Progress Toward Performance-Graded Emulsified Asphalt Specifications

A Workshop

January 17, 2013
Washington, D.C.

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Preface

Two TRB e-circulars published in 2006 and 2007, *Transportation Research Circular E-C102: Asphalt Emulsion Technology* and *Transportation Research Circular E-C122: Asphalt Emulsion Technology: Review of Asphalt Emulsion Residue Procedures* covered various specific topics on emulsified asphalt technology. This third e-circular is a progress report on where the industry is presently. It is the outcome of a workshop, Progress Towards Performance-Graded Emulsified Asphalt Specifications, sponsored by the Characteristics of Asphalt Materials Committee at the 92nd Annual Meeting of the Transportation Research Board. The reviews and research from various practitioners address topics that aim to define the beginnings of new test methods that eventually will redefine how the industry determines specifications and performance of emulsified asphalts.

Appreciation is expressed to the authors of the six papers for their contributions and to Robert McGennis of The HollyFrontier Companies and Darren Hazlett of the Texas Department of Transportation for providing valuable editorial input.

—Delmar R. Salomon
Pavement Preservation Systems, LLC
Characteristics of Asphalt Materials Committee, Chair

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Workshop Overview

GAYLE KING
GHK, Inc.
Workshop Organizer

When the Utah Department of Transportation (DOT) adopted the first performance grade (PG) binder specifications in 1996, the control of asphalt quality through performance specifications took an important step forward. Unfortunately, a performance-based system comparable to SuperPave® has never materialized for the application and use of asphalt emulsions. Although the need for and value of pavement preservation fills the headlines of paving journals around the world, the asphalt emulsions used for most preventive maintenance applications are controlled using antiquated test methods rather than those that may be found in a modern asphalt laboratory.

THE NEED

The lack of performance-based specifications for asphalt emulsions has not gone unnoticed. The Office of Pavement Preservation and Construction at FHWA, in conjunction with AASHTO and others, hosted a Forum on Pavement Preservation in Kansas City in 1998 and later hosted a series of three national workshops that culminated in the creation of research needs statements detailing deficiencies in the pavement preservation sector that require further study. The final document from that effort, Transportation System Preservation Research, Development, and Implementation Roadmap (January 2008), divided 38 prioritized research needs into six preservation sectors: asset management, design, construction, materials, maintenance contracting, and performance. The materials section includes seven research needs, two of which are directly related to creating performance specifications in support of pavement preservation applications. The first, Mechanical Binder Properties to Predict Surface Treatment Performance, carries an estimated price tag of $1.5 million, while the second project, Performance Grading System for Asphalt Emulsions, defines a $4.5-million anticipated effort. A third performance-related materials need, Triggers for the Timing of Surface Treatments, comes with a $10-million cost estimate.

Although the need for research to support the important industry trends toward preservation has been defined, finding the substantial resources needed to accomplish this task has proven elusive. Just these three performance-related materials projects are estimated to cost $16 million, which represents 40% of the anticipated cost to complete the entire Preservation Roadmap. A SHRP-scale research program supported by line-item legislation in the highway bill is unlikely in today's political environment. Progress can only come in smaller steps, using money and research time wherever it comes available. Unfortunately, such efforts are slow, yielding good research ideas but little coordinated strategy to accomplish goals on a scale needed for broad-based emulsion performance specifications. Experience from the SHRP–SuperPave era has shown that FHWA-supported expert task groups can combine the expertise of numerous academic researchers with the agency and industry knowledge needed to develop practical test methods and performance-based specifications. Hence, FHWA's Office of Preservation and

Construction created the Emulsion Task Force (ETF), a group of technical experts tasked with advancing the "Materials" needs from the Roadmap. The ETF operates as the materials arm of the Pavement Preservation Expert Task Group, so any test methods or specifications reviewed and accepted by the ETF are forwarded to either the AASHTO materials or maintenance committees for approval and adoption as AASHTO standards. Researchers with funding in appropriate subject areas work with the task force as members or friends, knowing that ETF approval of their work products can lead to fairly rapid adoption as AASHTO standards.

Although the ETF is the primary standard-bearer for performance-based emulsion specifications at the moment, ASTM has also been actively working to improve emulsion test methods and specifications.

TRB WORKSHOP

TRB Characteristics of Asphalt Materials Committee sponsored a 2013 workshop, Progress Towards Performance-Graded Emulsion Specifications, to update the research community regarding activities and research related to the topic. All presenters were invited and no formal papers were required. However, the timeliness of this workshop generated so much interest that each speaker was later invited to submit a paper to create this TRB e-circular.

The agenda for the workshop was as follows.

Session I: Goals and Progress: PPETG, Emulsion Task Force, and ASTM Activities

8:00 a.m.	The Need for Performance-Based Emulsion Specifications: The FLH Study Michael Voth, *Central Federal Lands Highway Division*
8:30 a.m.	Emulsion Task Force Activities and Emulsion Straw Man Specification Arlis Kadrmas, *BASF*
9:15 a.m.	ASTM Update Delmar Salomon, *Pavement Preservation Systems LLC*

Session II: Emulsion Performance Tests, Field Tests, and Validation

10:15 a.m.	Performance Tests for Emulsion Residues Hussain Bahia and Andrew Hanz, *University of Wisconsin*
10:40 a.m.	Application-Specific Performance Tests for Chip Seals Richard Kim, *North Carolina State University*
11:05 a.m.	Field Validation of Emulsion Performance Specifications Amy Epps-Martin and Aishwarya Vijaykumar, *Texas A&M University*
11:30 a.m.	Chip Seals: Field Sampling and Testing Mike Farrar, *Western Research Institute*
11:45 a.m.	Using Vacuum to Accelerate Residue Recovery Gerald Reinke, *MTE Services*

Summary of Presenters and Topics

The first session of the workshop was organized to present the need for materials research in the area of pavement preservation and to provide an overview of ETF and ASTM activities tasked with creating and validating performance-based emulsion test methods and specifications. The second session focused on specific research programs working to accomplish these goals.

**NEED FOR PERFORMANCE-BASED EMULSION SPECIFICATIONS:
FEDERAL LANDS HIGHWAY STUDY**

Mike Voth

From his position at Central Federal Lands Highway Division (CFLHD), Michael Voth has been a staunch supporter of emulsion performance specifications. In his workshop presentation, Voth noted the problems he faces when specifying asphalt emulsions for use on national park roads spanning extreme climates from Death Valley to Rocky Mountain peaks. For example, CRS-2P and CRS-2L enable him to select polymer type, but a single grade with one very broad range for residue penetration gives no flexibility to vary material by climate.

Working in cooperation with FHWA's Office of Pavement Preservation and Construction, CFLHD placed a series of chip seals and microsurfacing projects on pavements in national parks and monuments across the United States. Through the National Center for Pavement Preservation (NCPP), they collectively funded research to evaluate SuperPave tools and test methods and new emulsion residue recovery methods to determine their applicability for future performance-based specifications. CFLHD monitors each project annually for performance and makes materials and performance data available to other researchers needing validation for their own work.

Using residue dynamic shear rheometer (DSR) data collected from various chip seal and microsurfacing projects, the FLH study and the CFLHD lab found numerous examples where emulsion residues placed in hotter climates were considerably softer than comparable residues placed in colder climates. Assuming that a performance-based specification would require equal binder moduli at the highest pavement temperatures, the polymer-modified emulsion residues supplied to Death Valley and Utah Park chip seal projects appear to have a combined discrepancy of five PG grades.

The CFLHD experience emphasizes the need for development of performance-based test procedures and the field validation of specification limits for accepted testing protocols. The FLH study proposed a draft straw man performance specification for chip seals that included the AASHTO PP 72, Method B, thin-film residue recovery procedure. The ETF subcommittee has revised the original draft straw man specification and proposal of changes will continue. Although the FLH study evaluated Method B in some detail, researchers recognized that the amount of residue provided by thin films was not sufficient to conveniently run bending beam rheometer (BBR) and direct tension test (DTT) tests as found in PG binder specifications. The ETF challenged other research teams to develop alternative DSR test methods to evaluate the rheology of the residue at low temperatures. As reported in other papers in this e-circular, Farrar and his team adapted the DSR 4-mm plate method for emulsion residues, whereas University of Wisconsin (UW) developed algorithms to predict low-temperature rheological properties from

intermediate temperature DSR frequency sweeps. Both DSR methods now appear to be viable alternatives to BBR. The direct measurement of $G*$ and phase angle using the 4-mm plate will likely be preferable for specifications, but the interpolation using extrapolation from intermediate temperature data will be a quality control (QC) alternative for older rheometers that lack the capability to collect data at lower temperatures.

Most of the data from the FLH project was previously published, so no written paper is included for Voth's presentation. Much of the technical data was previously reported in the following publication:

> King, G., H. King, L. Galehouse, M. Voth, et al. Field Validation of Performance-Based Polymer-Modified Emulsion Residue Tests: The FLH Study. Presented at International Conference for Pavement Preservation, Newport Beach, 2010.

Project reports and data from the FLH studies are available on the NCPP website or by request from Voth or King.

ASPHALT EMULSION RESIDUE RECOVERY UPDATE

Arlis Kadrmas

Kadrmas chairs the ETF subcommittee overseeing the recovery and testing of emulsion residues. In this e-circular, Kadrmas provides a description of the issues facing the committee in efforts to select a new emulsion residue recovery procedure. He includes some of his own research data using the DSR to compare residue properties from the different recovery methods. As he points out, AASHTO PP 72, Procedure B, is currently the ETF subcommittee's preferred procedure against which it will compare all other recovery procedures.

ASTM PROGRESS IN PERFORMANCE-GRADE–BASED EMULSIFIED ASPHALT SPECIFICATIONS

Delmar Salomon

Delmar Salomon chairs the TRB Characteristics of Asphalt Materials Committee that sponsored this workshop, and he is an active member of ASTM committees supporting testing and specification of asphalt emulsions. Although all ETF activities are intended to support AASHTO specifications, ASTM continues its own efforts to advance the specification and testing of asphalt emulsions. In his review of ASTM activities, Salomon focuses on recent developments regarding residue recovery and the measurement of emulsion viscosity.

**DEVELOPMENT OF A MORE RATIONAL SYSTEM FOR
SELECTING EMULSIONS FOR SURFACE TREATMENTS**

Hussain Bahia and Andrew Hanz

Hussain Bahia's group at the UW–Madison has received significant financial support from the Asphalt Research Consortium (ARC) to develop performance-based test methods for asphalt emulsion residues. This research has produced a number of new test procedures, many of which use the DSR in novel ways pertinent to the performance of emulsion residues in surface treatments. Bahia and Andrew Hanz present a comprehensive review of the following performance-related techniques developed at UW:

- Bleeding: MSCR adapted to test emulsion residues;
- Raveling and moisture damage: bitumen bond strength (BBS) test ;
- Polymer elasticity: elastic recovery in a DSR;
- Low-temperature rheology: DSR frequency sweeps at intermediate; temperature and algorithms to predict BBR-equivalent results;
- Cracking resistance: linear amplitude sweep test (LAS); and
- Residue recovery and pressure aging vessel (PAV) aging procedures.

APPLICATION-SPECIFIC PERFORMANCE TESTS FOR CHIP SEALS

Richard Kim

Richard Kim has had a long-standing contract with the North Carolina DOT to improve chip seal performance. Kim's paper here is a comprehensive review of the results from that effort. His work evaluates such critical performance elements as curing time for traffic, chip retention under traffic, and chip seal designs. One example of Kim's work is the use of an MMLS-3 lab scale traffic simulator to understand chip seal performance under traffic. He then evaluates accelerated laboratory test methods such as the sweep test to determine if they can be validated as tools for performance specifications. Kim has published a number of papers at TRB and elsewhere that describe the individual test protocols in more detail.

Several years ago, TRB Characteristics of Asphalt Materials Committee submitted a research needs statement to further develop performance specifications for chip seals and microsurfacing. Kim is now principal investigator for NCHRP Project 9-50, which resulted from that effort. As such, Kim continues to focus his research on application-specific tests that evaluate the performance of asphalt emulsion in the presence of aggregates. Bahia is leading the binder residue testing portion of that same study. Interested readers are encouraged contact Kim for more information, pending publication of the final report from NCHRP Project 9-50.

FIELD VALIDATION OF EMULSION PERFORMANCE SPECIFICATIONS

Amy Epps-Martin and Aishwarya Vijaykumar

With strong support from Darren Hazlett of Texas DOT, research teams at Texas A&M University System directed by Epps'Martin have been developing performance-based chip seal tests and specifications for many years. Work from this effort included new recovery procedures (stirred-can method) and recommendations for specification limits using results from DSR frequency sweeps. After Epps'Martin joined the ETF, some elements of her research changed to reflect the collective thinking of the larger industry panel represented there. Epps'Martin's team is now proceeding using a large sampling of Texas chip seal projects to establish failure criteria for the chosen test methods and validate recommended specifications. A formal paper covering this presentation was submitted through conventional TRB channels and was accepted for publication in *Transportation Research Record: Journal of the Transportation Research Board, No. 2370: Asphalt Materials and Mixtures 2013, Volume 1*. It will not be reproduced in this e-circular.

FIELD SAMPLING AND TESTING OF DEATH VALLEY CHIP SEAL EMULSION RESIDUE: CASE STUDY

Mike Farrar

The Western Research Institute (WRI) dedicated significant financial resources from their ARC and Fundamental Properties Studies to advance the research needs of the ETF. Farrar's short presentation to the workshop focused on WRI's experiences with field sampling of chip seal binders; however, he offered to expand his written paper to include a comprehensive review of WRI's work in this area. Farrar used data collected from the FLH field project in Death Valley to demonstrate the breadth of new capabilities now available to researchers and practitioners. Subjects of the enclosed paper include:

- Field sampling methods for chip seal residues, including new extraction–recovery methods;
- Introduction of the DSR 4-mm plate method as an alternative to BBR when evaluating emulsion residues at low pavement temperatures;
- Evaluation of polymer morphology before and after extraction and the influence of polymer morphology on rheological properties;
- In-place aging of chip seal residues, including rheological changes and carbonyl gradients within the binder layer and chemical changes within the polymer;
- Recommended improvements to thin-film residue recovery procedures, including possibility for PAV (pressure-aging vessel) aging of residues in a simple aging test (SAT); and
- Use of intermediate temperature DSR capabilities such as LAS and Glover–Ruan parameters to evaluate binder brittleness before and after aging.

The extension of the DSR 4-mm plate method to emulsion residues is particularly important to the goals of the ETF, because the rapid thin film recovery method favored by emulsion experts does not provide enough residue for BBR or DTT testing.

TECHNIQUES FOR ACCELERATING RECOVERY OF ASPHALT EMULSION RESIDUES AT 60°C USING THIN-FILM PROCEDURES

Gerald Reinke

Like the previous presentation, Reinke's presentation to the workshop was a short summary of a single topic: the use of vacuum to accelerate the recovery of emulsion residue in thin films. As a leader in the industry effort to develop rapid ambient temperature recovery methods, Reinke agreed to expand his enclosed publication to include a much broader range of research on the subject conducted primarily at MTE Services. Some of the more important elements of the paper include the following.

- Use of vacuum ovens to accelerate the recovery of emulsion residues in thin films. It appears to be possible to reduce the recovery time from the 6 h required by AASHTO PP 72, Method B, to 2 h by replacing the forced draft oven with a vacuum oven without negatively impacting residue properties.
- Use of shear in the DSR to break the emulsion and then evaluate the residue in a single step. Although this recovery method turned out to be impractical due to DSR instrument time, this work has interesting implications for researchers. Among other things, Reinke's paper provides extensive evaluations of minimum moisture contents at which accurate rheological data can be expected.
- Evaluation of high float emulsion residues as recovered using thin-film vacuum recovery. The float property does indeed exist in emulsion residues recovered at ambient temperature. Thin-film recovery does produce enough material for a float test. Reinke further discusses the need to replace the float test, along with ideas for a DSR rheological test that might capture the gel-like properties exhibited by high-float emulsion residues.
- Extensive evaluation of residue rheological properties using DSR, including the 4-mm plate, MSCR (multi-stress creep recovery), and BYET (binder yield energy test). One interesting extension of the 4-mm plate DSR work is the determination of the glass transition temperature (Tg) of the emulsion residue using the first derivative of the relationship between G^* and temperature.
- Determination of the impact of HCl concentration from the emulsion soap on residue rheology. As exemplified by polyphosphoric acid (PPA) and other asphalt additives, it is well known that certain inorganic acids can modify the molecular structure, hence the rheology, of asphalt. Reinke's paper shows that HCl concentration in the soap of cationic emulsions also affects the rheology of the emulsion residue. Residue properties may not match those of the base asphalt because HCl or tall oil salts (high float) may cause significant rheological changes.

Asphalt Emulsion Residue Recovery Update

ARLIS KADRMAS
BASF Corporation

The use of latexes and polymers in asphalt emulsion applications to improve field performance created a challenge for the recovery of the residue using traditional oven evaporation or distillation techniques (260ºC). Those techniques recovered the residue at temperatures that deteriorated the polymers used and therefore the procedures were adjusted to lower temperature techniques (177ºC or 204ºC) to reduce the breakdown of the polymers used in the asphalt emulsion manufacture. A vacuum distillation procedure at 135ºC was also introduced to lower the temperature to recover the residue. These techniques use temperatures that are still much higher than the field application temperatures for these products. Recent evolution of lower-temperature evaporative techniques at 60ºC can provide a residue that is at summer field conditions and allows material to be evaluated through improved rheological techniques.

The following report shows the development of recovery techniques that are similar to field conditions, and the test results that compare those techniques to the higher-temperature recovery procedures. An update of further developments to improve the time to recover the residue and possible aging issues is also provided.

ASTM D6997 DISTILLATION TECHNIQUE

The distillation apparatus is shown in the Figure 1. The temperature of this procedure for latex modified asphalt emulsions is usually 177ºC or 204ºC. This is lower than the 260ºC that was

FIGURE 1 Distillation apparatus.

originally developed for this procedure for asphalt emulsions, but still much higher than the pavement temperatures that the asphalt emulsions are subjected to when applied to pavement preservation applications.

ASTM D7403 VACUUM DISTILLATION TECHNIQUE

An attempt to lower the distillation temperature using vacuum was developed as ASTM D7403. Figure 2 shows an example of the setup for this test procedure. The vacuum allowed the temperature to be lowered to 135ºC, but the emulsion had to be frozen prior to placing in the still to allow the material to not boil over during heating. This procedure was not used by agencies to any extent, but still provided a step change in the process to get a residue at closer to pavement conditions that asphalt emulsions are subject to during application and curing.

WHY CHANGING TO LOWER TEMPERATURE
RECOVERY TECHNIQUES IS NOW POSSIBLE

The existing tests on emulsion residues require the pouring of the asphalt for the testing, which can be done at the temperatures from the distillation. Most of the current specifications for asphalt emulsion residues currently use a penetration range at 77ºF to evaluate the residue rheology. The absolute viscosity values are tested at 140ºF. For latex- or polymer-modified emulsion residues, the elastic properties using tests such as elastic recovery or forced ductility are tested at 39.2ºF or 50ºF.

The use of the DSR for asphalt grading in hot-mix applications has opened the possibility of samples to be placed on this device for testing without having to be poured. The ability to test the residue at multiple temperatures, at various stress or strain levels, and various frequencies give this technique an added advantage. This has allowed the development of evaporation techniques at, or near, pavement temperatures due to the lack of the necessity to have to have the sample in a liquid form to pour into a test apparatus.
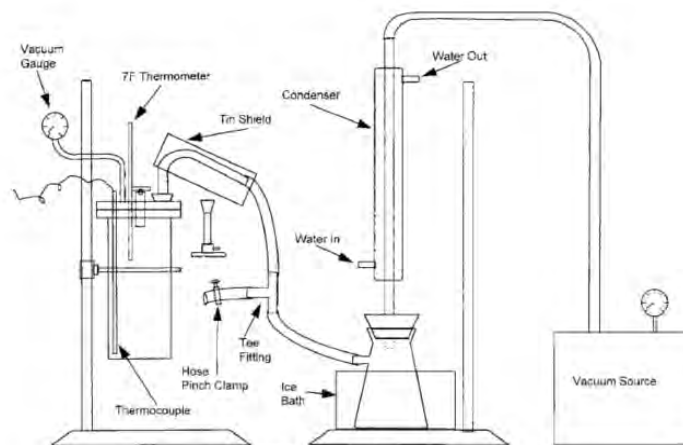
FIG. 2 Connection Apparatus Schematic

FIGURE 2 An example of the setup for this test procedure.

TWO RECENTLY APPROVED LOW-TEMPERATURE
EVAPORATION TECHNIQUES AND RESULTS

AASHTO PP72, Method A, is similar to ASTM D7497 and utilizes a 1.5- to 2.0-kg/m^2 film of asphalt emulsion on a silicone mat. The film is evaporated at 25ºC (or ambient room temperature) for 24 h and then placed in a 60ºC oven for 24 h. The procedure was developed from European standard EN13074. The initial 24 h at near-ambient room temperature is to encourage evaporation without a skin forming on the surface of the asphalt emulsion. The film thickness is very close to that used in a chip seal application. A picture of a silicone mat with the film of emulsion is shown in Figure 3. One issue with this procedure is the length of time that is necessary for drying the residue prior to testing. Also, there is a question on the appropriate aging of the residue during this procedure.

AASHTO PP72, Method B, was developed to reduce the length of time required to get a residue from an asphalt emulsion using Method A or D7497 low-temperature recovery technique. The technique was developed by the Texas DOT and utilizes the same 60ºC evaporation temperature, but a thinner film (0.015-in. drawdown) for evaporation. The procedure takes 6 h rather than the 24+24 h of Method A or D7497 procedure.

A picture of this film and removing it from the silicone mat can be seen in Figure 4. This film is a similar thickness to that used in a fog seal or tack coat application of asphalt emulsions. There is still a question on the length of time being too long as well as how this ages the emulsion residue.

The results comparing the two low-temperature evaporation techniques can be seen in Table 1. The DSR results show a small decrease in the results when using the Texas evaporation method.

The testing of unmodified asphalt emulsion samples is important to get an understanding of the test procedure prior to continuing with latex-modified asphalt emulsions. Tables 2 and 3 compare unmodified emulsions with Method A and the distillation technique.

FIGURE 3 A silicone mat with the film of emulsion.

FIGURE 4 A thinner film (0.015-in. drawdown) for evaporation, being removed from the silicone mat.

TABLE 1 Results Comparing the Two Low-Temperature Evaporation Techniques

Temperature (°C)	CRS-2L		CRS-2P	
	ASTM D7497	Texas Method	ASTM D7497	Texas Method
52	7.69	6.07	12.12	9.8
58	3.47	2.73	5.76	4.58
64	1.65	1.32	2.94	2.27
70	0.81	0.67	1.56	1.16

TABLE 2 Comparison of Unmodified Emulsions with Method A and the Distillation Technique

		Original DSR Testing on Residue from Evaporation (25°C 24 hour & 60°C 24 hour) @ 10rad/sec @ test temperature									
		52°C		58°C		64°C		70°C		76°C	
Emulsion	% L/P	g*/sin delta	delta	g*/sin delta	delta	g*/sin delta	delta	g*/sin delta	delta	g*/sin delta	delta
PGXX-34 base	0.0	2.0485	84.74	0.93585	86.14						
PG58-28 base	0.0			2.408	84.92	1.119	86.45	0.546	87.55		
PG64-22 base	0.0			6.4015	82.25	2.854	84.40	1.324	86.16	0.6475	87.49

TABLE 3 DSR Results from the Original Base Asphalts Used

Asphalt Grade	% L/P	Original DSR Testing on Base Asphalt							
		52°C		58°C		64°C		70°C	
		g*/sin delta	delta	g*/sin delta	delta	g*/sin delta	delta	g*/sin delta	delta
PGXX-34	0	0.9009	87.775						
PG58-28	0			1.501	86.82	0.7054	88.05		
PG64-22	0			3.1355	85.515	1.419	86.955	0.6913	88.025

The DSR results of the evaporation technique are slightly higher than those of the distillation procedure for each temperature tested. This has proven to be quite consistent based on multiple test results. There was initial concern that there may be residual moisture that affected the results, but there was determined to be no remaining moisture in the samples.

The DSR results from the original base asphalts used can be seen in Table 3 for comparison to those in the Tables 1 and 2.

The distillation results are obviously closer to those of the original asphalt DSR results. The purpose of a recovery procedure is to mimic the residue in the field and not the base that was used. There are emulsifiers and reacting agents that are used in the emulsification process that may affect the residue in the low-temperature recovery technique that is closer to field conditions.

DEVELOPING RESEARCH IN RESIDUE RECOVERY TECHNIQUES

To lower the time of the recovery of Method B, various vacuum methods have been tried including using the PAV vacuum degassing apparatus as well as a vacuum oven. Along with decreasing the time, the effect on the aging of the specimens is being looked at as well.

CONCLUSIONS

It is clearly shown that the use of evaporation techniques at temperatures close to application conditions can be accomplished if using the DSR for testing. The accumulation of data from various asphalt emulsions using AASHTO PP72 along with current agency testing techniques will allow for specifications using these techniques in the future. Comparison to field performance to validate the specifications recommended will also be an important step to move forward with the low-temperature residue recovery techniques.

Development of New Viscosity and Residue Recovery Standards at ASTM International

DELMAR R. SALOMON
Pavement Preservation Systems, LLC

The purpose of this review is to give an update of the current work on viscosity and residue recovery that has taken place at various laboratories and developed into standards at ASTM. Emulsified asphalts are complex fluids and their testing is complicated.

Two of the most important tests for emulsified asphalt are viscosity and residue recovery. Reviews on emulsified asphalt technology and, in particular, the residue recovery procedures are published in two TRB circulars (*1*, *2*). Many of the ASTM traditional test methods overlap with AASHTO test methods, and in many cases can be used interchangeably (*3*). This underlines an ongoing technical collaboration between these two organizations in a time of scarce resources and in relation to this most important topic for the pavement industry.

In the past few years, two new standards have been developed at ASTM. One is a faster and more automated viscosity procedure using a digital rotational paddle viscometer (ASTM D7226). The other is a simple and automated emulsified residue recovery procedure using a moisture analyzer balance (MAB) (ASTM D7404). There are other residue recovery procedures but they are variations of distillation and oven evaporation procedures that are not automated and involve large sample size.

ASTM (*4*, *5*) and AASHTO (M140, M208) have specifications for emulsified asphalt. However, there is no ASTM standard for polymer-modified emulsified asphalt, whereas AASHTO does have the CRS-2L and CRS-2P (*6*) specifications. The selection for various applications of emulsified asphalt is outlined in ASTM D3628 (*7*).

VISCOSITY

The workability and flow properties of emulsified asphalts are controlled by viscosity and the amount of asphalt. For example, having the appropriate viscosity avoids drilling or run-off in chip seal applications that are widely used in pavement preservation treatments in the United States. The emulsified asphalt must be thin enough to be sprayed, yet thick enough as not to flow from the crown or grade of the road. For mixing-grade emulsified asphalt the viscosity will affect its workability and the resulting film thickness on the aggregate.

The Saybolt Furol viscometer (*8*) has been the workhorse test method for many years in the emulsified asphalt industry. Figure 1 shows the equipment along with the process for viscosity determination of emulsified asphalt. Measurement is fast (typically 5 to 6 min), but the sample preparation and cleanup easily occupy more than 90% of a technician's time (minimum of 1 h) to determine the viscosity of a sample. Moreover, ancillary equipment is needed such as ovens and water bath for sample preparation.

Over the past 2 years, several ASTM interlaboratory studies were performed for both the Saybolt and Paddle viscometers (Figure 2). Now a new test method using a rotational paddle viscometer is an ASTM standard (*9*).

(a)

(b)

(c)

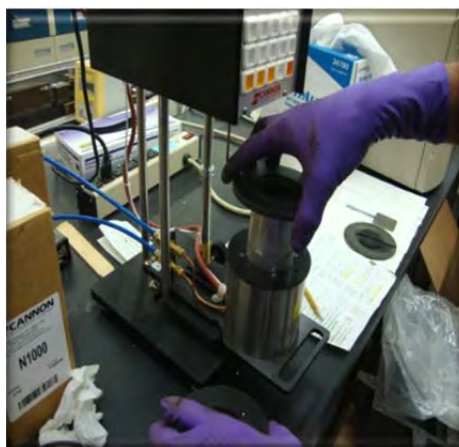(d)

FIGURE 1 From left to right: (*a*) Saybolt viscometer; (*b*) start of measurement; (*c*) measurement; and (*d*) end of measurement.

FIGURE 2 From left to right: (*a*) digital rotational paddle viscometer; (*b*) sample loading; (*c*) placing paddle and temperature probe into the sample; (*d*) temperature probe and paddle; and (*e*) digital measurement of viscosity and temperature.

Two ASTM Interlaboratory Studies (ILS) for Saybolt and the digital paddle viscometers were performed using emulsified asphalt samples typically used in the industry, including polymer-modified emulsified asphalts. Precision statements were obtained from the participation of 17 laboratories and were followed by a second ILS involving another 15 laboratories. Participant make-up was 29% users, 47% producers, and 24% independent laboratories. Current precision statements for Saybolt are shown in Table 1 and for the digital paddle viscometer in Table 2.

The two emulsified asphalt specification standards (*4, 5*) will now include an optional table for specifying the use of the rotational paddle viscometer to determine the viscosity of the emulsified asphalt. The paddle viscometer, as shown in Figure 2, shows that the temperature probe is actually inside the sample as oppose to reading the temperature from a water bath that is transferring heat to the sample. Also, there is no ancillary equipment needed for emulsion preparation or extensive cleanup required. This translates into lower operating costs and a smaller equipment footprint.

EMULSIFIED ASPHALT RESIDUE RECOVERY

There is no general consensus on a procedure for the residue recovery of emulsified asphalt. This is particularly true when it comes to the recovery of residue from polymer-modified emulsified

TABLE 1 Saybolt Furol Viscometer

Test Temperature [°C (°F)]	Viscosity (s)	Repeatability (% of the mean)
Single-Operator Precision		
25 (77)	25 to 200	6.7
50 (122)	75 to 400	10.8
Multilaboratory Precision		
25 (77)	25 to 200	22
50 (122)	75 to 400	88

TABLE 2 Rotational Paddle Viscometer

Test Temperature [°C (°F)]	Viscosity (mPa-s)	Repeatability (% of the mean)
Single-Operator Precision		
25 (77)	25 to 200	8.2
50 (122)	100 to 1,000	12.9
Multilaboratory Precision		
25 (77)	25 to 200	22
50 (122)	100 to 1,000	64

asphalt. There is general acceptance that any high-temperature (>120°C) recovery procedure for polymer-modified emulsified asphalt, such as the commonly used distillation or oven evaporation test methods (*10*, *11*) are not suitable. New procedures have been developed and proposed (*12*, *13*, *14*). It has been shown that high recovery temperatures and extended recovery times degrade the polymer significantly, hence measurement of residue properties give results unrelated to binder performance. Once a consensus is obtained for a recovery procedure the rheological tests will follow. In this section we discuss an extension of the ASTM D7404 test procedure, recovery by a MAB to include testing the residue by a DSR. This procedure is referred to as MAB–DSR.

It is accepted that the measurement of rheological properties of asphalt binders reveal something about their performance. Therefore, one of the objectives is to transition to rheological tests once a consensus is obtained for a residue recovery procedure. The evolution of the MAB–DSR procedure has been discussed with a more recent development (*15*, *16*).

Figure 3 shows good correlation between the 3-h oven evaporation procedures versus the 20-min MAB procedure.

The operation of MAB–DSR procedure is shown in Figure 4. The recovery is performed in the silicone mold of the DSR and then transferred and placed directly on the parallel plate of the DSR, similarly to placing hot binder for performance grading. Figure 4*b* shows recovery in an aluminum pan then transfer to the DSR silicone mold, while Figure 4*c* eliminates that step and places the emulsified asphalt sample directly into the silicone mold (typically about 1.7 g of sample). Figure 4*d* shows the residue in the silicone mold, which is taken directly to the DSR (Figure 4*e*) where the binder high-temperature PG is obtained.

The graph shows a plot with the equation $y = 1.0191x - 0.0162$ and $R^2 = 0.96$. The y-axis is labeled "Residue By Evaporation (D 6934)" ranging from 60.0% to 76.0%. The x-axis is labeled "Moisture Analyzer Balance (D 7404)" ranging from 60.0% to 76.0%.
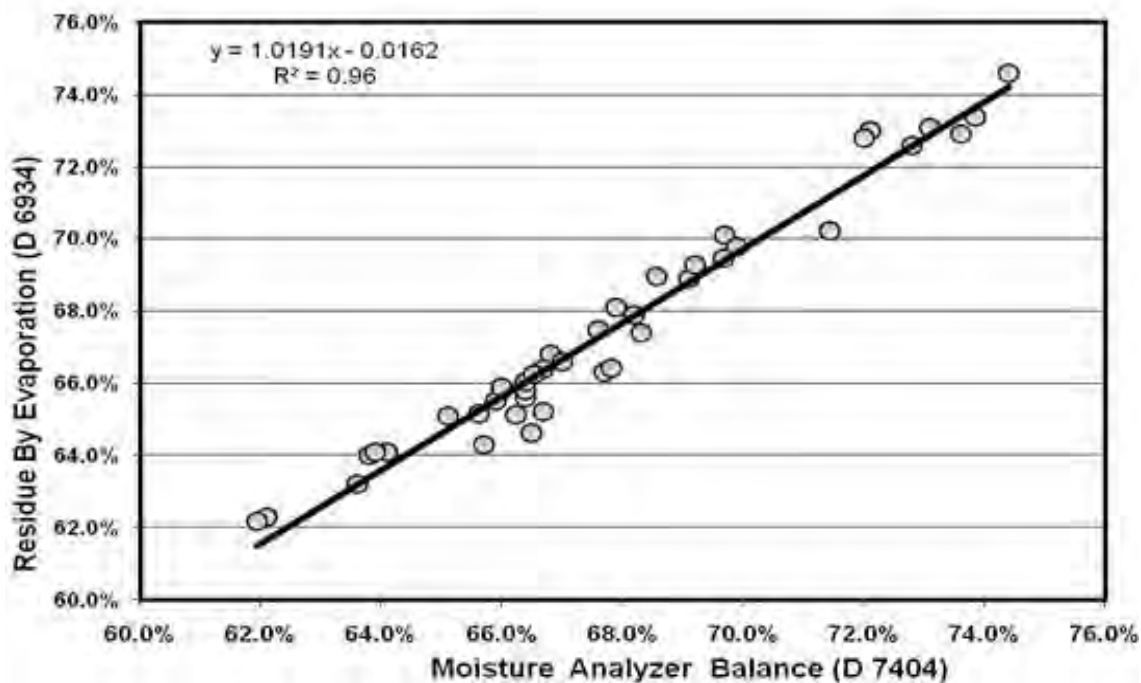
FIGURE 3 Correlation of two ASTM procedures.

(a)

(b)

(c)

(d)

(e)
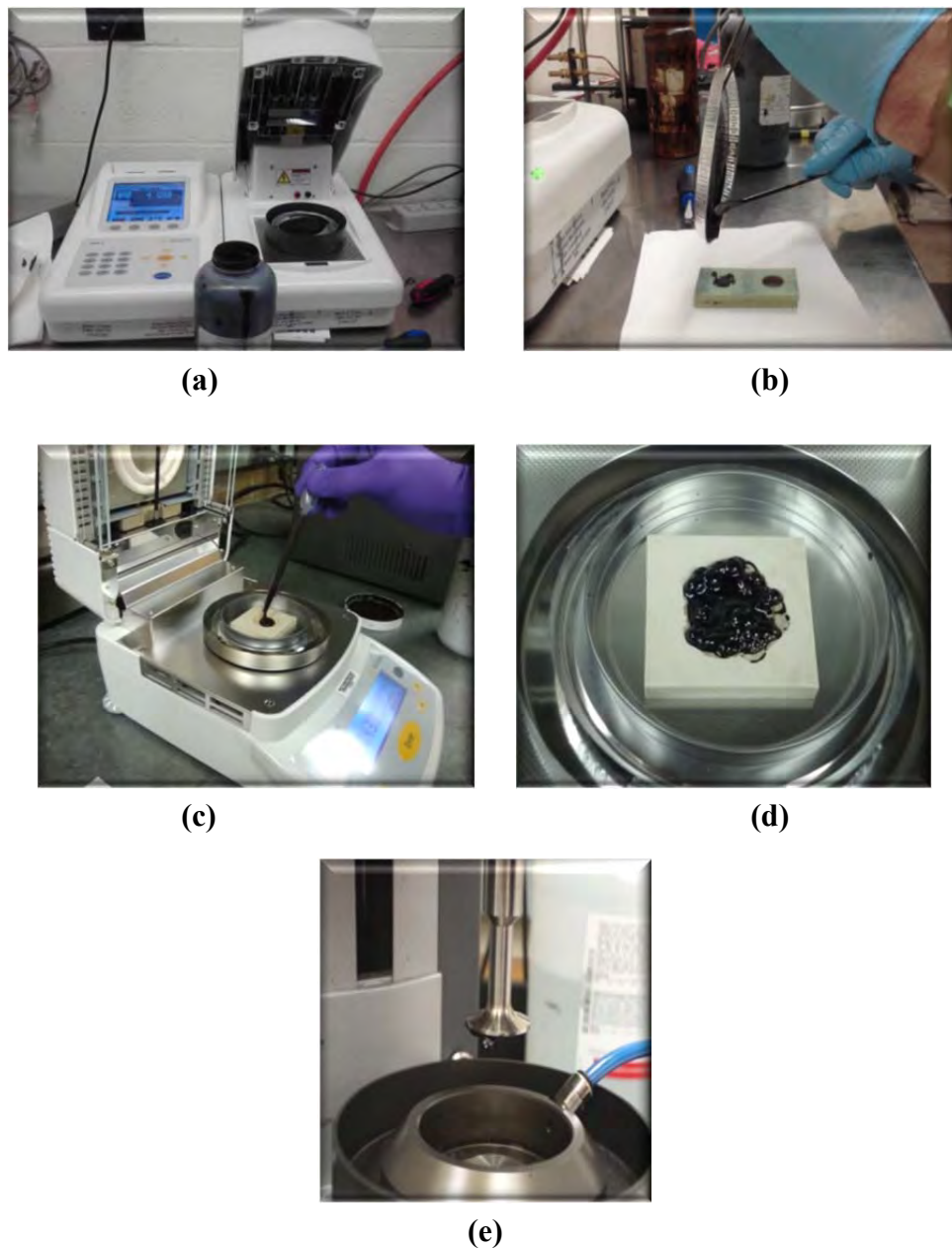
**FIGURE 4   MAB–DSR: (*a*) moisture analyzer balance; (*b*) recovery
and transfer to DSR mold; (*c*) direct recovery in silicone mold;
(*d*) recovered residue; and (*e*) residue placed on DSR.**

CONCLUSIONS

A new test method for measuring emulsified asphalt viscosity has been developed and is now a standard in ASTM. Furthermore, the specifications now include an option to use the paddle viscometer. New precision statements were obtained for both viscometers.

An extension of the MAB procedure now includes the recovery, with minimum sample manipulation and determination of the high-temperature performance grade of the emulsified

asphalt residue. Recent work, using the based binder used to make the emulsions, show that the recovered binder from the emulsified asphalt, if unmodified, has a similar shear modulus as the based binder. Further work on the MAB–DSR is continuing to optimize recovery conditions for unmodified and polymer modified emulsified asphalt and to determine other rheological properties for the recovered binders.

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Development of a More Rational System for Selecting Emulsions for Surface Treatments

ANDREW J. HANZ
PETRINA JOHANNES
PREEDA CHATURABONG
HUSSAIN U. BAHIA
University of Wisconsin, Madison

To promote sustainability and optimize use of funding State Highway Agencies (SHAs) are integrating pavement preservation concepts into management of their road way network. Pavement preservation alternatives include chip seals, micro-surfacing, and slurry seals, traditionally these treatments have been successfully placed on low volume roads. However, as advancements in emulsion formulations continue and use of surface treatments on medium and high traffic facilities becomes more prevalent, there is a need to improve the methods used to select and specify emulsions. The objective of this study was to develop an emulsion testing framework that captures properties related to critical distresses observed for surface treatments that is capable of considering the effects of service conditions including traffic, environment, and aging. The proposed framework recommends use of the DSR and bitumen bond strength (BBS) test to evaluate high, intermediate, and low-temperature performance. The testing protocols were applied to recovered and PAV-aged emulsion residues from emulsions formulated with different emulsifier chemistries and types of modification. Results indicate that the proposed test methods can characterize material performance and are sensitive to changes in emulsion formulation, however, additional research is needed to further develop test methods and to establish the relationship between laboratory-measured residue properties in order to define specification limits.

INTRODUCTION

Increasing construction costs and the motivation to reduce the environmental and social impacts of pavement construction have resulted in integration of pavement preservation concepts into state agency pavement management systems. Effective pavement management strategy is based on allocating funds both to reconstruct pavements that have failed and to conduct preventative maintenance on relatively new roadways to delay the onset of distress. As a result, over time the roadways in need of full reconstruction are reduced and the service life of existing pavements is extended (*1*). Numerous pavement preservation alternatives exist including, chip seals, slurry seals, and microsurfacing. Traditionally these treatments have been placed on low-volume roads, however recent advancements in emulsion technology, including increased use of modified emulsions, and the need to extend service life of existing pavements have resulted in use of surface treatments on roadways with average annual daily traffic (AADT) levels of 5,000 and in some cases 20,000 vehicles per day (*2*). Given application to more highly trafficked roadways there is a need to modernize the tests used in evaluation and selection of materials to prevent premature failure.

By definition, surface treatments are intended to impact the functional characteristics of the pavement by improving surface texture and providing an impermeable barrier to protect underlying pavement layers (*3*). A survey of state agencies and users identifies bleeding and raveling as the two most common distresses observed for chip seals. Specifically, these distress modes were identified by 33 and 22 states for aggregate loss and bleeding respectively (*4*). Furthermore, bleeding was recognized as the most common chip seal distress by 81% of the survey respondents, followed by raveling (67%), and both raveling and bleeding occurring at the same frequency (49%) (*2*). In regards to slurry seal and microsurfacing these modes of failure are also recognized as both resistance to raveling and bleeding are addressed in current ISSA guidelines (*5*). While limited tests on surface treatment systems exist to select emulsions, many of the tests are empirical and no relationships between emulsion residue properties and performance have been established.

Use of a performance-related system for selection of materials is the most direct way to decrease the probability of premature failure. In current practice, emulsion properties are specified by AASHTO M316 and AASHTO M140 standards. These standards use penetration and limited mechanistic testing as a basis for approving materials. These tests are not related directly to performance, because the effects of temperature, aging, and stress are not considered. As a result in current practice, state agencies take a high risk approach by relying on estimation from empirical tests that are not specific to climate and traffic conditions, and include limits based on past experience to select emulsions. This shortcoming extends to decisions on when to use modified emulsions. For example all of the 27 states that reported regular specification for use of modified emulsions indicated that the reasons for selection were qualitative and based mostly on field observation. Specific reasons include improved cold weather performance, increased aggregate retention, reduced bleeding, and improved constructability (*4*).

To advance the current state of practice, this paper summarizes recent efforts to develop a framework for emulsion selection based on the critical distresses and conditions observed in the field. The approach leverages existing technology developed in the SHRP program by proposing use of the DSR at a range of temperatures and stress levels, and also introduces the BBS test as a means to incorporate consideration of emulsion–aggregate compatibility. The effects of aging are also considered through evaluation of material properties on both recovered residue and PAV residue properties. Specific test methods and conditions were defined based on the functional requirements of surface treatments to improve texture through demonstrating resistance to raveling and bleeding and to protect underlying layers by assessing cracking potential.

BACKGROUND

Initial efforts to characterize emulsion residue properties using performance-based testing were presented in the surface performance grading (SPG) system (*6*). The system involved application of the SuperPave binder grading tests with adjustments made to account for the use of emulsion and its application at the pavement surface. To represent field conditions, the SPG-modified SuperPave standards by defining the high pavement temperature at the surface rather than at 20 mm below. Due to higher variation in surface temperatures, this modification required a reduction in the temperature increment between grades to 3°C. Adjustments were also made to aging methods and high- and low-temperature specification limits (*6*). A validation study was conducted after 1 year in service by comparing SPG results to the environmental conditions in

various areas of Texas and to distresses observed in visual field surveys. Results indicate that for 76% of the materials used the SPG agreed with the climatic grades required by analysis of surface temperatures in the LTTP database (*7*). The results of these research efforts contributed significantly to the advancement of emulsion technology by presenting improved methods to characterize the material and linking results to field performance.

The concept of emulsion selection based on the critical failure mechanisms that impact the functionality of chip seals was first introduced by Bahia and coworkers (*3*). This work proposed that surface treatments serve two main functions: to provide skid resistance and to protect underlying areas by providing an impermeable barrier. These functions are compromised by loss of surface texture at high and intermediate temperatures due to bleeding or raveling and loss of impermeability at intermediate and low temperatures due to cracking. The modes of distress considered are similar to other research which identified aggregate loss, bleeding, and cracking as important considerations in chip seal design and life-cycle cost analysis (*2*, *8*). The study also recognized that seal performance is not only a function of emulsion residue properties, but also depends on external factors that impact shear, tensile, and fatigue behavior. Specifically these factors include, surface hardness, stiffness of underlying layers, traffic speed and loading, and climate in terms of both temperature and moisture. As a result, a testing framework was proposed that addresses critical modes of distress and considers the effects of these external factors through application of tests at a wide range of temperatures and modes of loading (*3*, *9*). The main difference between this framework and the SPG approach was rather than direct application of test methods used for grading of hot applied binders, new tests were proposed intended to better simulate the distresses experienced by surface treatments.

The use of chip seal performance properties as a basis for emulsion selection was further advanced in the FLH by definition of test methods to evaluate performance properties of polymer modified emulsions (*10*). To characterize modified emulsions the study piloted a low-temperature residue recovery method (ASTM D7497 Method A) and proposed a variety of test methods. The testing protocols used in the project were developed in conjunction with the FHWA ETF, with similar test methods integrated into studies by Hanz and coworkers (*11*) as well as Hoyt and Epps Martin (*12*). The testing framework recommends use of the DSR and BBR to characterize emulsion residue properties after recovery and PAV aging. Tests included the MSCR test at high temperatures, frequency and strain sweeps at intermediate temperatures, and the BBR to evaluate low-temperature performance. Results indicate that the proposed test methods were able to differentiate between emulsion types, showing dramatically lower performance properties for one emulsion; in the field the same material experienced premature distress (*10*). A follow up to this initial study was published in 2012, although significant differences in emulsion residue performance properties were found in laboratory testing, to date the field sections generally did not demonstrate high levels of distress. Exceptions include bleeding observed at intersections and isolated areas of heavy chip loss due to ditch-line construction and plowing operations (*13*).

DEVELOPMENT OF PERFORMANCE EVALUATION FRAMEWORK

The concept driving the proposed performance evaluation framework is definition of the functional requirements of surface treatments and the conditions that cause distress as a means to simulate in-service conditions in the laboratory. This relationship is provided in Table 1.

As shown in Table 1 distresses occur under varying service conditions and at various stages of service life. Therefore, it is necessary that the emulsion selection system includes testing conditions that represents a range of temperatures and loading conditions and also includes various types of material conditioning to evaluate the effects of moisture and aging. A schematic of this concept is presented in Figure 1.

The proposed emulsion evaluation framework leverages the use two devices, the DSR and BBS test, to simulate various forms of surface treatment distresses. Selection of these devices was based on the concept that bleeding and cracking distresses are attributed to failures within the emulsion residue, whereas raveling can occur both due to a failure within the asphalt binder and at the asphalt–aggregate interface due to failure of the bond in the dry condition or due to the presence of moisture. The DSR was applied to evaluate emulsion residue related distresses, the BBS test was selected to evaluate the bond at the emulsion residue–aggregate interface.

The effects of aging were considered by testing both the emulsion residue and the emulsion residue after PAV aging. The emulsion residue recovery method selected was the low-temperature evaporation procedure specified in ASTM D7497 Method B, which involves drawing the emulsion film down to 380 microns and conditioning in a forced draft oven at 60°C for 6 h. The low-temperature procedure was selected based on recent recommendations made by the ETF and research indicating that emulsion residue recovery under these conditions did not compromise the polymer network of modified emulsions (11). Long-term aging was conducted using the PAV at the film thickness specified in AASHTO M320; minor modifications were made to the sample size to accommodate the low quantity of emulsion residue obtained from the selected recovery method. The specific test methods used and justification for their selection are provided in Table 2.

TABLE 1 Functional Requirements of Surface Treatments and Related Distresses

| Functional Requirement	Applicable Treatment			Related Distresses
	Chip Seal	Microsurfacing	Ultrathin Overlay	
Skid resistance	X	X	X	Bleeding at high temperatures; raveling at intermediate temperatures
Protect underlying pavement structure	X	X	X	Cracking due to load–thermal stress and aging.
Rut filling	NA	X	X	Instability caused by temperature–traffic

NOTE: NA = not applicable.
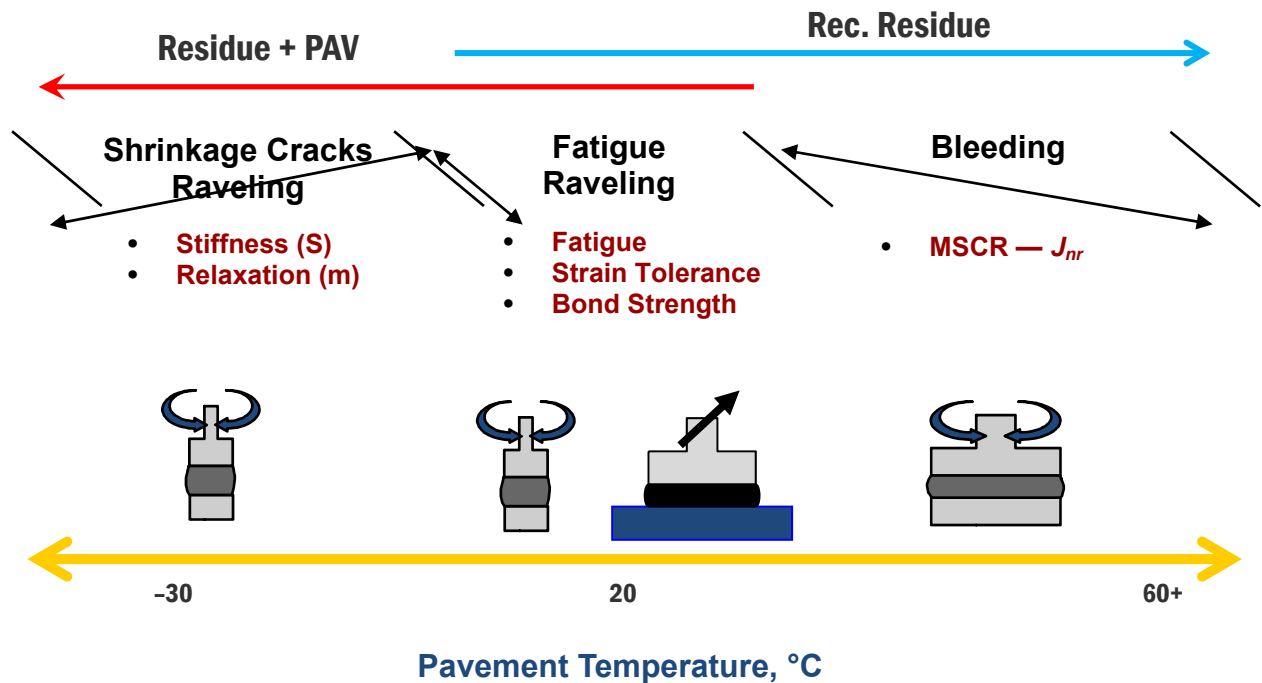
FIGURE 1 Schematic of the performance-grading framework.

TEST METHODS

Resistance to Bleeding

Bleeding can occur both due to construction- and materials-related failures. Aspects of construction that cause bleeding include used of an excessive application rate or embedment of the aggregate into a soft pavement surface (*3*, *14*). Regarding materials properties, bleeding is related to emulsion residue performance at high service temperatures and is caused by viscous flow of the emulsion to the treatment surface due to softening related to temperature or stress. To simulate this behavior in the laboratory the MSCR test was selected. For MSCR testing the loading scheme and number of cycles specified in AASHTO TP70 were maintained. The standard requires testing at the climatic grade temperature at stress levels of 0.1 and 3.2 kPa. In this study, a range of test temperatures was used and stress levels were modified by replacing 0.1 kPa with 1.0 kPa and adding a third stress level of 10 kPa to better represent the softening due to temperature or increasing stress realized in the field.

Resistance to Raveling

Potential mechanisms for raveling of in-service chip seals include adhesive failures at the emulsion residue–aggregate interface or cohesive failure within the emulsion residue. The study aimed to address both of these failure mechanisms by use of the BBS test and application of a strain amplitude sweep test to evaluate strain tolerance.

TABLE 2 Summary of Proposed Emulsion Evaluation Framework

| Engineering Property	Test Method Output	Justification
Tests on Emulsion Residue		
Resistance to bleeding	MSCR–AASHTO TP-70; J_{nr}, % rec. stress difference	Evaluate effects of stress and temperature
Resistance to early raveling	BBS Test–AASHTO TP-91; bond strength, BSR (wet–dry)	Evaluate integrity of bond with aggregate and effects of moisture
	LAS Test–AASHTO TP-101; strain @ max. stress	Evaluate binder strain tolerance as a measure of binder resistance to raveling
Elasticity	Elastic recovery in the DSR; % elastic recovery	Define presence and effect of modifiers on elastic response
Tests on PAV-Aged Emulsion Residue		
Resistance to late raveling	LAS Test–AASHTO TP-101; strain @ max. stress	Evaluate effect of aging on binder strain tolerance
Resistance to fatigue cracking	LAS Test–AASTHO TP-101; cycles to failure (N_f)	Establish relationship between predicted fatigue life and strain
Resistance to thermal cracking	DSR frequency sweep to estimate BBR; S (60) and m (60)	Identify formulations susceptible to brittleness at low temperatures

NOTE: max. = maximum.

The BBS is a pneumatic adhesion test adapted from the paint and coatings industry (ASTM D4541) that has recently been accepted as a provisional AASHTO Test Method (TP-91).

The device and procedure were developed in recent research to evaluate moisture damage in conventional binders and the rate of curing of fresh emulsions (*15*, *16*). The test involves subjecting a pull stub adhered to an aggregate substrate to a normal force created by increasing pneumatic pressure. The bond strength is defined as the maximum pull off pressure exerted by the machine. The device and a schematic of the loading mechanism are provided in Figure 2.

In this study the bond strength in the dry condition and the change in bond strength after moisture conditioning, as defined by the ratio of wet to dry bond strength (BSR) were used as evaluation parameters. Moisture conditioning was conducted by submersion in a 40°C water bath for 24 h. In addition the mode of failure was determined through visual examination of the failure surface to determine if failure occurred within the emulsion residue or at the emulsion residue–aggregate interface. The value of including visual inspection of the failure surface is presented in Figure 3; results clearly indicate use of different additives impacts the mode of failure. While these results are presented for hot-applied binders, similar concepts hold for emulsions as there is potential for interaction between different emulsifiers and aggregate mineralogy to influence bonding.

Emulsion residues are also required to demonstrate adequate strain tolerance, as the thin film between the asphalt–aggregate surfacs is subjected to high strains, particularly under slow or heavy traffic loading. The strain amplitude sweep conducted during the LAS test was used to
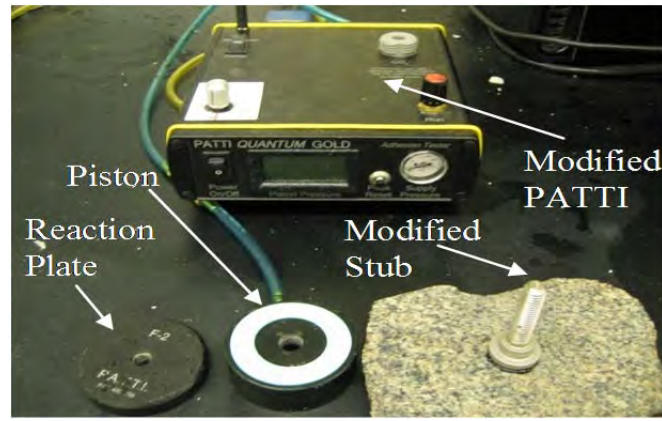
(a)
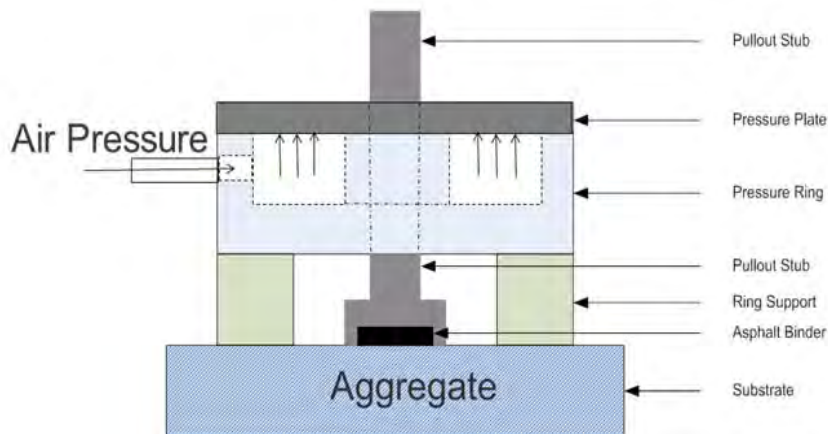
(b)

**FIGURE 2 (*a*) Binder bond strength testing device and
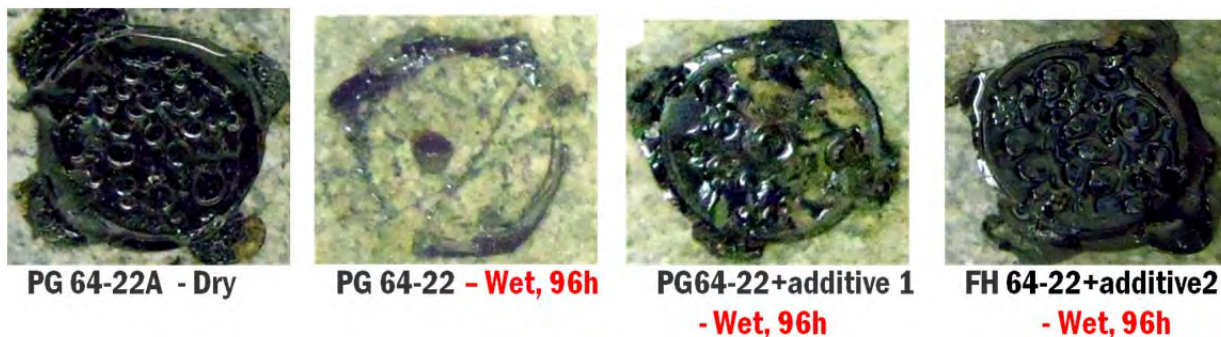(*b*) schematic of loading mechanism.**

FIGURE 3 Effect of additives on BBSbinder bond strength failure modes.

evaluate this behavior (*17*). During the test strain is increased linearly from 1% to 35%; at each strain the sample is subjected to 100 cycles of loading and the resulting stress is recorded. In preliminary work, all testing was conducted at 19°C and a frequency of 10 Hz strain tolerance was evaluated as the strain at maximum stress. Current research is underway to evaluate the effect of temperature on strain tolerance. An example of the determination of strain at maximum stress based on the LAS loading scheme and the effect of emulsion type is presented in Figure 4.

Elastic Recovery in the DSR

Current protocols in evaluation of polymer-modified asphalts and emulsions require the elastic recovery test specified in AASHTO T301. The test is not directly applicable to emulsion residues because the testing geometry requires excessive heating of the samples to mold the material into a dog-bone–shaped specimen and the selected residue recovery procedure does not provide sufficient material. To address these issues the elastic recovery test in the DSR (ER–DSR) proposed by Clopotel was selected (*18*). The test simulates the AASHTO T301 procedure by subjecting an 8-mm DSR sample to a strain rate of 2.32%/s for 120 s followed by imposing a controlled stress condition of 0 Pa for 30 min. Percent recovery is calculated as the ratio of the strain after recovery to the strain immediately after loading. Previously presented results have shown strong correlation between the ER–DSR and the standard elastic recovery test, however, the ER–DSR test consistently under predicted conventional measurements of elastic recovery by approximately 15% (*18*).

Resistance to Cracking

Cracking at intermediate and low temperatures compromises the ability of chip seals to serve as an impermeable barrier. Loss of this functionality has the potential to degrade the strength and
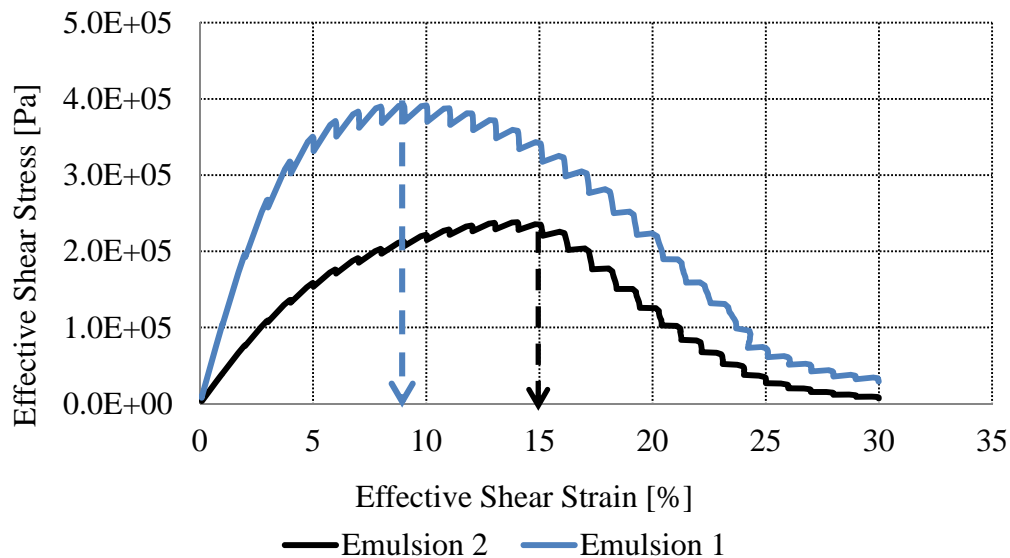

FIGURE 4 Determination of strain at maximum stress from AASHTO TP-101 LAS output and effect of emulsion type.

stiffness of the pavement layer through allowing exposure to moisture, prompting the need for premature full rehabilitation. At intermediate temperatures the mechanism driving failure is fatigue cracking, which was evaluated in this study through use of the LAS test. The procedure as specified in AASHTO TP101 involves measurement of undamaged properties using a frequency sweep followed by the strain amplitude sweep previously mentioned. Experimental data are modeled using viscoelastic continuum damage (VECD) concepts to develop the relationship between fatigue performance and damage intensity. Based on this relationship a fatigue law is defined that provides cycles to failure (N_f) as a function of applied strain (*17*).

Low-temperature performance was evaluated using estimates of the BBR parameters of stiffness and *m*-value after 60-s loading. Practical considerations prevented direct measurement of BBR performance due to the small amount of residue recovered and the need to minimize heating of the sample. Instead, low-temperature creep properties were estimated from intermediate temperature shear properties using well-known interconversion methods (*19, 20*). The equations for inter-conversion are presented below.

$$S(t) \approx \frac{3G^*(\omega)}{[1+0.2\sin(2\delta)]} \tag{1}$$

where

$\quad S(t)\ =\ $ creep stiffness at time, *t*, Pa;
$G^*(\omega) =\ $ complex modulus at frequency ω, Pa; and
$\quad\ \delta\ =\ $ phase angle at frequency ω, Pa.

$$m = \frac{d(\log G^*)}{d(\log \omega)} \tag{2}$$

where

$\quad m =\ $ slope of G^* versus frequency plot at a given frequency;
$\quad \delta\ =\ $ phase angle;
$\quad G^* =\ $ complex modulus; and
$\quad \omega\ =\ $ frequency (rad/s).

The shear parameters (G^*, δ) required to provide the estimates of stiffness and *m*-value were obtained from a master curve based on data from a frequency sweep (1 to 150 rad/s) conducted at DSR temperatures of 5°C, 10°C, and 15°C. Previous studies have shown a strong correlation and equivalency between measured and predicted values for both stiffness and *m*-value for both asphalt binders and emulsion residues (*21*).

PRELIMINARY RESULTS AND ANALYSIS

Resistance to Bleeding

The J_{nr} at 3.2 kPa versus temperature relationship for anionic and cationic emulsions are provided in Figure 5*a* and 5*b* and respectively. As a frame of reference the J_{nr} limits associated

with standard ($S = 4.0$ kPa^{-1}) and heavy ($H = 2.0$ kPa^{-1}) traffic levels in AASHTO MP19 are included in the figures. Results indicate that both sensitivity to temperature and the effectiveness of modification are dependent on the emulsifier chemistry used. Significant differences in performance of the anionic series emulsions were observed as values of J_{nr} at 64°C ranged from 2.0 to 6.0 kPa^{-1}. In applying the AASHTO MP19 criterion, the failure temperature for the HFRS-2 (S grade) is approximately 58°C, failure temperatures for the modified emulsion (H grade) are 58°C and 62°C for the latex- and polymer-modified products, respectively. Conversely, the cationic emulsions perform similarly regardless of emulsion modification, achieving an S grade at approximately 62°C. These results demonstrate the value of incorporating performance-based criterion in formulation and selection of emulsions as based on current practice it is assumed that modified emulsions perform similarly, regardless of type of modification and further that they improve performance relative to unmodified products. The variation in stress sensitivity at two stress differences for modified and unmodified emulsions at 64°C is presented in Figure 6; similar results were observed at 52°C.

Results presented in Figure 6 indicate higher stress sensitivity for modified emulsions, regardless of emulsifier chemistry. This behavior is attributed to the presence of a third component in the residue in the form of either a latex or polymer network. As stress increases the integrity of the network deteriorates, thus causing a higher change in J_{nr} relative to conventional products. These results highlight the importance of selecting the appropriate service conditions in performance grading as there is potential that the effectiveness of modified emulsions could diminish under high stress conditions, causing decreased bleeding resistance due to stress softening. An example of this is provided in Figure 5b for the cationic series emulsions, as similar performance is observed at a stress level of 3.2 kPa, based on variations in stress sensitivity at higher stress levels there is potential that the unmodified product will demonstrate improved performance relative to emulsions modified with polymer or latex.
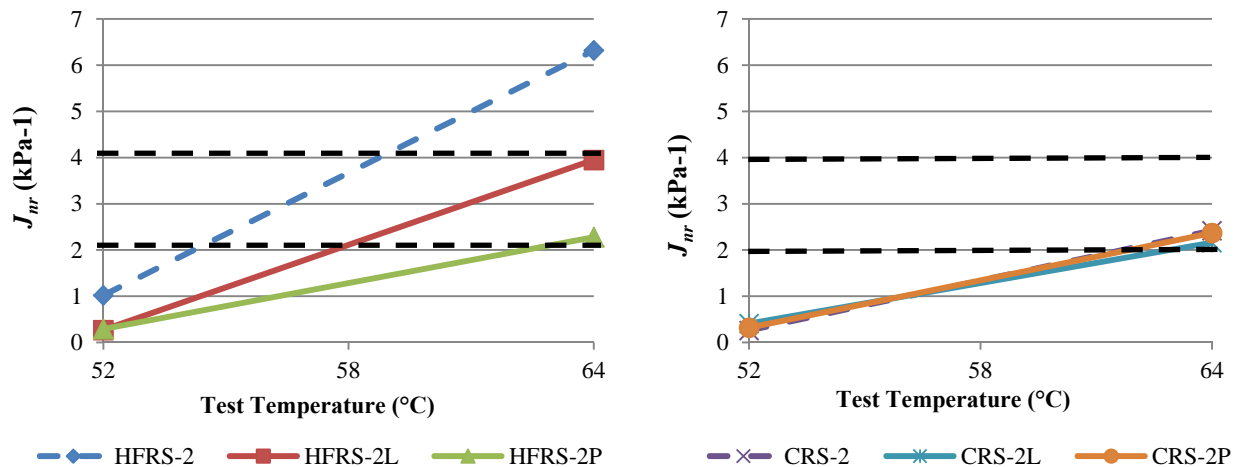

FIGURE 5 Effect of emulsion modification and chemistry on J_{nr} versus temperature relationship.
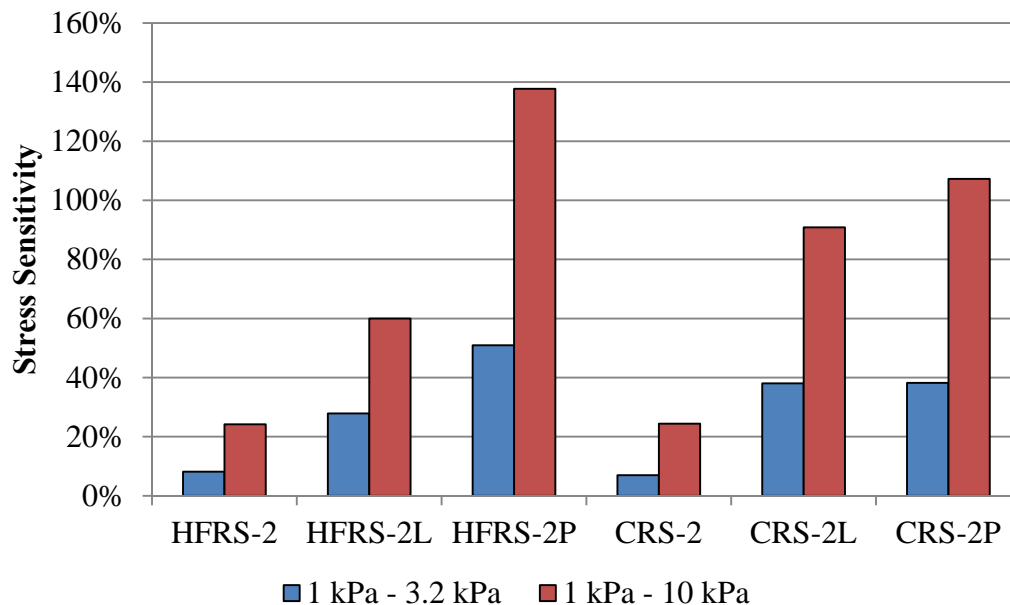
FIGURE 6 Effect of modification and stress level on stress sensitivity (64°C).

Resistance to Raveling

In-service raveling is characterized as dislodging of the aggregate chip from the surface of chip seals or degradation at the surface of slurry seal–microsurfacing treatments due to traffic loading. Failure mechanisms include a loss of bonding at the emulsion residue–aggregate interface or failure within the emulsion residue due to the applied strain from trafficking exceeding the strain tolerance of the material. The BBS test was applied to evaluate the integrity of the bond at the emulsion residue–aggregate interface and how it was influenced by moisture. All tests were conducted on a granite aggregate substrate from north central Wisconsin. Results are presented in Figure 7. As a measure of moisture damage, the BSR defined as the ratio of wet-to-dry strength is provided as a secondary axis. Results for RTFO-aged based binders are also provided.

Results presented in Figure 7 indicate that both dry and wet bond strength are sensitive to emulsion type and modification with ranges in bond strength of 150 and 100 psi for the dry and wet conditions, respectively. In regards to the effect of emulsion type, higher values of bond strength are observed for conventional emulsions relative to modified emulsions with the CRS-2 emulsion achieving the highest value of bond strength. All values of bond strength are lower than those of the base binders, some by a factor of 2. Based on the use of residue recovery for emulsions and RTFO aging for the base binders it is unclear if these differences are attributed to material properties or different aging conditions. Results also indicate that the presence of the emulsifier and use of modification improves resistance to moisture damage as BSR values of most emulsions are higher (improved moisture resistance) compared to the base binder by approximately 10% and use of modified emulsions for a given chemistry improves moisture
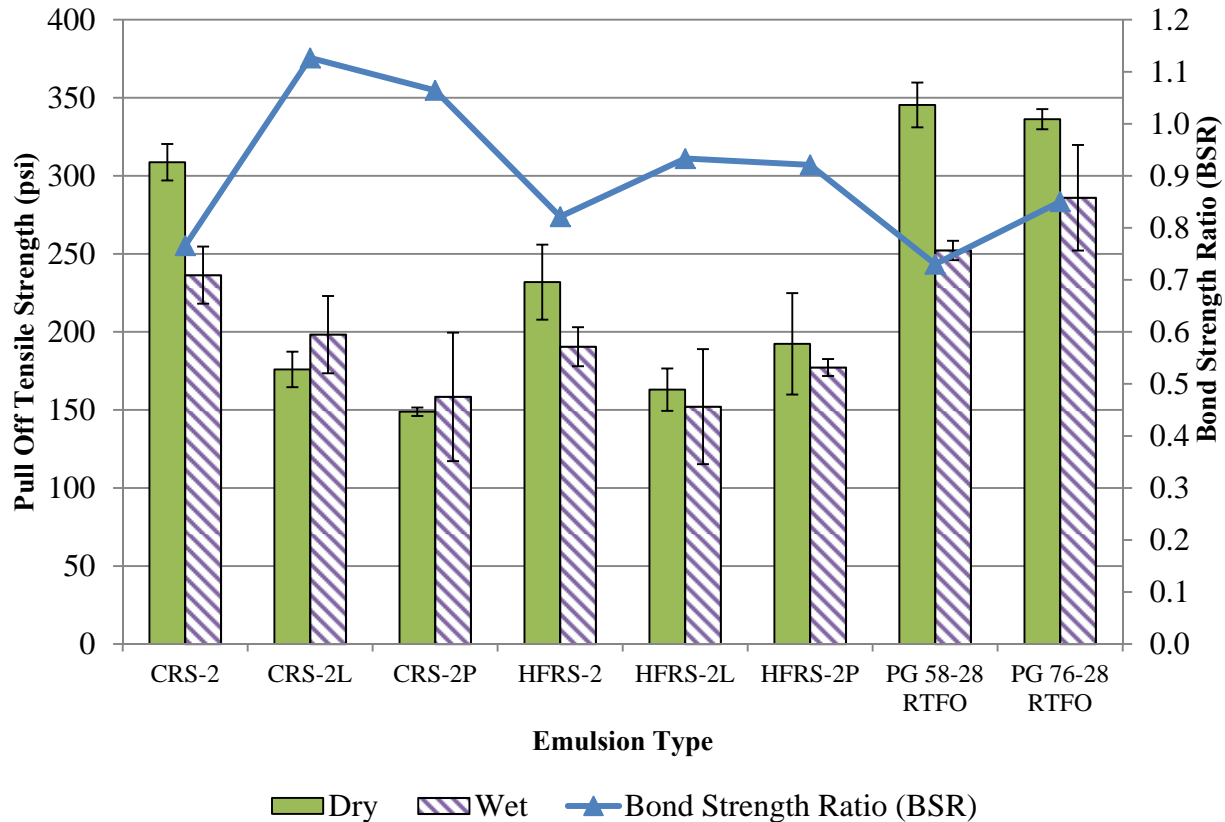
FIGURE 7 Evaluation of raveling resistance at emulsion–aggregate interface: effect of emulsion type, modification, and moisture on bond strength at 22°C.

damage resistance relative to unmodified products. Results of strain tolerance evaluation as presented as the strain at maximum stress are provided in Figure 8 for both recovered and PAV-aged emulsion residue. The PAV aging condition was included to assess the effects of embrittlement due to aging on strain tolerance.

The ability of modified emulsions to improve strain tolerance and thus reduce raveling caused by failure of the emulsion residue is clearly demonstrated in Figure 8 as use of latex modification results in increases in strain at maximum stress ranging from 2% to 6%. Furthermore, polymer-modified systems demonstrate increased strain tolerance relative to latex-modified emulsions for both emulsifier chemistries. Differing effects of aging were observed for conventional and modified emulsions as conventional products had slight increases in strain tolerance while the decrease in strain tolerance for modified emulsions ranged from 1% to 3%. In all cases, regardless of aging condition the modified emulsions demonstrated strain tolerance equal to or higher than that of modified emulsions, indicating potential improvements in raveling resistance when modified products are used.
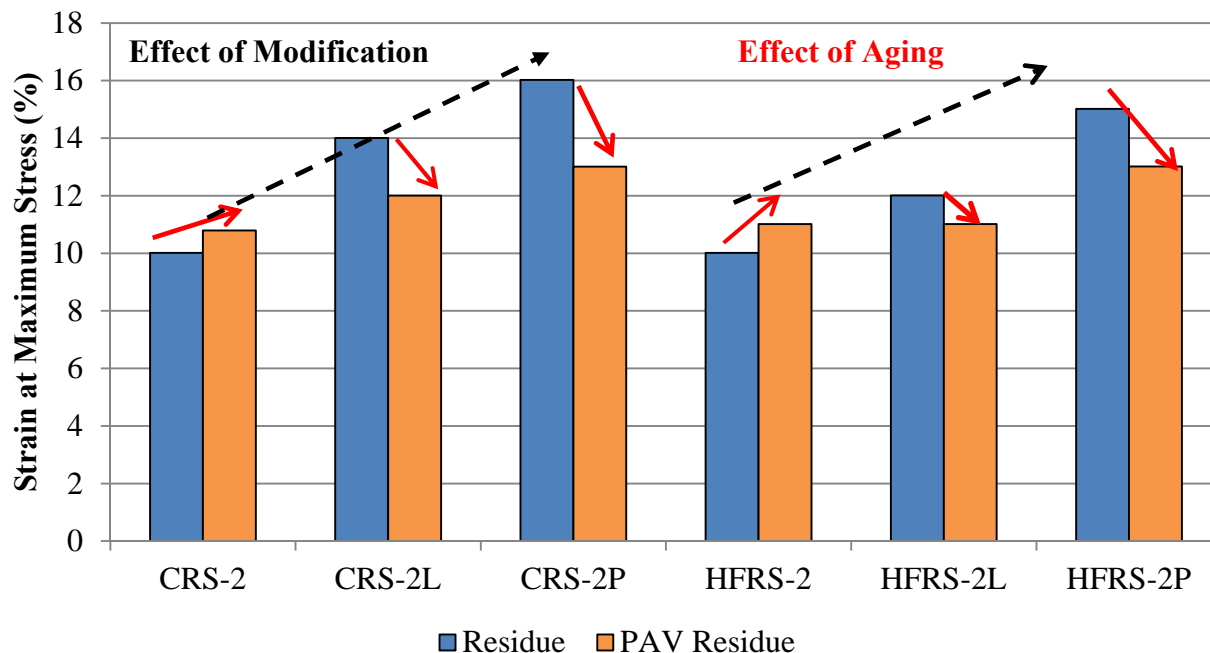
FIGURE 8 Evaluation of raveling resistance due to binder strain tolerance: effect of emulsion type, modification, and aging on strain tolerance.

Elastic Recovery

The ER–DSR test as developed by Clopotel (*18*) was selected to provide measurement of a material property currently used to evaluate modified emulsions. Results for tests conducted at a temperature of 25°C provided in Figure 9 indicate that polymer modification provides a greater increase in elastic recovery than use of latex relative to unmodified emulsions. Specifically, use of polymer and latex modification increased elastic recovery by approximately 35% and 15%, respectively. Furthermore, sensitivity to emulsifier chemistry was not observed and performance of the unmodified and modified emulsions was similar to that of the base binders.

Resistance to Fatigue Cracking

The LAS test as specified by AASHTO TP101 was used to evaluate the effect of emulsion modification on the relationship between applied strain and fatigue life through application of VECD analysis on the stress versus strain relationship presented in Figure 4, using procedures developed by Johnson and Hintz (*17*, *22*). As indicated in Figure 10, for the anionic series of emulsions, modification has a little to no effect on estimated fatigue life as all three emulsions demonstrate similar cycles to failure versus strain relationships, with the polymer-modified emulsion demonstrating marginally higher values of fatigue life. Similar results were observed for the cationic series of emulsions. In this study, all tests were conducted at 19°C and investigation of additional test temperatures is proposed to improve the understanding of the effects of emulsion modification on estimated fatigue life.
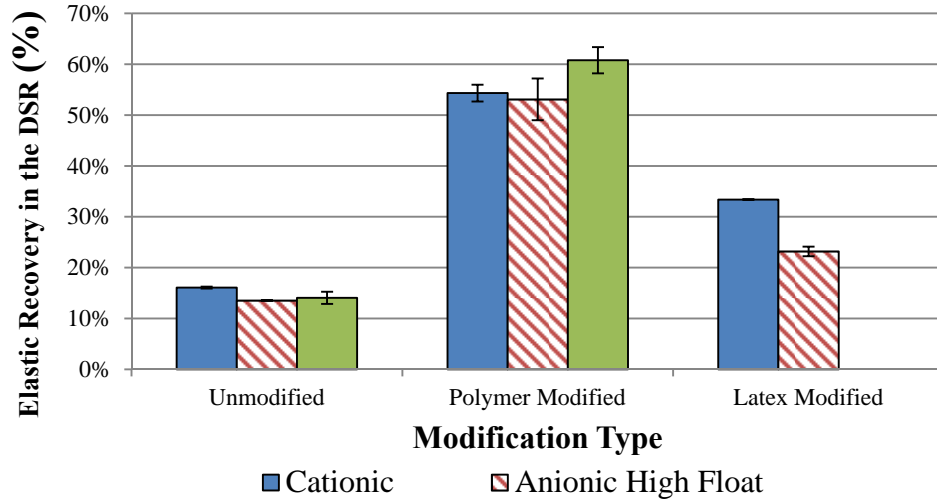
**FIGURE 9 Summary of ER–DSR results for unmodified
and modified emulsions and base asphalts.**
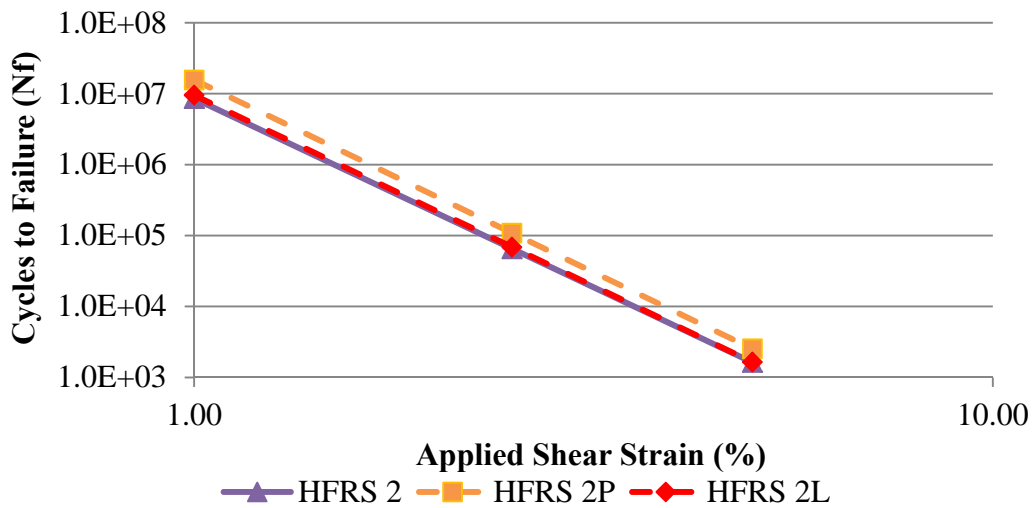
**FIGURE 10 Effect of modification on emulsion residue
fatigue resistance to thermal cracking.**

Low-temperature properties were estimated using interconversion of shear properties measured at intermediate temperatures. The effect of emulsion type on estimated $S(60)$ and the low-temperature continuous grade are provided in Figure 11 and Figure 12, respectively. Grading of the base binders are also included as a frame of reference. Results indicate that low-temperature properties are sensitive to emulsion type as ranges in stiffness of approximately 200 MPa and low-temperature continuous grades of approximately 4°C are observed. In general, modified emulsions demonstrate improved low temperature properties relative to conventional products for both emulsifier chemistries.
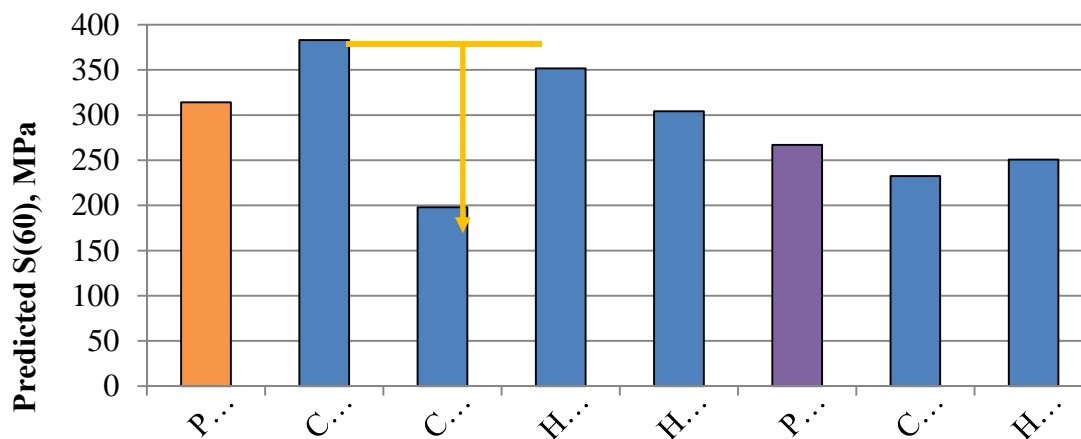
**FIGURE 11 Effect of emulsion type and modification on predicted
low-temperature stiffness at –12°C PAV-aged emulsion residue.**
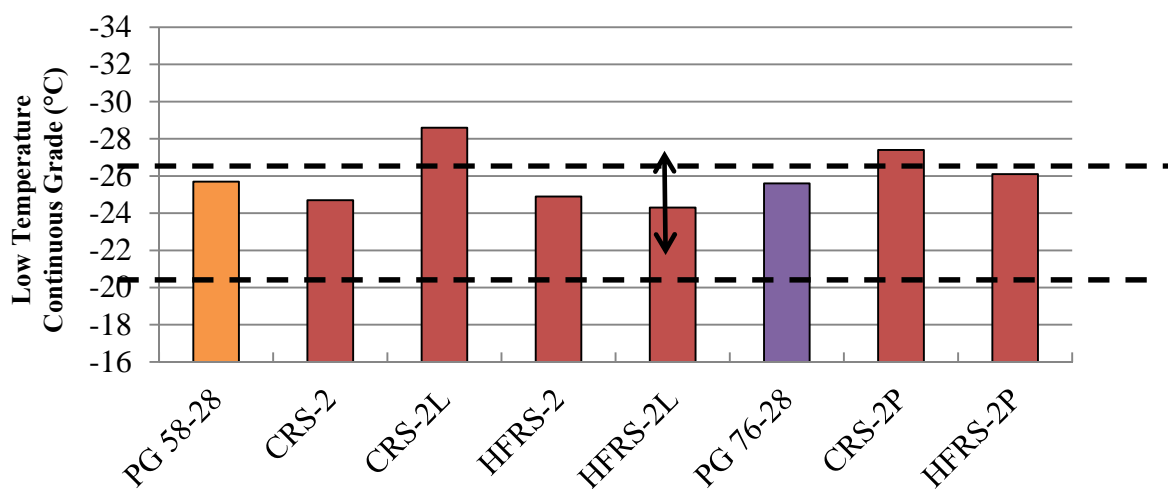
**FIGURE 12 Effect of emulsion type and modification on
predicted LT continuous grade–PAV-aged material.**

SUMMARY OF FINDINGS AND RECOMMENDATIONS

This paper summarizes an approach for improved evaluation of emulsion and residue performance properties based on the functional requirements of surface treatments and the service conditions experienced in the field. Based on the concepts and results presented the following findings and recommendations are stated:

1. To advance the use of emulsion technology in surface treatments it is necessary to implement a framework based on the functional requirements of surface treatments. Current practice relies on past experience and empirical test methods conducted at a limited range of temperatures and loading conditions. There is an opportunity to improve emulsion

characterization by leveraging use of existing technology to evaluate the effects of temperature, stress, and aging on material properties.

   2.   The performance evaluation framework proposed in this study shows promising potential to differentiate between emulsion types and to quantify the effects of modification. Particular sensitivity to materials type is observed at high and intermediate temperatures.

   3.   Differing effects of modification are observed between polymer modifiers for cationic and anionic chemistries. These results support the need for performance-related specifications as the effectiveness of modification is formulation specific. Therefore, the current practice of expecting improved performance through only specifying a modified emulsion is insufficient. Modification effects can be different and should be specified based on specific climactic conditions and distress mechanisms.

   4.   Additional research is needed for further development of test methods. Specific examples include the effects of test temperature on fatigue life as predicted using the LAS test, and validation that the residue recovery and PAV procedures used represent the condition of the emulsion residue in the field while in-service.

   5.   To fully develop the concept of implementing a performance based methodology for emulsion selection comparisons of residue properties to the performance of laboratory prepared surface treatment specimens and field performance are required. Establishing the relationship between residue and seal performance would quantify the significance of the range in performance properties observed in this study and promote development of the specification limits required to properly select materials.

   6.   While the example data set provided in this study is limited to emulsions used in chip seals, the concepts and test methods presented could be applied to other surface treatments as well. In further development of this framework it is necessary to define application based performance limits. In addition, it is necessary to extend this concept to fresh emulsion properties through development of test methods and specification limits for emulsion viscosity, storage stability, and curing.

ACKNOWLEDGMENTS

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Performance Evaluation of Chip Seals

Y. Richard Kim
Javon Adams
Jeong-Hyuk Im
North Carolina State University

Jaejun Lee
Chonbuk National University, South Korea

Over the last decade, great emphasis has been placed on pavement preservation nationwide. Pavement preservation treatments are an effective means of improving surface quality and extending the service life of pavements. The design of pavement preservation treatments has undergone significant developments over the past two decades, particularly in South Africa, France, Australia, New Zealand, and the United States. In the United States, these treatments have become increasingly important as tools for highway agencies to use in response to the aging and deterioration of the nation's road network. Various types of surface treatments, when combined, constitute a group of pavement preservation treatments that state agencies typically employ. One of the most cost-effective preservation treatments is the chip seal. A chip seal is an asphalt surface treatment formed by applying emulsified asphalt and aggregate. Chip seal surface (ChipSS) treatments provide a durable surface layer that protects the existing pavement surface from water infiltration and environmental aging–oxidation effects. Additionally, a safety benefit of chip seal surface treatments is that they add skid resistance to the road surface by increasing the roughness of the surface. Most of the research detailed herein discusses findings and advancements made in the field of chip seal design and construction from a series of research projects conducted at North Carolina State University (NCSU).

ASPHALT SURFACE TREATMENT PERFORMANCE TEST METHODS

The following descriptions provide a brief summary of the test methods utilized in this paper to perform various types of analyses on both asphalt emulsion materials and surface treatment mixtures. These test methods are used to evaluate several asphalt surface treatment material types, environmental conditions, and seal–treatment alternatives to improve the quality and overall performance of the surface treatment in practice.

Third-Scale Model Mobile Loading Simulator

The third-scale model mobile load simulator (MMLS3) simulates the traffic loading conditions experienced by asphalt surface treatments under field traffic. The MMLS3 applies repeated wheel loads to the asphalt surface at a constant and accelerated rate (990 wheel loads applied every 10 min) and causes the surface treatment to respond similarly to its response in the field. The machine itself consists of a rotating drum that drives a train of buggies across a set of test samples mounted beneath the machine. The train includes a total of eight buggies, four of which have third-scale wheels (relative to standard dual-tire wheels). A maximum of three samples

(356-mm length per sample) can be secured underneath the MMLS3 for testing at one time. The cumulative sample length of 1,067 mm is the effective loading length for the MMLS3. With a wandering width of 178 mm, the effective MMLS3 loading area is 7,468 mm. For chip seal surface treatment testing, three samples can be mounted simultaneously for testing. The MMLS3 test is used to measure the aggregate loss, bleeding, and rutting performance of the surface treatment samples. Figure 1 presents the MMLS3 test equipment and procedure. The one departure from the picture shown in Figure 1*d* is that in actual testing the top of the MMLS3 temperature chamber would be covered to maintain the test temperature.

For MMLS3 testing, specimens are fabricated through a procedure that closely simulates the surface treatment fabrication in the field. This fabrication procedure is detailed later in this paper. The test method allows for complete temperature control between approximately 10°C and 60°C using an environmental chamber and cooling–heating unit. First, specimens are secured underneath the MMLS3 machine using screws, or clamps, to affix the specimens to the base as shown in Figure 1*a*. Then, the test temperature is set on the heating unit control box (to ±1°C). After allowing adequate time for the temperature to reach its target and for the specimen temperature to stabilize, testing can begin. Wandering simulates the natural wandering of traffic across the surface treatment under field loading.

FIGURE 1 MMLS3 test preparation: (*a*) installation of specimens on steel base; (*b*) side view of MMLS3; (*c*) positioning the MMLS3 in the temperature chamber; and (*d*) complete MMLS3 test setup.

ChipSS Sweep Test

The ChipSS sweep test method is a derivative of the ASTM D7000 test method and offers a different sample fabrication procedure from the ASTM D7000 test method. The ChipSS sweep test measures raveling performance in the same manner as the ASTM D7000 method and utilizes the same ASTM D7000 specified test equipment as well. However, the ChipSS sweep method is designed to improve upon the specimen fabrication procedure outlined in the ASTM specification by removing aggregate segregation issues that can occur when the ASTM D7000 procedure is used to fabricate specimens for aggregate spreading. The distinguishing characteristic of the ChipSS sweep test is that sample fabrication is performed using the ChipSS aggregate spreading device. This sample fabrication procedure using the ChipSS aggregate spreader is discussed later in this paper.

Vialit Adhesion Test

The Vialit adhesive test uses both gravity and impact to measure the aggregate retention capabilities of a chip seal. The test method standard is published as British Standard EN12272-3 (*1*). A sample is fabricated on a Vialit plate, which is a 203-mm-x-203-mm square stainless steel plate. Like the MMLS3 test samples, the Vialit test samples are cured at 35°C for 24 h before testing. Prior to the test, but after curing the samples, a flip-over test (FOT) is conducted (ASTM D7000). The purpose of the FOT is to remove any excess aggregate from the surface. In this procedure, the sample is turned at a 90° angle, and the entire area of the specimen is brushed lightly once with a soft-bristle brush. This process simulates the sweeping and removal of excess aggregate in field construction. After the FOT, the samples are weighed and conditioned to the proper test temperature. Once a sample has been fully conditioned, it is turned 180° and placed face down in the Vialit adhesion apparatus, shown in Figure 2. A steel ball (500 ± 5 g) is then released from its resting position so that it falls vertically 500 mm and strikes the back of the sample plate. The steel ball must be dropped three times from the elevated resting position within a 10-s time limit for a test to be considered valid. After all of the drops have been completed, the sample is reweighed to determine the amount of aggregate that was lost during the test.

Bitumen Bond Strength Test

The BBS test is a pneumatic adhesion test adapted from tests used in the paint and coatings industry (ASTM D4541). The BBS test device and procedure have been modified and developed in recent research to evaluate the curing rate of asphalt emulsions (*2*) and moisture damage in conventional and modified hot asphalt binders (*3*, *4*).The test also has been recently accepted as a provisional AASHTO test method (TP91). The BBS test protocol requires a bond to be prepared between an aggregate substrate and the binder (emulsion, residue, or hot binder) under controlled temperature and humidity conditions. A 20-mm diameter pull-out stub with a thickness of 0.8 mm is affixed to the binder. The pull-out stub thickness helps to control the film thickness of the binder on the aggregate substrate. The pull-out stub is subjected to a normal force using a pneumatic adhesion tensile testing instrument (PATTI) quantum gold testing unit produced by Semicro and M. E. Taylor Engineering, Inc. The maximum force required to detach the pull-out stub from the binder is recorded by a computer equipped with a LabView-based software interface for data collection and analysis. The parameter measured is the bond strength, defined as the maximum pull-off pressure exerted by the machine. Figure 3 shows the device used in the BBS test.

FIGURE 2 Image of the Vialit test apparatus.

Three-Dimensional Laser Profiler

Figure 4 shows the three-dimensional (3-D) laser profiler developed at NCSU. The laser profiler is used to capture macrotexture surface data that can be used to measure texture depth as well as embedment depth, which are critical parameters of surface treatment performance. The laser measures the distance between the laser sensor and the pavement surface in both the longitudinal and transverse directions of the pavement and produces a 3-D map of the pavement surface texture. The laser in this device is a point laser that captures a single point at a time as the device travels in both the transverse and longitudinal directions. The laser profiler dimensions are 20 in. wide by 24 in. long. This small size makes it practical for transport and field use. That is, this compact 3-D laser scanner is sized small enough for efficient transport, but is still large enough to capture the entirety of the average effective wheel path width.

Figure 5 displays a visual representation of the 3-D macro-texture data obtained from scanning a field test section that has been trafficked under real field traffic conditions for two days after the initial construction of the test section. These data are representative of all the data obtained from the scan and have not been processed at all prior to being graphed. Figure 5 illustrates the ability of the 3-D laser profiler to capture the macro-texture of each aggregate particle in addition to that of the overall wheelpath. Such accuracy is essential in determining the surface texture and the mean profile depth (MPD) changes that occur under traffic loading over time. Figure 5 is color-coded based on height to depict the wheel path more clearly. The laser data obtained from the 3-D laser profiler have been used to complete a variety of macrotexture-based analyses that are related directly to the performance of chip seal surface treatments.
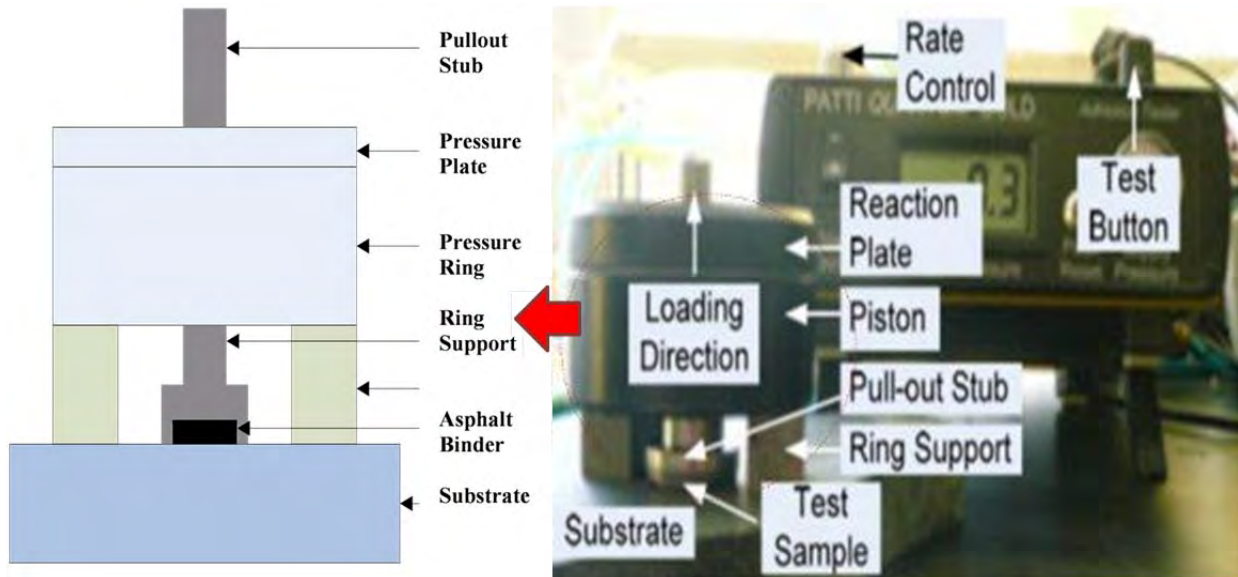
FIGURE 3 The BBS test apparatus for measuring the force required to remove a pull-out stub affixed to an aggregate substrate.

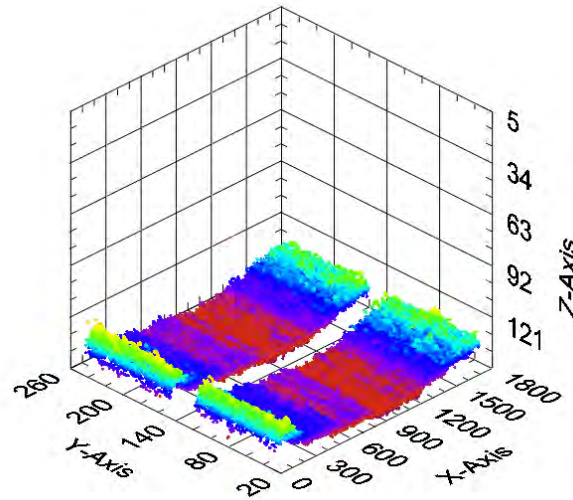**FIGURE 4 3-D laser profiler prototype.**

FIGURE 5 3-D laser profiler graph of a chip seal pavement.

LABORATORY CHIP SEAL SPECIMEN FABRICATION USING CHIPSS

The process of fabricating chip seal samples in a laboratory setting involves simulating the chip seal construction process as closely as possible. The NCSU research facilities provide various tools to replicate the chip seal sample construction process.

The first step in constructing chip seal specimens in the lab is to obtain a felt disk in the desired size and shape of the chip seal specimen to be fabricated. In the case of MMLS3 samples, these felt disks are 12 x 14 in. on which 7- x 12-in. samples are fabricated. A measurement of 7 in. is used for the width because this width is the transverse wandering distance that the MMLS3 covers during testing. Therefore, specimens should not exceed this width because otherwise the specimen would not be fully trafficked across the whole specimen area. In order to make the 7- x 12-in. samples on the felt disk, a template is created and placed on top of the sample during the emulsion spraying process to ensure consistent dimensions and rates for each sample replicate. The template ensures that the emulsion reaches only the desired area. In addition, in order to apply the emulsion to the felt disk in a manner that simulates the emulsion being sprayed from the truck in the field, a paint spray gun is used. It is recommended that this paint gun is capable of applying emulsion at a rate of 5.4 gal/h or higher (ideally higher than 7.2 gal/h to spray polymer-modified emulsion, which is more viscous than unmodified emulsion). Lastly, a weight scale is used to keep track of the amount of emulsion that has been sprayed onto the felt disk to ensure accurate emulsion application rate (EAR) during sample fabrication. Figure 6 shows the emulsion spraying process.

Following this step, the felt disk with newly applied emulsion is then positioned underneath the ChipSS. Figure 7 shows the automated aggregate spreader and the process of spreading the aggregate onto a felt disk after the initial application of emulsion. Figure 7*d* shows the aggregate that is spread by the ChipSS on top of the felt disk. After the application of the emulsion onto the felt disk (within the 7- x 12-in. area), only the section of the felt disk covered by emulsion will retain the aggregate, and the excess is swept off by a small brush. Through this process a single layer of aggregate is obtained that completely covers the specimen.
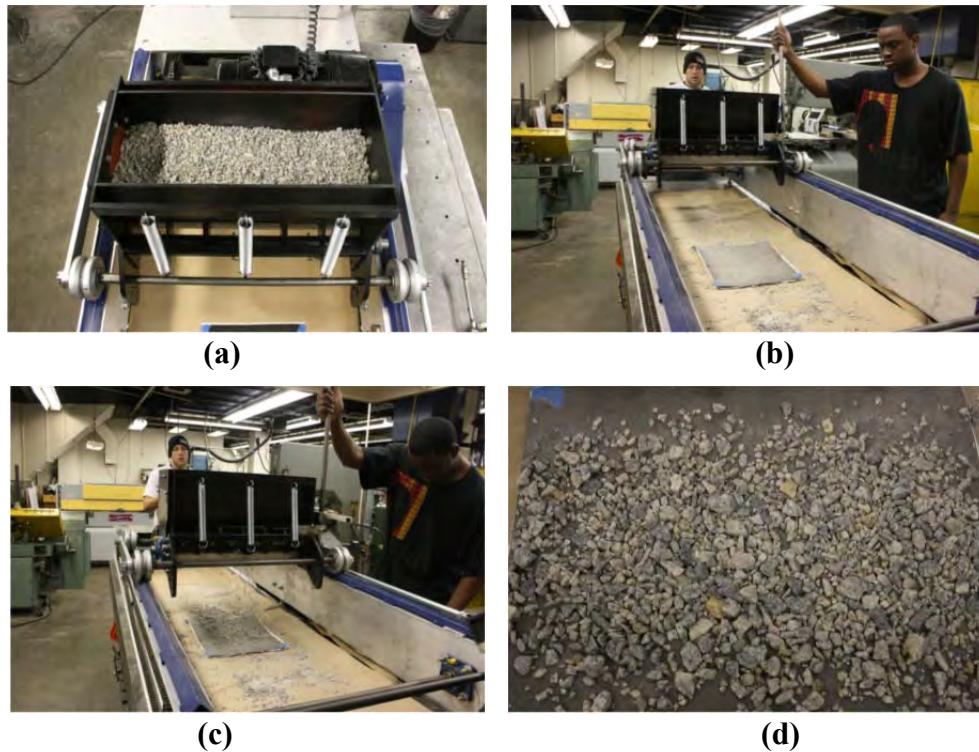
FIGURE 6 Chip seal emulsion spraying procedure.

**FIGURE 7 ChipSS aggregate spreading machine applying aggregate onto a felt disk: (*a*)
hopper filled with aggregate; (*b*) ready-to-spread aggregate on felt disk; (*c*) spreading
aggregate on felt disk; and (*d*) aggregate spread on felt disk.**

Prior to beginning the sample fabrication process, the aggregate spreading machine is
calibrated to drop the appropriate amount of aggregate that is required to achieve the desired
aggregate application rate (AAR) for the sample being fabricated to ensure that the desired rate is
applied. The AAR is controlled by two parameters: the box speed and the drum speed. The box

speed is the speed at which the box moves across the sample, and the drum speed is the speed at which the rotating drum (located inside the aggregate hopper) rotates and releases the aggregate. At lower box speeds, more aggregate is dropped onto the sample, and vice versa at high speeds. At lower drum speeds, less aggregate is dropped out of the hopper, and vice versa for high drum speeds. Thus, the AAR can be controlled effectively during specimen fabrication.

　　　After the aggregate is applied, and the excess outside of the designated sample area is swept away, the aggregate weight is measured to determine the exact amount of aggregate that was applied, and then the sample is compacted. The compaction device is shown in Figure 8.

　　　The compactor shown in Figure 8 is used to compact the chip seal specimen directly after the aggregate is applied to the hot emulsion. More specifically, this compactor is used in conjunction with a thin rubber mat in order to replicate a combination roller (a combination of steel wheel and pneumatic tires), which has been found to be the most effective type of roller in the field. This procedure is necessary because the steel supplies great compaction force, while the rubber material helps minimize the breaking of chips that occurs when the steel wheel is used alone for compaction. The compaction procedure involves three compaction passes across the horizontal face of the sample, and then additional compaction passes perpendicular to the first three passes. Following compaction, the newly fabricated sample is then placed in the oven undisturbed at 35°C for 24 h to allow the sample to cure.

CHIP SEAL FIELD SAMPLE EXTRACTION

In order to conduct laboratory testing on the field-constructed samples, the method for extracting samples from chip seal field validation sections is developed. This sample extraction process allowed to perform tests to validate the material application rates that were applied during the

FIGURE 8 Chip seal sample compactor used to compact chip seal specimen.

field construction using the ignition oven test. That is, the residual asphalt binder and the aggregate rate applied could be determined from the ignition oven test results. In addition, the extracted field samples could be used for aggregate loss, bleeding, and rutting tests to assess surface treatment performance.

The refined sample extraction process is as follows (Figure 9). Prior to the start of construction, the first step is to affix all the felt disks and Vialit plates securely onto the existing pavement surface to ensure that they are not removed by passing traffic or during the construction process. It is helpful to use small flags on the side of the road to help locate the general location of the samples once the surface treatment construction is complete. Following construction, the process of field sample extraction can begin after at least one hour of curing, which is needed to prevent damage to the field samples during extraction. Following sample extraction, the sides of the sampling area are cleaned so that the construction crew can patch the damaged area effectively. It is important that the sampling area is located between, and not inside, the wheelpaths because patched areas inside the wheel paths could lead to performance problems once the road is opened to traffic due to inconsistencies in the road surface, especially as the repair work to the extracted areas is undertaken by hand, which can lead to variability in the quality of the repair. After the samples are extracted, they are placed on wooden boards to avoid being bent and then transported to a box truck where they are placed on secured racks and taken to the laboratory.
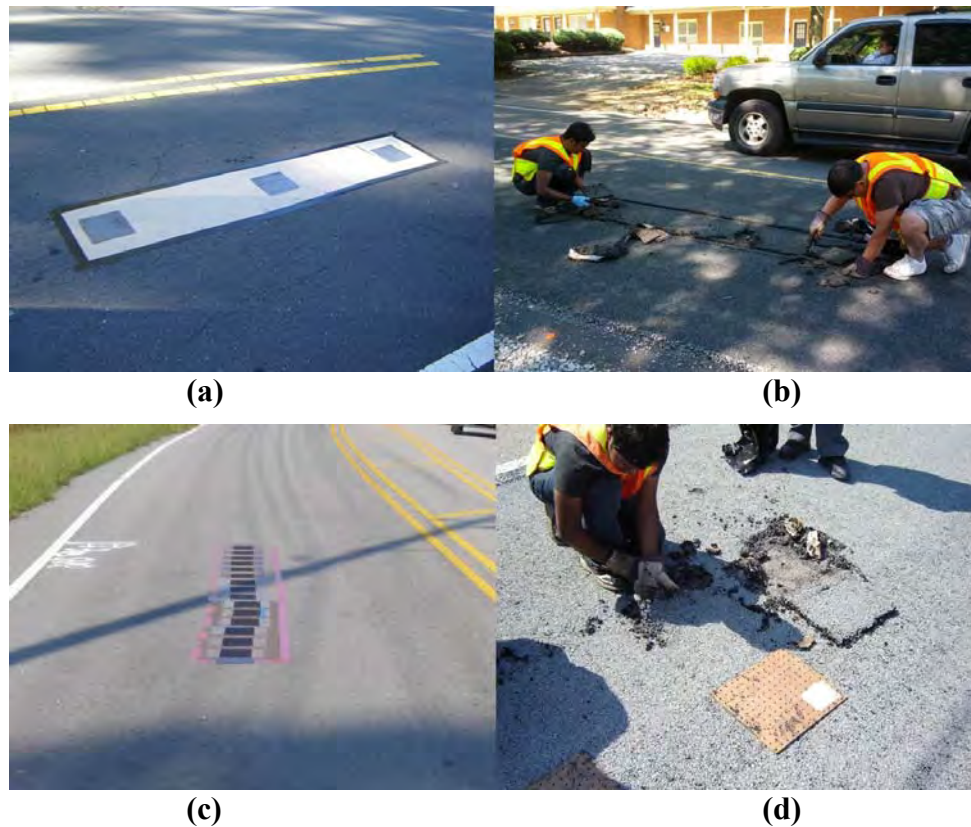

(a)   (b)

(c)   (d)

FIGURE 9 Field sampling: (*a*) Vialit sample template; (*b*) Vialit samples; (*c*) MMLS3 sample template; and (*d*) MMLS3 samples.

MMLS3's ABILITY TO SIMULATE THE FIELD TRAFFIC LOADING

Because MMLS3 is one of the main test methods for chip seal performance evaluation, it is important to evaluate how well the MMLS3 loading simulates the effects of traffic loading on chip seal performance. A field study was conducted for this purpose. Field test sections were constructed in Franklinton, North Carolina, by the North Carolina Department of Transportation (DOT) at NC-96 south of SR-1705 and at SR-1623 east of NC-96. Data were collected using traffic counters during each quarter over the course of 1 year. The data were utilized in combination with vehicle class data obtained during the traffic counts in order to determine the effective number of wheel passes experienced by the field sections. Wheelpath data from the field were obtained specifically for the lane where the test sections were constructed at each location. These same sections were scanned in the wheel paths using the 3-D laser profiler shown in Figure 4. The field scans were conducted at various times following construction of the chip seal to measure the changes in surface texture. These times ranged from immediately after sweeping to 1 week after sweeping.

Likewise, samples were extracted from designated sampling areas within these same field construction sections and were transported to the laboratory and tested under MMLS3 traffic loading. These samples were laser-scanned at different numbers of wheel passes during the 2-h MMLS3 accelerated loading tests.

The surface profile of the chip seal pavements and samples was represented by mean profile depth (MPD) defined by Transit New Zealand (*5*) as follows:

$$MPD = \frac{Peak\,level\,(1st) + Peak\,level\,(2nd)}{2} - Average\,level \tag{1}$$

The MPD represents the exposed texture depth of a chip seal surface treatment and is inversely related to the embedment depth. Essentially, as the EAR for a given single aggregate layer increases, the MPD will decrease, and where the EAR (or embedment depth) is decreased for a given aggregate structure, the MPD will increase.

Figure 10 schematically shows the various chip seal parameters that make up Equation 1. In the diagram, the MPD clearly indicates the roughness (i.e., macrosurface texture) and aggregate exposure depth of the chip seal. Roughness is important, because it provides the skid resistance and friction needed for vehicles to brake adequately. The aggregate exposure depth is also important because it is a function of the aggregate embedment depth, which is the most important factor that controls the aggregate loss and bleeding performance of chip seals. A low MPD value indicates the likelihood of bleeding and skid resistance problems. A high MPD value after construction indicates the possibility of excessive aggregate loss and, therefore, bleeding due to aggregate loss.

Figure 11 and Figure 12 present the results of these experiments in both the field and laboratory for granite and lightweight aggregate, respectively. For almost all the 11 field sections, the final field-trafficked MPD value is extremely close to the final MMLS3 lab-trafficked MPD value. This finding indicates that the MMLS3 traffic load translates similarly to the field traffic rate in terms of changes in texture depth over time. Also, because the complete amount of field MPD change occurs within the first week of field traffic, it can be said that a 2-h MMLS3 test simulates about a week of field traffic for a field traffic volume up to approximately 5,000 ADT. It is possible that this relationship holds at higher traffic volumes, but field sections
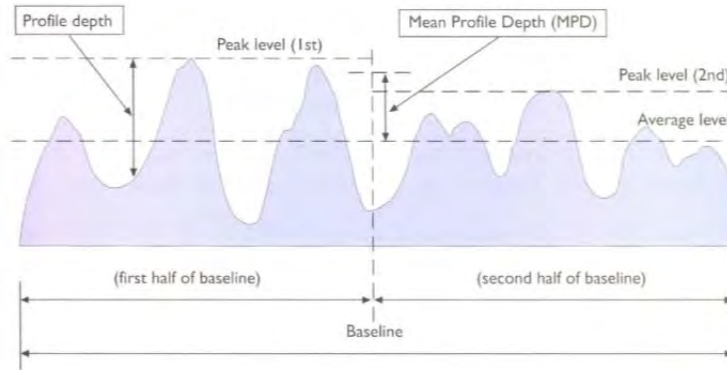
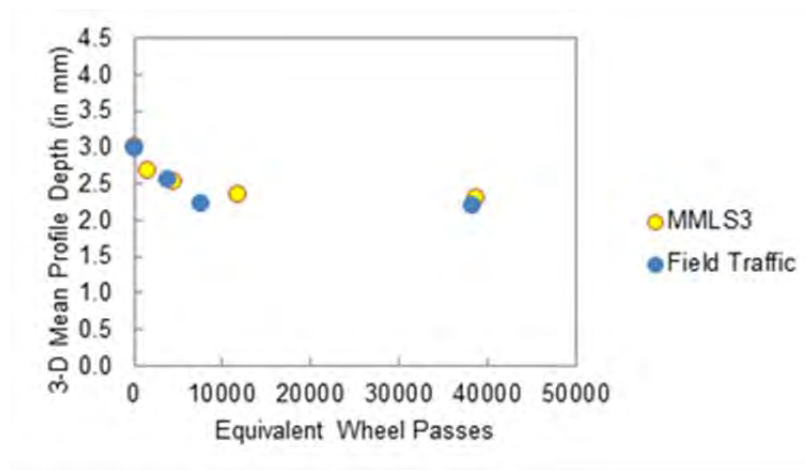**FIGURE 10 Schematic diagram of the MPD determination (5).**

**FIGURE 11 MPD for granite 78M aggregate and CRS-2L emulsion
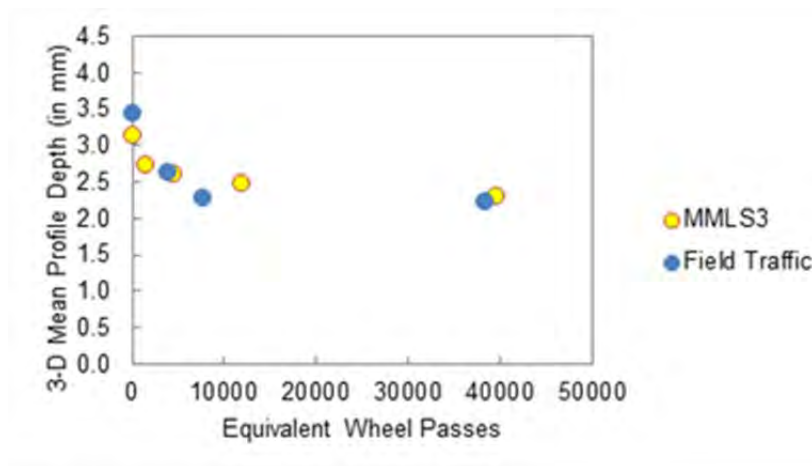section subjected to field traffic and MMLS3 loading.**

**FIGURE 12 MPD for lightweight aggregate and CRS-2L emulsion
section subjected to field traffic and MMLS3 loading.**

at higher traffic volumes were included in this study (*6*). In this study, it was found that the field and MMLS3 traffic loads show very similar changes in surface texture (MPD) in multiple field sections at various mix design rates and average daily traffic levels. The full results, including the material application rates for the various sections, are provided in the report for that research effort (*6*). The findings from the Kim and Adams (*6*) study confirm for the authors of this paper that MMLS3 traffic loading results provide a reasonable prediction of the changes in MPD as well as other performance parameters that are related to surface texture depth (such as skid resistance, aggregate loss and bleeding) as the chip seal is traffic-loaded over time.

OPTIMIZING AGGREGATE GRADATIONS FOR CHIP SEALS

For asphalt surface treatments such as chip seals, the uniformity of the aggregate gradation has been shown to have an effect on the overall performance of the surface treatment. The aggregate performance uniformity coefficient (PUC) is a performance indicator of aggregate gradation and gives an indication of the uniformity, or lack thereof, of the aggregate source being analyzed. In chip seal surface treatments, gradations that are more uniform perform better in terms of aggregate retention and bleeding resistance than those that are less uniform. Therefore, the PUC of the aggregate source affects the bleeding and aggregate loss performance of the chip seal surface treatment being constructed (*7*).

Performance Uniformity Coefficient Concept

The concept of the PUC is founded on principles that are based on McLeod's chip seal failure criterion (*8*). Essentially, McLeod's premise that 70% embedment is the ideal embedment for chip seal surface treatments is implemented in the PUC definition. The PUC is the ratio of the percentage passing at a given embedment depth (P_{EM}) to the percentage passing at twice the embedment depth (P_{2EM}) in a sieve analysis curve (*9*). The P_{EM} value represents the bleeding failure criterion, and the P_{2EM} value represents the aggregate loss failure criterion with regard to the gradation. The P_{EM} value is defined as the percentage passing that corresponds to 70% of the median particle size on the gradation curve. The P_{2EM} value is defined as the percentage passing that corresponds to 1.4 times the median particle size, with the median particle size defined as the particle size of which 50% of the gradation passes through the sieve. In the case of a chip seal, the P_{EM} value should be low, because a low percentage of the gradation passing at the bleeding failure criterion indicates that the aggregate particles in that range of the gradation are larger and less susceptible to bleeding than smaller particles would be. Conversely, for the P_{2EM} criterion, if the value is high, the percentage of the aggregate particles that do not meet the aggregate loss criterion is low, and therefore, less aggregate loss is expected.

Figure 13 visually displays the concept behind the PUC parameter. In theory, if the aggregate is embedded in emulsion up to 70% of its median (M) particle size, the particles that are smaller than 0.7 M will be submerged completely in the emulsion and, therefore, will experience bleeding. Ideally then, the smaller particles should be larger than 0.7 M to avoid bleeding. Conversely, the particles that are bigger than 1.4 M are likely to be lost when trafficked because they will be less than 50% embedded after trafficking. In this case, the larger the coarse aggregate particles, the more likely aggregate loss will occur. Thus, the closer the PUC value is
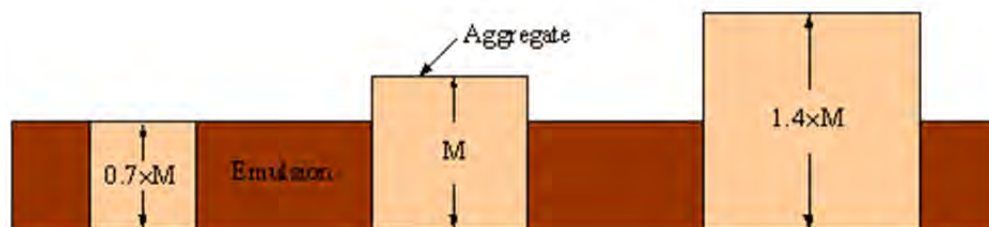
FIGURE 13 Visual explanation of PUC parameter.

to zero for a particular aggregate gradation, the more uniformly the aggregate is graded. In other words, the P_{EM} value that is closer to 0% and the P_{2EM} value that is closer to 100% indicate a uniform gradation that corresponds to improved chip seal performance; that is, these values indicate less bleeding and a smaller amount of aggregate loss, respectively.

Effect of PUC on Chip Seal Performance

The performance results displayed in Figure 14 and Figure 15 show aggregate loss and bleeding performance as a function of the PUC, respectively. The AARs and EARs are shown as a single material application ratio (AAR-EAR). As the AAR-EAR parameter increases, the condition of the chip seal treatment becomes drier by comparison. Figure 14 shows that for various chip seal treatment conditions, aggregate loss increases as the PUC increases and aggregate gradation becomes less uniform. A higher level of uniformity in the aggregate gradation makes for more consistent embedment of each aggregate particle, which improves retention.

Likewise, Figure 15 shows that more uniform aggregate particles prove to be more resistant to bleeding. This finding is best exemplified in Figure 15 at the AAR-EAR ratio of 76, where the ratio is low enough (meaning the chip seal is sufficiently wet) to increase the likelihood of bleeding. At this AAR-EAR ratio, a low PUC (highly uniform gradation) shows low levels of bleeding, whereas the higher PUCs (lower uniformity) show significantly greater amounts of bleeding. This phenomenon is partially due to the fact that a more uniform aggregate structure has less aggregate loss (which contributes to bleeding) and also less aggregate reorientation due to the consistency of the aggregate layer under traffic loading than is the case with a less uniform aggregate structure.

QUANTIFYING THE BENEFITS OF IMPROVED ROLLING OF CHIP SEALS

To improve the current chip seal rolling practice, it is necessary to quantify the benefits of possible changes to the compaction protocol. In order to investigate the benefits of the rolling protocol, aggregate retention and adhesion must be measured. In this study, the FOT and MMLS3 test are employed to evaluate aggregate retention, and the Vialit test is used to evaluate adhesion for different types of compaction operations. These tests are used to determine the optimal rolling protocol for chip seals based on the evaluation of various performance characteristics.
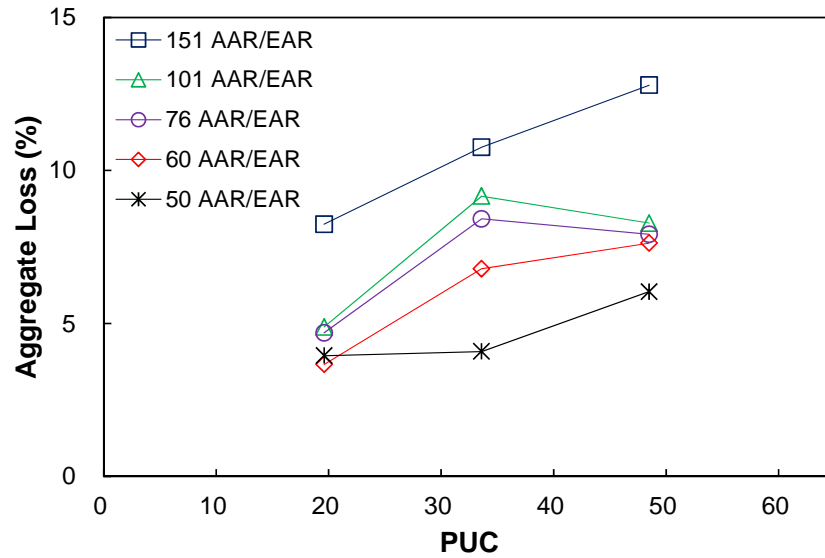
FIGURE 14 PUC versus aggregate loss as a function of material application rates.
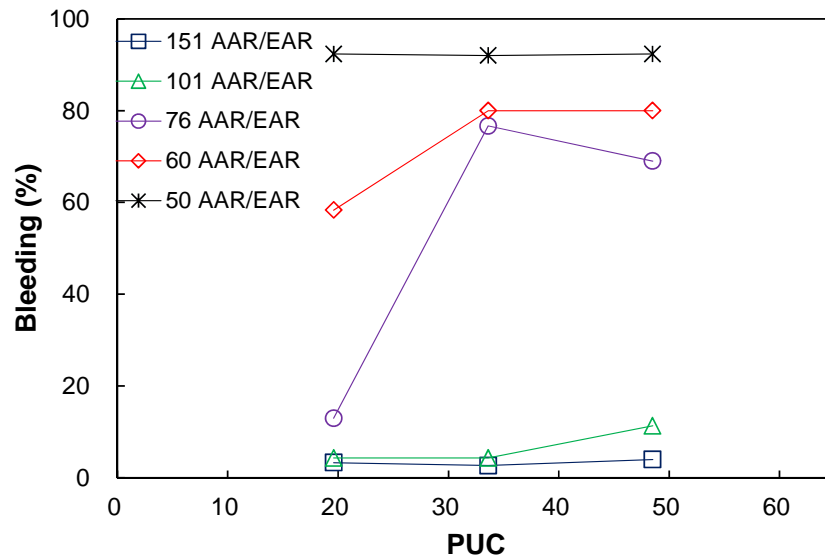

FIGURE 15 PUC versus percentage of bleeding as a function of material application rates.

Effect of Rolling Pattern on Aggregate Loss

Figure 16 shows the three selected rolling patterns that were included in the final compaction pattern evaluation to determine the optimal rolling pattern for chip seal construction. Details regarding the selection of these rolling patterns as well as other analyses are detailed in the FHWA/NCDOT research report, Quantifying the Benefits of Improved Rolling of Chip Seals (*10*). In each rolling pattern shown in Figure 16, the emulsion sprayer is shown at the top of the image above the aggregate spreader, followed by the pneumatic tire roller(s), and last, the

combination roller(s). The arrows indicate the pattern that each type of roller follows in the compaction effort. These rolling patterns were used on a single-seal field section constructed with granite 78 M aggregate.

Figure 17 shows the effect of each rolling pattern on aggregate loss performance. For this study, samples were collected following field construction using the field sample extraction procedure described earlier. Samples were tested using the three laboratory aggregate loss tests,
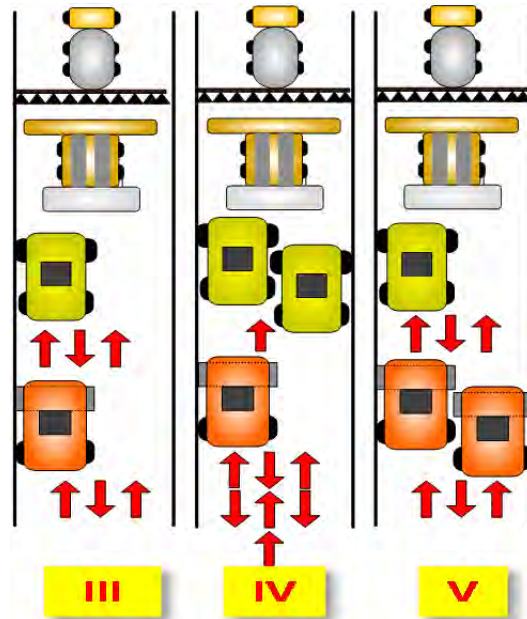

FIGURE 16 Rolling patterns with both two and three rollers selected for final rolling evaluation.
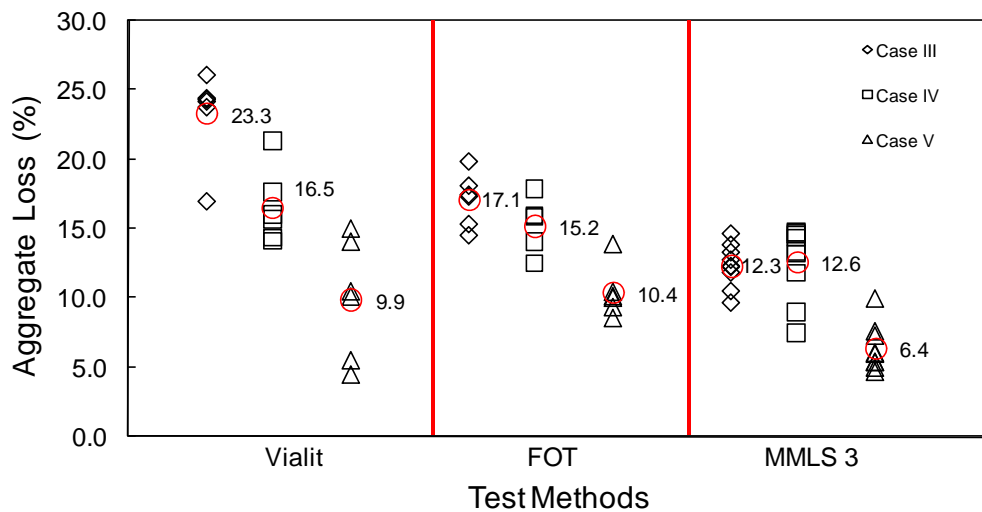

FIGURE 17 Effect of rolling pattern on aggregate loss performance.

i.e., Vialit, FOT, and MMLS3, to determine the impact of rolling pattern on the performance of the surface treatments. The results clearly show that Case V yields the best aggregate loss performance (i.e., most aggregate retention) of the rolling patterns involved in the study. The Case V rolling pattern is that which includes three passes of a pneumatic tire roller immediately after the aggregate is spread, followed by three passes of two combination rollers side by side.

Rolling the Bottom Layer of a Multilayer Seal: Effect on Performance

The optimal coverage distribution for the underlying layers of a multilayer chip seal (i.e., double or triple layer seal) is determined based on the results of the aggregate retention performance tests. Figure 18 and Figure 19 present the findings for the compaction of multiple layer seals, i.e., double and triple seal layers, respectively.

Figure 18 indicates that the double seal requires rolling the bottom layer for improved aggregate retention performance. The results show that aggregate retention in cases where the bottom layer is rolled is significantly better than cases where the bottom layer is not rolled.

Conversely, the results for rolling the bottom layer of a triple layer chip seal (Figure 19) yield a different finding. The triple seal shows no significant improvement in aggregate retention as a result of rolling the bottom layer of the seal. Therefore, the rolling operation may be eliminated for the bottom layer of a triple seal.

Thus, the overarching principle for multilayer chip seal surface treatments is that rolling the layer immediately below the top layer improves the aggregate retention performance of the top layer. Therefore, for a double seal, the bottom layer should be compacted for a single coverage, and for a triple seal, the second layer should be compacted as a single coverage without compacting the bottom layer.
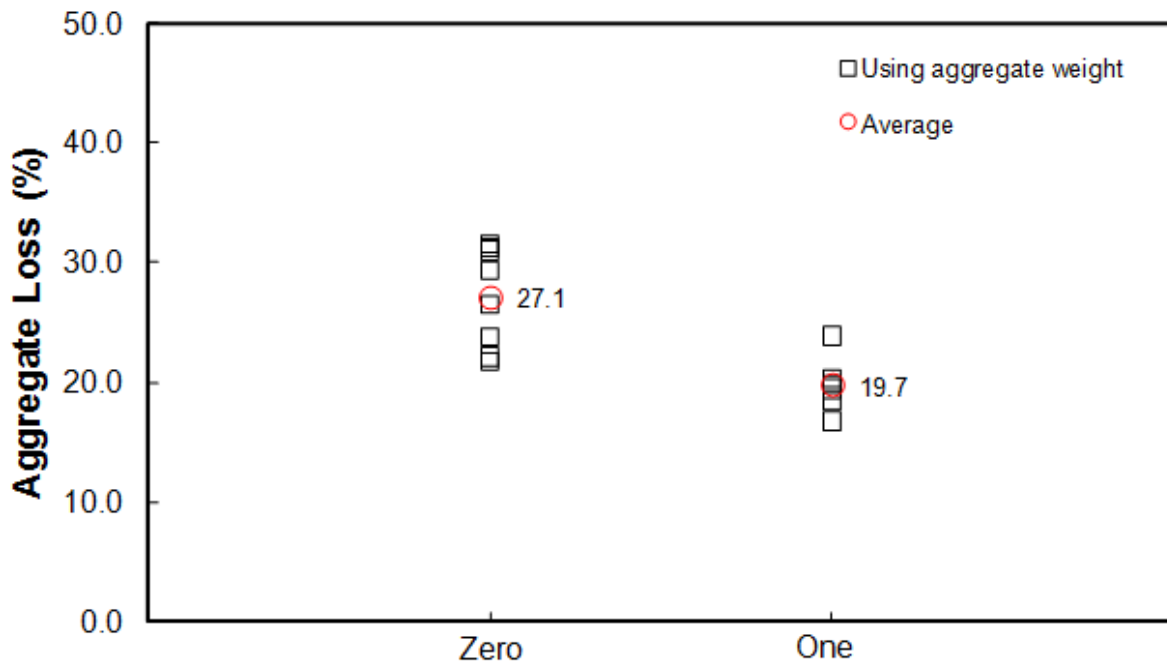

FIGURE 18 Rolling coverage distribution for the bottom layer of a double-layer chip seal.
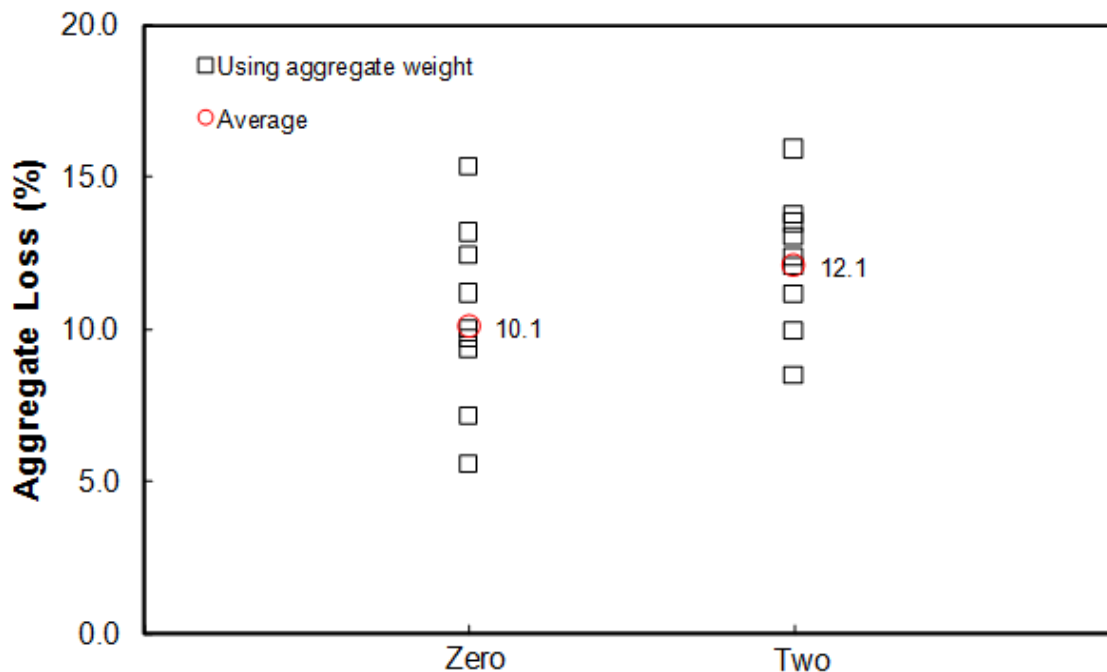
FIGURE 19 Rolling coverage distribution for the bottom layer of a triple-layer chip seal.

PERFORMANCE-BASED ANALYSIS OF POLYMER-MODIFIED EMULSIONS IN BITUMINOUS SURFACE TREATMENTS

Chip seals constructed with polymer-modified emulsion (PME) provide better initial and long-term performance and extend the overall service life of pavements longer than those constructed with unmodified emulsion (*11*). The use of polymer-modified chip seals, which have tougher, more-resilient surface characteristics than unmodified chip seals (*12*), may extend the use of chip seals to roadways that have higher traffic volumes than the low-traffic roadways for which unmodified chip seals typically are used. Experiments are conducted using both modified and unmodified chip seals in order to determine the performance differences between polymer-modified and unmodified surface treatment conditions that previously have not been fully investigated.

Effect of PME on Curing

The adhesion development of CRS-2 and CRS-2L emulsions was investigated as a function of curing time using the Vialit test. Granite 78 M aggregate was used in the fabrication of all the specimens included in this study. The aggregate loss percentages obtained for the two emulsions are plotted in Figure 20. Each data point in this figure represents the average of seven replicates. The figure shows the adhesion behavior of granite 78 M aggregate for 1, 2, 3, 1, and 24 h.
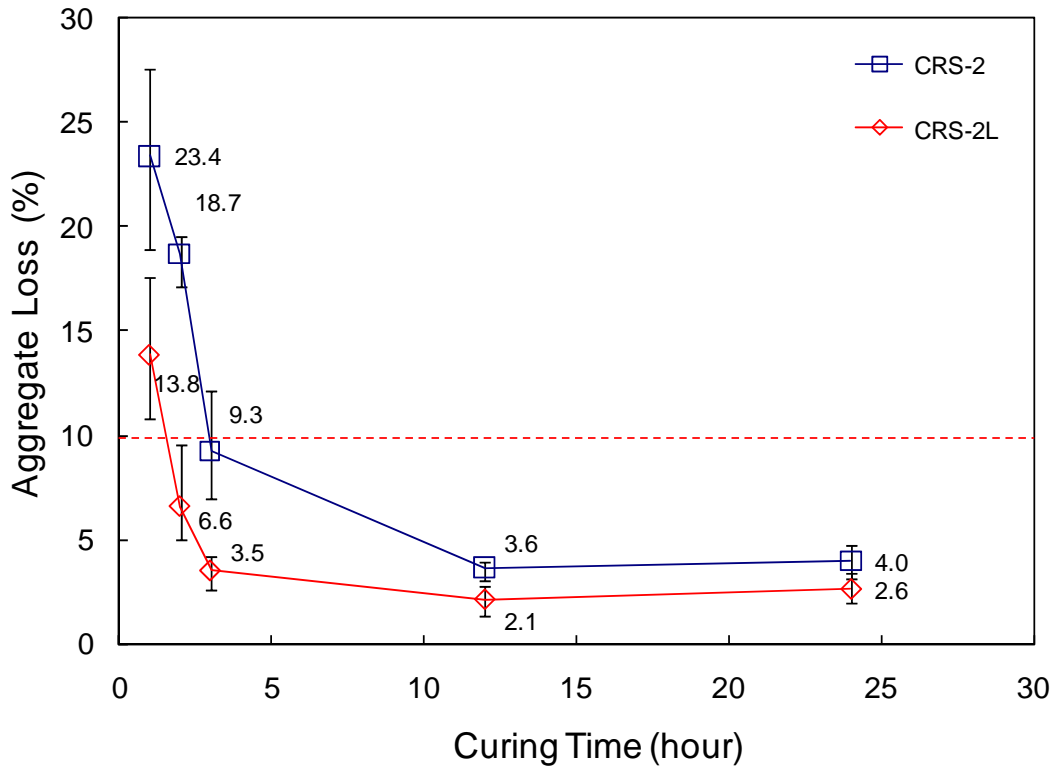
FIGURE 20 Aggregate loss results from Vialit testing as a function of curing time at 95°F.

As seen in Figure 20, the average aggregate loss of the CRS-2 emulsion is about 10% greater than that of the CRS-2L emulsion for 1 h of curing. However, the percentage of aggregate loss for both emulsions is greater than the 10% that is specified in the Alaska chip seal design guide as the maximum allowable aggregate loss (*13*). This finding implies that 1 h of curing at 95°F is not enough time for proper adhesion to develop between the aggregate and the binder, whether the emulsion is modified by polymer or not. Another observation from Figure 20 can be made at the 2-h curing time. The percentage of aggregate loss of the CRS-2L emulsion is less than 10% of the maximum allowable aggregate loss; however, the average aggregate loss of the CRS-2 emulsion is still over 10%. This finding indicates that the CRS-2L emulsion achieves proper adhesion within 2 h, which satisfies the maximum allowable aggregate loss specified in the Alaska chip seal design. However, 2 h is not enough time for the CRS-2 emulsion to pass the 10% loss criterion. All average aggregate loss values measured for samples that were cured for more than 3 h (3, 12, and 24 h) are below 10%. Also, the difference between the aggregate loss of the CRS-2L and CRS-2 emulsions is clearly reduced after 3 h.

Overall, these findings are critical because chip seals that cure in little time allow for earlier traffic opening and fewer construction-related delays than those that take a long time to cure. The overall trend, i.e., that the aggregate loss of the CRS-2L emulsion is less than that of the CRS-2 emulsion, as seen in Figure 20, indicates that the latex modification of the CRS-2L emulsion enhances the aggregate retention performance, and does so more significantly in the first 3 h of curing. The largest difference in aggregate loss between the CRS-2 emulsion and CRS-2L emulsion (i.e., 12% difference) occurs at 2 h, as seen in Figure 20.

Effect of PME on Low-Temperature Performance

The effect of low temperature on performance using both polymer-modified and unmodified emulsions is also investigated. Figure 21 presents these results. The results shown in Figure 21 indicate that latex modification improves performance at all temperatures, but this effect is most pronounced as the temperature lowers. The CRS-2 specimens all are above the 10% aggregate loss threshold for all tests below approximately 41°F (5°C), whereas the latex-modified CRS-2L emulsion shows consistent aggregate retention performance, with all tests yielding aggregate loss below the 10% threshold, even as the temperature decreases.

Effect of PME on Rutting Resistance

MMLS3 rutting tests were conducted for triple seals using both CRS-2 (unmodified) emulsion and CRS-2L (polymer-modified) emulsion at 104°F (40°C). The average rut depths of the triple seal are plotted in Figure 22 as a function of the number of MMLS3 wheel passes (N). It is noted that the *x*-axis in Figure 22 is in logarithmic scale. The dashed line shown in the figure indicates a failure criterion of 8-mm rut depth. The rut depth of 8 mm is used for the failure criterion because it is approximately one-third of an inch (25.4 mm), which is the common failure criterion for hot mix asphalt pavements, and thereby reflects that the MMLS3 is a third scale down. Significantly different rutting behavior is evident between the use of the CRS-2 and CRS-2L emulsions. The rut depth growth, as shown in Figure 22, definitely illustrates that the CRS-2 emulsion shows poor permanent deformation resistance as it crosses the threshold at significantly fewer wheel passes than the CRS-2L specimen.
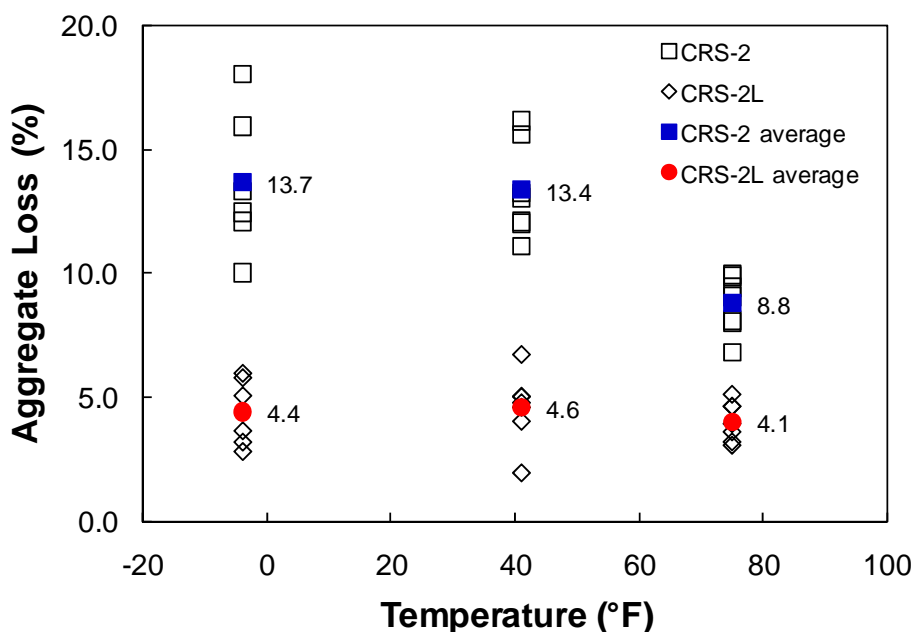

FIGURE 21 Effect of PME on low-temperature Vialit testing using granite 78M aggregate.
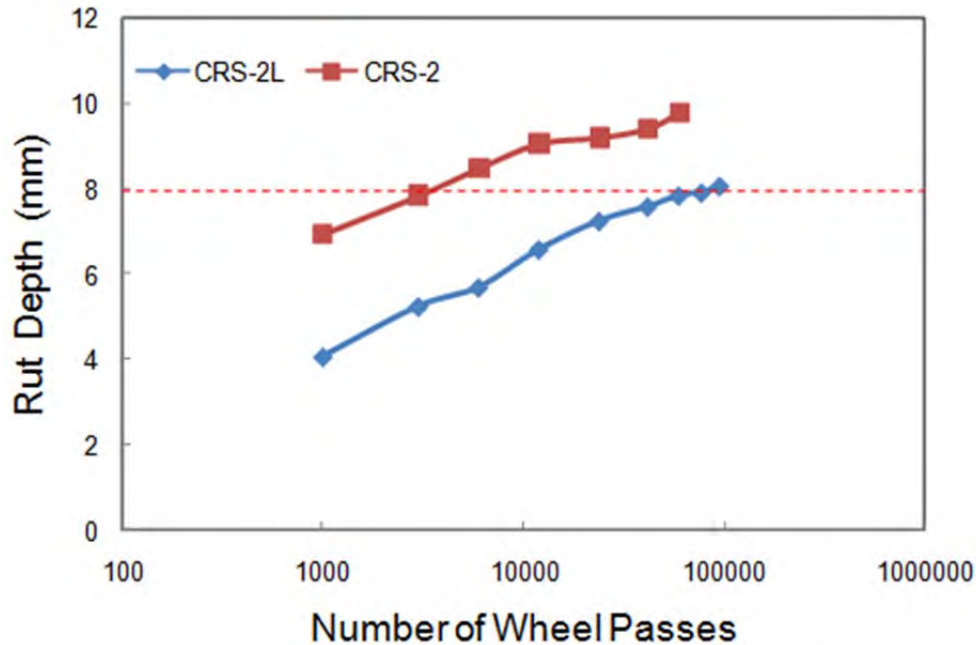
FIGURE 22 Comparison of rut depth growth at 104°F (40°C).

DEVELOPMENT OF A PERFORMANCE-BASED CHIP SEAL MIX DESIGN METHOD

The purpose of the NCSU mix design procedure is to provide a systematic method for engineers to design consistent chip seal mixes while ensuring satisfactory performance under field traffic and environmental conditions. The developed mix design seeks to provide effective quantitative methods and techniques for determining the appropriate AAR and EAR for the chip seal surface treatment to be constructed. The 3-D laser profiler and volumetric relationships are used to determine the optimal design for a surface treatment. This paper highlights key portions of the developed mix design method. Details regarding the mix design concept and validation are included in the report, Development of a New Chip Seal Mix Design Method (*6*).

Optimal Aggregate Application Rate Determination

The AAR to be used for the construction of chip seal surface treatments in the mix design procedure is determined using a modified board test analysis. The origins of the board test derive from the modified Kearby method that recommends using a 1-yd^2 board to determine the amount of aggregate that is required to fill a specific area with a one stone coverage of that particular aggregate (*14*). The aggregate should be spread evenly and in a well-lit area to ensure that a second layer of aggregate is avoided or, if detected, removed. It is important to fill all the empty spaces of the board to ensure that the proper AAR is determined and that the aggregate particles are packed tightly together as they would be in a chip seal surface treatment. Once a single stone coverage of aggregate is achieved, the aggregate is then weighed, and the AAR is calculated for that particular aggregate. The variability of the existing procedure is determined by conducting multiple tests, and 508 x 305 mm is recommended as the optimal size of the board used for the modified board test (*6*).

Optimal Emulsion Application Rate Determination

The method used to determine the optimal EAR for chip seal surface treatments involves a volumetric mix design procedure in conjunction with the 3-D laser profiler and modified board test to complete the volumetric analysis. Essentially, the goal of this analysis is to determine the amount of emulsion that is required to ensure satisfactory chip seal surface treatment performance in conjunction with the optimal design AAR, as determined by the board test.

In this procedure, the optimal AAR is applied and the board is scanned using the 3-D laser profiler shown in Figure 4. The first step in determining the EAR is to determine all the volumes associated with each phase of the chip seal (using air voids not yet filled with emulsion). The simple chip seal phase diagram (Figure 23) is composed of two phases, aggregate and air; that is, the diagram indicates two aggregate particles and the voids within the chip seal surface treatment. The air volume is split into two parts (above and below the dashed line in Figure 23): subsurface air voids and surface air voids. The optimal EAR is determined by considering all subsurface voids as voids that need to be filled with emulsion in order to retain the aggregate.

The total amount of emulsion needed to fill the available air voids, as needed, is calculated. It is assumed that by filling the available voids in the chip seal, the estimated embedment depth is about 50%. Most importantly, using this method that determines all the voids between the aggregate particles and also considers the overall gradation and size of the aggregate particles (via the laser scanning to determine total volume), the mix design procedure will adjust itself accordingly for different aggregate types and gradations. Figure 23 explains filling the subsurface void space to attain 50% embedment of the aggregate.

If the two circles shown in Figure 23 are assumed to be two uniform aggregate particles, and the space between them represents emulsion, it can be seen that if all the available subsurface voids are filled, 50% embedment of the aggregate is attained, with embedment being defined as the emulsion height divided by the aggregate height. In the realistic case where aggregate particles vary in size, this example becomes an approximation. However, the laser-based mix design procedure is designed in such a way to account for the size variation between each aggregate particle in the aggregate structure by lowering the emulsion rate for smaller (or flatter particles) and raising the algorithm to account for larger (or more cubicle) particles.
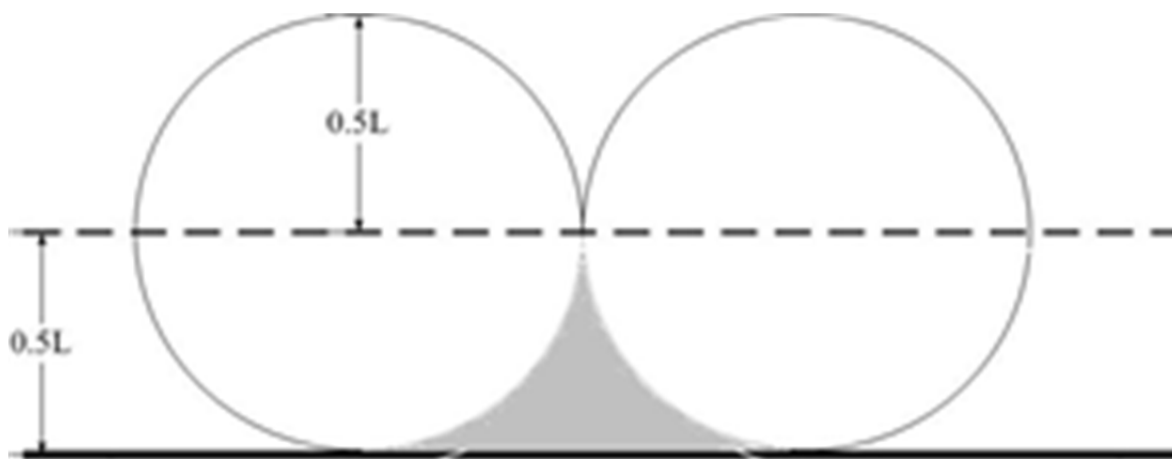

FIGURE 23 Explanation of 50% embedment concept.

The first step is to determine the total volume (the volume between the top red line in Figure 24 and the bottom of the board) of the chip seal surface treatment. This volume is found using the 3-D laser profiler to scan the aggregate during the modified board test procedure. Multiple scans for multiple board tests were performed for research purposes simply to ensure that the proper total was captured and that the variance from test to test was not a problem (*6*).

The total volume measured from the laser scans is made up of only the aggregate and air volumes. The aggregate weight and specific gravity are found during the board test; therefore, the aggregate volume can easily be determined. The subsurface air voids, which are to be filled with emulsion, make up the remainder of the total volume. Therefore, the aggregate volume can be subtracted from the total volume (from the scan data analysis) to determine the amount of emulsion that is needed to obtain the optimal embedment depth. Details regarding adjustments to the EAR for conditions that are based on the existing surface and aggregate absorption rates can be found in the full performance-based mix design report (*6*).

Validation: Performance Results

The performance-based mix design method was validated by performing the MMLS3 aggregate retention and bleeding tests on chip seals with three different gradations. Optimum AAR and EAR were determined for each of the three gradations using the modified board test and the 50% embedment concept implemented by the 3-D laser profiler.

EAR Versus Aggregate Loss Performance: Mix Design Validation Results

The aggregate loss versus EAR relationship is plotted separately for each gradation in Figure 25 for the granite 78M aggregate and the lightweight aggregate, respectively. The horizontal dashed line in each graph indicates the maximum allowable aggregate loss (10%) criterion established by the Alaska Department of Transportation (*13*). The figure shows that for all three gradations, as the EAR increases the aggregate loss decreases, which is the expected outcome. The more emulsion in the chip seal specimen, the better the aggregate retention performance, and the lower the aggregate loss percentage.

Figure 25 indicates that the design rates determined by the mix design yield aggregate loss results below 10% for the CRS-2L emulsion with granite 78M aggregate chip seal treatments. These results mean simply that the mix design successfully yields a design EAR that is above the minimum required for adequate aggregate retention performance for a wide range of aggregate gradations.
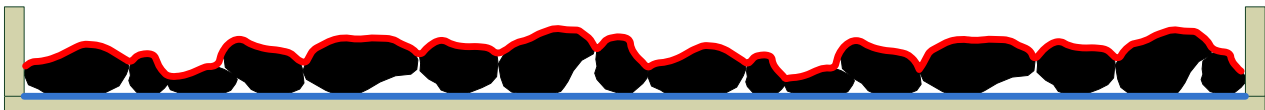

FIGURE 24 Aggregate board test (profile view).

EAR Versus Bleeding Performance: Mix Design Validation Results

The other critical performance measure used to validate the mix design is the bleeding performance obtained from the MMLS3 bleeding test. The bleeding test procedure simulates the bleeding of chip seal surface treatments during the summer months under field traffic loading. Essentially, for the mix design of a chip seal surface treatment, the optimal design should include as much binder as possible without causing bleeding problems. The bleeding test is conducted after the completion of the aforementioned 2-h aggregate loss test, and involves two hours of MMLS3 loading at a temperature of 50°C inside the temperature chamber. Prior to the start of the continuous MMLS3 loading, the samples are temperature-conditioned for 3 h at 50°C to ensure material temperature stability in the chamber. Figure 26 presents the results of the bleeding tests for specimens constructed with CRS-2L emulsion and granite 78M aggregate.
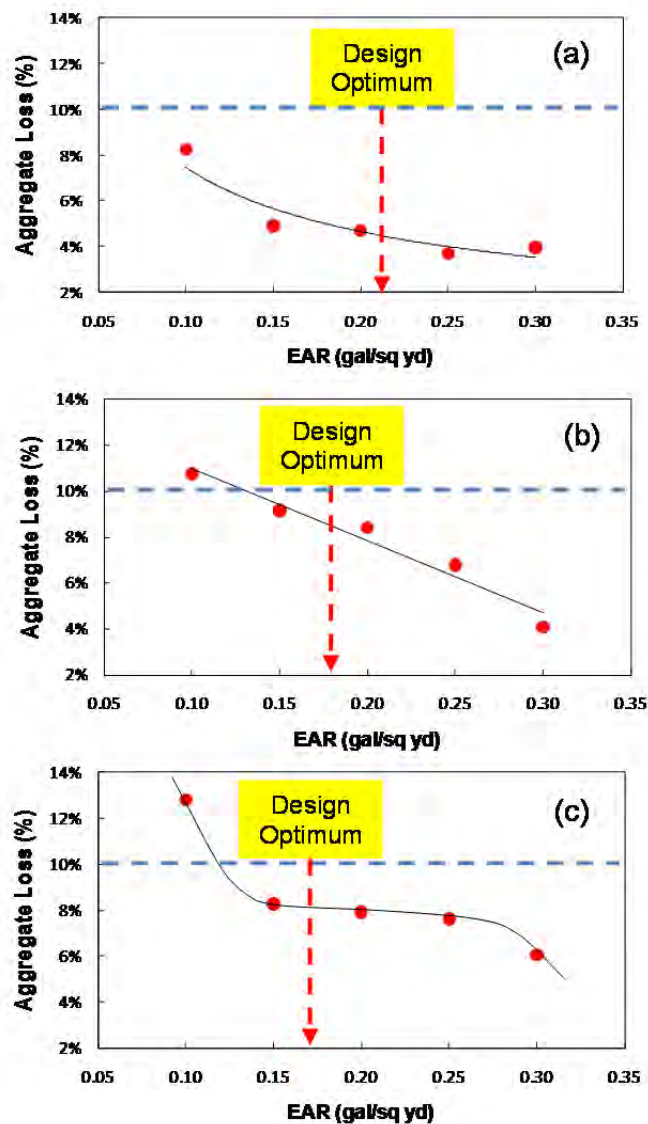

FIGURE 25 Mix design optimal EAR in MMLS3 aggregate loss tests for (*a*) Gradation A, where PUC = 17.1%; (*b*) Gradation B, where PUC = 33.3%; and (*c*) Gradation C, where PUC = 50.8%.
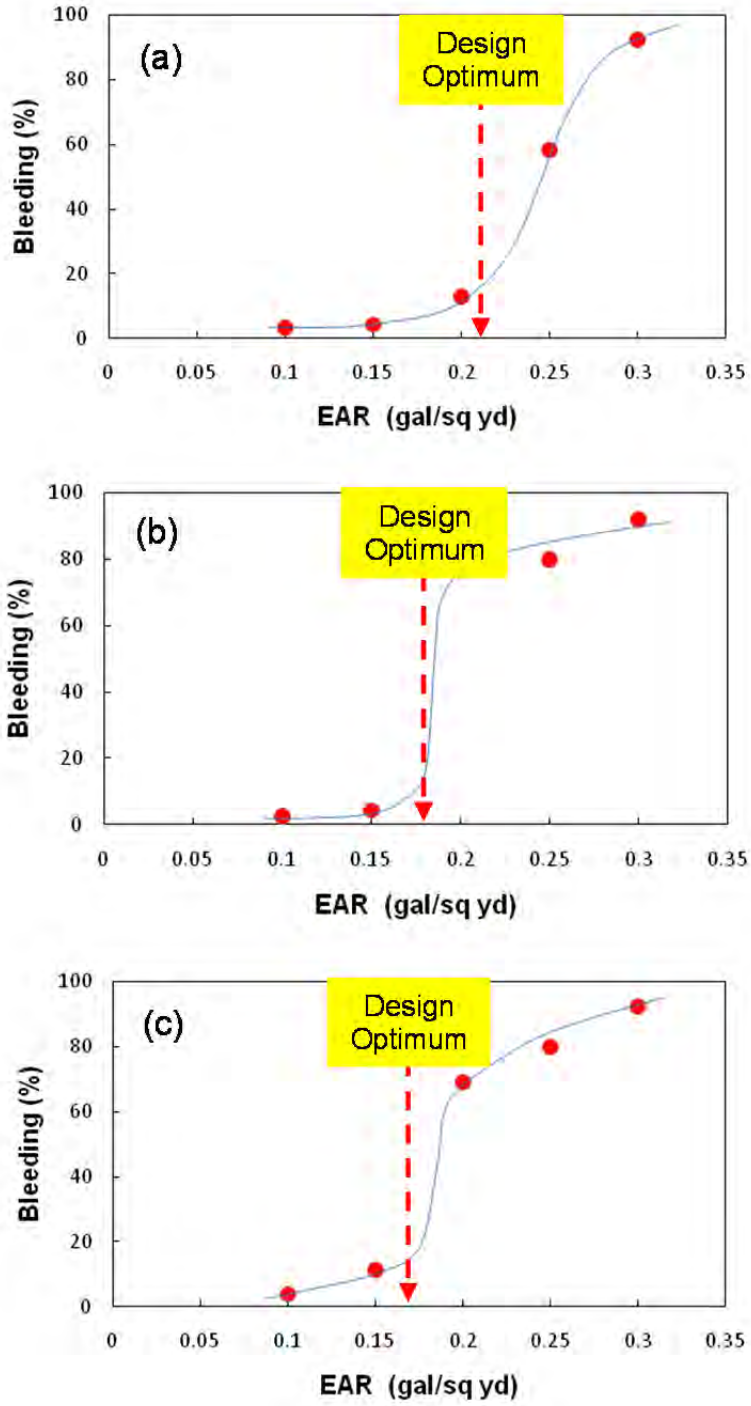
FIGURE 26 Mix design optimal EAR in MMLS3 bleeding tests for (*a*) Gradation A, where PUC = 17.1%; (*b*) Gradation B, where PUC = 33.3%; and (*c*) Gradation C, where PUC = 50.8%.

The bleeding performance results shown in Figure 26 indicate that as the emulsion rate increases, the likelihood of bleeding increases, as expected. The optimal EAR based on bleeding performance is determined to be the point immediately before the spike in bleeding occurs as the EAR increases. Figure 26 shows that the NCSU mix design provides a design EAR that results in acceptable performance with regard to bleeding performance. Therefore, the NCSU mix design performance tests validate that the design succeeds in minimizing the possibility of aggregate loss (as displayed in Figure 25) without increasing the likelihood of bleeding for the CRS-2L emulsion (as displayed in Figure 26) for multiple aggregate gradations.

HIGH-TRAFFIC VOLUME ROAD APPLICATION

In an effort to extend the use of chip seal surface treatments to roadways with higher traffic volumes than are used currently, a field study was conducted by constructing field sections for a variety of traffic volumes with ADT levels ranging from 5,000 to15,000 vehicles per day. The field sections were constructed using granite aggregate at the same target EARs and AARs for direct comparison. These field sections were scanned for changes in texture depth using the MPD parameter, and aggregate loss was measured.

Figure 27 indicates that the overall change in MPD, or the overall change in surface texture depth, varies as a function of ADT. As expected, a higher traffic volume leads to a higher change in surface texture (MPD), as the aggregate particles reorient to their least dimensions and are embedded deeper into the emulsion.
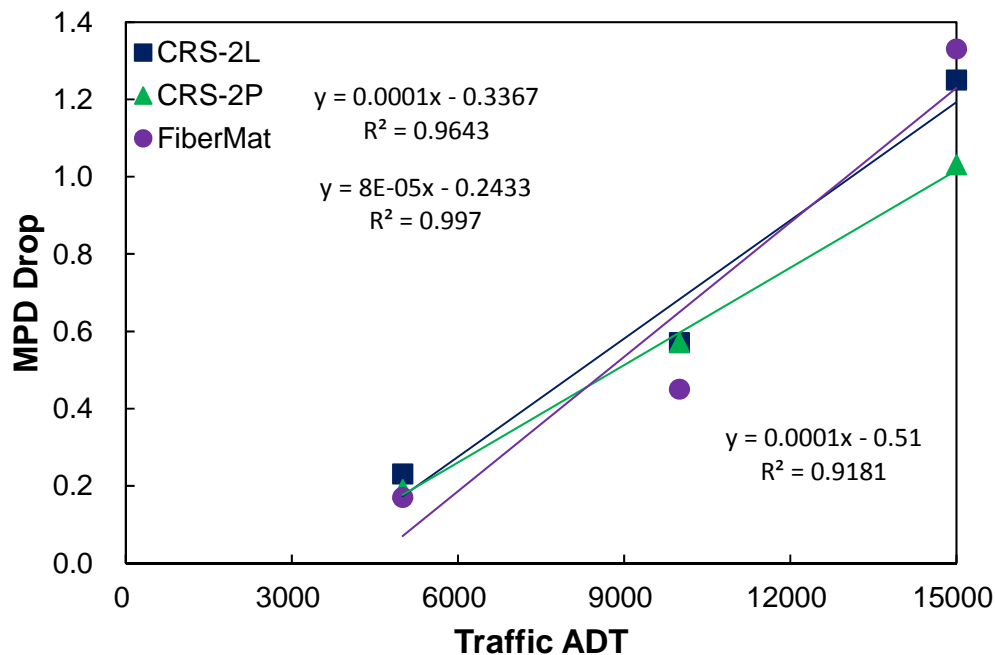
FIGURE 27 Relationship between overall MPD drop and field ADT.

Figure 28 shows that the overall decrease in MPD under traffic loading from time 0 (represented by the seal immediately after sweeping) to the time when the asymptotic MPD value is reached has a very strong relationship with the aggregate loss performance of chip seal treatments. This relationship indicates that the drop in MPD (which also has a relationship with ADT, as seen in Figure 27) can be used as a predictor of aggregate loss performance. These relationships are used to develop the summary data shown in Figure 29.

FIGURE 28 Relationship between the aggregate loss and the overall drop in MPD.

FIGURE 29 Field ADT versus percentage of aggregate loss for three chip seal sections.

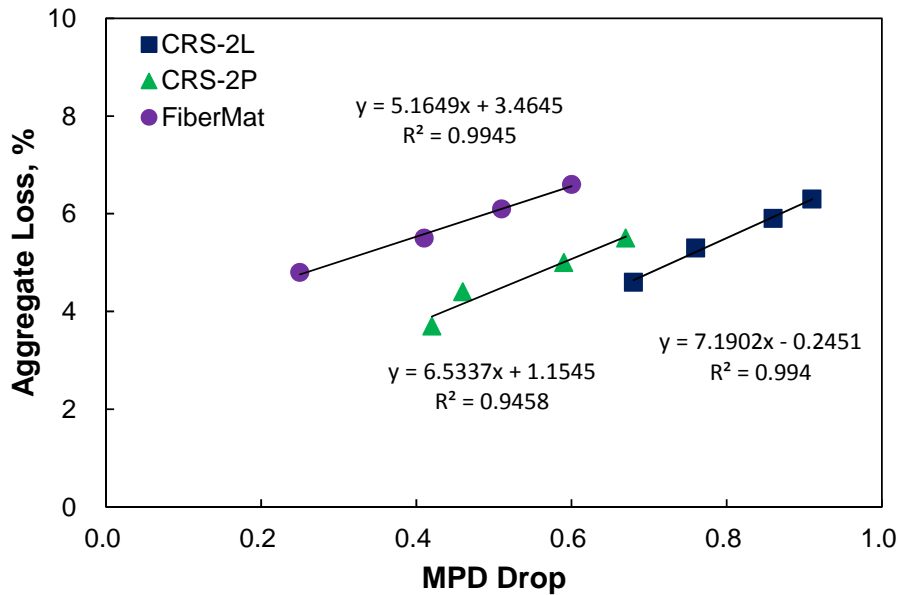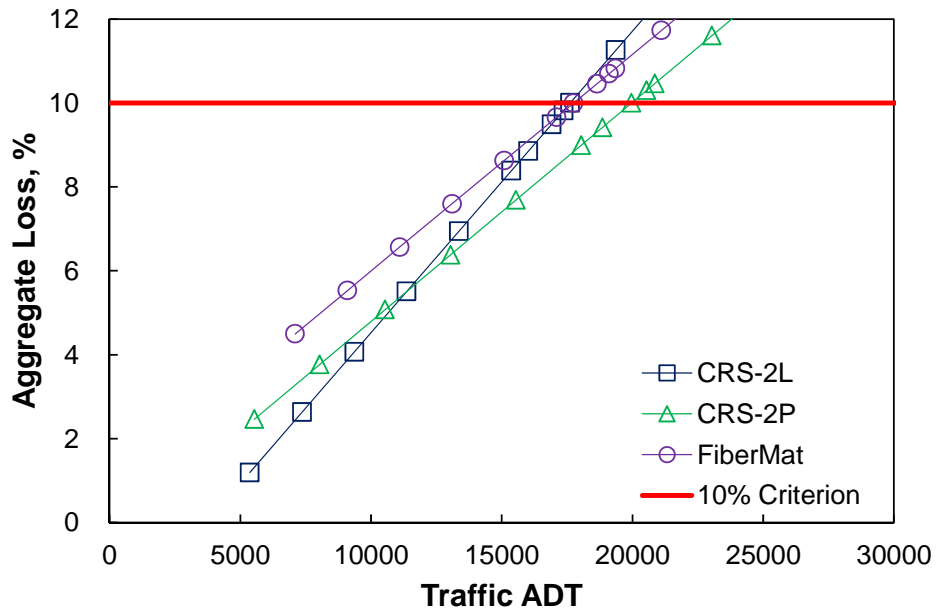Using the 10% aggregate loss criterion for chip seals, the data obtained from the field sections are used to estimate the ADT at which the aggregate loss is expected to be above 10%, based on the results obtained from the study. This concept is used to determine the critical estimated ADT at which aggregate loss is likely to occur in a typical chip seal treatment. Table 1 presents the estimated maximum traffic volume for each of the emulsion types included in the study.

RELATIONSHIP BETWEEN BOND STRENGTH AND AGGREGATE LOSS FOR THE DEVELOPMENT OF A PERFORMANCE-RELATED THRESHOLD VALUE

The Vialit and BBS testing were conducted on a variety of emulsion types, including both PME and unmodified emulsions, at a single temperature in order to determine the relationship between the aggregate loss of the chip seal mixture and the bond strength determined from emulsion material testing. In this study, each emulsion was cured for four total hours with BBS and Vialit testing at 1, 2, 3, and 4 h respectively. Figure 30 presents the results of these tests.

For each emulsion type shown in Figure 30, it can be see that as the emulsion cures, the bond strength increases accordingly. Likewise, as curing occurs, the overall aggregate loss

TABLE 1 Maximum Traffic Volume Estimate for High-Volume Chip Seal Sections

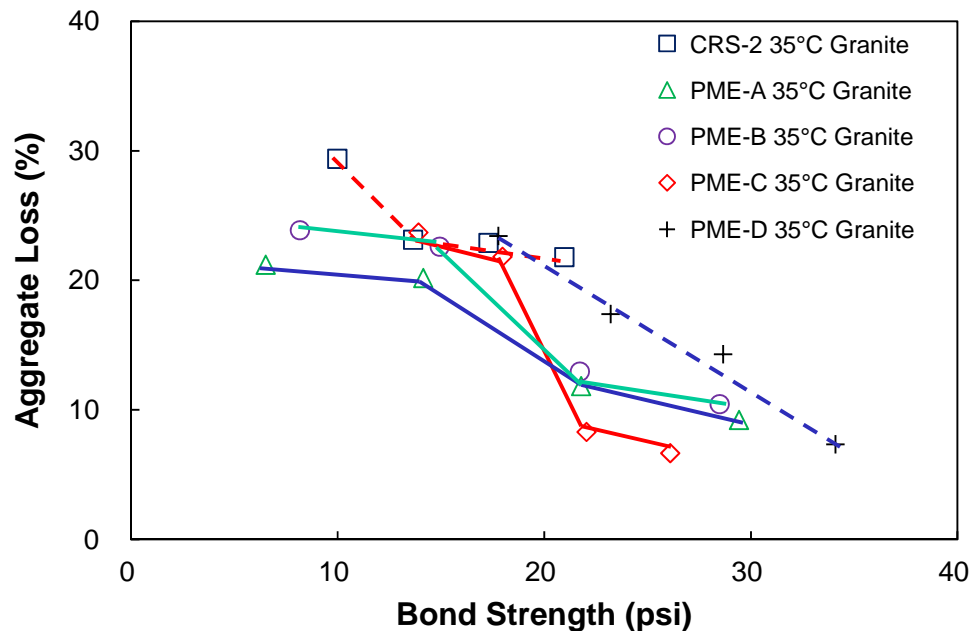| Sample Type	Maximum Traffic Volume (ADT)
CRS-2L	17,617
CRS-2P	19,996
CRS-2	17,750

FIGURE 30 Vialit test results at various curing times over 4 h of curing, shown as a function of bond strength from BBS testing.

decreases for each emulsion type. The figure also indicates that the unmodified CRS-2 emulsion develops less bond strength over the course of the four hours of curing than the PMEs. Likewise, it can be seen that the bond strength for each of the PMEs decreases near, or below, the 10% aggregate loss threshold, whereas the CRS-2 emulsion shows significantly inferior aggregate retention performance (i.e., more aggregate loss). Although the different PMEs reach the threshold at different rates, there appears to be a relationship between aggregate loss and bond strength that is sufficient to develop the critical bond strength from the emulsion testing to predict aggregate loss performance at 35°C.

The test results shown in Figure 31 indicate the sensitivity of bond strength to temperature as the bond strength increases as the temperature decreases and the binder stiffens. From this type of analysis for a wider variety of emulsions, a critical bond strength threshold can be determined at each temperature (e.g., 25°C and 35°C) that can be used to predict the performance of the surface treatment. This critical bond strength threshold would be based on the 10% aggregate loss threshold for chip seal surface treatments and would vary based on temperature.

CONCLUSIONS

This paper demonstrates that the performance test methods adopted for use in a series of chip seal studies at NCSU can evaluate the effects of various material and environmental factors on
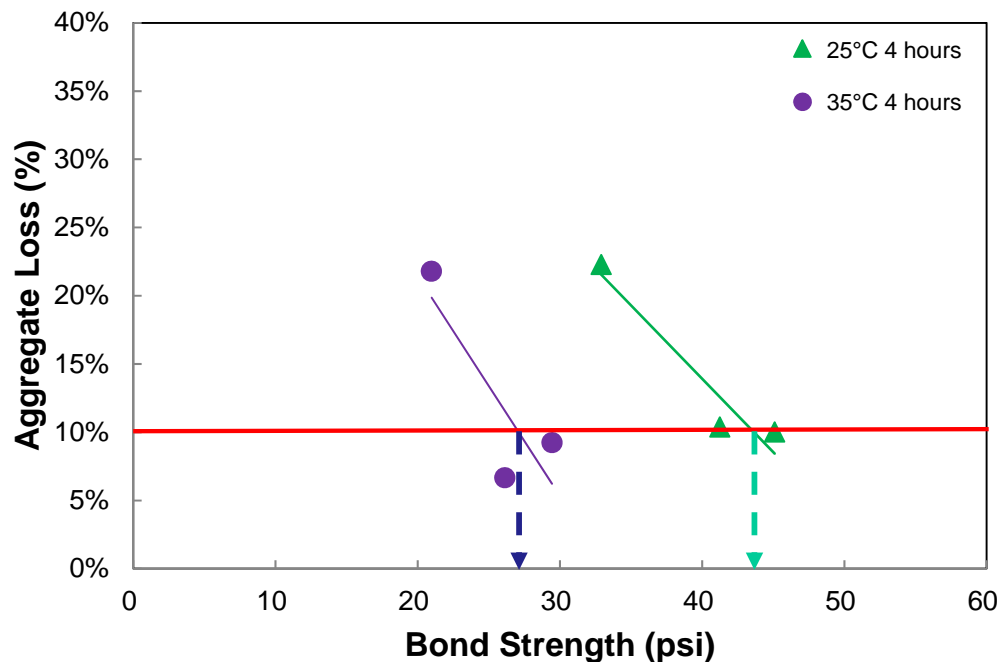

FIGURE 31 Bond strength versus aggregate loss at 4 h curing for multiple binders at two temperatures.

the performance of chip seals both qualitatively and quantitatively. The major findings and developments from these studies are as follows:

- The uniform gradation of aggregate is one of the most important factors for good chip seal performance. The PUC has been developed as a parameter to represent the degree of uniformity in aggregate gradations that can be used in chip seal specifications.
- The rolling pattern that yields the least aggregate loss is that which includes three passes of a pneumatic tire roller immediately after the aggregate is spread, followed by three passes of two combination rollers side by side.
- The overarching principle for multilayer chip seal surface treatments is that rolling the layer that is immediately below the top layer improves the aggregate retention performance of the top layer. Therefore, for a double seal, the bottom layer should be compacted for a single coverage, and for a triple seal, the second layer should be compacted as a single coverage without compacting the bottom layer.
- PMEs improve the performance of chip seals in terms of aggregate gradation, bleeding, and rutting. This improvement in performance is most significant at low temperatures.
- A new performance-based chip seal mix design method has been developed based on the findings of this research. This design method includes the modified board test to determine the optimal AAR and the 50% embedment depth concept, as implemented by the 3-D laser profiler, to determine the optimal EAR.
- A methodology has been developed to determine the maximum allowable traffic volume for a given chip seal. This methodology is based on the relationship between the reduction in MPD and the traffic level and the relationship between the percentage of aggregate loss and the MPD reduction.
- The BBS test has been demonstrated to be a means to specify emulsions to warrant the appropriate aggregate retention performance in chip seals.

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Field Sampling and Testing of Death Valley Chip Seal Emulsion Residue
A Case Study

MICHAEL J. FARRAR
QIAN QIN
R. WILLIAM GRIMES
JEAN-PASCAL PLANCHE
RYAN BOYSEN
JENNY LOVERIDGE
STEVE SALMANS
ALEC OTTO COOKMAN
Western Research Institute

This study presents the results of an investigation of emulsion residue recovered in 2012 from a chip seal placed in Death Valley, California, in 2008. This circular paper reports techniques used successfully to collect, extract, and measure the physicochemical properties of the emulsion residue as well as potential difficulties to be considered in each step. The results of the investigation are compared to the laboratory-recovered emulsion residue on the same chip seal project reported by King and Johnston (*1*).

INTRODUCTION

While there has been recent progress in developing performance-based emulsion residue test methods and specifications (*1–3*), there has been very limited chemical and rheological evaluation of the actual changes that occur in service under varying traffic and environmental conditions.

Chip seal emulsion residues are highly susceptible to oxidative aging because they are applied to the pavement surface, where they are exposed to high temperatures and an abundance of oxygen. Epps et al. (*4*) have shown that for chip seals constructed in Texas, the oxidative aging that occurs in 1 year on the road is roughly equivalent to the amount of aging that occurs during standard AASHTO R 28 PAV aging. The Epps et al. study compared laboratory-recovered and PAV-aged emulsion residues to extracted emulsion residues from the road, where the emulsion used in the lab was the same as that used in the field. The study used infrared (IR) spectroscopy to compare the extent of oxidative aging; application of IR spectroscopy rather than rheology in this sort of assessment makes the analysis relatively straightforward, because the extraction and recovery process does not change the carbonyl absorbance.

The Epps et al. study involved bulk extraction and recovery of the emulsion residue and did not show if there is an oxidative aging gradient within the emulsion residue layer. In this study, we have attempted to measure the oxidation gradient by applying a solvent wash technique to acquire IR spectra of the surface and bottom of emulsion residue field samples.

Determining the rheological properties of field samples of emulsion residue is challenging—particularly for styrene butadiene rubber (SBR) latex-modified emulsions, which are reported to form a honeycomb structure during low-temperature evaporative curing in the lab

and on the road (*5, 6*). Just as with recovering the emulsion residue from asphalt emulsion in the laboratory, recovering emulsion residue from field samples must be completed in a manner that causes the least possible disruption to the asphalt morphology and polymer network. A recovery method using dichloromethane (DCM) and a maximum temperature of 80°C during the extraction process showed promise as a way to obtain representative samples of emulsion residue from the road.

Another difficulty associated with emulsion testing is the relatively small amount of residue collected during field sampling. It is often impractical to collect sufficient emulsion residue to allow application of the BBR, which requires approximately 15 g to prepare one sample. This study used a recently developed 4-mm-diameter parallel plate DSR technique, commonly referred to as 4-mm DSR. The technique allows testing to as low as –40°C, requires only 25 mg of binder, and can be used to estimate BBR *m*-value and creep stiffness (*7, 8*).

EXPERIMENTAL

IR Spectroscopy

Researchers performed liquid cell IR analysis of emulsion residue in the transmission mode with a 1.0-mm solution cell with 50 mg asphalt per 1 mL carbon disulfide. Thirty-two scans were co-added on a Perkin Elmer Spectrometer 100 operated at a resolution of 4 cm^{-1}.

Attenuated total reflectance (ATR) was performed with a single bounce diamond ATR accessory coupled to a Perkin Elmer Spectrometer 100. Sixteen scans were collected at a resolution of 4 cm^{-1}.

Dynamic Shear Rheometry

Researchers performed dynamic shear tests with a Malvern Kinexus-controlled stress DSR and a TA Instruments ARES-controlled strain DSR. Storage and loss modulus master curves were developed from frequency sweep data using Rhea software developed by Abatech Consulting Engineers.

Scanning Electron Microscopy

Researchers performed the morphological study of latex modified emulsion residues on a Hitachi TM 1000 Tabletop Scanning Electron Microscope (SEM). The tabletop SEM uses an accelerating voltage of 15 kV and a backscatter electron detector to generate SEM images with a resolution of about 30 nm. Collecting images with this instrument requires minimal sample preparation, with no need for sample coating. Prior to imaging, researchers pretreated the samples by soaking the asphalt materials in methyl ethyl ketone (MEK) for 3 h at room temperature to reveal the polymer structure. After removal from the MEK, the imaging samples were dried at room temperature for approximately 3 h.

Emulsion Residue Extraction and Recovery

Researchers submerged approximately 30 g of field-scraped emulsion residue in DCM for 1 h. A syringe was used to pull off the supernatant, which was placed into Teflon centrifuge tubes. This process was repeated with each sample until the supernatant was almost clear in color. Tubes containing supernatant were centrifuged at 4,000 rpm for 25 min. A clean syringe was used to pull off supernatant to a point about 2 cm above the fine aggregate sediment. Supernatant was placed in pre-cleaned aluminum pans and allowed to evaporate under the hood for 2 h under a nitrogen sweep. The pans were then placed in a vacuum oven at 80°C for 4 h. IR was used to confirm that the solvent had been removed.

MATERIALS

This study investigated chip seal emulsion residue originally placed in 2008 on Highway 178 in Death Valley, California. The field sample was collected at MP 50.5 during August 2012.

FLH placed the SBR latex-modified chip seal and included the project in a major FLH study by King and Johnston (*1*), the object of which was to determine best practices and provide recommendations for the use of polymer-modified asphalt emulsions in surface treatments such as chip seals in varying climates.

The laboratory emulsion residue recovery method used in the King and Johnston study was an evaporative method consisting of 24 h at 25°C and then 24 h at 60°C in a forced draft oven. Some of the laboratory-recovered residue was then aged in a PAV under standard AASHTO R28 conditions.

This report gives the rheology results reported by King and Johnston to allow comparison between laboratory- and field-recovered emulsion residues.

To determine the effect of extraction and recovery on polymer-modified asphalt, this study used an SBR latex-modified emulsion supplied by the Utah DOT.

FIELD SAMPLING: COLLECTING CHIP SEAL EMULSION RESIDUE

Chip seal emulsion residue can be collected from the pavement surface using a variety of scraping tools, provided that the pavement surface temperature is sufficiently high. On a warm, sunny day where pavement surface temperatures are 50°C or above, sample collection is relatively simple and can be accomplished using a stiff putty knife or other scraper-type tool. The tool's cutting edge is forced through the chip layer by rocking the blade back and forth a bit or by striking the butt of the tool to work the edge down to the original pavement surface and under the emulsion layer. Once the edge is in contact with the underlying pavement surface, the tool edge can be slid across the old surface to cleanly remove the emulsion residue layer. Researchers successfully used the various scraping tools shown in Figure 1 (tile scraper, putty knife, and reciprocating saw with tile scraper blade) for emulsion residue collection at several field sites.

When the pavement surface temperature is below 50°C, scraper tools become ineffective. The cold material tends to be too stiff to penetrate and scrape with the force that can be reasonably applied with the hand tools shown in Figure 1. A simple pavement heater can be used

to warm the pavement surface for sampling on colder days. Researchers successfully used a 250-watt infrared heat lamp mounted in an upside down wastebasket (Figure 2) for this purpose.

With this heater, the pavement surface temperature increased from 0°C to 50°C in about 15 min on a cold and breezy day. Using a more efficient heater could easily decrease the amount of time required, but aside from the rather long warm-up time, this very simple device worked quite nicely.

Researchers also evaluated a hammer drill fitted with dust collection and a custom shallow-hole depth-stop as a possible means of extracting an emulsion sample when the pavement surface temperature is low. Figure 3 shows a hammer drill with a homemade dust collector system. Due to the conical shape of the drill bit tip and inconsistencies in the chip seal surface, it proved difficult to sample just the emulsion residue without getting part of the
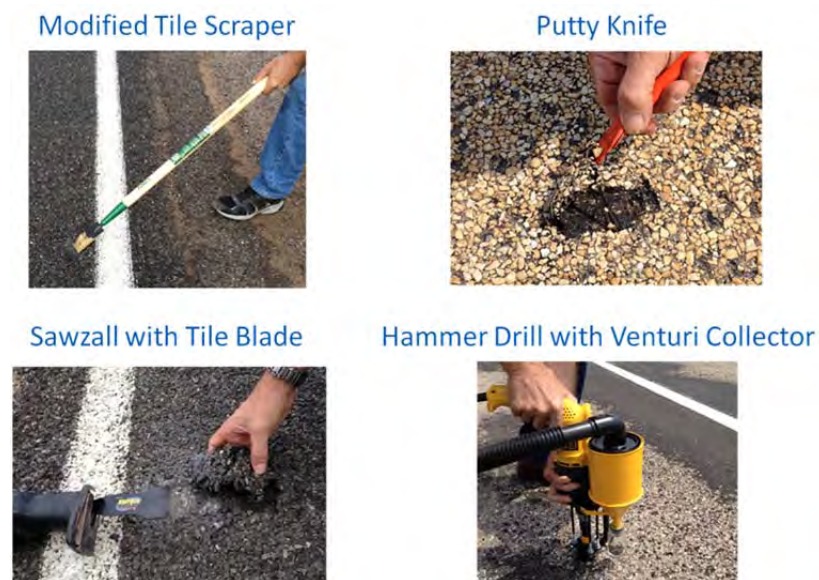

FIGURE 1 Emulsion residue sampling methods.

FIGURE 2 Portable pavement heating unit.

FIGURE 3 Homemade Venturi dust collection system.

underlying pavement surface. Since the heating and scraping technique can be conveniently used to obtain samples when the weather is cold, further development of this system was abandoned. This system may be useful when the interaction or penetration of the emulsion and the aged pavement surface is of interest.

Practical experience led to a strong preference for a more mechanized collection technique, due to the large force required with the manual scrapers; however, when using the reciprocating saw with scraper blade, the reciprocating motion of the saw, with a stroke of approximately 1 in., made sampling somewhat cumbersome. When scraping the pavement with this tool, the saw body tends to also reciprocate, causing unnecessary strain on the operator and excessive damage to the cutting edge of the tool bit.

Based on field experience, collection of a chip seal sample is best accomplished using a hammer drill (in nonrotational mode) with a chisel-type bit, commonly referred to as a demolition hammer when used in this mode. The hammer drill striker mechanism provides the mechanical force needed to penetrate the surface and subsequently scrape off a patch of emulsion residue. Due to the relatively small mass of the striker mechanism, very little motion is translated to the tool body; this reduces operator fatigue while maintaining contact between the sample and the chisel, which limits damage to the cutting edge. Figure 4 shows a cordless hammer drill with chisel attachment as recommended for chip seal sampling.

MEASURING THE OXIDATIVE AGING GRADIENT IN THE EMULSION LAYER BY IR

Oxidative Gradient Surface Wash

Researchers acquired IR spectra of the top and bottom of the emulsion residue using a solvent wash method. The samples used for this analysis were collected with the reciprocating tile blade equipment shown in Figure 1 and were essentially intact with an area of roughly 3 in.^{2}. The surfaces were washed with carbon disulfide as shown in Figure 5, to a roughly estimated depth of 0.3 mm. The liquid was centrifuged to eliminate very fine aggregate particles before the asphalt was recovered using a vacuum oven at 60°C. After drying, ATR single-bounce diamond IR spectra were collected.

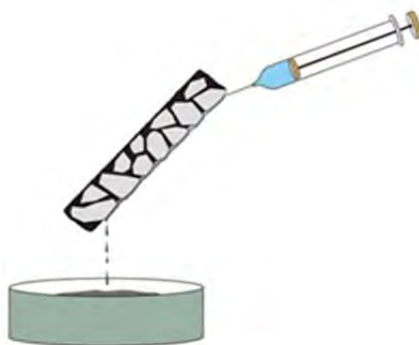**FIGURE 4 Cordless hammer drill with chisel.**

FIGURE 5 Surface wash technique with carbon disulfide.

The solvent wash spectrum shown in Figure 6 represents the top and bottom of the emulsion residue layer. The extent of oxidation at the top surface compared to the bottom of the emulsion layer can be observed by comparing the height and broadness of the carbonyl absorbance bands centered at ~1,700 cm^{-1} and the sulfoxide bands centered at ~1,030 cm^{-1}. The extent of polymer degradation at the surface and bottom can be assessed by comparing the bands at 966 cm^{-1} and at 699 cm^{-1}. The 966 cm^{-1} band corresponds to the SBR C-H out of plane bending of transalkene (present in the polymerized butadiene), and the 699 cm^{-1} band corresponds to the SBR C-H out of plane bending in monoalkylated aromatics (styrene) (*9*). Here we evaluate the bands at 966 cm^{-1} due to difficulty in interpreting the spectrum in the 699 cm^{-1} region.

Figure 6 shows that there is more carbonyl at the top surface compared to the bottom, which was expected. The difference is not large and suggests a relatively limited oxidative gradient. In terms of polymer degradation, there appears to be a slight gradient from the top to bottom based on the 966 cm^{-1} band. Thermooxidative reactions in styrene butadiene occur at
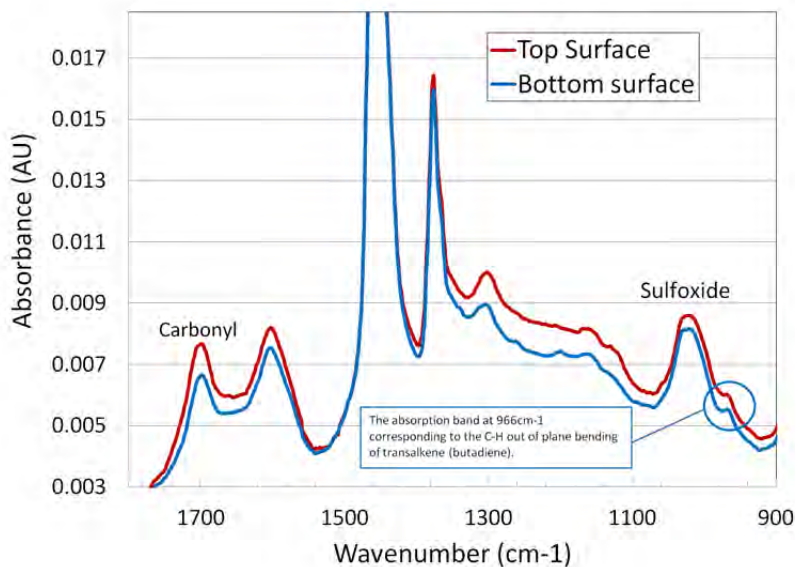
FIGURE 6 IR ATR: top and bottom emulsion residue spectra.

unsaturated bonds of the polybutadiene segments, causing not only chain scission but also macrophase separation between polystyrene-rich and polybutadiene phases (*10*); no oxidation of the polystyrene segments is expected.

Bulk IR: Liquid Cell

Figures 7*a* and 7*b* display liquid cell spectra of the bulk samples of recovered emulsion residue. Bulk samples refer to tests on the complete emulsion residue layer after extraction and recovery. The extraction was performed using DCM and a maximum temperature of 80°C.

The first thing to note is that all the DCM solvent was removed during the recovery process, at least to the level of the detection threshold of the IR instrument. That assessment is based on the lack of any noticeable peak at a wave number of ~ 1,260 cm^{-1}, which corresponds to DCM H-C-H out of plane wagging (a strong absorbance peak in the DCM spectrum).

FIELD EMULSION RESIDUE SOLVENT EXTRACTION–RECOVERY AND POLYMER STRUCTURE

Standard extraction and recovery methods such as AASHTO T319 are generally appropriate for the recovery of asphalt binder from unmodified or polymer-modified hot- or warm-mix asphalt; however, there are some unique aspects to be considered when extracting field chip seal samples of unmodified or polymer-modified emulsion residue—particularly in the case of SBR latex-modified emulsion residue, where there is a concern that the polymer structure that forms during emulsion curing may be significantly disrupted by the extraction solvent or the high heat involved during the recovery phase of the process.
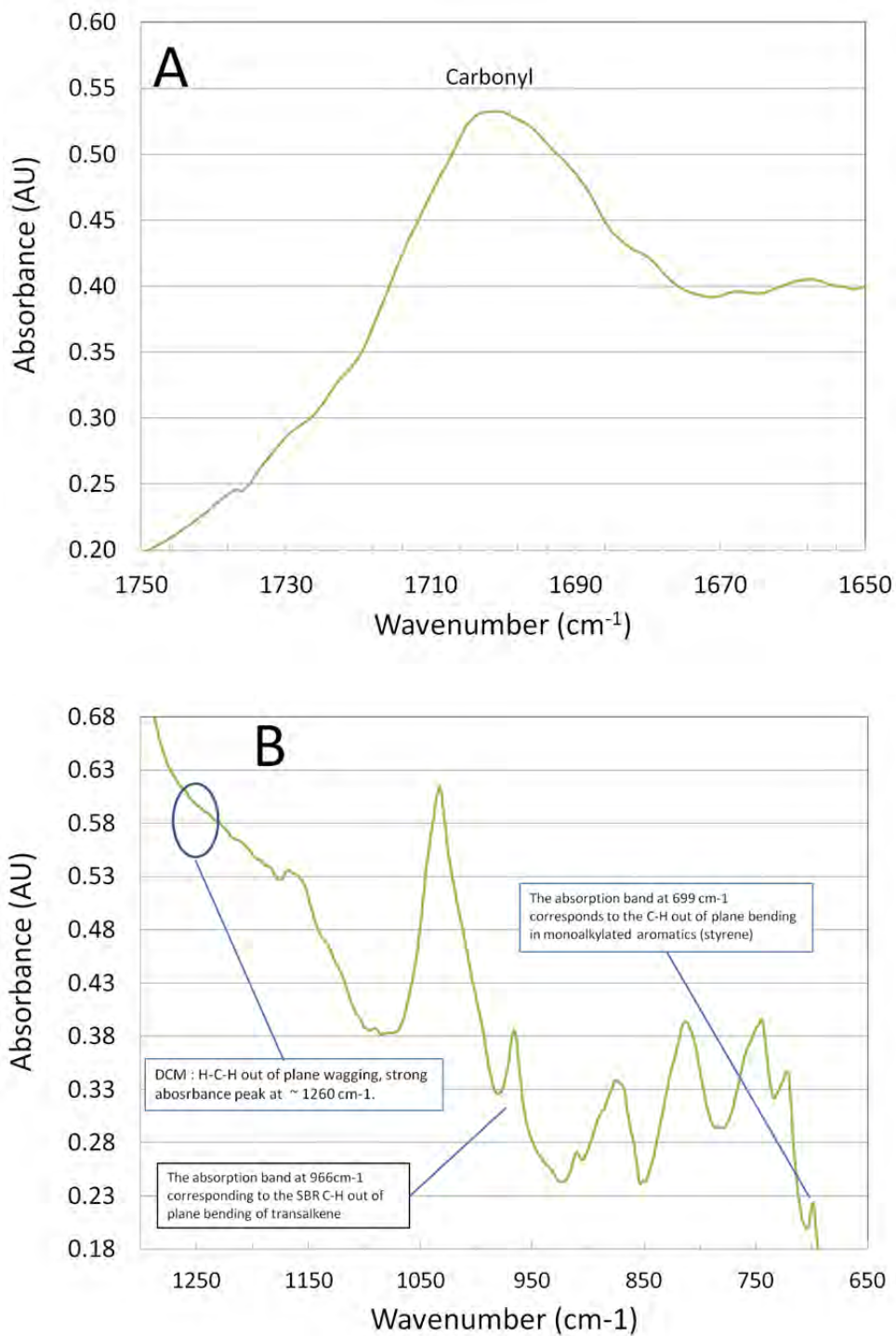
FIGURE 7 Liquid cell IR spectra–bulk sample of emulsion residue: (*a*) carbonyl region centered at about 1,700 cm⁻¹ and (*b*) approximate IR fingerprint region.

Takamura (*5, 6*) has shown that when water starts to evaporate from SBR latex-modified asphalt emulsion, dispersed latex particles in the emulsion migrate together and form a honeycomb structure surrounding the asphalt droplets. Takamura (*6*) further suggests and shows some evidence that this SBR honeycomb structure imparts elastic properties to the residue, allowing high strain from external stresses even at low temperature without causing major fracture.

In regard to laboratory recovery of emulsion residue, Hazlett (*11*) asks "How do we get emulsion residue representative of in-service binder?" The present study asks "How do we extract and recover emulsion residue from chip seal field samples representative of the in-service binder?"

The Death Valley chip seal was constructed using an SBR latex-modified emulsion, so the first step in evaluating the question was to confirm that there was in fact a honeycomb structure present in the emulsion residue field samples. Researchers considered and tried several methods to acquire an image of the honeycomb structure: atomic force microscopy (AFM), epifluorescence, and SEM. Along with these methods, we also tried different treatments to the emulsion residue to draw out the polymer structure. After several preliminary experiments, it appeared that the best method, along the lines proposed by Wolfe et al. (*12*), was using SEM and dissolution of the asphalt with MEK, which is a poor solvent for SBR. Gold or platinum coating was not necessary, as researchers used a backscatter electron detector that required only minimal sample preparation. Figure 8 displays the apparent honeycomb observed in the Death Valley emulsion residue using SEM and MEK treatment.

To evaluate whether or not the structure shown in Figure 8 is the honeycomb or perhaps an artifact, researchers recovered emulsion residue from an SBR latex-modified emulsion provided by the Utah DOT using the universal simple aging test (USAT) method (*13*). The USAT emulsion residue recovery method is comparable to AASHTO PP 72-11, Method B. Figure 9 shows the SEM image of the USAT-recovered emulsion with a honeycomb structure comparable to that in Figure 8.
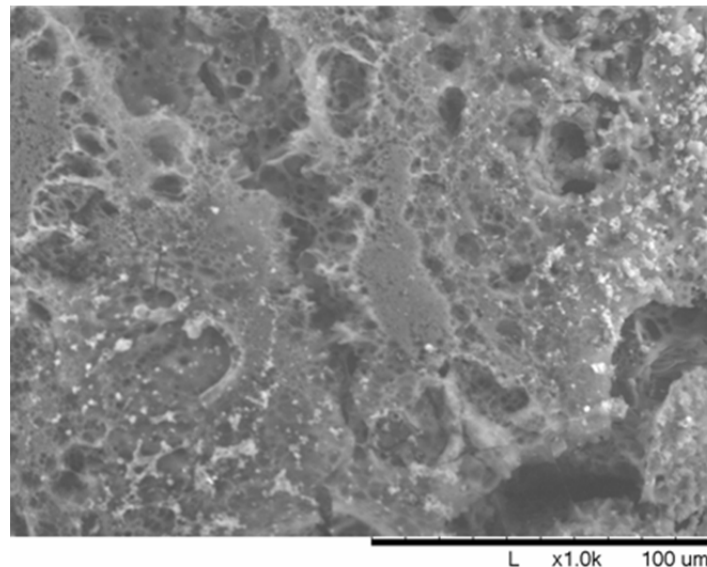

**FIGURE 8 SEM image: Death Valley MP 50.5 original scraping,
soaked in MEK before imaging.**

Figure 10 shows an image of the USAT-recovered material after dissolution in DCM and then removal of DCM under vacuum at 80°C. In this figure, there is a complete absence of the honeycomb structure.

Figure 11 shows the liquid cell IR spectra of the USAT SBR latex-modified recovered emulsion residue without any treatment and after DCM treatment. Aside from a slight variation in sulfoxide, which is not unusual, the spectra are essentially identical.
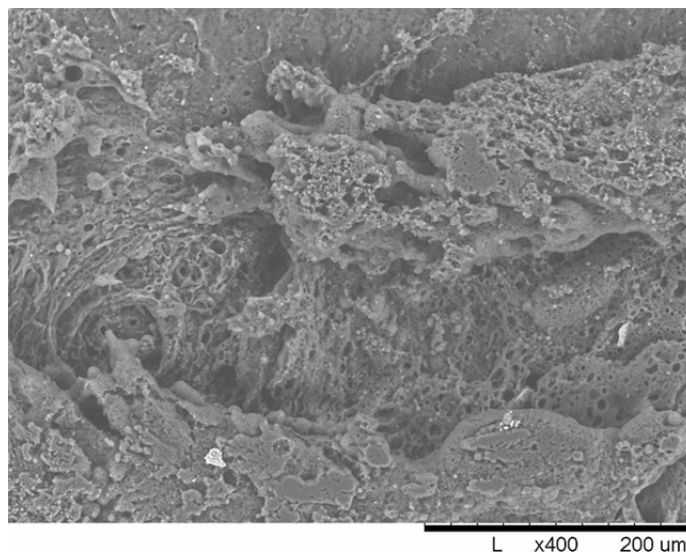

FIGURE 9 SEM image: CRS-2L (SBR latex-modified emulsion) recovered in the lab using the USAT method, soaked in MEK before imaging.
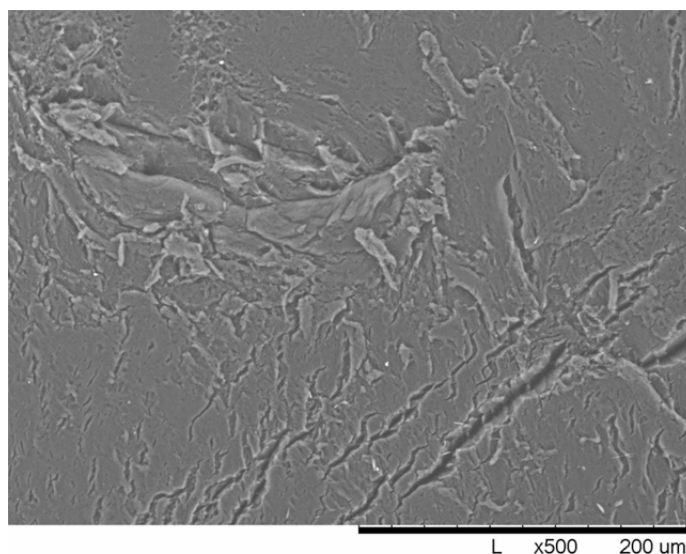

FIGURE 10 SEM image: CRS-2L (SBR latex-modified emulsion) lab-recovered, dissolved in DCM, then DCM removed with vacuum at 80°C, soaked in MEK before imaging.
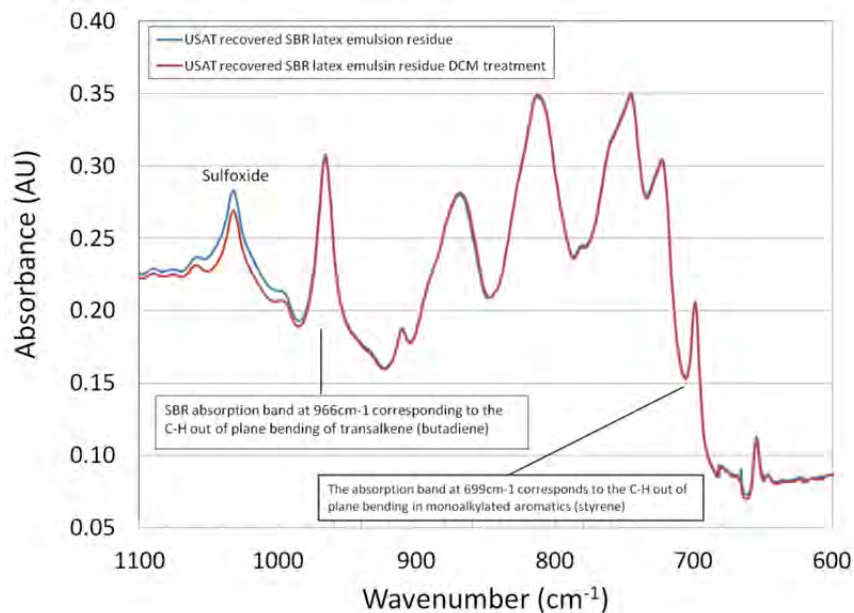
FIGURE 11 Liquid cell IR spectra.

The honeycomb appears to have been completely disrupted by the DCM extraction process; however, somewhat surprisingly, as shown in Figure 12*a* and 12*b* comparing 50°C and 70°C frequency sweeps, the linear viscoelastic properties between the materials shown in Figures 9 and 10 appear unchanged.

Figure 12*c*, a black space plot of the two frequency sweeps, further investigates this similarity in rheology before and after DCM treatment. A black space plot, also known as a van Gurp-Palmen plot (*14*), depicts the phase angle versus the corresponding absolute value of the complex shear modulus from the dynamic rheological data. The plot is typically used to measure the validity of time–temperature superposition (TTS) and thermorheological simplicity. The black space plot is one in a family of similar plots including the Cole-Cole, Han, and Wicket plots, which are independent of reduced frequency hence temperature.

It appears that the TTS principle roughly holds for the untreated and treated DCM samples; however, some failure of the TTS principle can be observed and is expected due to the presence of SBR latex polymer. It is particularly intriguing that the untreated and DCM-treated samples are essentially identical in black space. In the untreated sample, the latex and associated honeycomb structure could be considered essentially an immiscible blend with the asphalt, whereas after DCM treatment, one would expect a more homogeneous blend, and that appears to be the case in the morphology of the two samples shown in Figures 9 and 10.

The similarity in linear viscoelasticity between untreated and DCM-treated samples of SBR latex emulsion residue appears to also hold true for the nonlinear viscoelastic properties. Figure 13 compares the MSCR nonrecoverable creep compliance (J_{nr}) and percent recoverable strain at 64°C and 58°C (3.2 kPa).

Although dissolution in DCM and recovery under vacuum at 80°C appears to completely disrupt the honeycomb, the rheology remains essentially unchanged. This rather surprising result suggests that the field chip seal residue can be extracted with DCM and recovered under vacuum
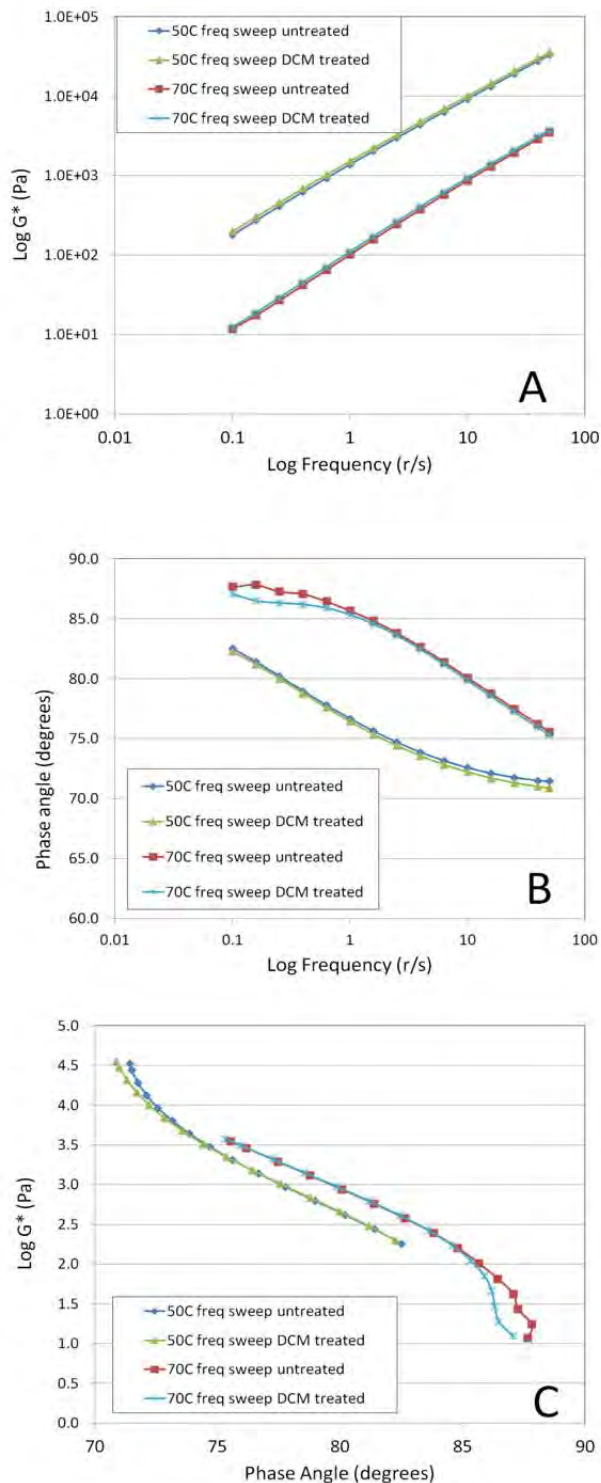
FIGURE 12 Frequency sweeps (50°C and 70°C): comparison of SBR latex-modified emulsion residue before and after dissolution in DCM and vacuum oven recovery, maximum temperature 80°C: (*a*) complex shear modulus; (*b*) phase angle; and (*c*) black space plot.

(a)

(b)
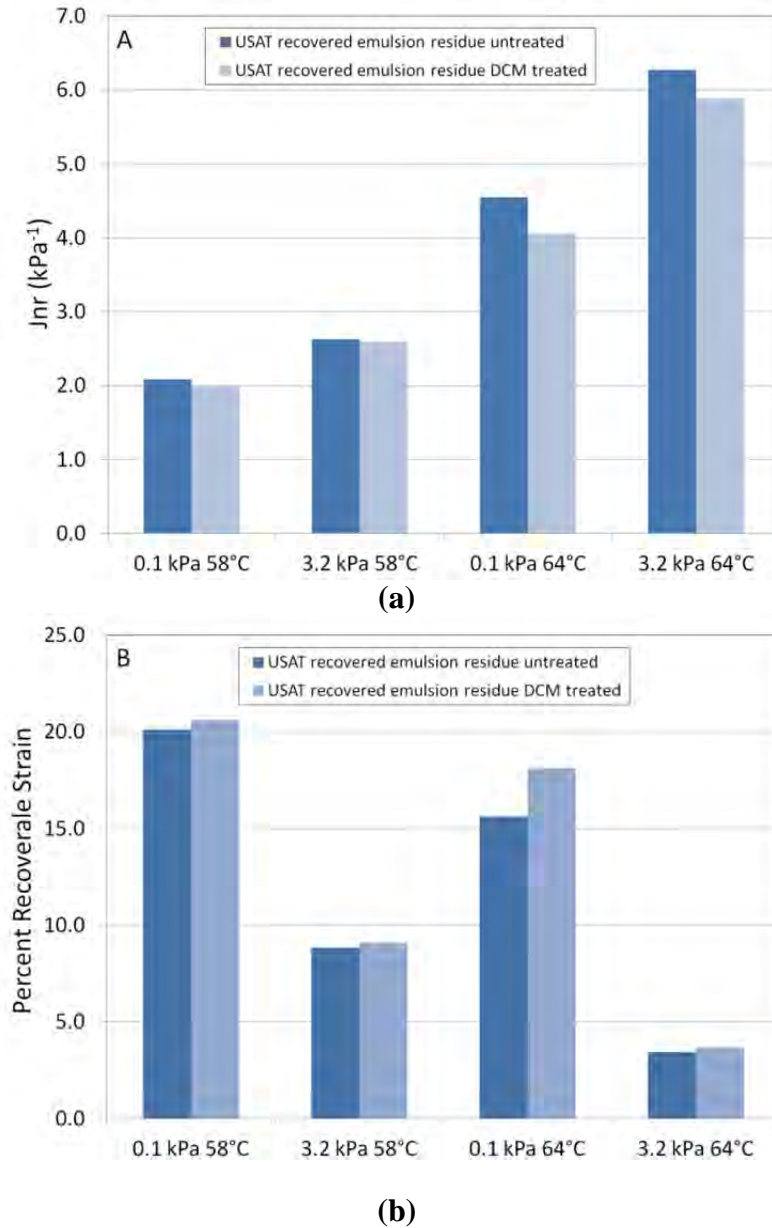
**FIGURE 13 Comparison of SBR latex-modified emulsion residue before and after
dissolution in DCM and vacuum oven recovery, maximum temperature 80°C:
(*a*) nonrecoverable creep compliance (*J*$_{nr}$) and (*b*) percent recoverable strain.**

with a maximum temperature of 80°C without unduly influencing the measured rheology. This
would seem to indicate that the measured linear and nonlinear viscoelasticity of the recovered
residue may be reasonably representative of the rheology of the actual residue on the road. This
also means that there is no direct link between the morphology and the rheological properties of
an asphalt binder, different morphologies can lead to the same properties. Obviously, since this is
a somewhat unexpected finding and is based on limited data, additional tests and analysis are
necessary to confirm the finding.

LINEAR AND NONLINEAR VISCOELASTIC PROPERTIES OF FIELD-RECOVERED EMULSION RESIDUE

This section presents the rheological properties of the Death Valley-recovered emulsion residue and compares those to the rheological properties of the same emulsion residue recovered in the laboratory as reported by King and Johnston (*1*). The rheological properties considered are

- Superpave DSR $G^*/\sin \delta$: Temperature where $G^*/\sin \delta = 1.0$ kPa, 10 r/s;
- MSCR: Nonrecoverable creep compliance (J_{nr}) and percent recoverable strain at 64°C and 0.1 and 3.2 kPa:
  - Strain sweep: 25°C, 10 r/s, 0.01 to 50% strain; and
- Superpave m-value and creep stiffness:
  - Temperature where m-value = –0.30 and
  - Temperature where creep stiffness = 300 MPa.

As mentioned previously, there has been considerable progress over the last several years in developing performance-based chip seal emulsion residue test methods and specifications based on rheological parameters developed for grading hot-mix asphalt (HMA) binders; however, the test methods and interpretation involved in establishing these parameters is evolving. The present study followed test methods along the lines used by King and Johnston (*1*) to allow a direct comparison between lab- and field-recovered emulsion residue.

$G^*/\sin \delta$

The parameter $G^*/ \sin \delta$ is a rutting factor in the Superpave PG Asphalt Binder specification. $G^*/\sin \delta$ is measured under oscillatory shear at low strain level in the linear viscoelastic range. Due to the low strain level, the PG high-temperature parameter does not accurately represent the ability of polymer-modified binders to resist rutting (*15*). The minimum $G^*/\sin \delta$ for unaged (original) binders under the PG binder specification is 1.00 kPa, and for rolling thin film oven-aged binders, the minimum is 2.2 kPa.

A minimum $G^*/\sin \delta$ has been proposed for surface treatment specifications to ensure that the laboratory-recovered unaged emulsion residue is stiff enough at high temperatures to resist aggregate loss and bleeding during the first season (*2*). Figure 14 shows the continuous grade temperature using a threshold of 1.0 kPa. This study shows a dramatic increase in the continuous grade temperature of the emulsion residue after aging on the road for several years.

MSCR Test

The MSCR test provides a means for determining nonrecoverable creep compliance (J_{nr}) and percent recoverable strain from repeated creep loading. Low J_{nr} indicates resistance to flow, which could be interpreted in terms of chip seal performance as resistance to bleeding. When grading HMA binders, a doubling of the J_{nr} represents a softening by approximately one full binder grade (*1*).

As shown in Figure 14, there was a dramatic increase in $G^*/\sin \delta$ from in-service oxidative aging. Similarly, in Figure 15 we observe a decrease in J_{nr} of well over an order of magnitude due to oxidative aging at the Death Valley site. As with the $G^*/\sin \delta$ test, the MSCR
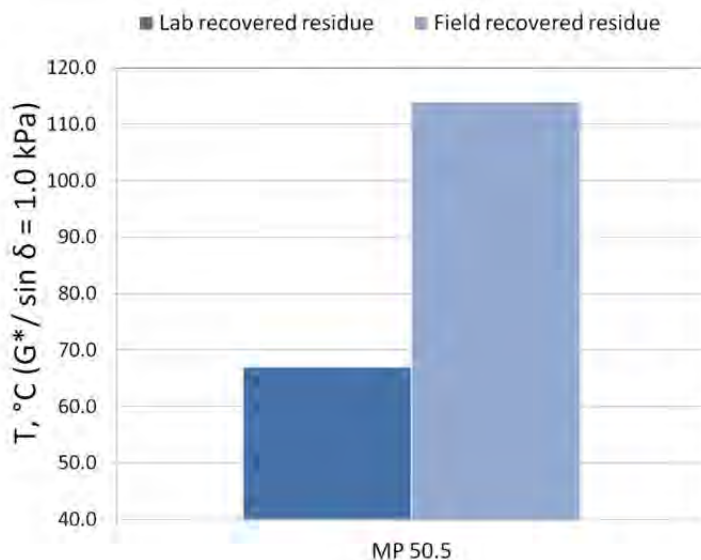
FIGURE 14 *G*/*sin δ = 1.0 kPa, continuous high-temperature grade (10 r/s).**
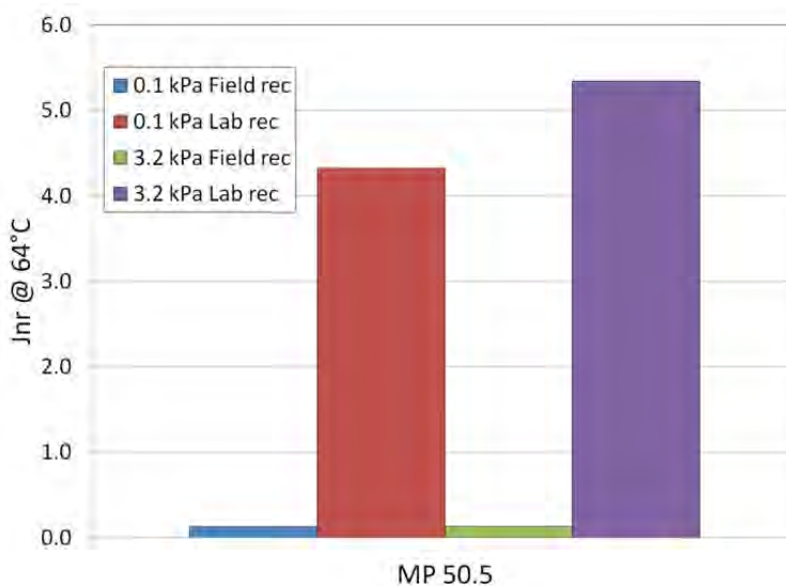
FIGURE 15 J_{nr} **unaged laboratory and field-recovered emulsion residue samples at 64°C.**

test, when applied to predict the potential for bleeding, is typically performed on the laboratory-recovered emulsion residue without further aging. Both temperature and stress levels are varied beyond AASHTO TP70 conditions to reproduce the softening due to temperature or increasing stress realized in the field (*3*). For this comparison of the unaged laboratory and field-recovered emulsion residues, J_{nr} at 64°C and two stress levels (0.1 and 3.2 kPa) are reported and displayed in Figure 15.

MSCR Percent Recoverable Strain

The part of AASHTO TP 70 dealing with percent recoverable strain (PRS) provides a means for determining the presence of an elastomeric polymer. While it is not quantitative, it is an alternative to the PG-plus elastic recovery test. The PRS from the MSCR test is used as an indication of the presence of polymer and is defined as:

$$PRS = 100 * \gamma_r/\gamma_p \tag{1}$$

where γ_r is the recovered strain and γ_p *is* the peak strain.

Wasiuddin et al. (*16*) reported a correlation between elastic recovery (AASHTO T301) and PRS for unmodified and PME residues. Wasiuddin et al. recommended a minimum PRS of 25 at 58°C and 0.1 kPa creep stress and a minimum PRS of 9 at 58°C and 3.2 kPa to indicate the presence of polymer.

This correlation was developed based on emulsion residue recovered using ASTM D7497, which requires 24 h at 25°C followed by 24 h at 60°C in a forced draft oven. Hanz et al. (*3*) found the ASTM D7497 evaporative recovery method produced residues with rheological properties similar to those of oven-cured or RTFO-aged base materials. PRS for aged conditions other than ASTM D7479 are not adequately developed.

Figure 16 plots the PRS for the Death Valley-recovered emulsion residues. As noted above, Wasiuddin et al. used a temperature of 58°C for the recommended minimum PRS of 25 at 0.1 kPa creep stress. Based on this data, we estimated a PRS threshold of 18 for 64°C and stress level 0.1 kPa (i.e., relevant to the MSCR test as conducted). Using the threshold of 18 suggests the Death Valley residue was polymer modified. As previously discussed, the IR spectra of the Death Valley emulsion residue also confirm the presence of polymer. [Note: There was insufficient DCM-extracted residue to complete the MSCR testing, so the MSCR data presented here is based on toluene–ethanol-extracted residue. In preliminary testing, we found very similar results when using either toluene–ethanol or DCM solvents, so we believe these data to be valid.]

Strain Sweep

Takamura (*6*) first proposed the strain sweep test, and while the original concept of the procedure—to evaluate the strength of emulsion residue under repeated high strain deformation—has remained relatively constant, the test procedure has evolved. Probably the latest iteration is to use the strain sweep part of the linear amplitude sweep (LAS) test. Hintz and Bahia (*17*) presented one of the latest versions of the LAS test at the 92nd Annual Meeting of the Transportation Research Board.

The present study performed the strain sweep test for the Death Valley field-recovered emulsion residue using the methodology in the King and Johnston (*1*) study: 8-mm parallel plates, 2-mm gap, 25°C, 10 rad/s, and strain from 0.01% to 50%.

The initial complex shear modulus of the field-aged emulsion residue (shown in Figure 17) is much stiffer than the laboratory PAV-aged recovered emulsion residue. This is not surprising, as PAV aging usually underpredicts field aging-related stiffening.

According to Vijaykumar et al. (*2*), the strain sweep test is capable of evaluating strain tolerance, or ability to resist aggregate loss, by monitoring the onset of nonlinear viscoelasticity. The field-recovered residue entered the nonlinear regime at roughly 2% strain. The unaged
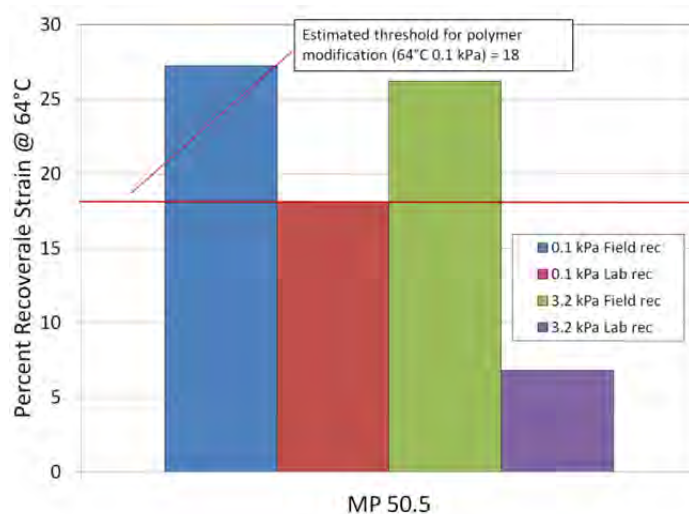
**FIGURE 16 PRS of unaged laboratory- and
field-recovered emulsion residue at 64°C.**
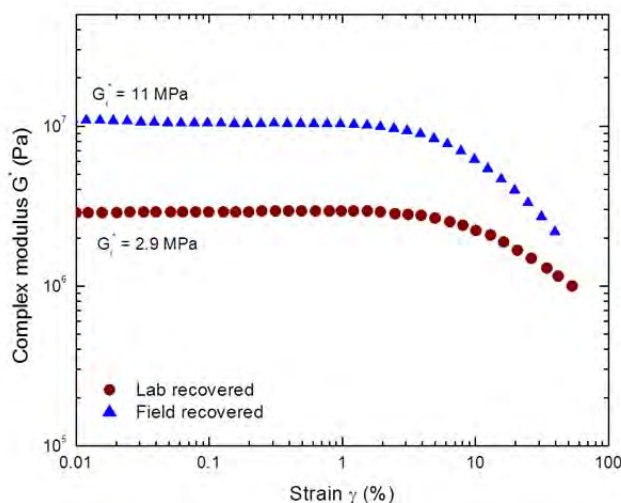
FIGURE 17 Linear sweep test.

laboratory residue entered the nonlinear regime at about 5% strain. The more rapid nonlinear response of the field residues might suggest a significant deficiency in terms of aggregate retention; however, although the field residues went nonlinear more rapidly, the strain level at which that occurred is still relatively high, and the material is probably not yet brittle.

The ductility model proposed by Ruan et al. (*18*) can be applied here to estimate the embrittlement threshold. Ruan et al. showed that the extensional flow of conventional asphalt binders, as measured by a ductilometer, can be qualitatively described with a simple elongation model using a viscoelastic Maxwell element. The model was developed for unmodified asphalt

and may not apply well for polymer-modified asphalt, still it might provide a very rough estimate of ductility (especially when ductility < 10 cm). The Ruan et al. model is of the form:

$$\text{Ductility} = 0.23 * \left\{ \frac{\frac{G'}{n'}}{G'} \right\}^{(-0.44)} \qquad (2)$$

where ductility is measured in centimeters at 15°C and an elongation rate of 1 cm/min with a ductilometer, and G' and η' are measured with a DSR at a reference temperature of 15°C and a frequency of 0.005 rad/s. Applying Equation 2, the ductility of the field-recovered emulsion residue is 4 cm, very close to the embrittlement threshold of 3 cm proposed by Ruan et al.

Low-Temperature Rheology (*m*-Value and Creep Stiffness)

Figures 18 and 19 show the continuous low-temperature performance grades of the field- and PAV-aged laboratory recovered emulsion residues. The low-temperature performance grades were determined using an iterative procedure to find the temperature at which *m*-value = 0.3 or $S(t)$ = 300 MPa.

The low-temperature rheological properties of the emulsion residues were measured at Western Research Institute using 4-mm diameter parallel plate geometry with a Malvern Kinexus rotational DSR. Frequency sweeps were performed at 15°C intervals over a temperature range of –30°C to 45°C (in some cases to 60°C) and an angular frequency range of 0.1 to 50 rad/s.

The test method requires only 25 mg of residue, but in actual practice, about 150 mg is necessary in order to load and trim the sample (still about two orders of magnitude less than the amount required to fabricate a BBR beam). Also, no specimen premolding is needed, and a relatively low temperature is used to load the samples into the rheometer.

The first frequency sweep was performed at 30°C after 20 min of conditioning at 30°C to ensure the specimen was at the test temperature. After performing the frequency sweep, the temperature was lowered to the next test temperature and the specimen again allowed to condition at that temperature for 20 min. The process was continued until reaching –30°C or the sample broke. The normal force was monitored continuously during the test and the gap adjusted to keep the normal force at or close to zero, which is essential to prevent stresses from building in the sample and possible rupture or loss of plate adhesion. During the development of the 4-mm DSR test, good reproducibility was found with data collected on 4-, 8-, and 25-mm diameter plates by different operators, which confirmed that reliable data can be obtained using small 4-mm diameter parallel plates (*7*).

BBR m-value and creep stiffness, $S(t)$, are estimated through a correlation with 4-mm DSR developed by Sui et al. (*8*). In the Sui et al. method, the slope and magnitude of the shear stress relaxation modulus, $G(t)$, master curve at 2 h and at the true low PG grading temperature are correlated with the corresponding $S(t)$ and *m*-values at 60 s and 10°C above the true low PG grading temperature from BBR measurements. The Sui et al. method was modified by measuring $G(t)$ slope and magnitude at 60 s and at 10°C warmer than the PG grading temperature.

Given the dramatic increase in continuous grade temperature (i.e., *G**/sin δ) shown in Figure 14, the decrease (or warming) of the continuous grade temperature in terms of *m*-value
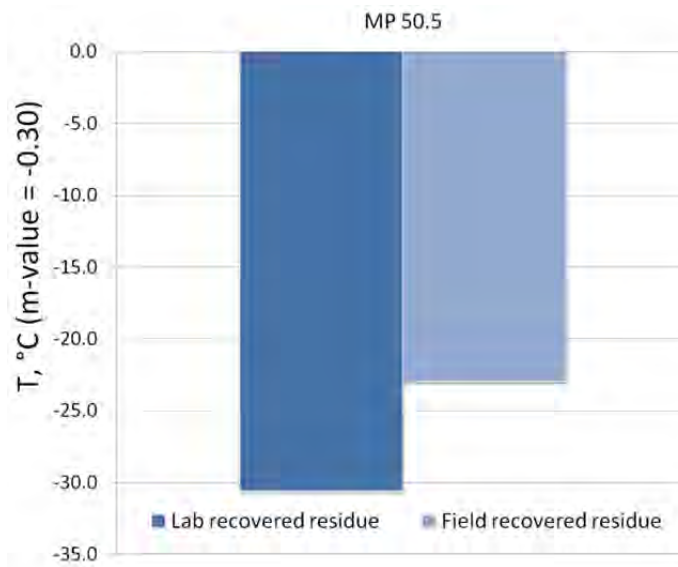
FIGURE 18 *m*-value = –0.30 continuous low-temperature grade.
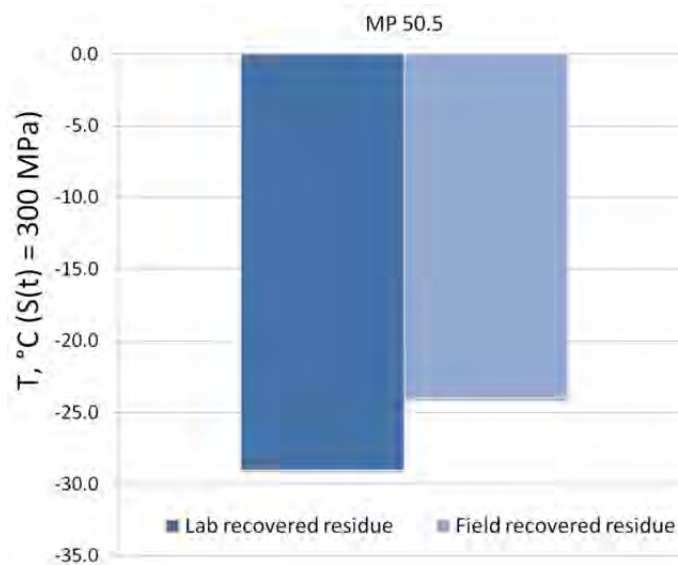
FIGURE 19 Creep stiffness = 300 MPa continuous low-temperature grade.

and creep stiffness seems to be minimal; however, it should be noted that the difference in G^*/sin δ continuous grade temperature between laboratory- and field-recovered emulsion residue is based on unaged laboratory residue, whereas the comparison for *m*-value and creep stiffness is between PAV-aged laboratory- and field-recovered residue. These results again confirm the somewhat limited field performance prediction power of the PAV, shown earlier through strain measurements.

CONCLUSIONS

- In the initial stages of this study, there was some concern about being able to collect field samples of emulsion residue from a chip seal, particularly several years after placement; however, it turned out to be relatively easy, and we demonstrate several ways to accomplish it. A cordless hammer drill (in nonrotational mode) with a chisel-type bit provides a convenient and simple collection method.
- To evaluate if there was an oxidative aging gradient within the emulsion residue, we developed an IR solvent wash method. The extent of oxidation at the surface compared to the bottom of the emulsion layer was determined by comparing the height and broadness of the carbonyl and sulfoxide absorbance bands. The solvent wash method also assessed the extent of polymer degradation at the surface and bottom. An aging gradient was found in the Death Valley residue, which was not large; however, there appeared to be a significant gradient in terms of polymer degradation.
- Extraction and recovery of SBR latex-modified emulsion residue using DCM and a maximum temperature of 80°C disrupts the polymer structure (honeycomb), but the rheology was unchanged. Since this is a somewhat unexpected finding based on limited data, additional testing and analysis are recommended.
- The IR indicated considerable oxidative aging had occurred in the field emulsion residue samples, and rheology reinforced that conclusion in terms of an increase equivalent to six PGs for $G^*/\sin \delta$ and a decrease in J_{nr} of well over an order of magnitude.
- The PRS may not be appropriate for estimating the presence of polymer in the field emulsion residue because of the large increase in PRS related to oxidative aging.
- The 4-mm DSR was successfully used to evaluate the low-temperature properties of the field emulsion residue in terms of BBR m-value and creep stiffness. The 4-mm DSR test only required about 150 mg of recovered residue. BBR testing is not appropriate for testing field emulsion residue because of the large amount of binder required (>60 g).
- PAV does not capture the whole field aging, since the field-extracted binder featured stiffer characteristics than the PAV-aged binder.

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Techniques for Accelerating Recovery of Asphalt Emulsion Residues at 60°C by Using Thin Film Procedures and Test Methods Suited to the Limited Amount of Residue Obtained to Characterize the Recovered Binder

GERALD REINKE
MARY RYAN
STEVE ENGBER
DOUG HERLITZKA
MTE Services, Inc.

Typical procedures to recover residues from asphalt emulsions require a distillation to 260°C $\pm$ 5°C (500°F) followed by a 15-min hold at that temperature (ASTM D6997). This procedure has served the emulsion industry well for many years, but the advent of PME more than 20 years ago lead to modifications of the standard distillation procedure. The realization that some polymers are degraded by the high-temperature distillation procedure and the subsequent loss of the elastomeric properties for which the emulsions were originally produced has resulted in the development over the years of several alternate procedures to obtain the asphalt emulsion residues in an ongoing effort to obtain residues that more closely match those of the base asphalt and especially do not result in the reduction of the elastomeric properties of the emulsion residue. The information reported in this paper utilizes a variant on existing procedures by heating a thin emulsion film at 60°C (140°F) but employing a vacuum (88- to 114-mm Hg pressure, 12 to 15 kPa) for 3 h to obtain the residue. Comparison testing of residues so obtained to base asphalt properties is disclosed showing generally little degradation or stiffening of residues compared to the base binder. Further testing using a newly developed 4-mm DSR test procedure (*1*) enables determination of the low-temperature PG grade of the emulsion residue without the need of obtaining the quantity of binder needed to perform a conventional BBR test on the residue. Also reported in this paper is a novel emulsion residue recovery procedure using a DSR to break, cure and test the emulsion residue stiffness and elasticity (where appropriate) in a single continuous process.

ABSTRACT UPDATE: JULY 2013

Subsequent to TRB Session 837, papers for individual presentations were requested. In the ensuing interval a substantial amount of additional work was conducted by MTE using the techniques presented in the original discussion. Incorporated into this report is a discussion of that additional work and therefore this document is up to date (as of July 2013) report on the vacuum recovery procedure and the test results of samples obtained with that procedure. The vacuum procedure has evolved to a level of 5- to 10-mm Hg (0.67 to 1.33 kPa) pressure and times reduced to 2 h. Several high-float emulsions have been evaluated specifically to determine whether or not high-float emulsion residues obtained at 60°C under vacuum will yield a material that will pass the float test. All additional work discussed in this paper utilized a thin film of

0.381 mm rather than the 0.318 mm originally employed. The material added since the presentation at Session 837 begins on page 13.

INTRODUCTION

Typical procedures to recover residues from asphalt emulsions require a distillation to 260°C $\pm$ 5°C (500°F) followed by a 15-min hold at that temperature (ASTM D6997). This procedure has served the emulsion industry well for many years, but the advent of PMEs more than 20 years ago lead to modifications of the standard distillation procedure. The realization that some polymers are degraded by the high-temperature distillation procedure and the subsequent loss of the elastomeric properties for which the emulsions were originally produced lead to using the residue from the oven evaporation procedure (ASTM D 6934) to obtain a less stressed residue because the emulsion is held at 163°C (325°F). Due to the length of time the residue is held at this temperature in an open beaker there is considerable opportunity for oxidative aging of the emulsion residue. Another procedure, the low-temperature vacuum distillation residue recovery was introduced (ASTM D7403) which required freezing the emulsion and then introducing the frozen sample into an emulsion still, pulling vacuum on the still to 88 kPa below atmospheric pressure ($\approx$ 13 kPa absolute based on atmospheric pressure = 1 atm or 101.3 kPa), heating the contents under this vacuum to 135°C $\pm$ 5°C (275°F) and holding for 15 min. Variants on these reduced temperature approaches have been stipulated within the United States by different agencies and have generally taken the form of following the normal (ASTM D 6997) distillation procedure but reducing the upper temperature to some lower value such as 204°C (400°F). In 2011 AASHTO approved PP72 (a provisional procedure) encompassing two procedures for residue recovery. Procedure A requires emulsion applied to silicone sheets at a rate equivalent to either 1.5 or 2.0 kg/m^2. The emulsion is cured for 24 $\pm$ 1 h in a forced draft oven at 25°C (77°F) followed by 24 $\pm$ 1 h at 60°C (140°F). Depending on the silicone rubber mat size quantities of emulsion as great as 125 g can be used. Procedure B utilizes a much smaller amount of emulsion and requires a wet film of 0.381 mm (0.015 in) applied to a silicone rubber sheet followed by a curing period of 6 h at 60°C (140°F). Within the last 2 years MTE has experimented with alternate procedures to obtain emulsion residues and test them to obtain the full spectrum of residue stiffness from very low [–35°C (–31°F)] to in service pavement temperatures. Additionally the MSCR test has been utilized to obtain elastomeric recovery values at a range of temperatures to demonstrate that a very modest amount of emulsion residue is sufficient to fully characterize these materials.

TESTING METHODS EVALUATED

DSR Thin Film Rheology Method

Beginning in early 2010 MTE began exploring procedures for obtaining emulsion residues at temperatures in the range of pavement surface temperatures, approximately 60°C. Having explored the proposed AASHTO procedure that became PP72 Procedure A, the conclusion was reached that the method required too much time and therefore would not be suitable to a production environment. Having also explored the procedure that became PP72 Procedure B it

appeared as though the 6 h of curing at 60°C resulted in recovered residue properties that were substantially stiffer than the based binder from which the emulsion had been produced. The approach developed by MTE was presented at the FHWA ETF meeting in Boston on July 25–27, 2010 (*2*). The procedure utilized a DSR with an environmental chamber, a cup, and plate geometry (Figures 1 and 2). The cup is 38 mm in diameter and the side walls are 5 mm high. The gap is zeroed at 58°C and 2 to 2.5 ml of emulsion is added to the cup. The gap is closed to 500 µm and a steady shear test at a shear rate of 50 s^{-1} is executed for 60 min. The gap is closed to 400 µm and another steady shear test at 50 s^{-1} for 120 min is executed. The purpose of the two steady shear steps is to break the emulsion by shearing the material within the thin gap. After these two steps the gap is closed to 250 µm and a DSR frequency sweep is performed from 5 to 100 rad/s (10 points/log decade) at 10% strain at 58°C. At the end of this frequency sweep step a MSCR test is conducted at stress levels of 0.050, 0.1, 1.0, 3.2, and 10 kPa. The sample is then conditioned for 30 min at either 52°C or 64°C depending on the type of emulsion being tested[1]. The frequency sweep and MSCR tests are repeated at the second temperature. In the work conducted at MTE the samples were held in the cup at 58°C for an additional 4 h and then the test sequences were repeated. The purpose of the 4-h hold and retest was to ascertain based on the repeated tests that the emulsion had been dehydrated to the point where a repeatable result had been obtained. A few example results will serve to show the utility of this testing approach. Figure 3 shows the results of testing an AMRL CSS-1h emulsion sample at 58°C and 64°C after the initial 4-h conditioning time followed by the 4-h additional waiting time and retesting.

Examination of Figure 3 shows that there is very minor variation between the two test temperatures after the additional 4-h conditioning. This result and similar results for other samples demonstrates that after 4 h the emulsion was dehydrated sufficiently to yield a
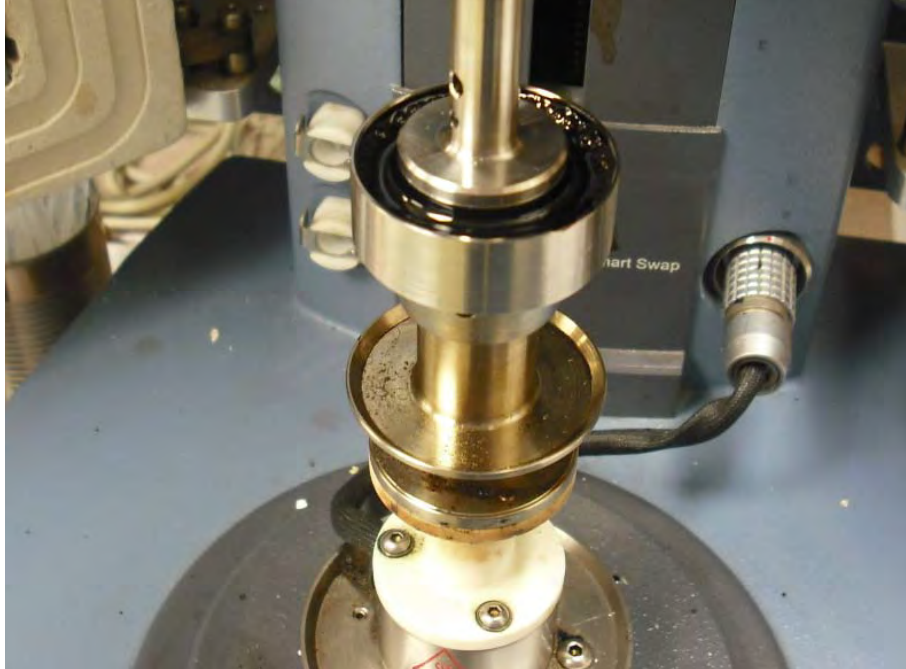

FIGURE 1 Cup and plate geometry for emulsion test.

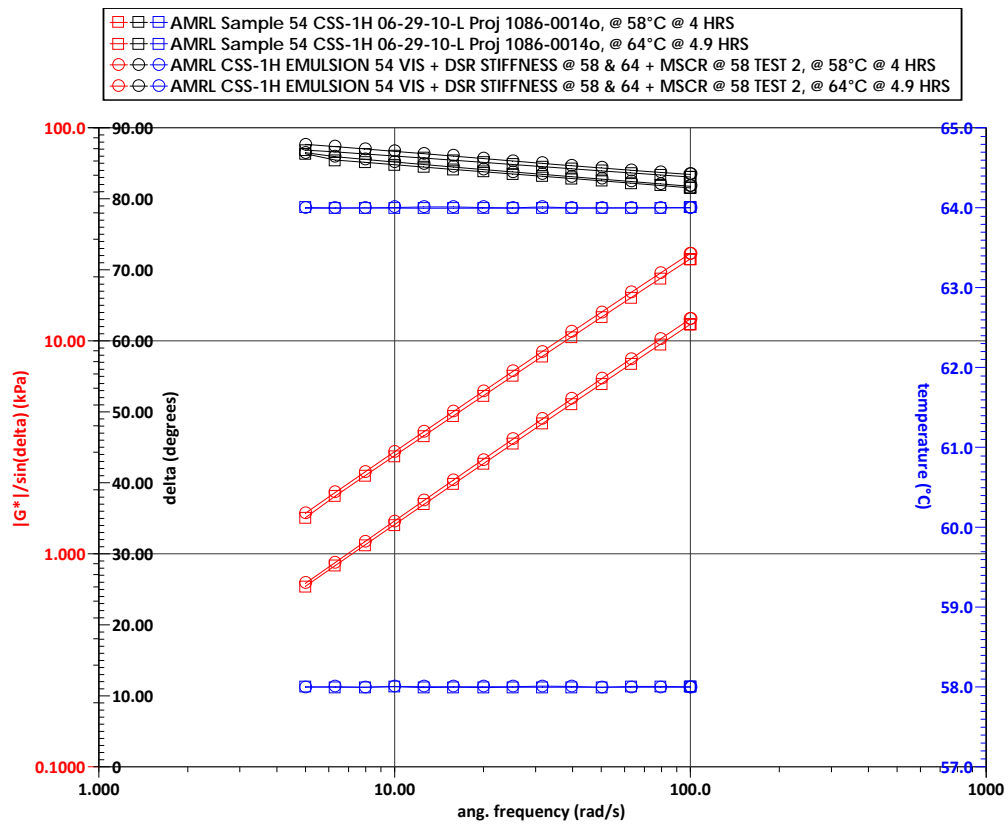**FIGURE 2 Cup and plate assembly with emulsion.**

FIGURE 3 AMRL CSS-1h emulsion test using DSR thin film method.

stable result. Subsequent testing of this same emulsion sample showed that after only 3 h of conditioning the DSR results at 58°C matched the results after the 4 h conditioning test. Figure 4 is a DSR thin film conditioning and test of a CRS-2P emulsion produced from a PG 64-34 PMA base binder. Initial conditioning at 4 .2 and 4.5 h for the two test temperatures followed by additional conditioning and retesting of the rheological properties shows virtually no change in the residue. Additionally the base binder was tested at both 58°C and 64°C to demonstrate that the DSR thin film procedure did not alter the base binder properties.

The DSR thin film emulsion testing demonstrated that shearing emulsion in a narrow gap is suitable to break the emulsion and when performed at a temperature approximating pavement surface temperatures the residue is dehydrated to yield stiffness properties comparable to the base binder. The other advantage to this procedure is that the residue is produced in the actual test equipment and therefore once the conditioning procedure is completed the residue stiffness can be obtained directly. Additionally the ability to determine the residue stiffness and also perform the MSCR test yields a measure of the residue elasticity as well. Unfortunately the strengths of using this procedure are also one of its weaknesses. Using a costly piece of equipment to condition the emulsion residue and monopolizing the DSR for several hours in so doing is not a cost-effective use of the equipment. The work described in the data presented in MTE's report did serve to demonstrate that a thin film shearing procedure, which in many respects mimics the interaction of aggregate particles being mixed with emulsion, is sufficient to break and cure the emulsion. Therefore even though the work MTE conducted showed promise it was abandoned in favor of a more economical approach.
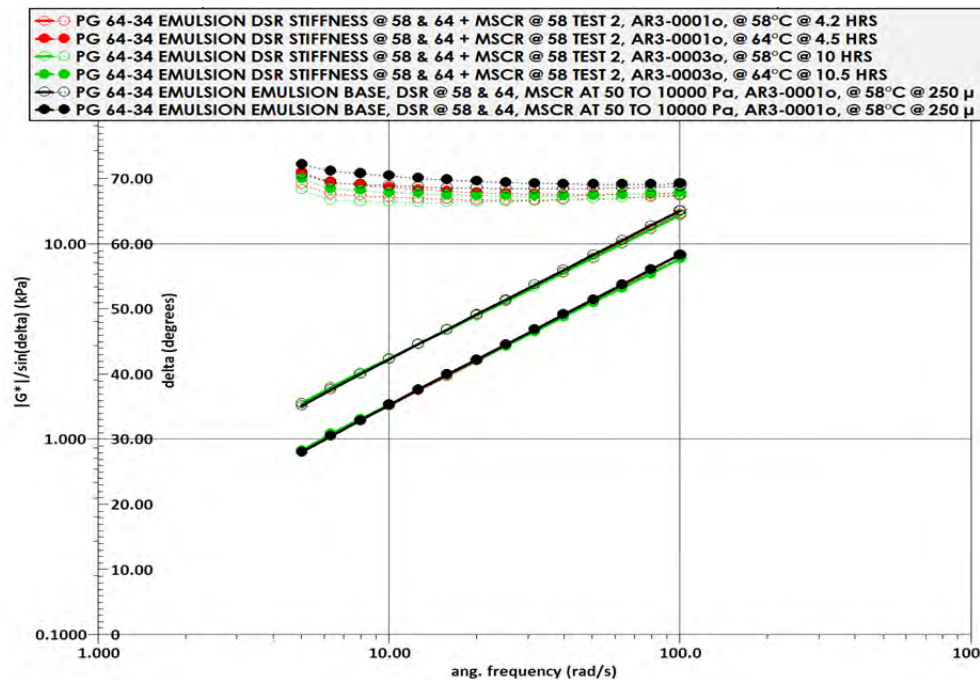

**FIGURE 4 CRS-2P emulsion, PG 64-34 base DSR thin
film emulsion residue and base binder test.**

COMPARISON OF 6-h 12.5-mil (318-µm) AMBIENT PRESSURE OVEN EVAPORATION TO OTHER APPROACHES

Procedure B of AASHTO PP72 requires curing a thin film of emulsion for 6 h at 60°C prior to testing. MTE evaluated this residue recovery procedure in comparison to other methods.

Figure 5 shows a comparison of the DSR stiffness at 58°C of PG 58-28 base binder and the stiffness values of the residue of a CRS-2 emulsion obtained according to two curing methods. One method was the DSR thin film recovery procedure discussed above and the other was a 5.5-h, 60°C procedure following PP72. The residue was only cured for 5.5 h rather than 6 h due to operator error, but as Figure 5 makes clear the reduction in cure time still resulted in a DSR stiffness for the 5.5-h residue that was 1 kPa greater than the base binder and 0.9 kPa greater than the DSR thin film residue. All DSR stiffness values were obtained at a gap thickness of 250 µm to match the gap thickness of residue obtained by the DSR thin film procedure.

The data shown in Figure 6 examines a polymer modified microsurfacing emulsion where the residue is obtained by conventional distillation, a 6-h, 60°C thin film curing procedure according to PP72 Procedure B, a 3-h, 60°C thin film curing procedure and duplicate runs of the DSR thin film residue recovery procedure; the base binder was not available for testing. The $G^*/\sin(\delta)$ stiffness values at 10 radians/s are shown on the plot. It appears that the high-temperature distillation degraded the polymer-modified base since the distilled residue has the
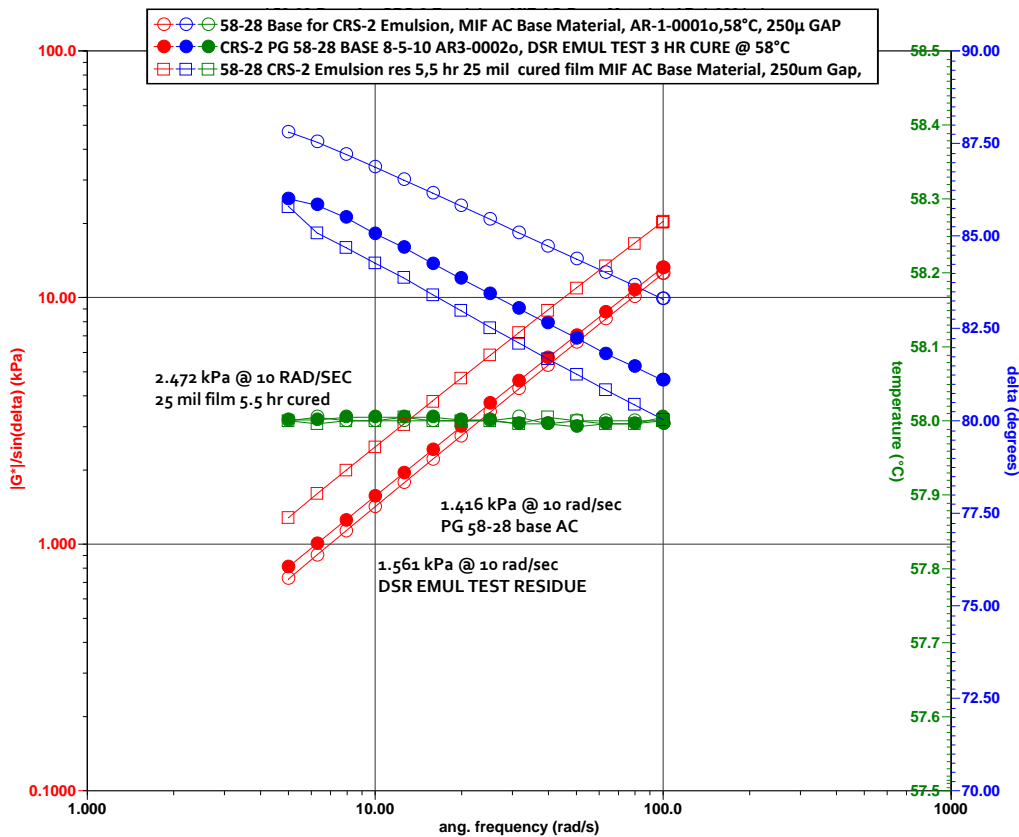

FIGURE 5 Comparison of DSR stiffness of base binder, DSR thin film residue and residue from 12.5 mil (318 µm) emulsion film cured at 60°C for 5.5 h.
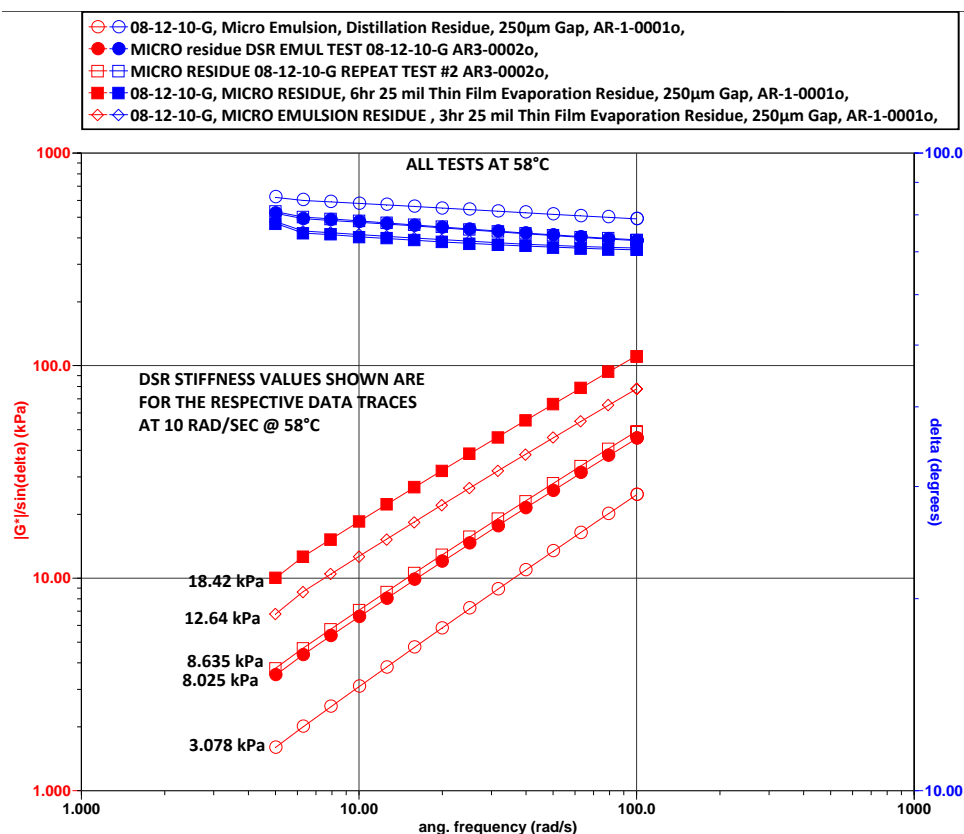
FIGURE 6 DSR stiffness properties of microsurfacing emulsion residue obtained by conventional distillation, 6- and 3-h, 12.5-mil (317-µm) 60°C residue recovery method and duplicate tests of DSR thin film residue recovery of 3 h duration.

lowest stiffness. The DSR thin film residues showed good repeatability for the procedure. If, as previous data in this report shows, the DSR thin film residue recovery methodology is a reasonable predictor of base asphalt stiffness; then both the 3- and 6-h, 60°C recoveries result in a 50% and 100% increase, respectively, in stiffness over the base binder. From the data shown in Figures 5 and 6 it appears as though the 12.5-mil (317-µm) thin film of emulsion cured for even 3 h at 60°C in a forced draft oven results in a substantial amount of residue stiffening. The data from Figure 5 shows that the DSR thin film residue is 10% stiffer than the base binder while the 5.5-h, 60°C, 12.5-mil residue is 75% stiffer than the base asphalt. If a residue recovery methodology is desired such that the residue properties will approximate the actual binder being used on the road, then an alternate approach is needed.

VACUUM OVEN EVAPORATION FOR 3 h OF 12.5-mil (317-µm) WET FILM AND TESTING

Two factors were considered when searching for an alternative to the 60°C forced draft oven residue recovery procedure. One factor of course was the obvious fact that a very thin film of asphalt binder subjected to even a moderate temperature of 60°C and abundant air flow is going

to readily oxidize. The other factor that is inherent in evaluating cationic emulsion residues is the presence of high quantities of hydrochloric acid. For some emulsions such as microsurfacing emulsions the amount of hydrochloric acid can equal or exceed the amount of emulsifier on a weight basis. It is well established that a strong protic acid can have a stiffening effect on asphaltic materials and this stiffening is compounded in the thin film curing procedure and the abundant amount of air available. During the DSR thin film residue recovery procedure, while conducted at 58°C, the majority of the asphalt sample that is ultimately tested is occluded from oxygen because the sample is trapped between two steel surfaces in a thin gap of 400 µm which is further squeezed to 250 µm prior to testing. As already stated the DSR thin film procedure is impractical for several reasons, but this impracticality does not alter the fact that the procedure does a good job of producing emulsion residues that closely match the base binder. Using that fact as a guide the next approach that suggested itself was to use the 12.5 mil (317 µm), 60°C thin film residue recovery procedure but to perform the conditioning in a vacuum oven. The procedure adopted is outlined in Figures 7 through 10.

The drawdown bar according to the manufacturer produces a wet film approximately ½ the cut thickness indicated on the bar. Therefore the 25-mil cut produces a wet film of nominally 12.5 mil (317 µm). This tool produces a sample of approximately 90 mm in width and for the testing performed a silicone rubber sheet of 3-mm nominal thickness and 280 to 300 mm in length was used.[2] Any convenient width can be used; the sheets shown in Figure 8 are 200 mm wide. To facilitate sample handling the silicone sheets were placed on flooring tiles that would fit in the vacuum oven. A Fisher Isotemp Model 282A vacuum oven was employed although any suitably sized vacuum oven will suffice. The vacuum oven was held at 3.5 to 4.5 in. of Hg (≈11 to 15 kPa) absolute for 3 h. For the Fisher Isotemp oven three samples at a time can be dehydrated.
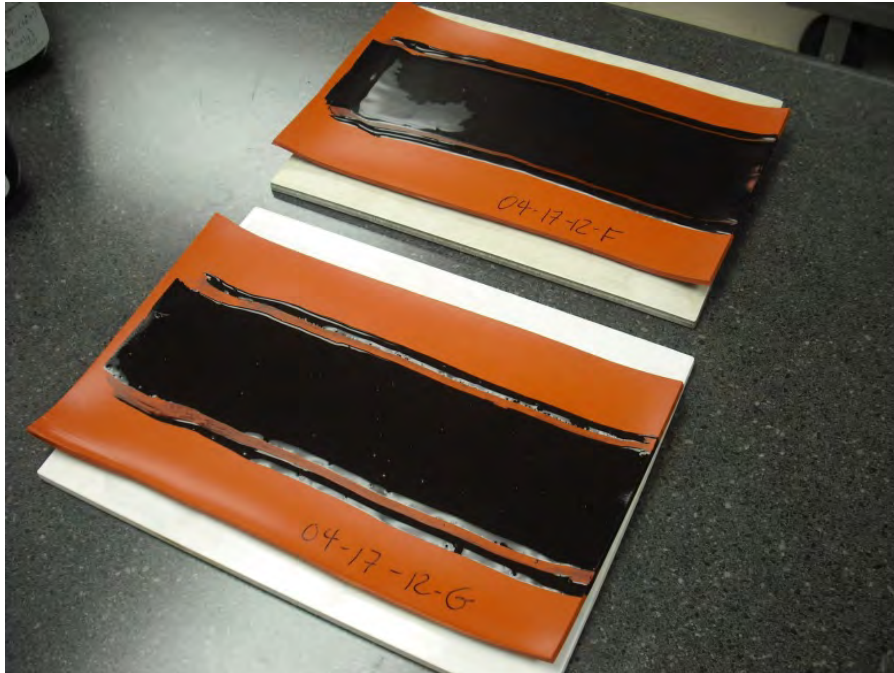

FIGURE 7 Thin film drawdown bar.

FIGURE 8 Emulsion samples at 25 mil.

FIGURE 9 Sample in vacuum oven.

FIGURE 10 4 mm plate–plate fixture.

To evaluate the vacuum oven procedure compared to the 6-h, 60°C thin film ambient pressure curing procedure a study was undertaken to obtain the residues from four emulsions produced at different emulsifier levels and consequently differing amounts of hydrochloric acid. The results of this testing are shown in Figure 11.

The base asphalt for this study was a polymer-modified PG 64-28 and EM-1 through EM-4 had decreasing levels of hydrochloric acid. The G^*/sin (δ) data for the base asphalt was not obtained after blank conditioning and therefore the base asphalt data is the stiffness of the asphalt at 58°C. The data in the plot shows that the 6-h ambient pressure residues are stiffer than the 3-h vacuum residues and a general trend for both residue recovery procedures that more hydrochloric acid results in a stiffer residue. It is clear that the amount of acid impacts the stiffness of the residue regardless of the curing procedure employed. However, when comparing the properties of EM-4 residue, which had a low level of acid the stiffness increase for the vacuum residue was 7.7% while the increase for the 6-h ambient pressure residue was nearly 40%. That difference in stiffness is due solely to the difference in curing time and the presence of oxygen in the residue recovery environment.

Only about 4 to 5 g of residue is obtained from one sheet of emulsion cured in the vacuum oven procedure. This is sufficient to run a DSR test at several temperatures and also to perform a MSCR test at a single temperature from a single sample. To obtain low-temperature properties of the emulsion residue from these residue recovery procedures requires the utilization of new test method that was introduced by WRI in 2010 at the 89th Annual Meeting of the Transportation Research Board. Sui (*1*) and Farrar (*3*) have pioneered this work. Approximately 0.2 g of binder is required to perform a test that will determine the rheological properties of the emulsion residue from –40°C to as high as 60°C. From these data is possible to determine the complex shear modulus mastercurve at any temperature in this range and it is possible to also determine the low-temperature performance grade of the emulsion residue. MTE has also
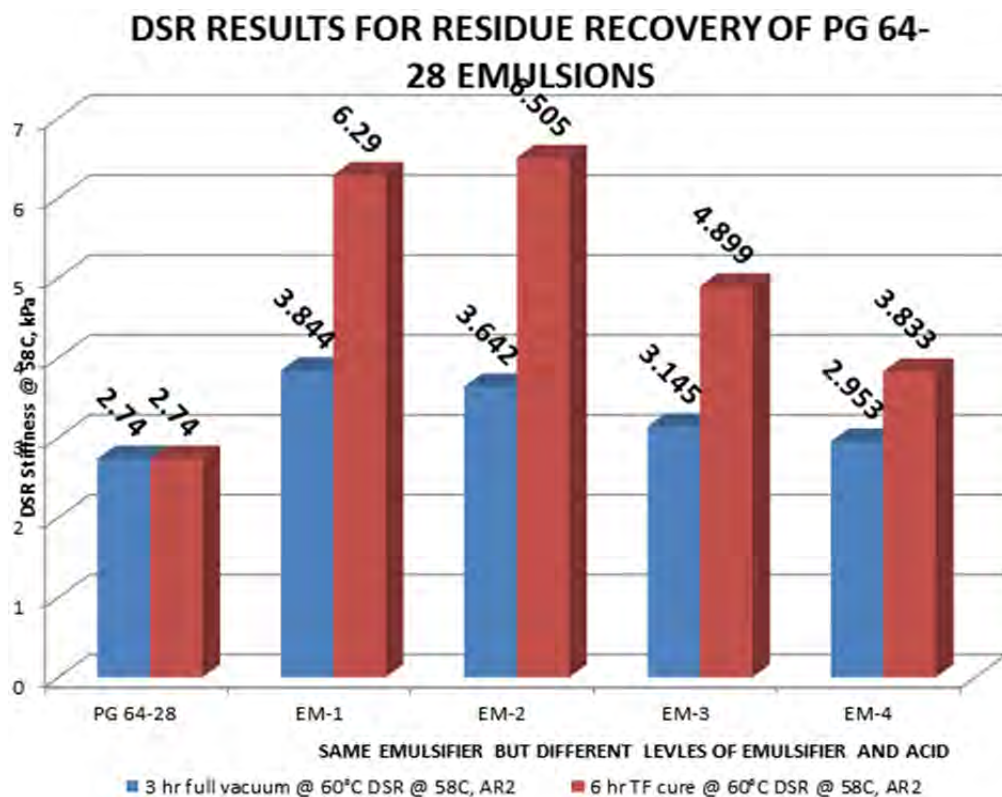
FIGURE 11 *G*/*sin (δ) for four emulsions at different hydrochloric levels

extended work on using the DSR to determine the glass transition temperature (*Tg*) of binders using binder torsion bars presented at AAPT in 2001 (*4*) to utilize the 4-mm DSR geometry to determine the glass transition temperature of emulsion residues obtained using the vacuum recovery procedure.

Using the 4-mm DSR fixture to determine *Tg* of the binder is a relatively simple procedure. The sample is loaded onto the plates at 30°C to 40°C depending on the anticipated stiffness of the residue (emulsions produced from highly modified binders or stiff PG grades would require the warmer temperature). The sample is trimmed at 0°C or +10°C, again depending on the anticipated binder stiffness and the gap is set to 2 mm. It is important when trimming the sample to lock the upper tool in place. There is so little resistance between the 4-mm diameter plates that trimming at low temperatures without locking the upper tool can result in pulling material out from between the plates or fracturing the sample. The test consists of a temperature ramp of 0.3°C/min from the starting temperature to –30°C or –40°C, again depending on the anticipated low-temperature grade of the binder. Because the emulsion residue has not been aged either through the RTFO or the PAV, the low-temperature grade of the residue will be about 6°C lower than the designated low PG grade of the base binder. While the temperature is ramping down data is taken at a sampling interval of 10 s, using 0.05% strain and a frequency of 0.05 radians/s. The parameter that is monitored is the loss modulus of the binder. A typical data trace for a *Tg* test is shown in Figure 12. The peak in the loss modulus data curve is a good approximation of the binder glass transition temperature. To obtain the most accurate result a low frequency is required, but one must balance the time to perform the test against minor refinements in accuracy as the frequency is decreased. There are several ways to assess the

temperature at which the G″ value reaches a peak. Simply "eyeballing" the data will give you a reasonable value, however a more rigorous approach is to fit a fourth-order polynomial to the log G″ data as a function of temperature, determine the first derivative and then find the exact value where the first derivative equals zero. For some tests the data in the region of the peak can be noisy and using the more analytical approach takes subjectivity out of the determination of the *Tg* value.

Data generated by the 4-mm DSR test can also be used as an alternative to the BBR test. The details of the analysis are provided in Sui (*5*) and a typical test result and analysis will be provided here. At this point in time the 4-mm DSR test and analysis procedure has not been adopted by ASTM or AASHTO, but based on work at MTE the test method and the analysis procedures provide results that match classical BBR results to within 2°C or closer. That level of agreement does not currently meet the repeatability between test methods that one would expect to designate the two procedures as equivalent. However, given the significance of being able to obtain emulsion residues at temperatures approximating real world curing conditions the ability to generate low-temperature failure criteria to within 2°C of the BBR result is important.

Figure 13 shows the temperature frequency sweep data for the residue recovered from an emulsion produced from a PG 76-22 using the vacuum procedure at 60°C for a 3-h period. The analysis procedure requires determining the relaxation modulus at two temperatures, one below the failure stiffness of the binder and one above the failure stiffness of the binder. This is similar to the requirement to determine the precise low-temperature PG grade using the BBR. RHEA software developed by Abatech (*6*) is used to obtain the relaxation modulus from the complex modulus data. Figure 14 shows the relaxation modulus plot at –30°C. The RHEA software allows determination of the relaxation modulus at 60 s as well as the slope of the relaxation modulus plot at 60 s. This process is analogous to the calculations performed in analyzing the
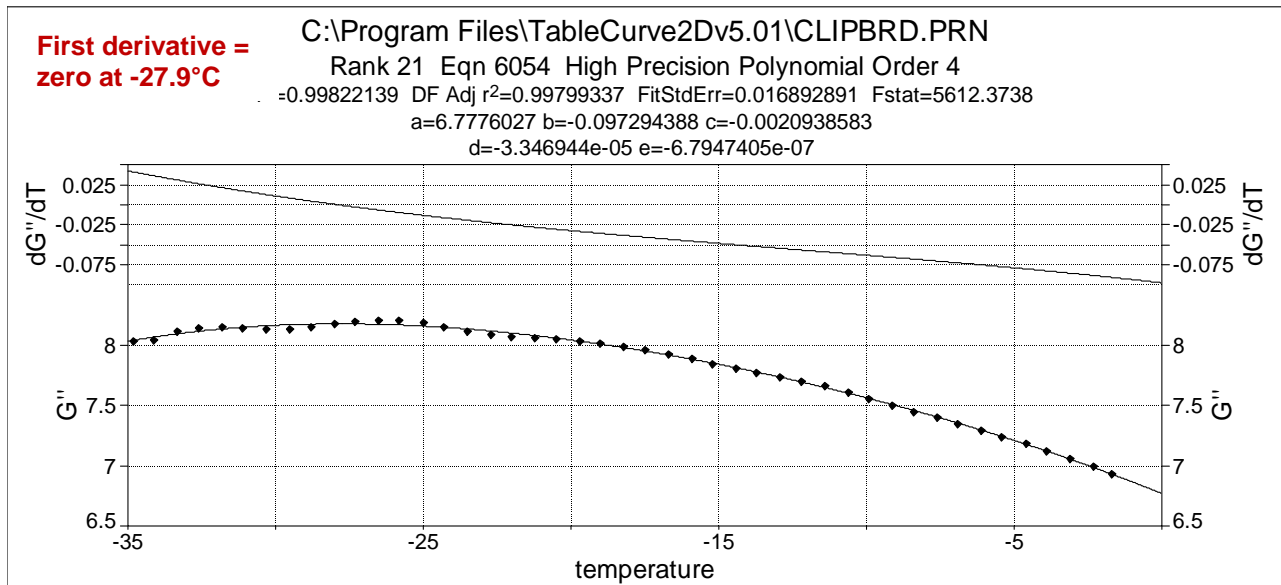
FIGURE 12 *Tg* data trace and determination of *Tg* value (the *R*, *R*², and standard error values do not reflect the actual precision of the data; the data plotted here are meant to illustrate the procedure for determining the *Tg* value based on G″ data).

BBR data where the $S(t)$ value is determined at 60 s and the slope of the creep stiffness curve is determined at 60 s.

The relaxation modulus and the slope of the relaxation modulus are determined at 60 s mastercurve at temperatures bracketing the failure criteria. In the case of the data for this sample those temperatures are –30°C and –20°C. In a manner similar to the BBR calculation the critical temperatures are determined based on the target values as reported by WRI of $G(t) = 143$ MPa and slope equal to –0.280 and then 10°C is subtracted from those critical temperatures to obtain the predicted failure temperature. That analysis is shown in Table 1.
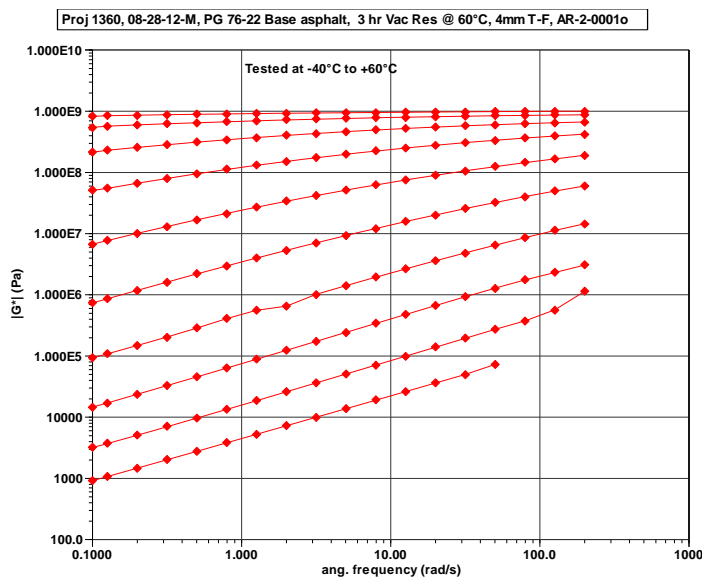
FIGURE 13 Temperature frequency data for 3-h vacuum residue of emulsion produced from a PG 76-22 base binder.
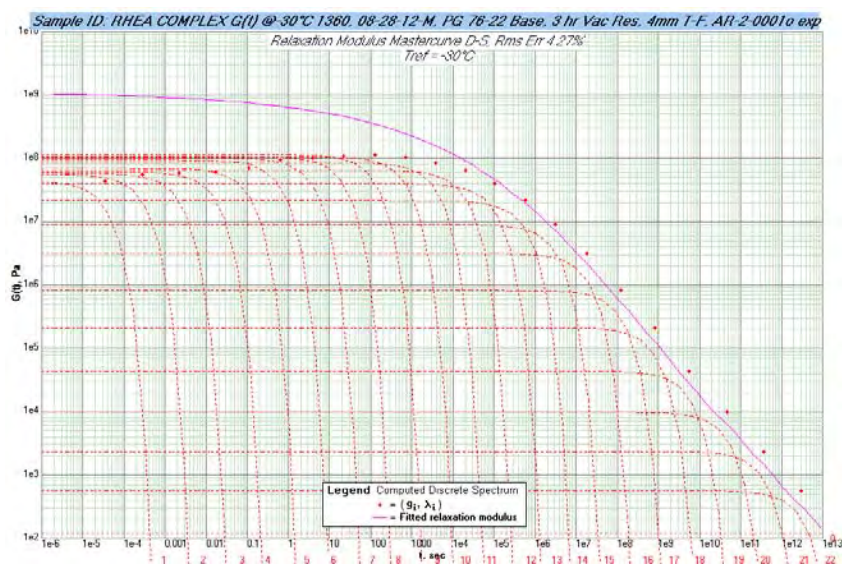
FIGURE 14 Relaxation modulus plot using Abatech RHEA software.

Data from RHEA can also be imported into Excel as a text file which then enables comparison plotting of the relaxation modulus mastercurves. Figure 15 is a plot showing both re laxation modulus plots on the same set of axes as well as a line indicating the 60-s value where the slope is determined.

TABLE 1 Critical Temperatures for 3-h Vacuum Recovered Residue for PG 76-22 Emulsion

Temp	$G(t)$, MPa	m, creep	log [$G(t)$]
–30	413.07	–0.161	2.62
–20	93.57	–0.3833	1.97
Target spec value	143	–0.28	2.16

Slope	Intercept	Critical Temperatures	Critical Temperature, –10°C	
–0.064	0.68	–22.9	–32.9	$G(t)$ grade
–0.022	–0.83	–23.7	–33.7	m, creep grade

FIGURE 15 Plot of relaxation modulus vs reduced time for –30°C and –20°C.

**TABLE 2 Critical Temperatures for 4-h Vacuum Recovered
Residue for PG 76-22 Emulsion**

Temp.	$G(t)$, MPa	m, creep	log [$G(t)$]
–30	465.85	–0.145	2.67
–20	164.29	–0.3023	2.22
Target spec. value	143	–0.28	2.16

Slope	Intercept	Critical Temp.	Critical Temp., –10°C	
–0.045	1.31	–18.7	–28.7	$G(t)$ grade
–0.0157	–0.62	–21.4	–31.4	m, creep grade

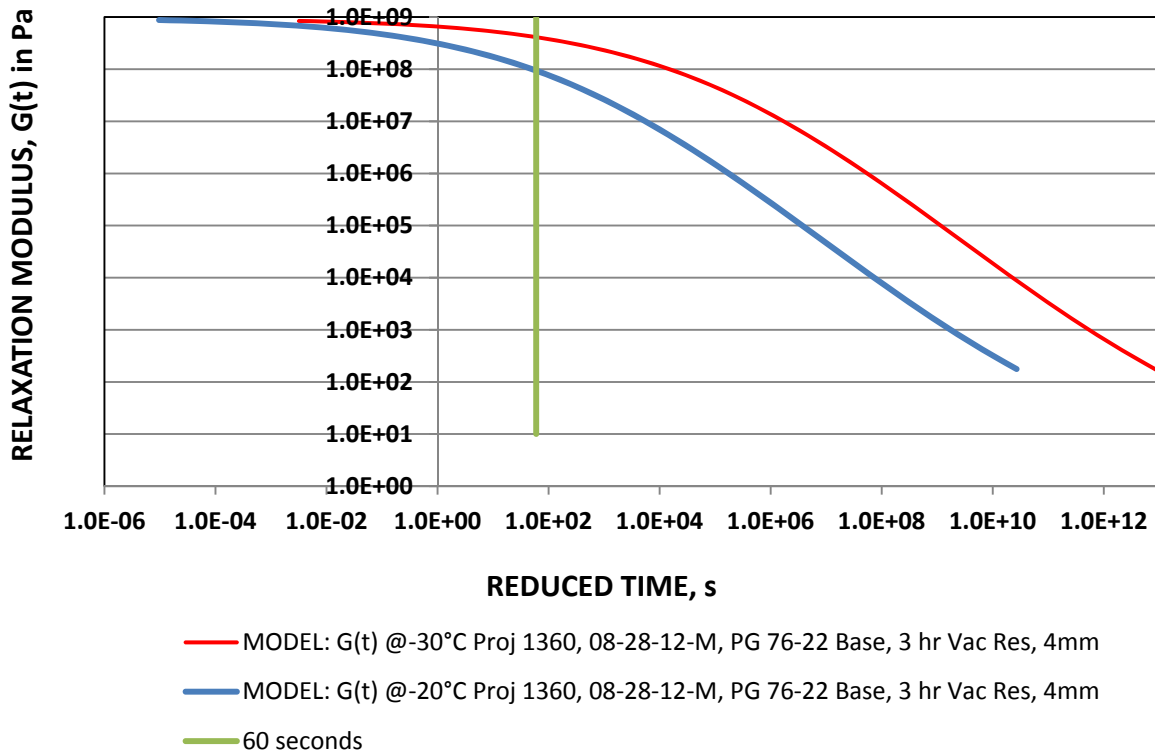A 4-h vacuum recovery was also performed for purposes of this paper to provide the impact of one additional hour under vacuum. Those results are shown in Table 2. Clearly the additional time has a substantial impact on the predicted low temperature critical values. It is also worth noting that the *Tg* value of –27.9°C shown in Figure 12 is for a test performed on the same 4-h vacuum cured residue. A determination of the glass transition temperature using the 4-mm DSR test will generally give a very good approximation of the limiting stiffness temperature for the recovered residue.

ADDITIONAL WORK AT HIGHER VACUUM LEVELS

Regardless of the thin film residue recovery methodology there appears to be a small amount of residual moisture left in the residue. This is true for the vacuum residue recovery procedure as well as the Procedure B of PP72 (the 6-h ambient pressure conditioning procedure). Residue testing comparing the vacuum residue recovered binders to the base binder shows that a residual moisture content of less than 0.8% produces rheological test results comparable to the base binder. This conclusion has not been rigorously investigated at MTE although it is the practice to determine the residual moisture of each sample gravimetrically by heating a sample of vacuum residue (approximately 0.6 to 1 g) at 150°C for 1 h and determining the loss. Higher levels of vacuum have been investigate to determine if the residual moisture content can be reduced, however the residual moisture is typically in the 0.2% to 0.7% range.

Figure 16 shows plots of complex modulus mastercurves for the residues of a recent AMRL emulsion proficiency sample (59A, SS-1h) obtained using a 60°C thin film vacuum procedure at a vacuum of 5 to 10 mm Hg for 2, 2.5, and 3 h; the PP72 6-h 60°C thin film evaporation procedure at ambient pressure, and the residue from a standard emulsion distillation to 226°C (500°F). All of the 60°C thin film residues had some residual moisture ranging from 0.31% to 0.62% but as the data in Figure 16 shows the five resulting mastercurves overlay nicely. The slightly lower stiffness modulus of the 2.5-h vacuum at high frequency is most likely testing variability for the –40°C isotherm of that sample.
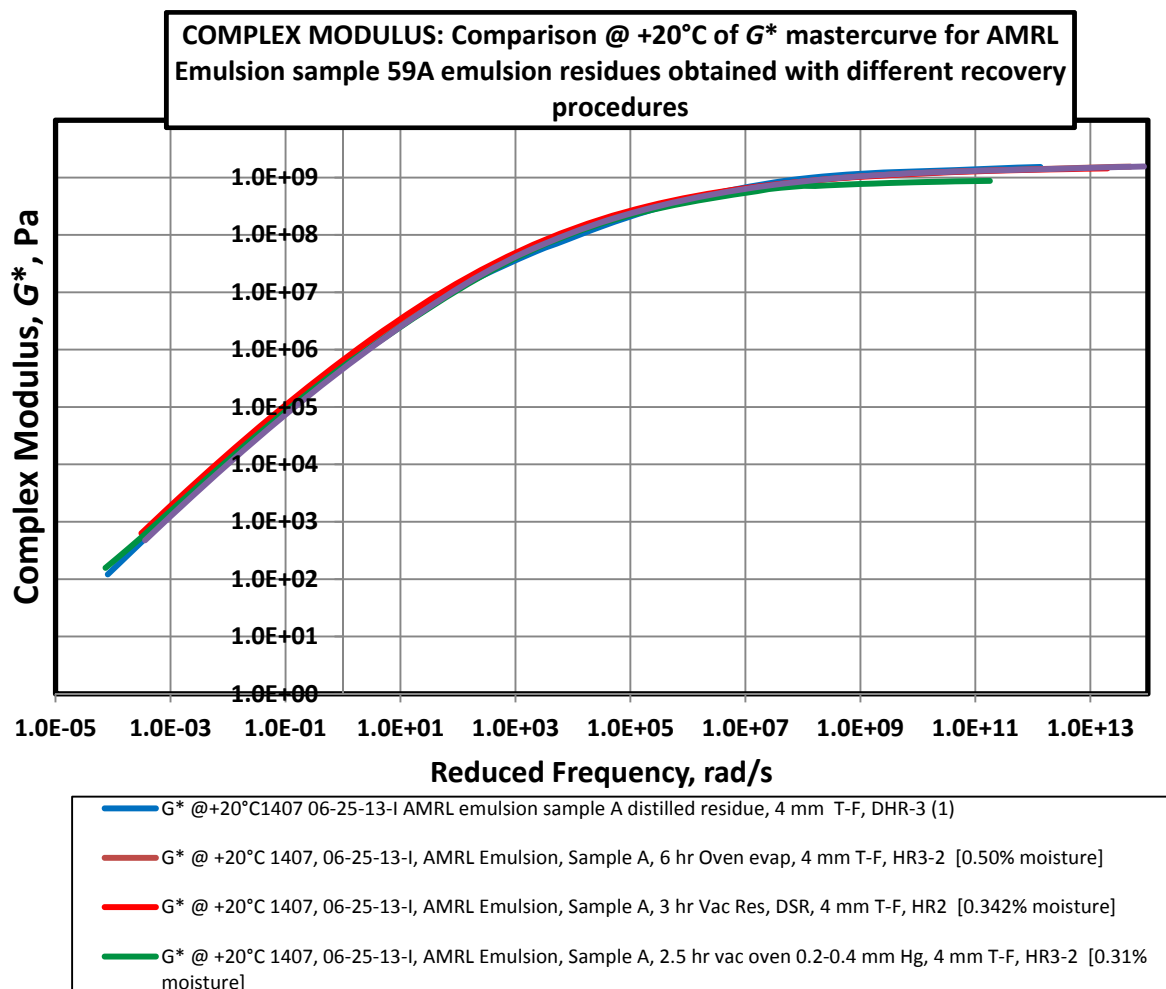
COMPLEX MODULUS: Comparison @ +20°C of *G** mastercurve for AMRL Emulsion sample 59A emulsion residues obtained with different recovery procedures

Legend:
- G* @+20°C1407 06-25-13-I AMRL emulsion sample A distilled residue, 4 mm T-F, DHR-3 (1)
- G* @ +20°C 1407, 06-25-13-I, AMRL Emulsion, Sample A, 6 hr Oven evap, 4 mm T-F, HR3-2 [0.50% moisture]
- G* @ +20°C 1407, 06-25-13-I, AMRL Emulsion, Sample A, 3 hr Vac Res, DSR, 4 mm T-F, HR2 [0.342% moisture]
- G* @ +20°C 1407, 06-25-13-I, AMRL Emulsion, Sample A, 2.5 hr vac oven 0.2-0.4 mm Hg, 4 mm T-F, HR3-2 [0.31% moisture]

FIGURE 16 Compare modulus of emulsion residues recovered by vacuum, ambient pressure 6-h evaporation and 500°F distillation.

The results shown in Figure 16 are for a nonmodified binder and therefore the results may not appear too surprising. The results shown in Figure 17 are for a highly modified PG 76-22 base binder and a CSS-1HP emulsion made from the PG 76-22. Vacuum recovery was performed at 2 and 3 h of conditioning time at 60°C and a vacuum level of 5 to 10 mm Hg. The mastercurve comparison plots at 20°C show very nearly identical results between all three samples with slightly higher modulus at the lower frequencies for the base binder. Table 3 summarizes the low-temperature stiffness and creep values for the three samples obtained from the 4-mm DSR data as discussed earlier in this report. The low-temperature grade of the emulsion residues is slightly better than the base binder; this could be the result of the emulsifier loading and the presence of acid used to react the emulsifier. There is research showing that the use of acid can improve the low temperature grade of some binders. The MSCR recovery data for the base binder and the 2-h cured vacuum residue are nearly identical.

A series of emulsions were provided from an emulsion producer. These included CRS-2 and CSS-1h and the base binders from which those emulsions were produced. Thin film vacuum recovery was performed on the emulsions for 2 h at a vacuum of 5 to 10 mm Hg (0.2 to 0.4 in. Hg)
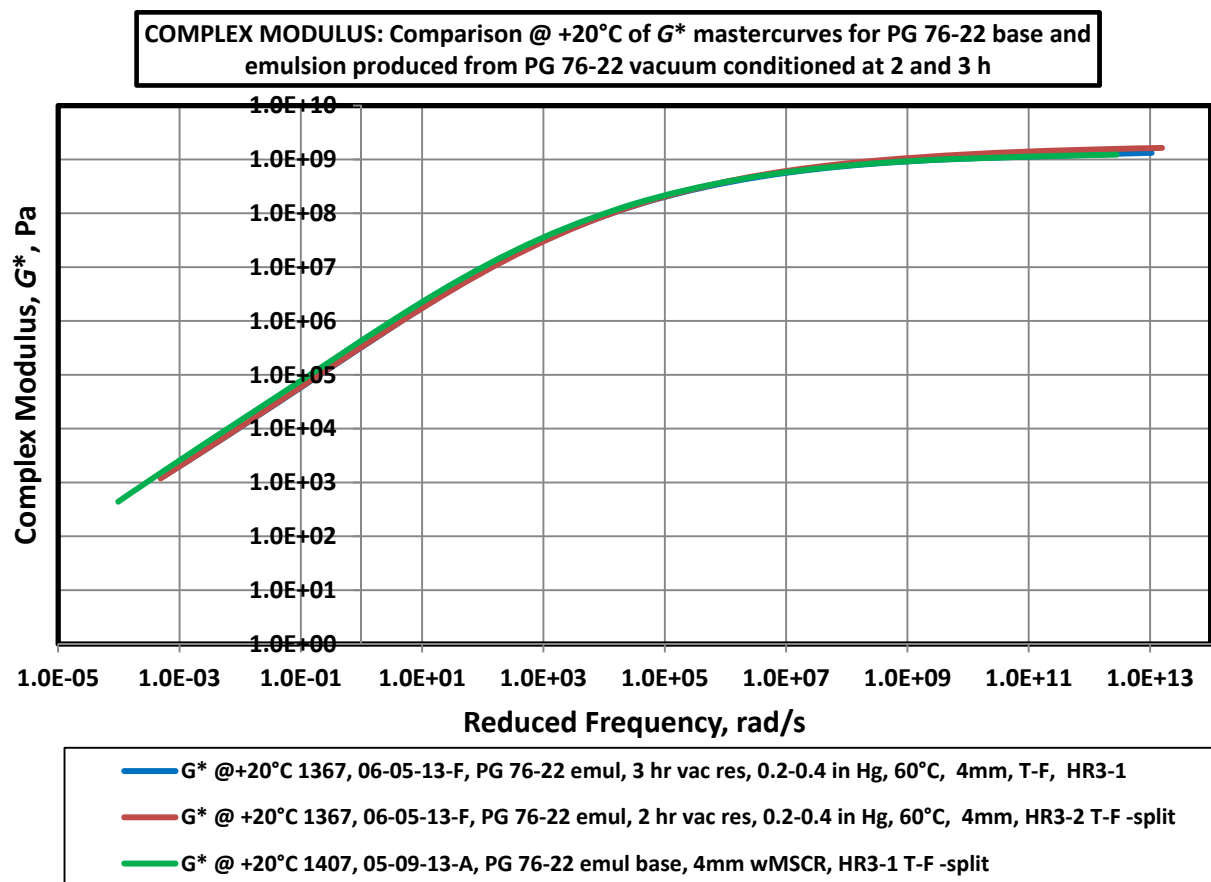
FIGURE 17 Comparison of complex modulus of PG 76-22 base binder and residue of emulsion obtained at 2 and 3 h of recovery at 60°C and 5 to 10 mm Hg vacuum.

TABLE 3 Low-Temperature Properties of 76-22 Base Binder and Emulsion Residue from 2- and 3-h Vacuum Recovery Procedure

Low Temperature Grade Results Based on 4-mm DSR			
Binder	$S(t)$ grade, °C	m grade, °C	MSCR % recovery at 3.2 kPa at 25°C
PG 76-22 base binder	−32.8	−32.9	77.4
PG 76-22 emulsion 2-h vacuum cured	−33.6	−35.4	76.5
PG 76-22 emulsion 3-h vacuum cured	−34.7	−35.5	No test performed

followed by 4-mm DSR testing of the residues. The base binders were also tested using the 4-mm DSR. The results of the rheological testing are shown in Figure 18 where the mastercurves of the base binders and their respective vacuum recovered residues are nearly identical.
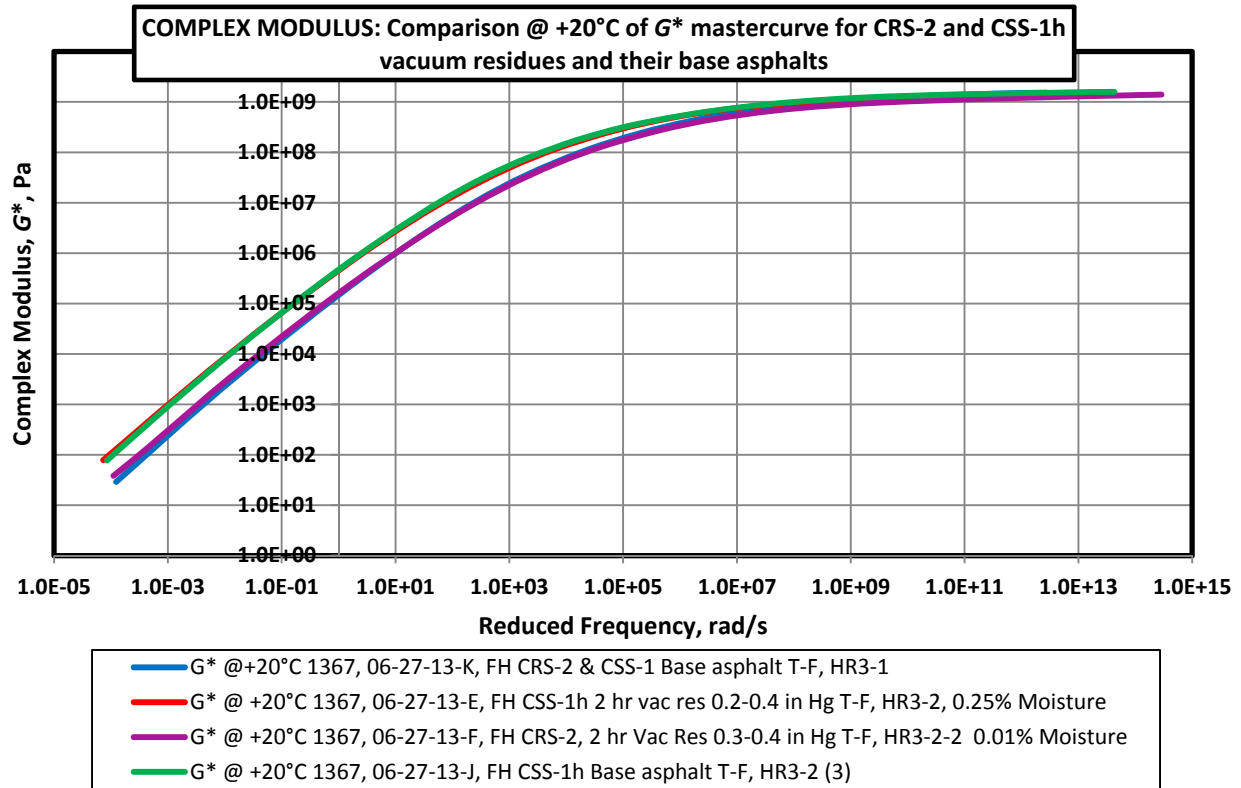
FIGURE 18 Comparison of modulus vacuum recovered residues of CRS-2 and CSS-1H and the base binders from which the emulsions were produced.

HIGH-FLOAT RESULTS

High-float emulsions were received from three suppliers. The grades varied; some were polymer modified, some were HFRS, some contained high levels of solvent. Table 3 summarizes the float test evaluations performed on these samples. For a wide variety of high-float emulsions the residues obtained using the thin film vacuum recovery procedure exhibited passing float results. It is certainly possible to obtain residues that pass float, but a substantial amount of the residue obtained from the vacuum recovery procedure is used to fill the float thimble. A valuable rheological investigation would be to determine a rheological test and a parameter that would be unique to the gel structure obtained in a high-float emulsion. To perform the float test the material removed from the silicone rubber sheet is put into a ½-oz. ointment tin. The tin is gently warmed on a hot plate and stirred using a glass capillary tube. As there is some foaming due to the presence of residual water it is advised to wait until most of this foaming has ceased. The float is poured and the test proceeds as normal. Any sample required for rheological testing is removed before the sample is warmed for the float test. The presence of low levels of residual moisture does not seem to affect the float test result, but it was noted that residual moisture in excess of 1% resulted in samples that either failed or were marginal on the test. With many high-float samples it is difficult to know how much of the gravimetrically determined residual moisture is actually solvent. For example the HFE-1000 residue showed 2.47% loss after vacuum conditioning, but it passed the float test handily; my comment on that sample in Table 4.

An evaluation of the vacuum residues of several of the high-float emulsions are shown below as an illustration of how the residue properties are affected by the vacuum recovery procedure compared to the distillation procedure. The data for the HF 150P (Figure 19) shows the distilled residue to be the least stiff compared to the 2- and 3-h vacuum residues. This could be due to some solvent loss under vacuum, which based on the low temperature properties determined from the 4-mm DSR test seems to be a reasonable assumption.

The low temperature results determined from the 4-mm DSR test procedure for the samples shown in Figure 18 are in Table 5.

Testing of the HFRS-2P emulsion provided some additional insights into the behavior of high-float emulsions when subjected to vacuum curing. A sample of HFRS-2P was conditioned for 2 h

TABLE 4 High-Float Emulsion Float Test Results on Thin Film Vacuum Residues

Material	Vacuum Oven Time, h	Vacuum Range	Residual Moisture, %	Float, s	MSCR % Recovery @ 25°C, 3.2 kPa, 4-mm DSR
HFE-90, Supplier 2	2	7.6–12.7 mm Hg	0.92	1,800, little movement	
HFRS-2, Supplier 2	3	7.6–20 mm Hg	0.36	2,531, bulged but did not sink	
HFRS-2P, Supplier 2	2.75	5–10 mm Hg	0.51	3,600, slight movement	67.9
HFRS-2P, Supplier 2	2	5–10 mm Hg		2,761	69.5
HFRS-2P, Supplier 2	3	5–10 mm Hg	0.29	2,160, bulged but had not failed at this point	69.5
HF-250S, Supplier 1	3	15–25 mm Hg	0.76	3,600, no movement	
HF-150P, Supplier 1	3	15–25 mm Hg	2.24**	1,903, no bulge	59.8
HF-150P Supplier 1	2	15–25 mm Hg	4.36**	2,476, bulged quite a lot	53.3
HF-150P, Supplier 1	400°F distillation	na	na	Not tested	64.4
HFE-1000, Supplier 2	2	5–10 mm Hg	2.47, but I think most was solvent loss	1,980, minor bulging	
HFRS-2, Supplier 3	3	5–10 mm Hg	0.23	1,040	
HFRS-2, Supplier 3	Boiled water off sample	NA	Dry	813	

** These moisture contents are probably not reflective of the true moisture content. Based on the odor of the emulsion there was some solvent present. However the low values of the MSCR recovery relative to the 400°F distillation indicates there was sufficient moisture present to weaken the binder structure. Also note that the vacuum level was substantially higher than the finally settled optimum range.
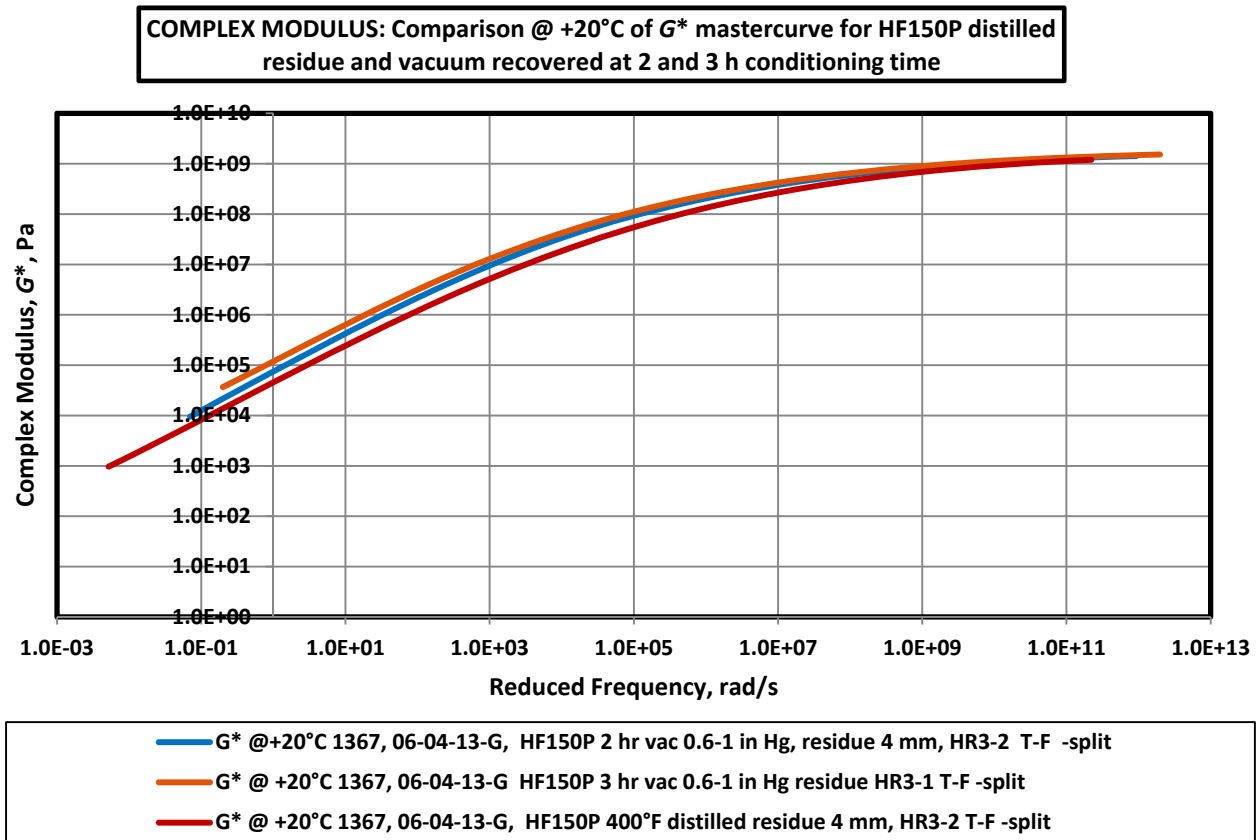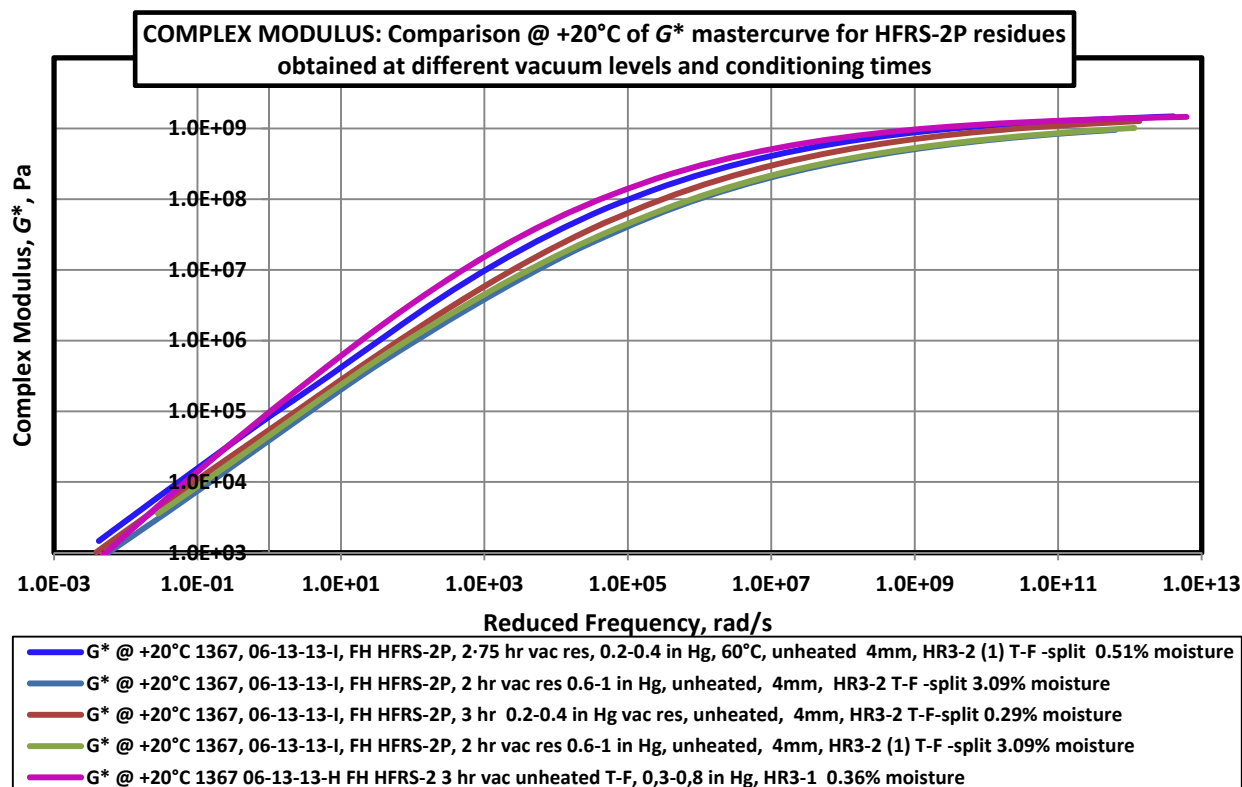
FIGURE 19 Complex modulus comparison of 2- and 3-h vacuum residues versus the modulus of the 400°F distilled residue for HF 150P.

at a fairly high vacuum of 15 to 25 mm Hg (0.6 to 1 in. Hg) and the resultant sample had residual moisture of 3.09%. Normally a sample with a residual moisture level that high would be discarded, but it was already on the DSR. Subsequently vacuum residue recoveries were performed at 2.75 and 3 h at a vacuum level of 5 to 10 mm Hg (0.2 to 0.4 in. Hg) and 4-mm DSR tests were performed on those samples as well. Figure 20 shows the original 4-mm DSR test of the 3% moisture sample and a repeat of the same sample. Those two results overlay and are the green and turquoise, lowest data lines on the plot. In addition to providing evidence of the repeatability of the 4-mm DSR test the data also provides graphic demonstration of how the presence substantial levels of water in the binder will weaken the binder. The 2.75- and 3-h vacuum residues show greater stiffness than the 3% moisture samples and the HFRS-2 residue is the stiffest of all the samples, most likely due to loss of some solvent. Whether or not solvent was added to the HFRS-2P is not known.

TABLE 5 Low-Temperature Properties of HF 150P Residues

Sample Description	Stiffness Critical Temp., °C	*m*-Value Critical Temp., °C
Proj 1367, 06-04-13-G, HF 150P 400°F distilled residue 4 mm	–47.3	–47.8
Proj 1367, 06-04-13-G, HF 150P 2-h vacuum residue 4 mm	–42.2	–43.3
Proj 1367, 06-04-13-G, HF 150P 3-h vacuum residue 4 mm	–39.9	–41.4

FIGURE 20 Comparison of complex modulus results for HFRS-2P residues obtained by vacuum curing at different vacuum levels and conditioning times.

Examination of the plots in Figure 21 discloses one behavior that seems atypical for an asphalt binder. All of the high-float residues tend towards the same stiffness frequencies at less than 0.1 radians/s regardless of the stiffness in the 1.0E+05 range. Typically asphalt stiffness does not drop off as rapidly as the data shown in Figure 19. For comparison in Figure 20 the CRS-2P vacuum residue from the same producer who provided the HFRS-2 and HFRS-2P has been plotted. Note that for the CRS-2P residue the rate of change of stiffness from 1.0E+05 to 0.1 radians/s is much less than for the high-float residues. There is a possibility that looking at a ratio of binder stiffness between those two frequencies could be a rheological method of determining whether or not a binder is a high-float material.

ADDITIONAL CHARACTERIZATION TESTS

There are several advantages of using any thin film emulsion residue recovery procedure most notably the ability to characterize polymer modified emulsion residues as nearly as possible as they will exist on the road. Also important is that using the vacuum procedure discussed in this report a residue can be obtained that closely matches the binder on the road within 2 h or possibly 3 h. With a vacuum oven capable of handling multiple samples two or more residues can be obtained in the same time period. However, none of these advantages can be realized until the emulsion specifications move away from penetration, ductility, force ductility, toughness and tenacity and elastic recovery; all of which require large amounts of binder to perform. This report has demonstrated that from a single 5-g residue sample several residue characterizations are possible. Having a means of obtaining the residue quickly is not a benefit until tests are adopted and specifications drafted based on those tests. The 4-mm DSR test can characterize low and intermediate temperature binder stiffness and thereby yield the low temperature grade of the emulsion residue. With the 4-mm geometry a MSCR recovery can be obtained at 25°C. The low temperature and MSCR result can be obtained on a single sample in one test sequence. Professor Hussain Bahia and his colleagues at UW-Madison have devised several tests to replace the classical ductility, force ductility, and elastic recovery tests with tests that have been moved to the DSR (7). One of these tests, the binder yield energy test (BYET) is designed to replace the
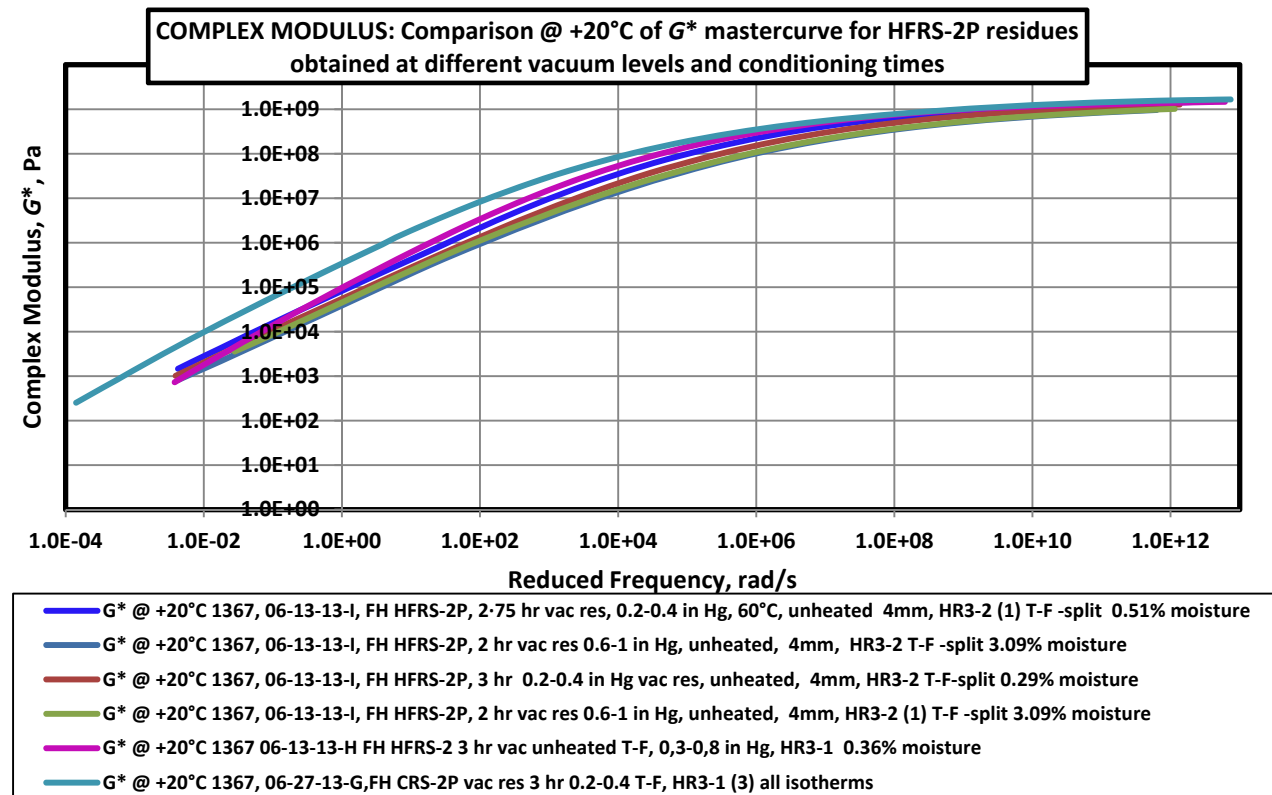

FIGURE 21 Overlay of CRS-2P modulus on high-float modulus data

force ductility test is performed at the specified Force Ductility temperature (typically 4°C or 25°C). Figure 22 is an example of the BYET test performed on a vacuum emulsion residue sample. This data is for illustrative purposes only. Bahia recommended using the ratio between the peak stress and the stress at 2000 seconds as a replacement for the force ductility test (*8*).

Alternative methods of quickly obtaining emulsion residues are being explored. Use of the Core-Dry has been explored as a vacuum device to obtain emulsion residue. WRI has proposed and is investigating their SAT (*3*) plate as a tool to obtain emulsion residue in a vacuum oven. MTE has investigated both of these approaches and has found they will provide residue with the appropriate properties. Both yield lesser amounts of residue (approximately 2 g) per conditioning plate. Depending on the testing to be performed that amount is conceivably sufficient. Figure 23 shows data comparing the modulus and MSCR results from the 2-h vacuum recovery procedure discussed in this report and the 2-h residue using the SAT plate to the properties of the original base binder. The results show very good reproducibility of the results for all three samples.
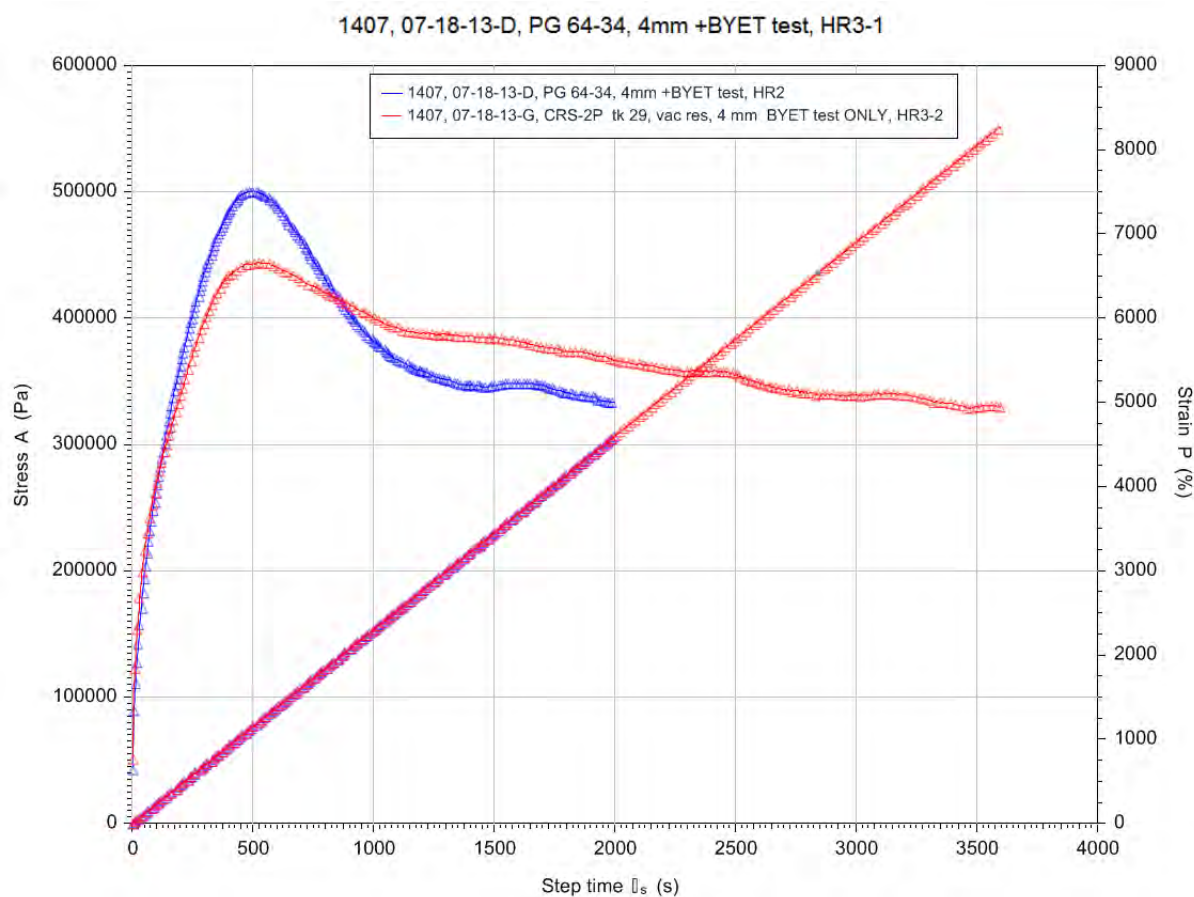

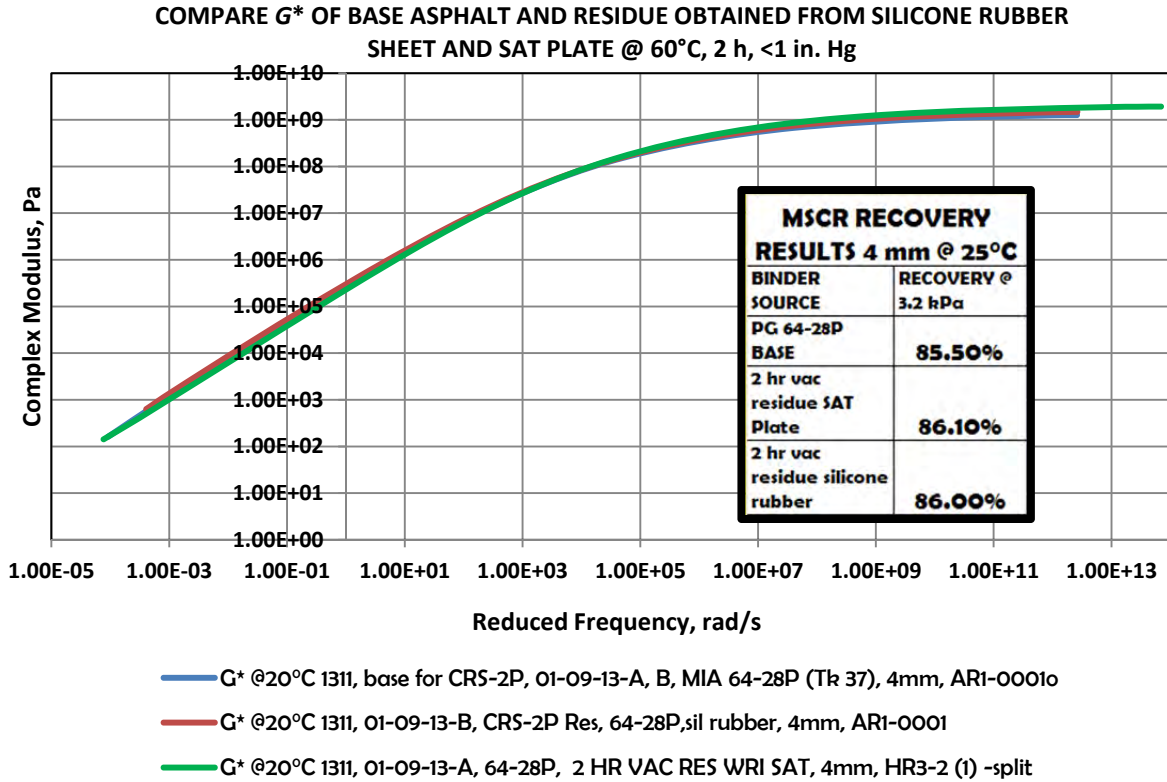**FIGURE 22 Example of the BYET test performed using a 4-mm DSR tool.**

COMPARE *G** OF BASE ASPHALT AND RESIDUE OBTAINED FROM SILICONE RUBBER SHEET AND SAT PLATE @ 60°C, 2 h, <1 in. Hg

Legend:
——— G* @20°C 1311, base for CRS-2P, 01-09-13-A, B, MIA 64-28P (Tk 37), 4mm, AR1-0001o
——— G* @20°C 1311, 01-09-13-B, CRS-2P Res, 64-28P,sil rubber, 4mm, AR1-0001
——— G* @20°C 1311, 01-09-13-A, 64-28P, 2 HR VAC RES WRI SAT, 4mm, HR3-2 (1) –split

MSCR RECOVERY RESULTS 4 mm @ 25°C

BINDER SOURCE	RECOVERY @ 3.2 kPa
PG 64-28P BASE	85.50%
2 hr vac residue SAT Plate	86.10%
2 hr vac residue silicone rubber	86.00%

FIGURE 23 Comparison of vacuum recovery using silicone sheet or SAT plate to the properties of the base binder to make the emulsion.

CONCLUSIONS AND SUGGESTIONS

Several approaches to recover emulsion residues at temperatures in the range of 60°C have been explored in this report. Based on the data comparisons it does appear that shorter times of recovery with reduced exposure to oxygen of the thin asphalt films required to dehydrate the emulsion are necessary to produce recovered residues that approximate the base binder from which the emulsion was produced. Since any procedure that is finally adopted will become part of an emulsion producers quality control program reducing the time to obtain the residue is essential. A 2-h, 60°C, 5- to 10-mm Hg vacuum recovery procedure appears to accomplish the goals of speed and causing the least aging to the recovered residue. Further, more in depth investigation of high-float emulsions may be needed to determine if 3 h is required for the residues to achieve float. I believe it will be necessary to arrive at binder stiffness data for solvent containing high floats that accounts for the greater solvent loss in the vacuum oven than in the emulsion still. This fact does not mean changing the high-float grades or formulations just a set of residue binder properties characteristic of the vacuum residues. Determination of a rheological test that can be correlated to the high-float residue gel characteristic is needed, but until such time as that test is developed it is possible to run conventional float tests on the small amount of binder recovered in the vacuum procedure.

Work reported in this paper identified a single comparison of a residue from 4 hours under vacuum to the same material after 3 h under vacuum indicates that there is a critical time

beyond which it isn't prudent to continue conditioning the emulsion. Obviously this concept needs to be studied in more depth.

There is a critical need to get the 4-mm DSR test method as well as the rheological approaches to supplant classical ductility and elastic recovery tests into the hands of agencies and producers alike. These are the critical tools that can provide low temperature property data, binder stiffness in the service temperature range and elastomeric property information using very small quantities of emulsion residue. Currently available procedures for determining high-service temperature binder stiffness and use of the MSCR test procedure to determine nonrecovered compliance and elastomeric properties of thin film recovered residues should allow agencies to eliminate the need to perform elastic recovery or other elasticity identifier tests now performed on distilled emulsion residue.

Many of the conditioning procedures discussed are new and some of the rheological test procedures are not well known, especially in the emulsion production community. The only way to move forward towards actually testing the binders being placed on our roads is to begin to investigate and adopt these procedures. MTE is currently working on a ballot item for ASTM to standardize a practice for performing the thin film vacuum oven residue recovery procedure.

NOTES

1. For chip seal type emulsions test temperatures of 52°C and 58°C might be appropriate for slurry seal or microsurfacing, or tack emusions test temperatures of 58°C and 64°C or perhaps higher might be appropriate.
2. Silicone rubber sheeting obtainded from McMaster Carr 40A Durometer and 1/8-in. thickness

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The **Transportation Research Board** is one of six major divisions of the National Research Council. The mission of the Transportation Research Board is to provide leadership in transportation innovation and progress through research and information exchange, conducted within a setting that is objective, interdisciplinary, and multimodal. The Board's varied activities annually engage about 7,000 engineers, scientists, and other transportation researchers and practitioners from the public and private sectors and academia, all of whom contribute their expertise in the public interest. The program is supported by state transportation departments, federal agencies including the component administrations of the U.S. Department of Transportation, and other organizations and individuals interested in the development of transportation. **www.TRB.org**

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