Advanced Methods for Quantifying Emulsion Setting and Adhesion to Aggregates

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Abstract:

An important emulsion property consideration in chip seal applications is the time required for the development of adequate binder properties. During application, the emulsion goes through breaking and setting stages, leaving behind the residual binder. The setting time of an emulsion is dependent on its composition, the ambient conditions, and the physical and chemical characteristics of the surfaces it is in contact with (15). The surfaces include both the substrate and surface of the aggregate chips. In a practical sense, setting rate of the emulsion directly affects the appropriate timing of opening to traffic. For example if a chip seal is opened to traffic before full setting, the binder will be too soft, allowing excessive deformation and removal of the aggregates under traffic load (early raveling). Conversely, overestimating curing time is detrimental to the public due to excessive user delays.

This paper is focused on using two test methods to quantify the time dependence of emulsion setting in terms of critical performance properties. In the first method the Dynamic Shear Rheometer (DSR) is used for conducting strain-sweep testing in which the complex modulus (G^*) and phase angle (δ) are measured over a range of strains after different setting times and different temperatures. The DSR procedure allows evaluation of the increase in resistance to flow and permanent deformation, and strain tolerance as the emulsion sets. The impact of aggregate mineralogy is also evaluated in the test through curing of the emulsion on granite and limestone substrates while sampling for the DSR testing.

The second test is used to measure development of emulsion adhesion with time using a pull off tensile test. This test is conducted as a function of time as emulsion sets on two different mineral surfaces: granite and limestone substrates.

The objective of these tests is to introduce more precise and scientific tests for better understanding of emulsion setting and its dependence on the aforementioned chemical and physical factors. Such understanding should allow more reliable design and construction practices of chip seals. Estimation of the time at which a given emulsion has developed adequate adhesion, resistance to deformation, and strain tolerance is expected to lessen the severity of various early forms of chip seal distress under traffic loading. These advanced tests could improve chip seal performance and broaden the appeal of surface treatment applications.

INTRODUCTION

As use of surface treatments becomes more prevalent, specifications and test methods related to performance in terms of construction and in-service properties are needed to enhance the reliability of surface treatment design. The key function of surface treatments is to extend the service life of the underlying pavement by providing increased skid resistance and an impermeable layer to protect the pavement from oxidation and moisture. There are three key distresses that hinder the ability of the surface treatment to perform its intended function, namely, raveling, bleeding, and fatigue cracking. Proper consideration of key construction properties such as storage stability, emulsion breaking and setting, emulsion viscosity, and development of adhesion/cohesion mitigates the potential for these distresses to occur, providing a more reliable design (7). Evaluation of emulsions are currently specified using various standards such as the ASTM standards, the European Norms, and French Standards. Review of specific examples of these standards will be presented in subsequent sections of the paper (1, 2, 3, 8). Most of these standards include empirical measures that are successful in evaluating emulsion chemistry and simplistic ranking in terms of performance, but have limited relationship to the mechanical properties necessary to achieve optimum field performance. Current research efforts have identified a clear need to combine fundamental test methods and practical experience to provide a more quantifiable method for characterization of emulsions. Definition of critical emulsion construction properties and advanced methods to test them has the potential to improve the current state of practice through transitioning to more performance based emulsion testing and specifications.

The first objective of this study is to provide an overview of the mechanisms of breaking, setting, and development of adhesion and the test methods used currently to evaluate them. The second objective is to introduce application of two existing asphalt binder test methods for evaluation of emulsions' setting and development of adhesion. Specifically, the Dynamic Shear Rheometer (DSR) and Pneumatic Adhesion Tension Testing Instrument (PATTI) are used to measure changes in rheological properties and adhesive strength as a function of time and substrate mineralogy. In terms of rheological properties, the development of stiffness and strain tolerance over time is quantified. These measurements provide insight as to when the emulsion has attained adequate stiffness, strain tolerance, and aggregate adhesive strength to allow the surface treatment to be opened to traffic. Furthermore, the methods of adhesion testing presented in this paper provide a protocol that could be used to evaluate aggregate/emulsion compatibility.

BACKGROUND

Breaking and Setting

Emulsion technology provides a low temperature delivery system for use of asphalt binders in paving applications, after the product is applied, the suspended asphalt droplets must revert back to a continuous asphalt film to suit its intended purpose. The transition of the emulsion to an asphalt film is defined as breaking and setting. In general, there are four steps to the emulsion breaking and setting process (9):

1. Destabilization: Physical or chemical change in emulsion system draws binder particles closer together.
2. Flocculation: Asphalt droplets begin to agglomerate as emulsifier is absorbed into the aggregate surface.
3. Coalescence: Continuous asphalt film is formed. Asphalt residue begins to exhibit tackiness and stiffness.

4. Evaporation: Water completely evaporates allowing for full development of stiffness and adhesion of base binder.

Breaking and setting are separated using the steps listed above. Breaking is a more rapid process which involves destabilization of the emulsion and flocculation of the asphalt particles. Setting is a slower process which involves coalescence of the floccules and evaporation of the water from the continuous asphalt film (15). Asphalt droplets are suspended in the aqueous emulsion matrix due to electrostatic surface charges imposed on them by the emulsifier, causing the particles to repel each other (9). If the amount of energy introduced to the system is greater than electrostatic forces between the droplets, the emulsion will destabilize, allowing the particles to flocculate, hence breaking the emulsion. After breaking the emulsion residue forms a non-tacky brittle solid (15). The setting process begins as the floccules coalesce, forming an asphalt film with mechanical properties more consistent with those of the base asphalt binder. The emulsion has completely set when all of the water has evaporated from the asphalt surface. Theoretically, after complete setting the properties of the emulsion residue are similar to those of the base asphalt.

The energy required to promote breaking and setting is introduced to the system by both chemical and physical means. The rate of emulsion breaking and setting is dependent on such chemical factors as the type of emulsifier used and the mineralogy of the seal aggregate and substrate (7). Aggregate mineralogy and emulsifier type are critical factors because of their effects on emulsion behavior. Introduction of aggregate to the emulsion induces a change in pH due to reactions between the acid in the emulsion and the aggregate and absorption of the emulsifier into the aggregate surface. The magnitude of these reactions is dependent on mineralogy, if the emulsion and aggregate are chemically compatible, the emulsion will destabilize, initiating breaking (9). However, if the emulsifier and aggregate are not compatible, breaking will be significantly delayed, causing both construction and performance issues. Breaking behavior is also influenced by physical factors such as climate and mechanical rolling. These physical factors initiate breaking by two mechanisms: forcing emulsion particles closer together, or increasing the rate of the reaction. Mechanical rolling decreases the space between suspended asphalt particles, promoting breaking to occur. Climatic conditions of temperature, humidity, and precipitation can significantly affect the rate of emulsion breaking and setting. Conditions that prevent the water from evaporating from the emulsion such as, cold temperatures, high humidity, or rainfall delay the breaking and setting process. Conversely, higher temperatures or lower humidity can accelerate breaking and setting (7).

Adhesion

Proper construction of a surface treatment, such as a chip seal, depends both on selection of an emulsion to provide the intended breaking and setting behavior and the ability of the residual asphalt to properly wet and bond with the aggregate. In chip seals, the aggregate is spread on top of the emulsion film as it cures. The adhesive bond between the seal and the aggregate surface is developed through the electrostatic attraction between the asphalt and aggregate and the pH change previously mentioned. Pneumatic action provided by rolling also promotes adhesion through mechanical force and acceleration of emulsion breaking. However, proper wetting is still a function of aggregate/emulsifier compatibility due to aggregate mineralogy. Some aggregate surfaces are hydrophilic, meaning they have a higher affinity for water than asphalt. In this condition full asphalt/aggregate adhesion will not develop until the aggregate surface is completely dry due to the residual water occupying bonding sites on the aggregate surface. Complete drying of the aggregate could take a considerable amount of time, preventing timely opening of the chip seal to traffic (15). Premature trafficking of the surface treatment leads to excessive raveling due to the inability of the adhesive bond to withstand the strains associated with tire action over the chip seal. Proper selection of an emulsifier mitigates this problem by displacing water from the aggregate surface as the emulsifier is absorbed by the aggregate. Removal of the water allows for proper development of adhesion and timely trafficking. In general, cationic emulsifiers are preferred for acidic aggregate and anionic emulsifiers for basic aggregate (15).

Current Methods of Emulsion Evaluation and Opportunities for Improvement

Conceptually, current practice provides emulsion and residue test methods to evaluate properties critical to seal performance. These methods are listed in several ASTM standards, European Norms, and also some specialized French Standards. The tests are well selected in that they provide a framework to allow for specification of an emulsion based on application conditions and expected emulsion/aggregate compatibility. However, many of these test methods are empirical in nature, ranking material behavior in a laboratory test that is not well related to performance properties in the field (7). The following is a list of most pertinent construction properties and the corresponding test methods specified in the current standards for emulsified asphalts used in surface treatment applications. More detailed description of the current standards related to breaking, setting, and adhesion are provided in a subsequent section.

- Construction related properties currently measured on the emulsion:
  - Storage Stability: ASTM D6930
  - Demulsibility: ASTM D6936
  - Sprayability and Drain-out: ASTM D244 17-21 (3)
  - Breaking Rate: EN13075-1, T 66-017 for Cationic Emulsions and T 66-019 for Anionic emulsions (Tentative French Standards) (1,2)
  - Viscosity: ASTM D 7226-06

- Emulsified Asphalt/Aggregate Interaction:
  - Coating Ability and Water Resistance: ASTM D244 22-29 (3)

Application of these procedures in conjunction with industry expertise have allowed for the collaboration between industry and agencies to provide quality surface treatments for highly trafficked roads. However, in order for continued growth a transition must begin to incorporate technologies used in specifications for hot asphalt applications into emulsion specifications. Cost effective improvement on the current state of practice for surface treatment applications has the potential to provide more reliable design and improved materials selection for surface treatments, particularly for modified emulsions. Specific examples of the short comings of current standards are the lack of a test to quantify setting time and the qualitative tests used to evaluate breaking rate, asphalt/aggregate compatibility, and coating.

The current tests to evaluate breaking rate (1,2,8) involve addition of silica flour to the emulsion under stirring until the emulsion breaks, changing color from dark brown to black. The output of the test is the amount of silica powder required for break. The test is an indicator for ranking breaking rate of emulsions, however the breaking occurs in the emulsion due to effects of the surface area introduced by the silica filler, rather than due to a chemical break, as occurs with various mineral aggregates normally used in the field. The test also does not provide a direct measure of changes in critical material properties (10). A rheological test to evaluate breaking was developed by Dr. Delmar Salomon that involves use of the DSR to conduct a temperature sweep from 25°C to 85°C at 1 Hz and 1% strain (16). The test allows for determination of the gel point where the visco-elastic behavior of the emulsion transitions from viscous, liquid like behavior to elastic behavior. In terms of rheology, the gel point is defined as the temperature at which the storage modulus G' is equal to the loss modulus G". The test was found to be effective in ranking the relative breaking rate of the same family of emulsions (16). The test was not conducted for this paper but future work should involve comparing rankings between the silica flour test and rheological measurements to evaluate if the current standard is adequate or more fundamental measurements of breaking rate are needed.

The current test to evaluate asphalt/aggregate compatibility specified in ASTM D244 is also qualitative in nature. The test involves preparation of an aggregate emulsion mixture which is then rinsed and visually inspected for aggregate coating (3). Researchers and practitioners have identified the critical need for a better mechanical test to quantify asphalt/aggregate adhesion and emulsion compatibility in previous studies. This paper introduces the use of the Pneumatic Adhesion Tension Testing Instrument (PATTI) as a potential replacement for the current test method. The test has been used to quantify moisture damage in hot asphalt applications (12), but procedures and test methods are introduced in this paper for application in emulsions. The test has the capability to both evaluate aggregate/emulsion compatibility and development of adhesion as the emulsion sets, providing a useful tool in both design and construction.

Current specifications do not provide a test to quantify the setting time of an emulsion. A strain sweep procedure using the DSR is introduced to determine the evolution of rheological properties in terms of stiffness and strain tolerance as a function of time. The test has practical applications in terms of providing insight as to when the emulsion residue has attained adequate strength for opening to traffic.

The complexity of emulsion breaking and setting, and development of adhesion, in emulsion applications combined with increased use of modified emulsions emphasizes a clear need for more fundamental test methods to quantify construction properties of emulsions. Fortunately technology and potential test methods currently used for hot binder applications are available to ease the required transition. Integration of current procedures with advanced testing will lead to an economical specification that provides more reliable chip seal design methods and construction practices.

TEST METHODS USED IN THIS STUDY

A CRS-2 emulsion sample was placed on aggregate substrates of different mineralogies and subjected to open air curing conditions. Tests for characterization of rheological properties of the residue at different stages of curing were conducted using a Dynamic Shear Rheometer (DSR). The tests were selected based on two critical performance issues related to aggregate retention in chip seals: (a) resistance to permanent deformation and (b) aggregate retention. Motivation for the aforementioned tests and definition of the evaluation criteria used are discussed next.

Measuring Response to a Strain Sweep

The strain sweep testing procedure in the DSR involves applying different levels of shear deformation and measuring the corresponding stress. Based on the material response the complex shear modulus (G*) and the phase angle (δ) are calculated, providing rheological material properties for further evaluation.

The strain sweep protocol directly relates to distresses observed for in-service chip seals, namely aggregate loss and resistance to permanent deformation. These performance parameters are critical during the first year a chip seal is in-service. The thin film developed between the residue and aggregate shortly after a chip seal is constructed is subjected to high strains under traffic loading (15). If the emulsion residue has not developed adequate strain tolerance or stiffness, the applied strain will exceed the failure limit and the bond will fail causing loss of aggregate (raveling). Rheological residue testing in the DSR can simulate this type of distress through the use of a strain sweep. The concept of using emulsion rheological properties to estimate stone retention in chip seals and quantify its development with curing time was introduced by Kucharek (14). In the study Kucharek conducted strain sweep testing on a variety of emulsions and showed strong correlation with the ASTM D7000 Sweep Test, a test that measures chip seal performance (5). Results showed that higher stiffness and strain tolerance lead to better aggregate retention.

The procedure presented by Kucharek involved curing of 9 grams of emulsion in the trays used to prepare samples for the frosted marble cohesion test; the resulting emulsion film thickness was approximately 1 mm. The emulsion was then subjected to open air curing with samples taken after 2, 4, 6 and 24 hours (14). The original procedure was modified to consider the effect of aggregate mineralogy on emulsion setting rate by placing a similar film thickness on granite and limestone aggregate surfaces. A rubber O-ring of 3.6 mm in cross-section diameter was used to ensure a constant film thickness was maintained. Examples of the test set up are provided in Figures 1 and 2.

FIGURE 1: Preparation of Aggregate Surface for Emulsion Placement (7)

FIGURE 2: Placement of Emulsion on Aggregate Surface (7)

Emulsion residue was sampled from the aggregate surface for testing using the strain sweep procedure after 2, 4, 6, and 24 hours of setting time. Strain sweep testing was conducted at 25°C and a frequency of 10 rad/s on a 25 mm parallel plate geometry at strain levels of 1% - 20% on granite and limestone substrates. Strain was applied to the sample at a logarithmic rate. Prior to testing, the sample was heated to 45°C for 5 minutes to ensure adhesion to the parallel plates (14).

Measurement of Adhesion

In-service chip seal performance is a function of both setting behavior and the ability of the emulsion to sufficiently adhere to the aggregate surface of the chips. Thus, development of adhesion is equally important in design considerations to mitigate surface raveling. Currently specifications for both hot applied and emulsified asphalts lack a quantitative measure of adhesion. The need for a quantifiable adhesion test is more pronounced in chip seal applications using emulsions due to the impacts of development of adhesion and emulsion/aggregate compatibility on performance. Past research efforts have reported on applying adhesion tests used in the paint industry to evaluate the bond strength developed between hot asphalt binders and aggregate surfaces (12). In applying these test methods it must be stated that failure in an asphalt aggregate system occurs at the weakest point in the system, which could be a cohesive failure within the asphalt film, an adhesive failure between the asphalt aggregate bond, or a combination of adhesive and cohesive failures (13). These circumstances render the test a measurement of bond strength rather than direct measurement of adhesion. Examples of typical failures will be presented in subsequent sections.

Adhesion Testing Procedure

The Pneumatic Adhesion Tension Test (PATTI) was developed by the National Institute of Standards and Technology for use in the paint industry and has been adopted by ASTM standard

D4551: Pull of Strength of Coatings Using Portable Adhesion Testers (4). Schematics of the PATTI test are provided in Figures 3 and 4.

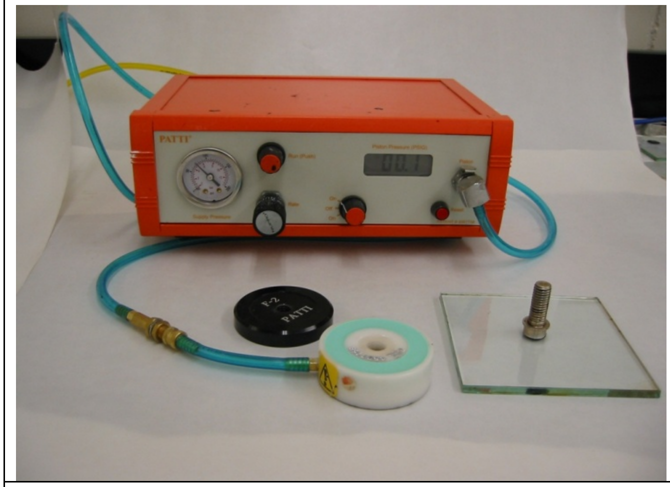

FIGURE 3: PATTI Testing Device (13)

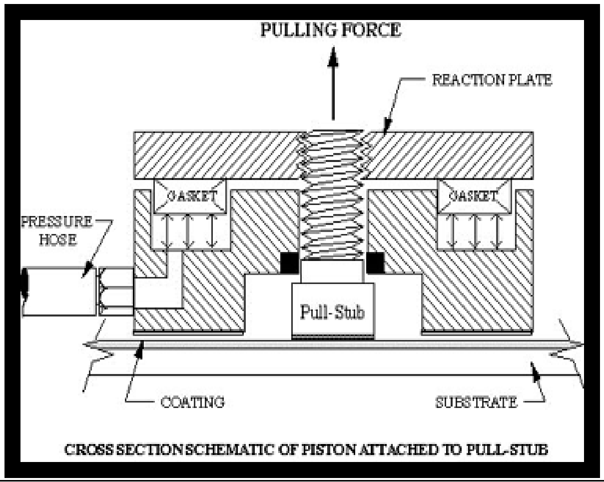

FIGURE 4: Schematic of Pull Stub and Pressure Chamber (13)

In the PATTI test, a pull stub is adhered to a substrate, and the loading mechanism provided in Figure 4 is attached. An air pressure is applied, creating a tensile force in the vertical direction until the stub has been separated from the substrate. The maximum air pressure is converted to a pull off tensile strength, the conversion factor depends on the reaction plate used in the device. The pull off tensile strength provides a quantitative parameter to evaluate bond strength, but cannot discriminate between adhesive and cohesive failure (12). Differentiation between adhesive and cohesive failure is conducted qualitatively by examination of the failure surface. A failure surface with asphalt remaining on the substrate is defined as a cohesive failure; conversely adhesive failure occurs when no asphalt remains on the substrate. Typical adhesive and cohesive failures are provided in Figures 5 and 6 (13).

FIGURE 5: Cohesive Failure (13)

FIGURE 6: Adhesive Failure (13)

The PATTI test has been used in hot asphalt applications as a measure to quantify asphalt/aggregate bond strength as it relates to moisture damage (12). The test was modified by FHWA and by UW Madison to reduce variability by controlling the loading rate and providing a system for more accurate measure of the maximum pressure. The success of past work and the reduction in variability due to the previously mentioned modifications led to consideration of the testing concepts for evaluation of emulsions in terms of both development of adhesion and aggregate/emulsion compatibility.

Procedures for Evaluation of Emulsion Bond Strength

Considerable modifications to previously published testing procedures for hot asphalts were required to make the technology suitable for evaluation of the development of bond strength in emulsions. Modifications were focused on control of the film thickness under the pull stub and measurement of bond strength as a function of recovery time. The ASTM evaporative recovery procedure currently being proposed for balloting (11) was used to provide emulsion residues after different curing times; specific sampling times will be presented in the experimental design. Film thickness was controlled by using forms to elevate the surface of the pull stub 0.5 mm from the surface of the substrate. Three replicates were tested to evaluate the repeatability of the test method and proposed procedure. The following is a general overview of the testing procedure:

1. Sample residue at prescribed curing time and place in 8 mm DSR mold.
2. Trim the sample in the mold using a heated spatula and adhere to the substrate.
3. Strip the mold from the substrate.
4. Heat the pull stub to 60°C and adhere to the substrate using forms to control the film thickness.
5. Allow system to cure for two hours to develop adequate bonding and conduct testing.

The research is in initial stages of testing; after adequate data is collected results will be analyzed and evaluated in terms of the ability to provide a repeatable test to quantify the development of bond strength. Different substrates will be used for evaluation of aggregate/emulsion compatibility and the effects of mineralogy on changes in bond strength as a function to time.

EXPERIMENTAL DESIGN

A preliminary testing plan was developed and executed to evaluate the potential for the proposed tests to provide meaningful information in terms of emulsion setting behavior and development of adhesion. Both testing procedures used a CRS-2 emulsion applied to granite and limestone substrates. The base binder for the CRS-2 emulsion was graded as a PG 58-28. As previously discussed, time intervals, recovery procedures, and number of replicates varied between the strain sweep and PATTI testing. A summary of the materials used and the experimental design for each test method are provided in Table 1.

TABLE 1: Summary of Materials and Testing Procedures

Materials		
Variable	**Values**	**Levels**
Emulsion Charge	Cationic	1
Emulsion Rate of Setting	Rapid	1
Emulsion	Neat	1
Base Binder	PG 58-28	1
Substrate	Granite	2
	Limestone	
Strain Sweep Procedure		
Setting Conditions	Recovery Time (hrs)	Procedure
25 C Open Air Curing	2	Strain Sweep (25C)
	4	
	6	
	24	
PATTI Procedure		
25 C Forced Draft Oven Curing	2	PATTI (Room Temperature)
	24	

RESULTS AND ANALYSIS

Results are presented separately for the strain sweep and PATTI results. The following analyses are focused on the ability of the proposed test methods to measure the development of rheological properties and bond strength as a function of time. The tests will also be evaluated for their ability to show the effect of substrate mineralogy on the aforementioned performance properties.

Strain Sweep Testing Results

Chip seal performance in terms of development of resistance to permanent deformation and aggregate loss were evaluated using the previously defined strain sweep procedure. The motivation of the experiment presented was to evaluate if the strain sweep was a viable test method to be implemented for more detailed evaluation of emulsion setting behavior. Results of the analysis are presented in Figures 7 – 9.

Figures 7 and 8 provide strain sweep results for emulsion cured on granite and limestone substrates at the prescribed setting times.

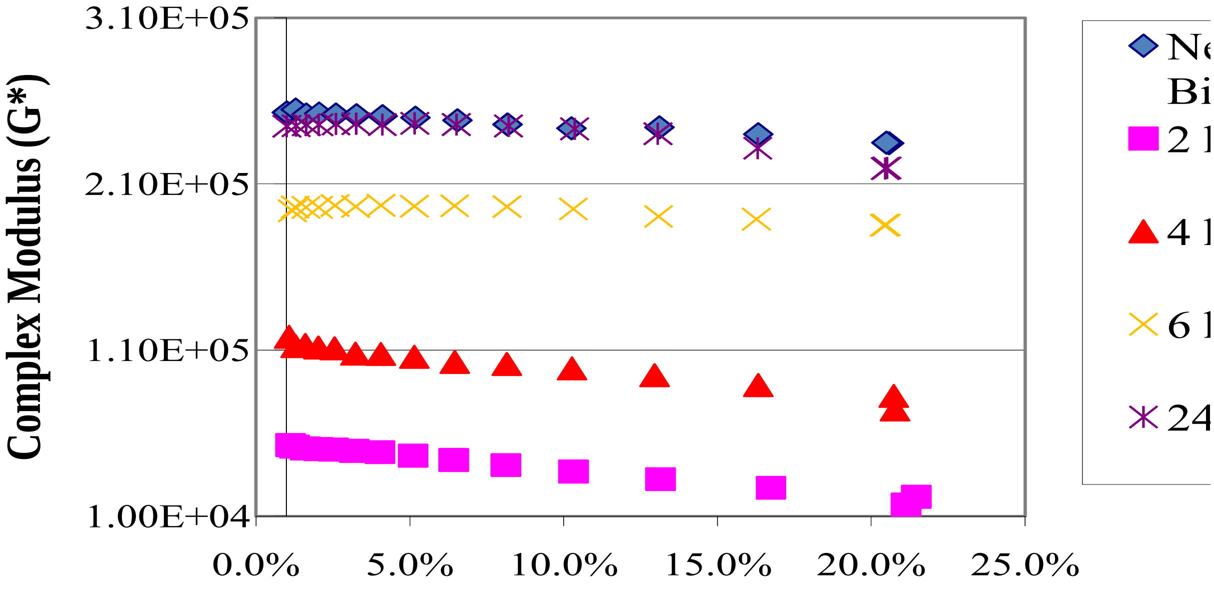

FIGURE 7: Strain Sweep Results at Different Recovery Times – Granite Substrate

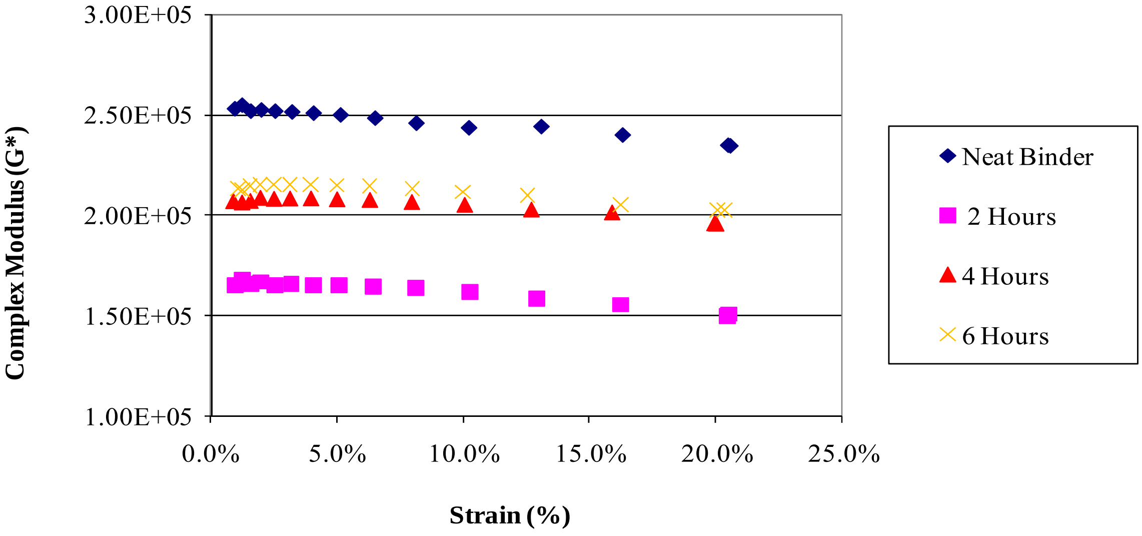

FIGURE 8: Strain Sweep Results at Different Recovery Times – Limestone Substrate

Figures 7 and 8 clearly show that the strain sweep procedure is able to indicate the development of both stiffness and strain tolerance as the emulsion sets and is also able to isolate the effect of substrate mineralogy on development of rheological properties. The data presented shows differing rates of increase in performance based on substrate mineralogy. Further discussion regarding the setting behavior on the granite and limestone surfaces are presented in Figure 9.

Figure 9 displays the range of measurements between the two replicates of each test as an indicator of the repeatability of the strain sweep procedure. The figure also provides a comparison of the differing rates of setting between the granite and limestone substrates.

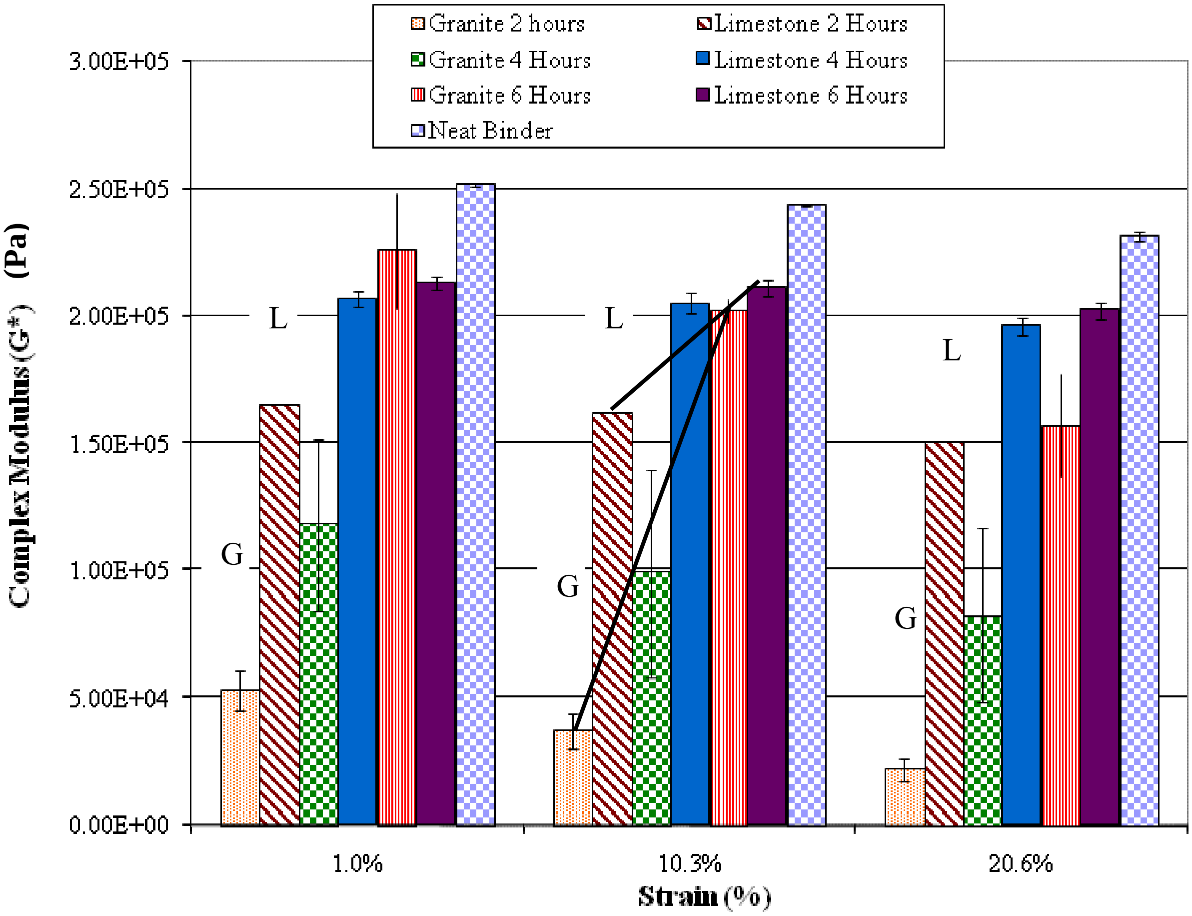

FIGURE 9: Variability and Comparison of Setting Rate on Granite and Limestone Substrates

The bar chart provided in Figure 9 represents average of two measurements taken at different curing times at low, intermediate, and high levels of strain. Results are provided for both the granite and limestone substrates. The order of the results is a comparison of the stiffness measured at a prescribed time of curing on each substrate. For example, the first two bars show the values of G* measured for the granite (G) and limestone (L) substrates after 2 hours of curing. The variability is shown by the error bars associated with each measurement. The error bars were developed using the standard deviation of the measurements, thus they represent the range of stiffness values (G*) measured for a given strain. The emulsion cured on the granite substrate shows higher levels of variability across all setting times. Based on the relative rates of curing this behavior is expected. Initial stiffness values for the granite substrate are considerably lower than values obtained for the limestone substrate, thus the emulsion on the granite substrate is unstable at early setting times due to the dynamic nature of the change in rheological properties. This concept is verified through examination of the variability of the results of curing on granite after 24 hours, which show a coefficient of variation of approximately 3%, indicating the material has stabilized. Unfortunately, results for curing on the granite substrate were not included in Figure 9 because test results for curing on limestone at this time interval were unavailable at the time of publication.

The relative rates of setting on limestone and granite substrates are indicated by the lines drawn from the stiffness measured after two and six hours of setting at the intermediate strain level. Results show rates of setting are highly dependent on substrate mineralogy. The emulsion on the granite substrate shows drastic, continuous development of stiffness with setting time. After two hours of setting the emulsion residue has a stiffness of approximately 37 kPa at 10% strain, the stiffness increases to 202 kPa after 6 hours of curing. The largest increase in stiffness occurs between 4 and 6 hours of curing, with stiffness measurements increasing from 99 kPa to 202 kPa. Conversely, the emulsion on the limestone substrate shows a considerably lower rate of development of stiffness for the same period of setting. The most significant development of stiffness on the limestone substrate occurs after two hours of setting with a measured G* of approximately 165 kPa. The stiffness after 4 hours of curing is 205 kPa, an increase of 40kPa. There is negligible strength gain between 4 and 6 hours of curing. Both substrates exhibit the same performance after 6 hours of curing. The differing early setting behavior of the emulsion on these two substrates is opposite of the behavior expected. Due to the cationic charge of the emulsion, it was expected that strength would be developed more quickly on the granite substrate due to the ability of the substrate to absorb the emulsifier (15). As provided in Figure 7, stiffness after 24 hours of curing on the granite substrate resembles that of the base binder, indicating full curing has occurred. It is hypothesized that the mineralogy of the limestone substrate allowed for early strength gain, but as indicated in the decreased rate of curing, chemical incompatibility between the emulsifier and the aggregate surface prevents full setting to occur in the same time frame. Further research is required to explain the difference in rate of setting and the hypothesis must be verified through testing of emulsion residue after 24 hours curing on the limestone substrate.

Adhesion Testing Results

A key performance parameter in chip seals is raveling resistance. As previously stated the ability of the emulsion aggregate system used for a chip seal is dependent on both the setting characteristics of the emulsion and the development of strong adhesive bonds to the aggregate spread as chips on the surface. Currently, there is not a test method available to evaluate adhesion of the aggregate surface to an emulsion. An initial experiment was conducted to evaluate the feasibility of implementing the PATTI test to address this gap in specifications. Results of PATTI testing were evaluated in terms of reliability and sensitivity to curing time and aggregate type. Initial testing results are presented in Figure 10.

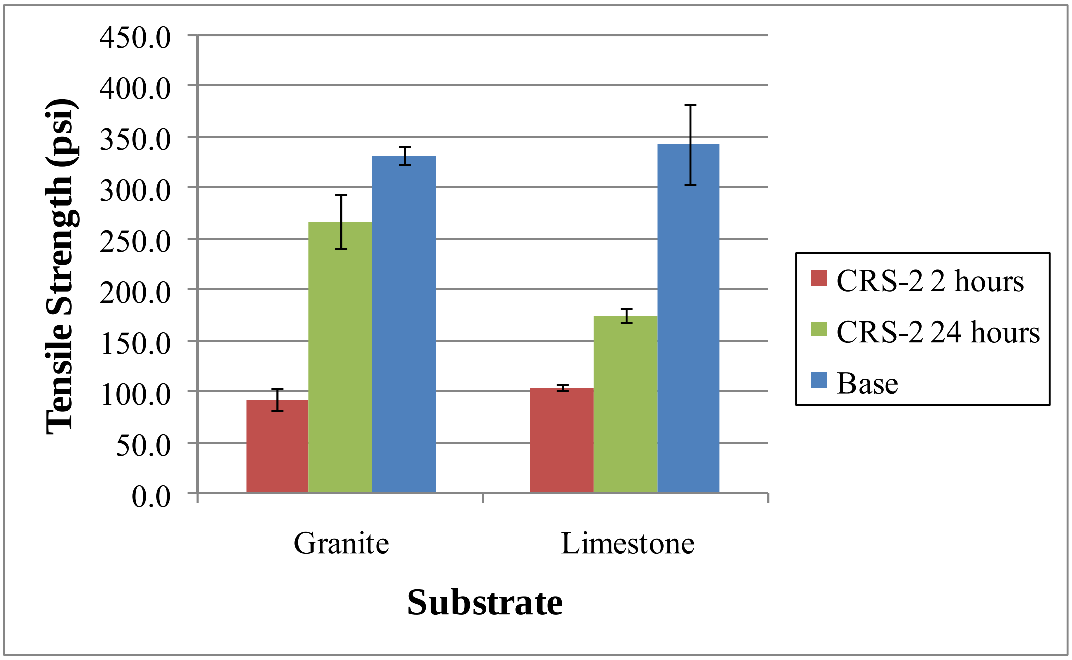

FIGURE 10: PATTI Testing Results for CRS-2 Emulsion at 2 and 24 Hour Curing Times

Figure 10 provides the Pull-off Tensile Strength (POTS) after curing the CRS-2 emulsion for 2 and 24 hours on the granite and limestone substrates, the POTS of the base binder adhered to the same substrates is also shown as a means of comparison. The range in measurements between the three replicates is shown by the error bars provided for each sample. Coefficients of variation for tensile strength measurements ranged from 1% to 12%, indicating that the procedure developed is adequate and the testing device used is a viable candidate for measuring the development of adhesion for emulsion residues. Results also show a difference in performance between both curing time and substrate type. Material cured on both substrates show a significant increase in tensile strength between 2 and 24 hours setting time, however both materials have not yet developed the adhesive strength of the base binder. The effect of substrate type on the rate of adhesion development is also shown by the differences in adhesive strength of the emulsion residue after 24 hours curing. Comparison of test results from the granite and limestone substrate after 24 hours of curing show that the material cured on the granite substrate has gained 80% of the adhesive strength, whereas curing on the limestone substrate resulted in 50% adhesive strength relative to the performance of the base binder. Observation of the failure surfaces of the aggregate and pull stub showed that there is potential that another failure mode must be considered in analysis of results. The presence of water in the emulsion residue prevents the pull stub from properly bonding to the asphalt, after testing at both 2 and 24 hours of setting time the surface of the pull stub had no asphalt on the surface. Conversely, tests on the base binder showed the asphalt had fully bonded to the surface of the pull stub. Testing after longer setting times should be conducted and compared with measurements of moisture loss to determine the relationship between the presence of this failure mode and remaining water in the emulsion residue.

SUMMARY OF FINDINGS AND RECOMMENDATIONS

A review of current practice of testing emulsions for paving applications was reviewed. Ideas for new, more advanced test methods were introduced to characterize emulsion setting behavior and development of adhesion, two critical construction properties for chip seal performance. The analysis of data collected in this study leads to the following summary of findings:

1. As the use of emulsions become more prevalent in high traffic applications there is a need for development of more fundamental testing procedures to characterize their construction and in-service properties. Evaluation of emulsions using tests more related to performance will allow for enhanced reliability of chip seal designs and applications.

2. Initial testing results show that both the strain sweep and adhesion testing procedures are able to characterize the development of emulsion rheological properties as a function of setting time. The success of these test procedures necessitates a more comprehensive experimental design that includes evaluation of modified and high float emulsions to address commonly used materials in chip seal applications. The effects of temperature and humidity on the rate of development of properties should also be considered to provide guidelines more related to field conditions.

3. While the DSR procedure is well known, a more comprehensive experimental study in terms of emulsion types, substrate mineralogy, and curing time should be conducted for evaluation of the feasibility of incorporating the PATTI, or another similar test, into practice.

4. Modifications to the strain sweep procedure should be investigated to improve the reliability of test results, especially at short curing times. Potential modifications include use of a forced draft oven to provide a more controlled curing environment and use of a linear loading rate to better show change in rheological properties as the material fails.

5. Analysis of the strain sweep data should be expanded to provide more detail in regards to development of resistance to permanent deformation and strain tolerance. It is suggested that the evaluation parameter for resistance to permanent deformation be defined as the value of $G^*/\sin\delta$ at 12% strain. Use of the Multiple Stress Creep and Recovery (MSCR) test to further characterize resistance to permanent deformation and discern the effects of modification at high pavement temperatures should also be considered (6). Strain tolerance should be evaluated in terms of both the linear-viscoelastic and failure limits of the emulsion over time. These limits are defined as a 10% and 50% reduction in G^* respectively.

6. The effect of substrate mineralogy should be investigated by comparing test results from emulsion cured on different substrates to curing on the silicone rubber mats used in the

draft ASTM recovery procedure (11). Furthermore, research should include a test to evaluate aggregate chemistry to transition from the qualitative comparison of setting rate on granite and limestone substrates to comparison based on a quantitative measurement.

7. The relationship between the individual measures of setting rate and adhesion and the entire chip seal system should be established by using the Sweep Test as specified by ASTM D7000 (5). Sweep testing should be conducted after the same curing times used for the strain sweep and adhesion testing procedures.

8. Rheological evaluation of setting time and development of adhesion using the proposed methods does not include consideration of the effects of compaction on the development of properties. Literature review indicates that compaction significantly increases the rate of both emulsion setting and development of adhesion (7). Change in Sweep Test results as a function of time and field projects should be examined to address the disconnect between evaluation using the laboratory testing protocols presented in this paper and practical applications.

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