it is therefore crucial to understand how the mix dries. We observed that the drying of bitumen emulsion in a porous medium by an airflow at the free surface of the sample is thirty times slower than if it were water. To identify the underlying drying mechanisms, we imaged water and bitumen separately and measured their spatial distribution in a porous sample during drying using magnetic resonance imaging (MRI). It appears that a dry front, an area without water, progresses inwards from the free surface, while the pores below remain saturated with water. During this process, the bitumen distribution surprisingly remains homogenous throughout the sample, i.e. there is no bitumen transport or accumulation. We show that the very low drying rate of the cold mix asphalt is only partly explained by the water vapor diffusion towards the free surface over the increasing length of the dry front. An additional phenomenon must be taken into account, namely the decreasing drying rate of the bitumen emulsion itself as it concentrates.

Analyzing a Boil Test to More Effectively Evaluate Moisture Damage

Andrew LaCroix

InstroTek

Currently, one method of evaluating moisture sensitivity of asphalt mixtures is the boil test (ASTM D3625), in which a sample of loose mixture is boiled in water for 10 minutes to observe visual stripping. Boiling accelerates the breaking of chemical bonds between the asphalt binder and the aggregate and can quickly provide an indication on compatibility of binder and aggregates used in the mixture. Since visual inspection of the mixture is not reliable, this presentation provides a more advanced test method for analyzing the binder and aggregate compatibility after a boil test.

Asphalt Base Variability: A Need and an Opportunity to Develop Innovative Characterization Tools

<u>Jean-Pascal Planche</u>, Joe Rovani, and Fred Turner (1); Frederic Delfosse, Sabine Largeaud, and Ivan Drouadaine (Eurovia)

(1) Western Research Institute; (2) Eurovia

The refining industry has been going through many phases of rationalization to maximize the crude oil supply flexibility while matching production models and tools, and meeting product market demands. For asphalt users like Eurovia, this has triggered concerns about the quality and consistency of the delivered asphalt binders, especially as current specifications appear insufficient to ensure satisfactory performance of the finished products.

In this context, the search for relationships and correlations between asphalt binder and mixture properties and the pavement performance has become very relevant. This study was dedicated to identifying and quantifying such relationships. It focused on unmodified binders from various origins and involved a standard asphalt mix, designed with these binders and one aggregate type. The mix characterization covered various performance characteristics from rutting to fatigue and thermal cracking resistance.

The binder compositional analysis included the advanced fractionation method SAR-ADTM that separates asphalt in multiple fractions, including three polarity dependent asphaltene subfractions. A chemometrics analysis using a new software was performed to identify and develop the correlations. Both tools were developed by the Western Research institute.

Significant correlations were found tying binder chemical composition metrics to binder mechanical characteristics and asphalt mix characteristics, and proving the concept viability.

Separation and Characterisation of Asphaltenes Based on Solubility Parameters and Their Influence on Basic Bitumen Properties

<u>Dawid D'Melo</u>, Rohit Gupta, and Sathish Subramani (1); Richard Taylor (2) (1) Shell India Markets; (2) Shell International Petroleum Company

Recent studies have investigated a method to separate the heptane insoluble asphaltenes into sub-fractions based on the solubility parameters of the asphaltenes. The current study utilised a hot extraction technique using cyclcohexane and toluene to separate asphaltenes into two sub-fractions, ACYC and ATOL respectively. These asphaltene sub-fractions were spectroscopically analysed, using X-ray diffraction and nuclear magnetic resonance, for structural and compositional details. The ATOL fraction was found to have a higher concentration of electronegative atoms as compared to the ACYC fraction, potentially explaining its insolubility in cyclohexane. Structurally, the ordering of molecules obtained using the two solvents were markedly different with different asphaltene stacking parameters and intermolecular distances. The influence of the asphaltene sub-fractions on basic bitumen properties, as a function of concentration, also showed that there was more interaction between maltenes and ATOL as compared to ACYC, leading to bitumen with a higher stiffness. The chemistry of asphaltene sub-fractions on performance of bitumen will be further investigated to enable tailoring of bitumen from the molecular level, allowing for bitumen to be manufactured with specific properties and maintaining quality.

Use of Ion-Mobility-Mass Spectrometry for Understanding Asphalt Aging and Composition

<u>Ashley Buss</u>, Nacu Hernandez, R. Christopher Williams, and Eric Cochran (1); and Andrew Hanz, Mary Ryan (2)

(1) Iowa State University; (2) Mathy Construction

Analytical chemistry innovations have advanced the ability to analyze complex chemical composition and molecular structures. The evolution of analytical chemistry applied to asphalt materials is summarized and new techniques are investigated for use with asphalt materials. The experimental plan includes analysis of test results from two asphalt binders in various aging states using a Waters Synapt G2 ion mobility-mass spectrometry (IM-MS) with an atmospheric solids analysis probe. Binders were tested in unaged and aged states, including: rolling thin film oven, 20-hour pressure aging vessel (PAV) and 40-hour PAV aged. The goal of the study is to determine if IM-MS ASAP techniques provide useful information about the chemical structure or components of asphalt materials as the asphalt is aged. The software package PetroOrg is used to analyze the IM-MS data to assign peaks, analyze mass to charge ratios with ion drift time, generates plots of double bond equivalents vs. carbon number, Kendrick mass and van Krevelen diagrams. Each binder for the four aged-states were fractionated into saturates, aromatics, resins and asphaltenes (SARA). The SARA fractions for the unaged- to 40-hour PAV aged-binders are compared with the IM-MS molecular mapping to estimate the chemical changes as the binder is aged.

Hole Distribution in Asphalt Films After the Dewetting of Asphalt Films

Nibert Saltibus (1); Nazimuddin Wasiuddin (2) (1) Sam Houston State University; (2) LA Tech University

This study makes use of a unique dewetting-based moisture damage test procedure consisting of a moisture conditioning procedure and quantitative analysis of the dewetting with the use of a microscope and the NI Vision (2012) software. A macro-scale dewetting analysis procedure was developed that considers all the holes in the sample. Three main asphalts, and two anti-strip additives were utilized. To further understand the dewetting phenomena within the asphalt films, the hole distribution in the film was analyzed based on the pattern and on the area density within the film. Therefore, the objectives of this study were to analyze the distribution of the dewetted holes in some of the dewetted asphalt films with approximately the same film thickness, pH, asphalt type, polymer modified asphalt, and additives.

Session 2 - Asphalt Modification

Chemical Effects from Epoxidized Plant Derived Oil Materials on Asphalt Binders Through Mass Spectrometry Utilizing Ion Mobility

<u>Joseph Podolsky</u>, Mohamed Elkashef, Nacu Hernandez, R. Christopher Williams, and Eric Cochran *Iowa State University*

Asphalt is a co-product derived during the refining of crude petroleum heavy fractions. Asphalt binders produced at refineries are becoming stiffer due to an ever-increasing demand for more expensive lighter fraction products such as gasoline, and jet fuel. To increase output of lighter fractions and heavy gas oils, refineries have gone through upgrades producing higher amounts of stiffer asphalt binders. Stiff asphalt binders are residuals produced in vacuum distillation (VD), solvent deasphalting (SDA), and residuum oil super critical extraction (ROSE) units to increase the output of more valuable heavy oil components. Materials used to soften/repair stiff asphalt binders for use in paving roadways are known as fluxes. Recent work at Iowa State University has shown that there is great potential for epoxidized plant oil materials to improve rheology of stiff binders and make them feasible economic alternatives to commonly produced base binders. To better understand the chemistry behind rheological improvements due to addition of epoxidized plant oil materials, several control binders (stiff and soft) as well as modified control binders with dosages ranging from 0.75% to 15% by weight of the binder were tested and analyzed using ion mobility mass spectrometry.

Determination of Recycled GTR Content on Rubberized Asphalt Mixtures Using an Automated Extraction Method

Ann Baranov (1); Joe Pitlik (2) (1) InfraTest USA; (2) S.T.A.T.E. Testing, LLC

S.T.A.T.E. Testing, LLC performed a blind study in conjunction with Asphalt Plus to evaluate the accuracy of the new Automated GTR Extraction method to determine the rubber content of a wide variety of mixtures. 19 mixtures were produced with a wide spread of rubber contents (0 to 14%) and mixing procedures were varied to include variables such as over-aging and over-heating of the material to alter the uptake of AC into the rubber. No data was provided to S.T.A.T.E. Testing, LLC prior to disclosure to Asphalt Plus of testing results. While data analysis is still underway, the correlation between batched rubber content and measured rubber content exceeds 85% prior to elimination of outliers. Detailed breakdown of the data is under way. Follow-on work will incorporate lessons learned during the testing to refine the testing technique and prevent the loss of rubber from the top of the testing chamber. Final analysis will include a formal procedure for specimen preparation and machine settings to promote best practices and rapid turnaround of samples for analysis and accurate quantification of rubber content of HMA.

Hybrid Polymer Modifiers for Improved Blending in Asphalt

<u>C.J. DuBois</u>, James de Garavilla, George Prejean, Sara Reynoso, and Felipe Sanchez DuPont Performance Materials

To improve the process speed to manufacture polymer modified asphalt (PMA), a series of 12 oils of different chemical compositions in 45 different states were compounded with different reactive elastomeric terpolymers (RET). The goal was to develop hybrid polymer modifiers that would dissolve faster in asphalt, at lower temperature, and with less shear than traditional polymer modifiers. Stability of the raw material, speed of dissolution into different asphalts at various times and temperatures, and performance properties in PMA blends will be discussed. In addition to providing an improved PMA product, these improvements are targeted to meet sustainability goals such as reduced dependence on fossil fuels, improvements in productivity, protection of the environment, creation of sustainable solutions, and growth in emerging markets while also providing a commercially viable product at a reduced cost per ton of modified asphalt.

Experience from the Round Robin Testing on Selected Functional Bitumen Characteristics for the Second Generation of European Bitumen Standards

<u>Guillaume Rousseau</u> (1); Jan Valentin, Ondrej Dasek, Vaclav Valentin, Vaclav Neuvirt, Radek Cerny and Petr Bures (2)

(1) ValoChem SARL, France; (2) Czech Republic

The second generation of European standards for bituminous binders will include in larger extent performance-related characteristics, which have been gradually implemented in the practice. With respect to their regular use, these characteristics have been seen as a new requirement. Nevertheless, the long-term experience with their determination and interpretation has been missing. Due to the fact, that especially polymer modified binders (PMB) have become a standard product for all types of asphalt mixtures, it is necessary to set the basic requirements for particular binders specified in the standard and based on their characteristics. The wide data collection overall Europe has been in process for several years. The main expectation is that laboratory data will give sufficient information for the proposal of first set of required parameters, which could be included in the standards. The Czech Republic has started to collect the experience and values based on round robin tests as well. This activity has been supported by SFDI (State fund for traffic infrastructure) and RSD (Road and motorway administration). The first stage of the project was launched in 2014 and 3 laboratories have tested two binders (paving grade and PMB) from different bitumen producers - virgin, after short term ageing (RTFOT) and long term ageing (RTFOT + PAV). Traditional analyses and performance analyses were used. The second stage of the project followed in 2015 and included already six laboratories. Four binders have been tested (one paving grade, three PMBs) in a similar frame as in the year 2014. Some conditions (e.g. testing temperature on DSR, BBR test etc.) have been added. The last stage in 2016 included six laboratories again and four binders (one paving grade, three PMBs). The last stage of the project included further extension of testing temperatures on DSR, different temperatures and further 10 shear stress set by MSCR test (8 kPa) have been added newly.

The evaluation of all results has led to the decision for wider data collection of all polymer modified binders, which are used in the Czech Republic. This data collection should be based on the draft of requirements, which can be expected for EN 14023. These data will be collected from all producers/traders on the market anonymously and evaluated after 3 years collection from a team of experts. This evaluation will lead to determining requirements for the standard specifications of PMB binders. If it will be decided in the Czech Republic that the performance-related characteristics shall be used in specifications of paving grade binders, a similar approach for data collection could be performed.

Fuel Resistance Binder

<u>Mohammed Memon</u> (1); Areeka Memon (2) (1) Consultant; (2) Medical Student, Virginia Tech.

Fuel Resistant Binder was designed to overcome the toxic issues associated with coal-tar while providing superior resistance to non-aromatic petroleum fuels and aliphatic solvents such as are found at airfields, truck depots, loading facilities, parking lots, and auto service centers. This binder generates an aggressive level of stiffness at high temperatures, and maintain cracking resistance at low temperatures. Resistant Binder can withstand enormous "point loads" exerted by aircraft or heavy vehicles as well as withstands premature aging or oxidation. This binder generates its strength in the pavement or structure by utilizing our specialized cross-linking system combining the adhesion properties of concentrated PMA and carbon black network structure of crumb rubber. The resistant binder can protect the sub-soils underneath from petroleum fuel contamination.

This kind of binder has an ability to resist fuel lies in a special blend of stone as an aggregate or other related recycled products like roofing shingles etc as a binder ingredients which also help give the modified binder an added strength when it is put under pressure by solvation or thermal or mechanical stress. The binder so produced

The Use of Gilsonite to Increase Performance Grade Properties of Asphalt Binders

can effectively be used in variety of applications. It has shown special ER, with a strong structural network.

Geoffrey Rowe (1); Bill Britton (2) (1) Abatech; (2) American Gilsonite Company

Historically, Gilsonite has been used with claims that it does not affect properties associated with thermal cracking. Often these claims have not been developed with theoretically designed experiments but rather on anecdotal evidence or empirical observations. Since the early 1990's the adoption of the Superpave specifications have led to a more considered approach to the use of modifiers in asphalt materials and much of the older testing with Gilsonite and empirical evidence of performance is typically considered less reliable. Consequently, as a result of this additional investigations have been performed to assess how Gilsonite can be used within this newer specification framework. Additional work was conducted to ascertain the performance change to a PG binder grade by adding amounts of Gilsonite Superpave Modifier (GSM) to binders from various sources and assess the changes in performance. The three binders selected for study represented materials used on the west coast and mid/central USA. Information on the binders chosen are given below. The work conducted enables the effect of GSM to be assessed as a binder modification tool in the production of binders to the AASHTO M320 and M332 specifications. In addition, information is presented on elastic recovery, ductility and assessment of ΔT_c .

High Performance Thin Bituminous Roofing Membranes

<u>Erica Jellema</u> and Sjaak Damen Kraton Polymers

Roofing membranes are to provide a building with a secure and durable waterproofing system. There are several different roofing products on the market, all with their own benefits.

In this work we report the development of bituminous roofing membranes combining benefits of both single-ply roofing materials and bituminous roofing felts. Compared to standard bituminous roofing, the new material has a flow resistance, a cold bend performance and a breaking/tensile strength well outperforming standard modified roll roofing. These improved properties allow making the felt 50% thinner and thus at least 50% lighter weight.

The key of this development is in the SBS content; from a standard level of 10-12% in bitumen it is increased to 20-25% of SBS. With a standard SBS grade this would lead to too high blend viscosities, however, a new SBS polymer has been developed to enable processing at such concentrations, while it can also be used with standard paving asphalt. The development and properties of the new materials will be discussed in this contribution.

The Circular Economy of Asphalt, New Additives from Old Resources

Domenic Di Mondo

GreenMantra Technologies

The ability to recover and generate new products from a resource at the end of its initial service life is the core definition of the circular economy. This beneficial reuse moves us past the "take, make, and dispose" industrial model into a more sustainable society. In the asphalt industry this not only means integration of recycled asphalt pavement or shingle, but also the use of on-purpose materials produced from spent resources such as waste plastic, glass, or rubber. One example is the role higher molecular weight polyethylene and polypropylene waxes derived from spent plastic products, such as containers and bags, can play in polymer modified asphalt (PMA). The presentation will review the evaluation of these products in PMA roofing formulations to improve resistance to deformation caused by force or heat, and the use of these products to adjust various physical and chemical properties of asphalt. The perception that cost and performance are sacrificed for environmentally responsible products is no longer true. Rather, improved performance and cost savings are the outcome from either using or designing products focused on the circular economy.

Performance Modeling of a Highly Modified Asphalt Pavement

Robert Kluttz (1); Richard Kim (2); Raj Dongre (3); Buzz Powell (4); and Richard Willis (5) (1) Kraton Polymers; (2) North Carolina State University; (3) Dongre Lab Services; (4) NCAT; (5) NAPA

This presentation reports on performance modeling of a thin, highly modified asphalt pavement versus conventional asphalt pavement at the National Center for Asphalt Technology. The sections were subjected to 20 million ESALs over a period of five years and the thinner, highly modified section outperformed the others in both permanent deformation and cracking. The performance results demonstrate that substantial thickness reduction is possible while retaining and even improving long term performance.

Equally important, material properties of highly modified mixes can be used to adjust the global calibration coefficients in AASHTOWare/r/ Pavement ME Design software so that appropriate pavement thickness can be determined through rational design. This methodology has been put into practice and so far the performance results on commercial projects have validated the design predictions.

The only significant distress in the highly modified pavement was hairline top down cracking. As ME Design does not yet adequately address top down cracking, additional modeling has been conducted with the FlexPAVE/tm/ program, a 3D finite element model developed at NCSU. The FlexPAVE program was successfully used to predict fatigue cracking performance trends of over 50 pavements. The presentation will compare performance predictions of both tools with field performance.

Session 3 - Binder Testing

Evaluation of MSCR Test Specification Parameters and Their Relationship to Pavement Performance

Raj Dongre (1); and Jack Youtcheff (2) (1) Dongre Laboratory Services Inc.; (2) FHWA (TFHRC)

The MSCR test was originally developed to quantitatively determine the differences in performance of well formulated (cured and cross-linked) and "dump and stir" methods of producing polymer modified asphalt (PMA) binders. The success of the test led to its implementation as a specification for asphalt binders (AASHTO M332). However, many State DOTs are concerned and reluctant to adopt the new MSCR specification due to various issues. To address these concerns, researchers at FHWA (TFHRC) have conducted a study to understand the impact of MSCR specification parameters (Jnr and %recovery). The variation in Jnr difference was also considered and a new parameter which showed relationship to performance was proposed at the TRB 2017 AFK 20 committee meeting.

In this presentation, impact of MSCR specification parameters on PMA formulations is discussed and the effect of % Recovery on polymer content and its implication to performance is considered. The difference in formulations based on DSR phase angle PG specification parameter and corresponding % recovery MSCR parameter will be explained. MSCR test protocol issues such as need for the initial test cycles, and identification of stress dependence of PMAs are covered and details on an alternative MSCR test protocol are offered.

Testing of Ontario's Asphalt Binders to Improve Cracking Performance

<u>Heather Bell</u> and Pamela Marks Ontario Ministry of Transportation

The Bituminous Section of the Ontario Ministry of Transportation (MTO) is responsible for developing and maintaining standards and test methods for bituminous materials used in the construction and operation of Ontario's provincial highway system.

MTO carries out specialized field and laboratory studies to evaluate construction practices and the performance of materials used on provincial highways. MTO implemented performance grading in 1997 and has only used Superpave mix designs since 2005. Rutting has all but disappeared; however, cracking is our primary concern.

MTO uses a PG plus specification to mitigate cracking concerns. Evolution of construction materials has driven the need for continued innovation in testing and acceptance. The presentation provides an overview of the test methods, examples of test data, analysis for procedures that predict field performance, and our chosen acceptance limits.

Innovative Roofing Asphalt Characterization: A Simplified Approach to Routine Rheological Testing

John Casola Malvern Instruments

This presentation will review some alternate testing techniques which relate to several commonly performed roofing asphalt tests. These methods will be discussed along with actual data presented from automated DSR determination of softening point, penetration, capillary and rotational viscosity. When implemented for quality control, intelligent software will be used for logical decision making which can be used for determination of pass/fail criteria, sorting of product by type or grade, provide escalation decisions to the operator for out of spec materials to company procedures, and compile custom reports.

Other techniques to investigate adhesive pull off, prediction of sag, as well as, high shear to simulate processing conditions will also be presented.

Effect and Significance of Ambient Pressure on the Aging of Asphalt Binders

<u>Dave Anderson</u> (1); Ramon Bonaquist (2); and James Rosenberger (3) (1) Consultant and Professor Emeritus, Penn State; (2) Advanced Asphalt Technologies; (3) Penn State University

AASHTO R-28, "Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel" (PAV) and AASHTO T-240, "Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)" (RTFO), make no provision for the effects of ambient barometric pressure (elevation) on the properties of the test residue. NCHRP Project 20-07/Task 400, "Effect of Elevation on Rolling Thin Film Oven Aging of Asphalt Binder", was developed based on reports from agencies located at elevations above 3,000 - 4,000 feet that variability exists between their test results and those from laboratories located at lower elevations. Statistical analyses performed as part of this project on data supplied by AASHTO-resources and the WCTG verified that the effect is of engineering consequence and that it is binder-specific, confirming an earlier analysis of the WCTG data. The binder-specific effect can be partially accounted for by the 135°C viscosity of the binder which appears to substantiate the importance of mixing during the RTFO test. Preliminary results indicate that the preferred method for accounting for elevation (ambient barometric pressure) is to increase the RTFO test time as a linear function of elevation.

Asphalt Binder Quality Assurance Test Method (ABQT)

Raj Dongre (1); and Jack Youtcheff (2) (1) Dongre Laboratory Services Inc.; (2) FHWA (TFHRC)

An innovative, simple, and easy-to-use test method for Quality Control and Assurance of asphalt binders was developed. This method, called the binder Asphalt Binder Quality Assurance Test (ABQT) has been discussed at and demonstrated at previous Peterson Conferences. The focus of this presentation is the considerable progress that has been made in the analysis of the creep recovery data obtained from the ABQT. Fractional single springpot with a dashpot in series (A. Sapporo et al 2014) was used to successfully model the creep and recovery data. A new proprietary software to predict PG Grade from the parameters derived from the creep and recovery curve has also been developed by DLSI. The software can successfully predict (greater than 95% success rate) the continuous PG Grade of a binder from the creep and recovery curve of asphalt binder conducted using the ABQT testing protocols. As the binder is tested in an unaged condition at 25°C, the estimation of its PG is quite rapid. Its use in binder quality assurance will also be discussed.

Study of Factors Affecting Low Temperature Strength and Performance of Asphalt Materials

Sang Soo Kim, Mohsen Radi and Moses Akentuna Ohio University

Characterization of asphalt materials has been commonly performed by measuring rheological properties, such as penetration, viscosity, or complex shear modulus. Certain distress type, such as rutting, may be adequately predicted by the rheological properties since the mechanism of the distress is largely a flow phenomenon. However, the mechanism of low temperature cracking involves both rheological and fracture phenomena. Among many factors potentially affecting the low temperature strength and the low temperature performance of asphalt materials, the cooling/loading rate, physical hardening, aging, and polymer modification were studied using the Bending Beam Rheometer (BBR), the Asphalt Binder Cracking Device (ABCD) and the Asphalt Concrete Cracking Device (ACCD). The test results showed that aging of asphalt materials lowered the low temperature strength of asphalt materials while SBS polymer modification significantly increased the low temperature strength and performance. A faster cooling rate and the physical hardening caused a significant increase in strength and their effects on the cracking temperatures were much smaller than the predicted values from the rheological properties without considering the strength changes.

Measurement, Tolerance and Effect of PAV Pan Warpage and Film Thickness on Physical Properties of PAV Residue

Dave Anderson

Consultant and Professor Emeritus, Penn State University

The long-term aging of asphalt binders is simulated with AASHTO R-28, "Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel" (PAV). Pan warpage has been reported as a problem but no acceptable means for measuring warpage has been adopted. The pans may be supported by a flat shelf, three points at the periphery of the pan, or a flat, hemispherical surface. Warpage at the periphery of the pan is not coplanar and therefore the resulting thickness from a warped pan depends on the design of the support system. A simple, low-cost, easy to use jig that can be used to measure the profile of the bottom of a pan for each of the support systems is discussed. The amount of oxidation and associated property change that occurs during PAV exposure depends upon the square of the thickness of the film, t, and a binder-specific coefficient, A. Measurements from several different pans exhibiting different degrees of warping are used in conjunction with this relationship to establish a recommended tolerance for pan warpage. Levelness and pan warpage requirements for reduced film thicknesses are also presented.

Inter-Laboratory Study of Performance Grade Testing of Crumb Rubber Modified Asphalt Binders

<u>Sallie Houston</u> (1); Shakir Shatnawi (2); Shauna Teclemariam (3); Gary Houston (1) (1) VSS Emultech; (2) Shatec Engineering Consultants, LLC; (3) US Oil Refining

An inter laboratory study (ILS) following ASTM guidelines and practices has been conducted through the Pacific Coast Conference on Asphalt Specifications to demonstrate the feasibility of using existing ASTM D 6373 Standard Specification for Performance Grade Asphalt Binder and related test methods to grade CRM modified asphalt binder. The study consisted of 16 laboratories including federal, state, academic, independent and industry laboratories. Data were collected testing three field produced CRM binders tested in triplicate. Some modification to current test methods are required to accommodate the larger particle size (0.6-1.4mm) of the CRM modifier to produce the material. Of particular interest in the study is the use of a 3mm gap size between the 25mm and 8mm parallel plates used on the dynamic sheer rheometer (ASTM D7175) and whether acceptable precision can be obtained.

This paper outlines the testing protocol, the material tested, data analysis and the statistical methods used to develop precision statements. The data precision statements are shown to be reasonable and include tests for high, intermediate and cold temperatures. Based on the positive outcome of this study, recommendations for testing modifications and future testing are also outlined.

Understanding Cracking Performance with Linear Visco-Elastic Parameters

Geoffrey Rowe

Abatech

The use of delta Tc and the Glover Rowe binder parameter have been shown to be related to cracking of asphalt mixes. These parameters, while strongly correlated, have differing relationships depending upon the type/family of materials being considered. PmB modification will significantly affect the relaxation properties at cold temperatures while generally having a better cracking performance. Other binder systems show differing relationships between observed cracking and new binder properties. This presentation reviews data from various tests and procedures considering point and shape parameters as captured by delta Tc and the G-R parameter. Certain materials types can be considered to lie in different areas of a plot of G-R versus delta Tc. The discussion will in addition include other concepts such as VET, Gc, tan delta, phase angle which have all been proposed as alternate parameters to consider. The concept of an aging index that captures these parameters is explored. Further consideration of the linear visco-elastic zone and the need to define fracture within this zone will also be presented for a range of conventional and modified binders.

Session 4 - Recycling - Effects on Chemical, Physical Properties and Performance - Rejuvenation Mechanisms

Materials for Extreme Rehabilitation Project

Frank Fee, LLC

This project involved the rehabilitation of a 35 year old concrete pavement on 1st Ave. in NYC. There were extreme constraints which included multiple layers of underground utilities, five lanes of very heavy traffic - required night paving, extensive cracking and utility trenches, and a curb reveal that required a maximum 1.5" overlay. It includes information on the special mix design and materials (HiMA) used for overlay and a description of the special pavement preparation and fabric application which was accomplished under the very restrictive time constraints. A present condition report will be presented for the four year old pavement.

Mix Performance Improvement of HMA Incorporating High Amounts of RAP Through the Addition of Bio-additives

Zahra Sotoodeh-Nia, Nick Manke, Chris Williams, and Eric Cochran (1); Emmanuel Chailleux, Erik Bessmann, Pierre Hornych, and Juliette Blanc (2); Davide Lo Presti and Ana Jimenez (3); Laurent Porot (4); Jean-Pascal Planche and Ryan B. Boysen (5); and Simon Pouget and François Olard (6) (1) Iowa State University; (2) IFSTTAR, France; (3) University of Nottingham, UK; (4) Arizona Chemicals, Netherlands; (5) Western Research Institute; (6) EIFFAGE Infrastructures, France

The use of reclaimed asphalt pavement (RAP) in asphalt mixture applications has been widely practiced in asphalt paving industry. The main challenge, however, is the aged RAP binder, which limits the amount of RAP in the mixture. An experimental study was conducted to evaluate the effectiveness of two bio-additives as rejuvenators, a bio-based material as a binder, and a bio-oil/GTR modified binder, on the properties of binder blends and their respective high RAP mixtures. Performance tests included performance grading (PG) of the binder blends, dynamic modulus, flow number, disc-compact tension (DCT) test, and flexural beam fatigue. PG results of the binder blends including the control blend indicate that the bio-additives could significantly soften the RAP binder. Dynamic modulus results showed softer mixtures at all temperatures, however, the differences were more significant at high temperatures. All alternatives ensured excellent rutting resistance at high temperatures, while providing superior fracture resistance at low temperatures and sound fatigue life at intermediate temperatures. Therefore, the main conclusion is that high amount of RAP could be incorporated in HMA along with appropriately selected additives/binders to soften the aged RAP binder without compromising the performance of the mixture.

The Crossover Temperature: Significance and Application towards Engineering Balanced Recycled Binder Blends

<u>Lorena Garcia Cucalon</u> (1); Fawaz Kaseer (1); Edith Arámbula-Mercado (1); Amy Epps Martin (2); Nathaniel Morian (3); Sara Pournoman (3); Elie Hajj (3); and Charles Glover (2) (1) Texas Transportation Institute; (2) Texas A&M University; (3) University of Nevada Reno

Large quantities of asphalt-based recycled materials can be incorporated in asphalt paving mixtures by using recycling agents (RA). Previous studies demonstrated that restoring to the required performance grade (PG) by inclusion of RA may not be sufficient to guarantee long-term durability. In this study the crossover temperature was used to characterize the viscoelastic properties of asphalt binders at the intermediate temperature range of asphalt pavements in-service. The crossover temperature was determined from mastercurves constructed using the dynamic shear rheometer (DSR). Durability thresholds for crossover temperature with aging were proposed based on correlation to the Glover-Rowe parameter. In combination with high PG, crossover temperature was used to evaluate rheological balance: resistance to early rutting and long-term cracking. A wide variety of materials were considered in the study including two grades and three sources of base binders, three types and three sources of recycled materials, and four types of RA products (tall oil, vegetable oil, modified vegetable oil, and bio based oil) at different dosages by weight of total binder in the mixture. Practical recommendations are provided in terms of appropriate materials selection (base binder, recycled binders, and recycling agents) towards engineering long-lasting binder blends with large quantities of recycled materials.

Determine the Correct Mixing and Compaction Temperatures for Using High RAP Content in HMA

<u>Huachun Zhai</u>, Alejandro Rosales and Erin Russell *Idaho Asphalt Supply*

In the current specifications, the mixing and compaction temperatures for RAP mixed were based on the PG grades of the virgin binders. Although this was not a critical issue when the RAP content was low (<15%), as the RAP content becomes higher, there is a concern that the mixing and compaction temperature based on virgin binder grade may not provide sufficient blending and coating to achieve the optimum strength for the mix.

To determine the proper mixing and compacting temperatures, a PAV aged asphalt was used to represent the RAP binder. A single gradation of aggregate was selected to be mixed with the PAV aged binder to eliminate the variances in RAP. The virgin aggregate was also limited to one single gradation. Several different grades of asphalts were used as the virgin binders. Several RAP contents between 15 to 40% were selected. Three sets of mixing and compaction temperatures were used: virgin grades, target grades (based on blend chart) and RAP grades. The compaction efforts were evaluated based on the change of the density and shear stress. The effects of the different mixing and compaction temperatures were evaluated based on various mix tests (Hamburg, TSR and IDT strength).

Influence of RAP and RAS Addition on the Mixture and Binder Performance - Three Years of Loading History and Evaluation

Adrian Andriescu (1); X. Li (1); and J. Youtcheff (2) (1) SES Group and Associates; (2) FHWA

TFHRC is evaluating the effect of high RAP and RAS addition on lab and field performance. Discussed here are data generated over the past 3 years. This includes data from comprehensive (rheological and fracture mechanics) testing of original and recovered from lab and field samples from 0 to 3 years old.

Two base binders were used. Aging progression is largely independent of the base binder use. The binder performance of RAS and higher RAP mixes showed an accentuated aging progression in the top layer, while the bottom layer remained largely unchanged. The parameters delta T and strain tolerance showed significant degradation during this short aging. Most notable was the observation for the RAS containing mixtures having the lowest strain tolerance at the initial time and upon aging the percentage loss in strain tolerance was among the highest.

Chemical Characterization of a Rejuvenated Binder Using a Soybean-Derived Material Through Thermal Desorption / Gas Chromatography-Mass Spectrometry (TD/GC-MS)

Mohamed Elkashef, R. Christopher Williams, and Eric Cochran *Iowa State University*

Recently, asphalt rejuvenation of reclaimed asphalt pavement (RAP) binders is gaining a lot of interest. A number of rejuvenators derived from natural oils have been successfully used in asphalt. It is still however not clear how many rejuvenators interact with the asphalt binders and their constituents to restore their unaged properties. One of the key issues that remain unanswered is whether the rejuvenators are chemically altering the binder's structure. In this study, a Gas Chromatography-Mass Spectrometry (GC-MS technique) coupled with thermal desorption (TD) and cryotrapping is used to examine the chemical structure of a rejuvenator before and after it is blended with a binder. A soybean-derived rejuvenator is blended with a binder at a dosage of 6%. The modified binder was both RTFO and PAV aged to assess its durability. The performance grade of the modified binder is determined using DSR and BBR testing. The unaged and PAV aged modified binder are studied using TD/GC-MS, in order to examine the nature of the chemical interaction between the rejuvenator and the binder, both before and after aging.

Thermal Analysis and Colloidal Stability of Asphalt Modified with Engineered Bio-Based Additives

<u>Hassan Tabatabaee</u>, Todd Kurth and Tony Sylvester *Cargill Industrial Specialties*

Increasing incorporation of highly aged bituminous material into pavements on one hand, and the potential deterioration of the quality of soft bitumen has accentuated the need to engineer the properties of bitumen with various modification and "Rejuvenation" technology. While it is understood that any modified formulation will need to adhere to high standards of compatibility and long term stability and durability, consensus on the definition of "Rejuvenation" and the associated mechanisms does not exist. The present study utilizes chemical fractionation, thermal analysis, and thermo-rheological analysis for to evaluate the impact of an engineered bio-based additive from a chemically modified vegetable-oil source. Chemical fractionation and colloidal instability indices were determined using an "Iatroscan". The viscoelastic response was analyzed via thermo-rheological parameters derived from 4-mm Dynamic Shear Rhoemeter master curve modeling. The Glass Transition and thermal analysis of the samples was conducted using a Differential Scanning Calorimeter (DSC), while the rate of asphalt physical hardening was analyzed in accordance to MTO Method LS-308 Extended Bending Beam Rheometer test.

Compatibility of Recycled Binder Blends with Recycling Agents: Rheological and Physicochemical Evaluation of Rejuvenation and Aging Processes

Lorena Garcia Cucalon (1); Gayle King (2); Fawaz Kaseer (1); Edith Arambula-Mercado (1); Amy Epps Martin (3); Fred Turner (4); and Charles Glover (3) (1) Texas Transportation Institute; (2) GHK, Inc.; (3) Texas A&M University; (4) Western Research Institute

Public and private sectors promote increased use of recycled asphalt materials into new asphalt pavements. However, concerns surface with respect to premature pavement embrittlement and cracking from utilization of these aged materials. Various recycling agents (RA), or rejuvenators, can be used to offset these concerns. Rejuvenation mechanisms can be inferred depending on RA type, yet there is a lack of supporting experimental evidence. This study provides fundamental insight on the mechanisms of rejuvenation through extensive rheological and physicochemical characterization by considering experimental variables such as RA type, type and proportion of recycled materials, type of substitute binder, and short- and long-term laboratory aging. Physicochemical aspects of recycling and rejuvenation were evaluated by differential scanning calorimeter (DSC) and SAR-ADTM (Saturates, Aromatics, Resins – Asphaltene Determinator). Rheology characterization was conducted in terms of performance grade (PG) plus evaluation of long-term durability by the Glover-Rowe (G-R) dynamic shear rheometer (DSR) function after laboratory short- and long-term aging. Tracking of oxidation products was performed using Fourier transform infrared spectroscopy (FT-IR). Rheological and physical characterization indicated that rejuvenation was effective. Chemical characterization. Benefits from rejuvenation were gradually lost with long-term aging.

Notes

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