

TEXAS TECH UNIVERSITY Multidisciplinary Research in Transportation

# Laboratory Evaluation of Constructability Issues with Surface Treatment Binder

Sanjaya Senadheera, Jorge Prozzi, Andre Smit, Ambarish Banerjee, Andrew Tubb, Lei Niu

Texas Department of Transportation

Report #: 0-5893-1 www.techmrt.ttu.edu/reports.php



March 2012

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**Technical Report Documentation Page** 

1. Report No.	2. Government Accession	No. 3.	Recipient's Catalog No.	Ŭ
FHWA/TX-12/0-5893-1				
4. Title and Subtitle		5	Penort Date	
Laboratory Evaluation of Constructability Issues with Surface		th Surface C	october 2011	
Treatment Binder				n Codo
		0.	Performing Organization	n Code
7. Author(s)		8.	Performing Organization	Report No.
Sanjaya Senadheera, Jorge Proz	zzi, Andre Smit, Aml	oarish 0	-5893-1	
Banerjee, Andrew A. Tubb, Le	i Niu			
9. Performing Organization Name and Add	ress	10	). Work Unit No. (TRAIS)	
Texas Tech Center for Multidis	ciplinary Research in	1		
Transportation		11	. Contract or Grant No.	
Texas Tech University		0	-5893	
Box $41023$				
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Texas Department of Transport	ation	T	echnical Report and Pe	nou Covereu
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Austin TX 78763-5080		14	I. Sponsoring Agency Co	de
1145011, 111 / 0 / 05 0 000				
15. Supplementary Notes	:4 4 <b>T D</b>		1.1	
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Highway Administration.				
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which EA will achieve stiffness and h	ond strength with aggreg	ate to be able to oper	the road for traffic.	This research
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others. This delay also depends on th	e climatic conditions. Th	ne ASTM D7000 Swo	eep Test, used in other	states to determine
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field test that determines mass loss an	d calculates the dilution	ratio in EA. This test	showed commendabl	e repeatability of
results. The second field test used Shell cups to determine the Saybolt-Furol Viscosity (SFV) of the binder. The				
repeatability of the test improved drastically when a water bath was introduced to control specimen temperature. Finally, six				
construction projects were used for field evaluation of the new field tests and to calibrate the statistical models that were				
developed. For the prediction model	s to be ready for widespr	ead use, additional te	sts are needed to incre	ease the model
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by

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> Research Project 0-5893 Research Report 0-5893-1

# Texas Tech Center for Multidisciplinary Research in Transportation Texas Tech University

# Center for Transportation Research University of Texas

Performed in Cooperation with the Texas Department of Transportation and the Federal Highway Administration

February 2012

Report 0-5893-1

# Acknowledgments

The authors thank the TxDOT Project Monitoring Committee (PMC) for their valuable guidance and suggestions during this research project. Special mention should be made regarding the efforts by Darlene Goehl P.E., Project Director from Bryan District and Dr. German Claros, RMC-1 Research Engineer of the Research & Technology Implementation Office for their leadership on this project, and of Gerald Peterson P.E. of the Construction Division for his support and accommodation of various testing activities at the Cedar Park Bituminous Binder Laboratory. Tracy Cumby, formerly with Lubbock District started as the Project Director for this study, but had to step down in the early part of the research study due to his new and expanded commitments in the Western Regional Office of TxDOT. The research team thanks him for his leadership in the early part of the study.

The research team also thanks Larry Miller, Carmen Iglehart, Rhonda Rowdy and Darell Vogsley, Kim Harris and Rita Schumacher for their help and support. In addition, employees of TxDOT Districts in Austin, Beaumont, Brownwood, Lubbock, Paris and San Antonio provided valuable support for the field portion of this research and their efforts are much appreciated. The authors also extend special appreciation to Cody Chambliss, Mac Shelby and Randy King who helped us organize the field monitoring program that was key to the success of this project,

Acknowledgements
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# **Chapter 1. Introduction**

## 1.1 Background

TxDOT depends greatly on sprayed seals for new road construction (surface treatments) and on preventive maintenance measures (seal coat). Procedures for pavement maintenance and preservation have shown to be successful and effective when done properly. TxDOT specification Item 316 defines surface treatments as one or more applications of a single layer of asphalt material covered with a single layer of aggregate. This treatment is typically called "chip seal" in other parts of the country. TxDOT also uses some other common treatments such as fog seal, prime coat, and tack coat. These treatments may be applied with a variety of materials, but in particular, experience has shown that emulsified asphalts, and to a lesser degree cutback asphalts, are very sensitive to the construction process and the conditions at the time of treatment application. The purpose of this research was to investigate the constructability issues of seal coat and surface treatment binders and develop guidelines for more effective binder use. At the beginning of the study, the research team was directed by the Project Monitoring Committee (PMC) to focus primarily on emulsified asphalt binders used in seal coats.

A typical surface treatment or seal coat construction is a simple and straightforward process. Nevertheless, their performance over the years has indicated some significant failures. These can be attributed to complexities arising from factors such as material selection, quality of materials, application of materials under undesirable conditions, unforeseen climatic events during construction and post-construction performance conditions. Senadheera et al. (2007) conducted a comprehensive constructability review of seal coat and surface treatment practices in research projects 0-1787 and 0-5169 respectively, and highlighted that a key factor in successful emulsion seals is the time lapse required between binder and aggregate applications.

Emulsified asphalt is a homogeneous, stable colloidal suspension of asphalt in water. In Texas, emulsified asphalts are specified in TxDOT Item 300, which includes limits to assess the quality of rapid-, medium-, and slow-setting emulsions. This refers to the rate at which the emulsion breaks, which can also be considered as an indicator of overall stability, or "pot-life," of the emulsion. The choice of emulsion for each application is a question of matching the reactivity of the emulsion with the reactivity of the aggregate and the environmental conditions. Aggregate reactivity is mostly associated with the very finest-size fractions which make the highest contribution to surface area. So a reactive rapid-setting (RS) emulsion is used with the low-surface area nonreactive aggregates used in chip seal, whereas a low-reactive slow-setting (SS) emulsion would be used for a dense cold mix which has a high content of very fine material passing a #200 sieve and, consequently, high reactivity. Environmental conditions also have to be taken into account. High temperatures accelerate the chemical reactions and physical processes involved in emulsion setting and, therefore, demand slower setting emulsions.

Curing of an asphalt emulsion, as stated by Holleran and Motina (2006), is the steady loss of water from the system and the stiffening of the total seal as cohesion increases. The

end result of the curing phase is a continuous cohesive film that holds the aggregate in place with a strong adhesive bond. For this to happen the water must completely evaporate, and the asphalt emulsion particles must coalesce and bond to the aggregate. Water evaporation can be fairly fast under favorable weather conditions, but high humidity, low temperatures, or rainfall can affect proper curing. The breaking of an asphalt emulsion is the flocculation and coalescence within the emulsion and the reaction with the aggregate surface (Holleran and Motina, 2006). The main mechanisms are:

- Closer proximity of the asphalt globules to each other, caused by evaporation of water
- More frequent contact between asphalt globules, caused by shear-induced motion
- Aggregate chemical interaction with the emulsion.

Asphalt emulsions are affected by storage and handling, which makes proper sampling and handling very important. The purpose of any sampling is to obtain samples that represent the true nature and condition of the material. The standard procedure is detailed in ASTM D 140, "Standard Methods of Sampling Bituminous Materials" (ASTM, 2007). The following precautions should be taken into account when sampling emulsified asphalt:

- Avoid contamination to ensure a representative sample
- Practice safety precautions, including the use of personal protective equipment
- Proper sample identification
- Timely delivery of samples to the laboratory (same day if possible) to protect and preserve the material properties as they are at the time of sampling.

TxDOT field personnel working on seal coat and surface treatment activities involving emulsified asphalt have reported the following construction-related problems.

- Emulsion curing period
- Breaking and curing variability
- Storage and transport instability
- Varying construction climatic conditions
- Improper dilution when diluted emulsion is used
- Improper binder application rates
- Not updating binder rates after recent changes in surface treatment design procedures

TxDOT specification item 300 specifies the numerous tests conducted on seal coat and surface treatment binders. The list of test methods identified in this specification is comprehensive. However, there are two issues that must be addressed in order to improve the quality of their construction. They are;

• Quality management (QC/QA) of surface treatment binders

• Testing of binders under conditions similar to those encountered during construction and service

There are significant challenges in addressing the two issues identified above. The aim of this research project was to develop improved testing and evaluation protocols for emulsified asphalt used in seal coats and surface treatments to ensure their quality as received in the field, and to adjust construction procedures according to emulsion constructability factors identified above.

In order to achieve the main research objective of investigating the constructability issues of emulsified asphalt binders in seal coats, the following technical tasks were identified in the work plan.

- 1. Literature Search and State-of-Practice Survey
- 2. Field/Lab Evaluation of Binder Curing/Breaking
- 3. Characterization of Binder Breaking/Curing Trends
- 4. Develop a Field Test to Assess Emulsion Quality
- 5. Evaluation of Emulsion Sampling/Handling Processes
- 6. Field Evaluation Program
- 7. Develop and Deliver a Training Course

The two research teams conducting this study, one from the Texas Tech University's Center for Multidisciplinary Research in Transportation (TechMRT) and the other from the University of Texas at Austin's Center for Transportation Research (CTR) divided the primary responsibility for each task. The two teams had joint responsibility for Tasks 1, 2, 6 and 7. The CTR team had primary responsibility for Tasks 3 and 4 whereas TechMRT had primary responsibility for Task 5. In this report, findings from Task 1 are reported in Chapter 2. Even though a State-of-Practice survey was included in the proposal, it was decided that findings from previous TxDOT research studies on the subject will be used instead of doing a new survey. Findings from Tasks 2 through 7 are described in Chapters 3 through 8 respectively. Chapter 9 outlines the key conclusions and recommendations from the findings. The two research products, the Binder Construction Toolkit (P1) and the Regional Training Material (P2) are included in Appendices A and B of the report respectively.

# Chapter 2 Constructability Issues of Emulsified Asphalt in Surface Treatment Applications

#### 2.1 Introduction

An emulsion is produced by scattering small droplets of one liquid into another liquid. Examples of emulsions in our day-to-day life are milk, butter, margarine, and paint. Emulsions are of different types (Figure 1): oil in water, water in oil, or multiple emulsions. In oil in water, water is in continuous phase and oil is in dispersed phase, whereas in water in oil, it is vice versa. In multiple emulsions, the disperse phase contains another phase which may not have the same composition as that of continuous phase (Saloman 2006).

Asphalt emulsion is formed due to the suspension of asphalt droplets (oil) into water (oil in water emulsion). It is the distribution of small asphalt particles in water, in which water acts as a continuous phase and asphalt particles are the dispersed phase. The distribution of particles and particle sizes in the emulsions is influenced by the machinery and procedures used at the manufacturing plant. The particle size and distribution of the emulsion droplets will affect some of the physical properties of emulsions like viscosity and storage stability (Furlong et al. 1999). The main ingredients of asphalt emulsion are asphalt, water, and emulsifying agent. Standard grade asphalt emulsions contain approximately 40-75% asphalt, 25-60% of water, 0.1 to 2.5% emulsifying agents and other minor components. Asphalt particles are in the range of 0.1-20 microns in diameter. Emulsions within this range of particle size are called macro asphalt emulsions.



FIGURE 2.1 Types of Emulsion (Saloman 2006)

The asphalt droplets in the emulsion have a small charge. The source of the charge is the emulsifier, as well as ionizable components in the asphalt itself. These small charges on the droplets normally provide an electrostatic barrier to their close approach to each other. However, when two droplets achieve enough energy to overcome this barrier and approach closely, they adhere to each other (flocculate). This flocculation may sometimes be reversed by agitation, dilution, or addition of more emulsifier. Over a period of time the water layer between droplets in a floccule will thin and the droplets will coalesce. The coalescence cannot be reversed. Factors which force the droplets together such as settlement under gravity, evaporation of the water, shear or freezing will accelerate the flocculation and coalescence process, as does anything which reduces the charge on the droplets. Lower viscosity asphalts coalesce more rapidly than high viscosity asphalts. Of course, eventually the emulsion droplets must coalesce after the asphalt emulsion has come in contact with the aggregate and placed on the roadway.

#### 2.2 Classification of Asphalt Emulsions

Asphalt emulsions are classified, based on the charge of the asphalt droplets, as anionic emulsion, cationic emulsion, and non-ionic emulsions. The charge of the particles is mainly due to the chemical nature of the emulsifying agents (surfactants) and ionizable components in the asphalt itself. Emulsifying agents are the chemicals used to induce electrical charge to asphalt particles. Emulsifying agents also keep the emulsion in stable condition and controls the emulsion setting and breaking mechanism. It acts as a surfaceactive agent and it contains large organic molecules with two different parts called head and tail. The head portion behaves as a polar hydrophilic component, and the tail portion as a non-polar lipophilic. Anionic and cationic classification mainly depend upon the charge that the head group adopts in water. Emulsified asphalts are categorized into three groups based on how fast they set or break: rapid setting (RS), medium setting (MS) and slow setting (SS). HF signifies a designation of a high float emulsion, which is anionic and specifically meet the criteria of the float test (AASHTO T-50 or ASTM D-139). Specifications for anionic and cationic emulsions are given by AASHTO and ASTM standards. Anionic emulsions (ASTM D 977) are named as RS, MS, SS, and three cationic emulsions (ASTM D 2397) starts with CRS, CMS, and CSS. Emulsions are subdivided into a series of numbers and alphabets "h and s" indicating viscosity of the emulsion and the hardness of the base asphalt cement (TAI 1989). Numbers 1 and 2 are used to designate the viscosity of the emulsion and the letter "h" designates harder base asphalt while letter "s" designates soft base asphalt. These anionic and cationic emulsions can be modified with polymers for better results in the field. These modifications are called polymer modified emulsions.

#### 2.2.1 Anionic Emulsions

Anionic nature is due to the presence of anionic emulsifying agents in the emulsion. Anionic emulsifier contains fatty acids, which react with bases such as caustic potash or caustic soda by forming a salt. Formed salt is the active emulsifier. The hydrophobic nonpolar tail aligns itself inward to the asphalt. The head part contains a group of atoms that chemically have positive and negative charge areas. The head is polar due to the presence of these two charged areas, and is soluble in water because of this polarity. In anionic emulsions, the positive head portion floats in the water by leaving the rest of the head negatively charged at the surface of the droplets (TxDOT, Undated). This conveys a negative charge to all the droplets. As like charges repel each other, all the droplets stay as individual asphalt droplets in the emulsion. Figure 2.2 shows the orientation of anionic emulsifying agent. Figures 2.3 and 2.4 show the asphalt particle in the anionic emulsion and the action of the emulsifying agent in imparting the net negative charge to the asphalt particle.

FIGURE 2.2 Orientation of Anionic Emulsifying Agent (TxDOT)



FIGURE 2.3 Anionic Asphalt Particle (VSS 2009)



FIGURE 2.4 Imparting Negative Charge to the Asphalt Particle (TxDOT)

#### 2.2.2 Cationic Emulsions

Cationic emulsions contain cationic emulsifying agents. The positive charge of the droplets is due to flotation of the negative part of the head in the water by leaving the positively charged head. This gives positive charge to all the droplets (TxDOT). As like charges repel each other, all the asphalt droplets remain as separate droplets in the suspension. Figure 2.5 shows the chemical structure and orientation of the emulsifying agent at the asphalt water interface and the positive charge imparted to each group (TxDOT). Figures 2.6 and 2.7 show the asphalt particle in cationic emulsion and the action of the emulsifying agent in imparting the net positive charge to the asphalt particle.



FIGURE 2.5 Orientation of Cationic Emulsifying Agent (TxDOT)



FIGURE 2.6 Asphalt Particle in Cationic Emulsion (VSS 2009)



FIGURE 2.7 Imparting Positive Charge to the Asphalt Particle (TxDOT)

#### Chemical Nature of Asphalt Emulsifiers

Asphalt emulsifiers are most commonly made up of renewable sources like natural fats, oils and wood. Due to the presence of both hydrophilic and lipophilic portions in their molecules, the emulsifiers will concentrate at the interface between water and asphalt. This reduces the energy required to emulsify the asphalt and prevents the coalescence of the droplets. Most emulsifiers contain a hydrophilic head group and lipophilic tail with 12-18 carbon atoms. They are classified into anionic, cationic, and non-ionic based on the dissociation of the head group in water. The charge of the asphalt droplets mostly depends on the charge of the head group. The size and sign of the charge on the droplets can be expressed as the Zeta potential of the droplet.

#### Polymer-Modified Emulsions

To achieve better results against temperature flexibility, fatigue resistance, and tensile strength many agencies recommend use of emulsions modified with polymers. Polymers are substances whose molecules have high molar mass and contains large number of repeating units called Monomers. Two types of polymers are used for modification of asphalt emulsions. They are elastomers and plastomers. An elastomer has a flexible rubber backbone and large side chains in its structure. Hence they are elastic in nature so that they recover their shape when stretched. Some examples for elastomers are styrene butadiene block copolymers (SB, SBS), styrene butadiene rubber latex (SBR), and natural rubber latex. A plastomer will deform into a plastic or viscous manner at melting temperatures and becomes hard at low temperatures. Plastomers have high modulus and act as a stiff material. Some examples of plastomers are ethylene vinyl acetate copolymer (EVA), polyethylene and polyolefin. Emulsions and polymers can be mixed thoroughly with the pre-blending process and can be tested and certified before application to the aggregate. The polymer additives do not change the chemical structure and chemical properties of the asphalt cement. Polymer modified emulsions are used to enhance the performance and durability, reduce the lifecycle cost, and to impart some early strength to the cold mixes. They only change the physical characteristics of the asphalt binder such as softening point and brittleness (Baughman 2008).

#### Asphalt Emulsion Manufacturing

The emulsions can be manufactured by mixing hot asphalt and water containing emulsifying agents, with the application of sufficient mechanical energy to break up the asphalt cement into droplets. A high-speed, high-shear mechanical device (colloid mill) is generally used to break up the asphalt into droplets. The colloid mill contains a rotor and stator. Figure 2.8 shows the schematic diagram of the asphalt emulsion manufacturing plant. The rotor contains teeth and it will be grooved to create a turbulent flow. The rotor revolves at a speed of 17-100Hz (1000-6000rpm), which produces asphalt droplets ranging from 0.001-0.01mm. Particle size analyzers are commonly used to characterize emulsion quality. Asphalt droplet sizes depend upon the mechanical energy density imparted by the mill. High energy input and a low asphalt viscosity results in smaller droplets. The heating temperatures of asphalt and emulsifying agents depend on the attributes of asphalt cement and the compatibility between asphalt cement and the emulsifying agent. A heat exchanger is used to reduce the temperature of emulsion leaving the colloid mill. Separate pumps are used to meter asphalt and the emulsifier solution into the colloid mill. Because the emulsifier solution can be highly corrosive, it may be necessary to use equipment made of corrosion-resistant materials. The produced emulsion is stored in storage tanks. In some situations solvents, such as gasoline and petroleum, can be added to provide fast, tardy, and slow hardening. Additives used in the manufacture of emulsified asphalt can vary between different manufacturers. The properties of emulsions also vary with different additives.

#### The Emulsifying Process

In the emulsifying process, heated asphalt is fed into the colloid mill where it is divided into tiny droplets. At the same time, water containing the emulsifying agent is fed into the colloid mill. The asphalt entering the colloid mill is heated to a low viscosity, and the water temperature is also adjusted to optimize emulsification. These temperatures vary and depend upon the emulsification traits of the asphalt cement and the compatibility between the asphalt and the emulsifying agent. Extremely high asphalt temperatures are not used because the temperature of the emulsion leaving the mill must be below the boiling point of water, unless a heat exchanger is used to cool the emulsion. The emulsion is then usually pumped into bulk storage tanks. These tanks may be equipped with mechanical agitation to keep the emulsion uniformly blended.



FIGURE 2.8 Asphalt Emulsion Manufacturing Plant (VSS 2009)

The methods of adding the emulsifier to the water vary according to the manufacturer's procedure. Some emulsifiers such as amines must be mixed and reacted with an acid to be water soluble. Others, such as fatty acids, must be mixed and reacted with an alkali to be water soluble. Emulsifier mixing is typically done in a batch mixing tank. The emulsifier is introduced into warm water containing acid or alkali and agitated until completely dissolved.

Asphalt and emulsifier solution must be proportioned accurately. This is normally done with flow meters, but monitoring the temperature of each phase and the mill discharge can also control the proportioning. If temperature regulation is used, the desired outlet temperature of the finished emulsion is calculated from the various emulsion ingredients and then used to control the asphalt content percentage.

Asphalt particle size is a vital factor in making a stable emulsion. These microscopic asphalt droplets are dispersed in water in the presence of the chemical surface active emulsifier (surfactant). The surfactant causes a change in the surface tension at the contact area between the asphalt droplets and the surrounding water, and this allows the asphalt to remain in a suspended state. The asphalt particles, all having a similar electrical charge, repel each other, which aids in keeping them suspended.

#### Tests for Asphalt Emulsions

#### Tests on Emulsions

Sound assessment of asphalt emulsion properties greatly help in selecting emulsion for the appropriate use. These properties can be measured using standard tests, which are specified in ASTM D 244 and AASHTO T59. The most commonly used tests are viscosity, storage stability, demulsibility, particle charge and sieve tests. These tests are used to find data for specification requirement, control the quality and uniformity of the product during manufacturing and use, and to predict and control the handling storage and field performance properties of the material.

The particle charge test is used to classify an emulsion, but only to find the cationic emulsions. The particle charge test is conducted by immersing electrodes in an emulsion sample, by connecting two electrodes with a controlled direct current electrical source. After a period of time, if the cathode has an appreciable deposit of asphalt, the emulsion is to be determined as cationic emulsion. This test is illustrated in the AASHTO Designation T59-01 and ASTM D244-95. Generally asphalt emulsion viscosity is measured with the Saybolt Furol viscosity test. The test temperature is varied depending on the type of emulsion. 25 °C will maintain to measure the viscosity of slow set emulsions, and 50 °C is for rapid setting emulsions. Results are reported in Saybolt Furol seconds (TAI 2004). To determine the rate of breaking of rapid setting emulsions when applied on aggregate and soils, the demulsibility test is conducted. The rate of breaking is

measured by adding cacl2 to the asphalt emulsion. This solution is thoroughly mixed with the asphalt emulsion and then allowed to pass through a No. 14 wire cloth sieve. This cloth preserves the asphalt particles that have coalesced. The amount of asphalt retained on the sieve is the measure of coalescence that has occurred. The demulsibility is expressed as (A/B) x100, where A is average weight of residue from three tests, and B is the weight of residue by distillation in 100g of the emulsified asphalt. The sieve test is used to determine the quality and stability of the emulsions. In this test the sample solution is passed through a No. 20 sieve and determines the percentage by weight of the material retained on the sieve. An excessive amount of asphalt material is retained on the sieve indicates that problems may occur in the application and handling of the material. The storage stability test is used to find the emulsion stability in storage. It allows a recommended volume of asphalt emulsion to stand in a graduated cylinder for a specific time. After collecting the two samples of emulsions from the top of the tank and bottom of the tank, they are placed in a beaker, weighted, then heated to evaporate water. The residue is then weighed, and these weights are used to find the difference between the samples of the upper and lower portions of the cylinder, which give the stability test results. Some agencies follow a 24-hour time period and some follow a 5-day period.

Coating ability and water resistance tests are used to determine the ability of a medium setting emulsion to check its ability to coat the aggregate, remain as a film on the aggregate during mixing, and resist the washing the action of water after mixing. This test is not suitable to rapid setting and slow setting asphalt emulsion. This is mainly to find the suitability of medium setting asphalt emulsions to mix with the coarse graded aggregate. The prescribed aggregate is coated with calcium carbonate dust and then mixed with the asphalt emulsion. About half the mixture is separated and placed on an absorbent paper, where the surface of the aggregate is observed for coating by the asphalt emulsion. The remaining mixture is sprayed with water and rinsed. Later this material is also placed on absorbent paper for inspection of the coating. The test is repeated and in this process the aggregate is coated with water instead of calcium carbonate dust, mixed and then inspected for the coating ability.

Choquet (1994) presented the European (RILEM) test method to measure the breaking index of emulsified asphalt. The method is similar to the TxDOT standard test method Tex-542-C, except that a mechanical mixer is used instead of manual mixing. The results from the RILEM test are used to rank the emulsions as rapid-setting when the breaking index (I) is from 65 to 80, medium-setting from 80 to 100, and slow-setting when I exceeds 100.

Asphalt binder residue is used to determine the quantity of the asphalt binder, which is used to examine the physical properties of the asphalt binder. Residue can be found either by distillation procedure or by evaporating procedure. In the distillation method asphalt, water, and oil are separated by heating the emulsion in an aluminum alloy still at a temperature of 260 °C for 15 minutes. In the evaporation method residue can be obtained by evaporating the emulsion in an oven at 163 °C for 3 hours.

#### Zahn Cup

The General Electric (or "Zahn") viscometer consists of a bullet-shaped steel cup with a small orifice in the bottom (Viswanath et al. 2007). The Zahn viscosity cup (Figure 9) is also known as Dip-Type viscosity cup because of the manner in which viscosity of a test sample is determined. It can be used to determine the viscosity of Newtonian paints, varnishes, lacquers, ink, and related liquids. For measuring viscosity, the cup is completely immersed in the liquid, withdrawn (hence its category "dip-type"), and the time for the flow through the orifice is measured. Although this type of cup can be used to measure viscosity cups are available in five sizes that can measure the oil viscosity in the range of 20 to 1600 centistokes. The cups are made of corrosion and solvent resistance materials. The volume of a cup can vary from 43 to 49 ml depending on the manufacturer.

Viscosity measurement using Zahn viscosity cups is generally made at 250 °C. For viscosity determination at other temperatures, a temperature correction curve or factor is determined for each liquid. The choice of cup for measuring viscosity depends on the efflux time which should be between 20 to 80 s. The cup is immersed in the container and is kept there for one to five minutes to allow thermal equilibration. The cup is lifted vertically from the container in a quick, steady motion. As the top edge of the cup breaks the surface, the timer is started and the cup is held about 6 in (15.2 cm) above the level of the liquid. When the liquid stream breaks at the base of the cup, the timer is stopped and the efflux time in seconds is noted. Zahn viscosity cups should be calibrated periodically, according to Test Method ASTM 4212-99 using standard fluids. However, it should be noted that the temperature control of Zahn viscosity cups is extremely difficult and this introduces errors in the calibration.



Figure 2.9 Zhan Cup

Shell viscosity cups are also Dip-Type viscosity cups and are available in eight sizes. They are made of stainless steel with a capacity of 23 ml and a 25-mm long capillary orifice at the bottom. A typical design of a Shell Viscosity cup is shown in Figure 2.10 below. The orifice size and the recommended size depend upon their intended use. The operational procedure and calibration method is same as that of Zahn viscosity cups.



Figure 2.10 Schematic of Shell Viscosity Cup

#### Tests to Examine the Asphalt Residue

Specific gravity, solubility, penetration, ductility and float tests are the common tests to run on the asphalt residue. Specific gravity of the asphalt binder can be determined by using displacement methods in water. Specific gravity does not determine any values, but it indicates the quality of the residual binder. Solubility test is used to measure asphalt portion of the asphalt residue. It is determined by dissolving the asphalt cement in trichloroethylene and filtering the soluble and insoluble components. The soluble portion in trichloroethylene represents genuine amount of asphalt binder and insoluble part represents the inorganic contaminants. Hardness of the residual binder is measured by conducting the penetration test. In this test we can measure penetration depth of a standard needle under a load of 100g for five seconds at a temperature of 25 °c. Ductility test measures the consistency of the asphalt binder. This test will be done by molding a briquette of asphalt cement under standard conditions and dimensions. The asphalt briquette is then placed in a water bath at specified test temperatures. It is pulled at a specified rate of speed until the thread connecting the two ends break.

#### Environmental Effects of Emulsions

Environmental Risk Management Authority conducted some experiments and stated some sense for the environmental danger with the continuous use of asphalt emulsions

(Ball et al. 2008). Emulsions are not classified as dangerous but they are biodegradable and can act as a waste pollutant. They are soluble in water and thus kept away from natural sources like dams, streams etc. The major potential environmental dangers of emulsion sealing in New Zealand are perceived to be, Spillage and/or runoff into waterways from tanker accidents or mishaps during spraying and Rain washing emulsion off the road before it has had time to set.

#### Accidental Spillage

Accidental spills occur during transportation or sealing. This type of failures can be avoided by taking some precautions such as blocking access to drains and water ways before sealing of emulsion. Sometimes spills result from tanker rollovers, but this is very rare case and the probability of this type of occurrences is 0.3% per year.

#### Storm Water Runoff

If a storm occurs before the emulsion has fully cured, the residual waters may be washed off by the rain into the surrounding soil or into the storm water system. The emulsifying agent is the only component that contributes to the Eco toxicity. The toxic danger is mainly due to the compounds present in the aqueous phase. Land Transport in New Zealand has conducted some experiments to investigate the effect of the emulsifier in determining the eco toxicity of the aqueous phase. They have considered four different kinds of emulsifiers for the testing. Based on some standards the Environmental Risk Management Authority (ERMA) has four categories for aquatic Eco toxicity, and the test result has been reported by using this classification. The classification is given in Table 2.1.

Category	Description
9.1A	Very ecotoxic in the aquatic environment
9.1B	Ecotoxic in the aquatic environment
9.1C	Harmful in the aquatic environment
9.1D	Slightly harmful to the aquatic environment or otherwise designed for
	biocidal action

 Table 2.1 New Zealand ERMA Aquatic Eco-Toxicity Categories (Ball et al. 2008)

Cationic emulsifying agents are highly toxic in nature. A calculation based on the published Eco toxicity data of an emulsifier, using an ERMA formula for mixtures suggested that emulsifier aqueous phases would fall into ERMA eco-toxicology Class 9.1C (eco-toxic in the aquatic environment), if it is assumed that the No Observed Effect Concentration (NOEC) is less than 1 mg/L, or into Class 9.1D (slightly harmful to the aquatic environment or otherwise designed for biocidal action).

## Setting and Curing Processes of Emulsified Asphalt

To accomplish their role as a binder for road materials, the asphalt emulsions should transform into a continuous asphalt film. The stability, and hence the success of an emulsified asphalt binder relies heavily on its ability to form films on aggregates. After the application, water needs to evaporate by leaving the asphalt cement on the applied surface by completing setting and curing process of emulsion. Setting and curing are two close related procedures. The flocculation of asphalt binder droplets is called setting, and the coalescence of asphalt droplets and the formation of a continuous adhesive layer of asphalt binder (through breaking) on the surface is called curing.

Film formation involves coalescence (without entrapment of water) of asphalt into a continuous film. Film formation is a function of kinetic factors such as temperature, viscosity, internal stability and thermodynamics of the system. Films form more slowly at low temperatures, with larger particles and at higher viscosities. Addition of solvents or coalescing agents will assist film formation, especially at low temperatures. Adhesion agents, doped into the asphalt, can also enhance adhesion (Hooleran, 1999).

The mechanism of chemical interaction between emulsified asphalt and aggregate is not fully understood, but the following general description appears to represent agreement among most researchers. When asphalt droplets come close to each other, the charge on the droplets prevents close approach of the droplets which could lead to flocculation. Over a period of time the water between the droplets will be squeezed out and the asphalt droplets will coalescence to each other. This process is called curing or coalescence (Saloman 2006). The water can be removed by evaporation, rolling, by absorption into the aggregate and weather conditions. Rapid setting emulsions will break quickly, where as medium and slow set emulsions may set after a long time. Figure 11 will show the sequence of setting and curing of the asphalt emulsions.

The stable emulsified asphalt particles are attracted to the surface of the aggregate, where the emulsifier from the bulk solution interacts with the aggregate surface charges. This changes the equilibrium of the emulsified asphalt system, and it is destabilized and the charge on asphalt particles is neutralized. This leads to deposition of asphalt on to the aggregate surface. The rate of film formation will determine the adhesion, and the emulsifier formulation will determine the breaking rate. The internal flocculation and coalescence will also have an effect. Within the forming film, the particles are in closer proximity and these mechanisms will accelerate. However, if this rate is too fast, water will be trapped and the binder film forming capability will be diminished. The bulk part of the emulsion remote from the aggregate surface will break by flocculation and coalescence. In seal coating, emulsions with high binder contents will accelerate the break in the bulk emulsion (Hooleran, 1999).



#### FIGURE 2.11 Stages in the Breakdown of the Emulsions (Saloman 2006)

The "curing" of an emulsion film is often confused with its breaking. Curing is simply the loss of water from the film and bulk emulsion. Therefore, curing rates depend on water content, rate of evaporation and diffusion of water through the curing binder. In systems with strong energy differences between the aggregate surface and the emulsified binder, an extra driving force is needed to push water away from the aggregate-binder interface. Cement is often used to enhance this phenomenon. The electrical charge on the aggregate surface may depend on the aggregate type and its mineral composition. Since both cationic and anionic emulsions are available, emulsifier choice relative to aggregate is a key issue (Hooleran, 1999).

Generally, the properties affecting the characteristic of emulsions are the type and content of binder, asphalt particle size distribution, and composition of the aqueous phase. Deneuvillers and Samanos (1999) studied the correlation between characteristics and properties of cationic bitumen emulsions in order to improve their behavior in the field. This systematic study investigated the effect of constituent parameters on emulsion properties. It also quantified the influence of median droplet size and standard deviation on three important properties, viscosity, breaking rate, and rate of cohesion building. The researchers showed that by controlling the standard deviation of droplet size, it is possible to control viscosity and the rate of cohesion build up. These two properties are particularly important for surface treatments. High viscosity prevents runs, and a fast rise in cohesion means the newly-dressed road can be opened to traffic sooner, with a reduced risk of aggregate loss (Deneuvillers and Samanos, 1999).

They correlated the breaking index of emulsions to the median diameter of their particles and found that, for slow-breaking emulsions, the breaking index decreased with the increasing median diameter. This was explained by the fact that, as the median diameter decreases the specific surface increases, and so does the number of bitumen particles. Deneuvillers and Samanos (1999) concluded that it takes more filler to make contact with all the particles and break all of the emulsion. For a given emulsion, the breaking index increases as the median diameter of the filler increases, for which the explanation is the same (it takes more filler with a smaller specific surface to break a given quantity of emulsion). The researchers also found that the relationship between breaking index and specific surface is not linear. They affirmed that the relationship between the developed surface area of the emulsion at the time of breaking and the breaking index is not constant. The specific surface is not the only parameter involved. Steric particle overcrowding is probably the cause of this phenomenon.

The properties of base asphalt used in an emulsion can have a significant influence on the properties of emulsified asphalt. Therefore, a sound knowledge of the asphalt cement composition and its effect on emulsions is important in producing good emulsified asphalts. Emulsion properties and stability are largely bulk properties, but the emulsion can be improved by creating finer particle size distributions. This makes interfacial properties more important and provides a chemical solution to stability problems. Polymer addition improves rheological properties of the binder, and processing and compatibility also play important roles in emulsion morphology and performance (Hooleran, 1999).

The desorption process of a surfactant molecule adsorbed on the bitumen surface influences not only the electrokinetic properties of bitumen particles under diluted conditions, but also the reactivity of emulsion during the breaking process. During mixing with aggregates, the desorption rate of the surfactant from bitumen, and the adsorption of bitumen on aggregate, could provide the kinetic control of the process. Moreover, surfactants with different reactivities with aggregates may desorb at different rates to different equilibrium states. The surfactant that leads to rapid setting (RS) emulsions desorbs from bitumen surface rapidly while the less reactive surfactant is attached to the stronger surface (Paez et al., 1999).

#### Setting (Breaking) in the Presence of Aggregate

If the asphalt emulsion is to perform its ultimate function as a binder, the water must separate from the asphalt phase and evaporate. This separation is called "breaking". For surface treatments and seals, emulsions are formulated to break chemically upon contact with rapid-setting and medium-setting emulsions; the initial placement of the asphalt emulsion droplets on the aggregate develops through electrochemical phenomena. For slow-setting emulsions, the mechanism is water evaporation. For dense mixtures, more time is needed to allow for mixing and placement. Therefore, emulsions used for mixtures are formulated for delayed breaking. A rapid-set emulsion will have a short breaking time (within 1 to 5 minutes after being applied), whereas a medium- or slow-set material may take considerably longer.

The type and concentration of emulsifying agent primarily control the rate of breaking. However there are other factors playing important roles in breaking of emulsion. In order to achieve optimum results, it is necessary to control all of the factors to meet the specific requirements of the field use of the asphalt emulsion. The supplier should have more information regarding the optimum use of its emulsion.

The formation of a continuous asphalt film from an emulsion involves flocculation and coalescence of the droplets and removal of water. Evaporation and absorption of the water by the aggregate may be the main breaking mechanism for very slow-setting emulsions, but in most cases, interactions between the aggregate and the emulsion contribute to the emulsion setting and it is not necessary for all the water to evaporate before curing takes place. The strength of interaction of emulsion with aggregate is in many cases sufficient to squeeze the water from the system. Clean water can be seen separating from the mixture. The speed of these setting and curing processes depends on the reactivity of the emulsion, the reactivity of the aggregate and environmental factors, such as temperature, humidity, wind speed, and mechanical action. Less viscous asphalt tend to give faster coalescence. It may take a few hours in the case of a chip seal to be reached.

A considerable amount of research efforts has been expended to clarify the mechanism of setting and curing of asphalt emulsion. Important factors are changes in pH caused by reaction of the aggregate with acids in the emulsion, adsorption of free emulsifier onto the aggregate surface, and flocculation of the emulsion droplets with the fines. The relative timescale of flocculation (setting) and coalescence (curing) depends on the system, but in general flocculation is the more rapid process in which some water can be expelled from the system and some cohesive strength develops, followed by a slower coalescence process which results in a continuous asphalt film phase. This asphalt phase must also adhere to the aggregate. Coalescence is an inversion process; the O/W emulsion is transformed into a W/O type which then slowly loses its internal water phase.

This inversion process is favored as the ratio of asphalt to water in the system increases. The tendency for an emulsion to invert can be determined in laboratory tests and has been related to curing behavior in the field.

Aggregates take up a characteristic surface charge in water which depends on the nature of the minerals, the pH, and the presence of soluble salts. So-called "acid" aggregates high in silica tend to take up a negative charge. In the presence of water, aggregates take up some charge depending on the nature of minerals it contains, pH and the presence of some soluble salts. Hence, the breaking mechanism of cationic emulsions and anionic emulsions differ from each other. Most aggregate surfaces possess negative charge (TxDOT Undated). Cationic emulsion consists of small, positively charged asphalt particles suspended in the aqueous phase. As mentioned above, the emulsifier molecule possesses an organic chain of carbon and hydrogen atoms with a number of amine and amide alkyl groups at one end of the chain. Emulsifier molecules at the end of the amine amide groups ionizes positively in presence of acidic environment, and due to the hydrophobic nature of the other end of the molecule it dissolves in the asphalt particles. Thus the positively charged ends point outwards from the asphalt particle surfaces, by giving positive charge to the surfaces. Hence the asphalt droplets have positive charge and the aggregate surface have negative charge. As opposite charges attract each other, the aggregate attracts the emulsifier particles immediately and the breaking process begins. The affinity of cations for aggregate is stronger than its affinity for water. So water is displaced and evaporates. Then the asphalt cement adheres to the aggregate and will act like a bonding agent. Setting process is faster with respect to cationic emulsions than anionic emulsions. A depiction of possible stages in setting of cationic emulsions is shown in Figure 2.12 (Saloman 2006). In case of anionic emulsions, asphalt droplets possess negative charge and the aggregate surface will also possess net negative charge. As like charges repel each other, the setting process may be continued by removing the water through evaporation. As water evaporates, the particles are forced to get closer until they can no longer be separated, and finally they coalesce in to large drops thus forming a sheet of asphalt on the road.



FIGURE 2.12 Possible Stages in the Setting of a Cationic Emulsion (Saloman 2006)
Some aggregates, like carbonates, and fillers, like cement, may neutralize acid in cationic emulsions causing the pH to rise and the emulsion to be destabilized. Anionic emulsions may be destabilized by soluble multivalent ions. We can consider two extreme cases of emulsion breaking. In the case where the charge on the emulsion droplets is quickly destroyed by pH changes, for example, then the emulsion very quickly flocculates and coalescence begins to occur at a slower rate. This rate is dependent on the viscosity of the binder, as well as environmental conditions; coalescence is slower with high viscosity asphalts and lower temperatures. At the other extreme where the emulsion droplets remain charged, loss of water, either by evaporation or by absorption of water into porous aggregate, eventually forces the droplets close enough for attractive forces to predominate, forcing out water and starting the coalescence process. The attractive forces between the droplets can generate significant cohesion even before coalescence occurs.

In a simplified process of the setting of a RS cationic emulsion where the aggregate does not contain significant fines, important stages in the setting process can be considered as follows:

- 1. Free emulsifier adsorbs onto the (oppositely charged) mineral surface, which neutralizes some charge on the surface while at the same time making the surface somewhat lipophilic. Too high a free emulsifier concentration in relation to the surface area of the aggregate can actually reverse the charge on the minerals and so inhibit the setting of the emulsion.
- 2. Minerals neutralize acids in the emulsion, causing loss of charge on the emulsion droplets, leading first to flocculation of the asphalt droplets and then to a slower coalescence of the droplets.
- 3. Water is absorbed by the mineral, as well as evaporates from the system.
- 4. Droplets in contact with the mineral spread on the surface, especially that surface made lipophilic by adsorbed emulsifier, eventually displacing the water film on the aggregate surface.

In the breaking of SS grades, where the aggregate contain high content of fines, Hetero-flocculation of the droplets of asphalt and the oppositely charged fines may occur, which is sufficiently strong to squeeze out water and form asphalt mastic. A similar situation is achieved in micro-surfacing where filler is intentionally added to initiate setting. Mechanical action, such as compaction or traffic, may squeeze the droplets together, promoting coalescence and squeezing water out of the coalesced film.

In practical situations too early coalescence of the asphalt droplets can hinder final curing by skin formation reducing the evaporation of water. Coalescence throughout the asphalt emulsion film, before water is trapped in the system, is promoted by smaller asphalt droplets with narrow size distribution. Too early coalescence of asphalt droplets in some systems can interfere with the formation of a composite binder formed from latex and asphalt, which depends on latex curing before asphalt.

## Factors Affecting the Setting Rate

Setting rate can be affected by aggregate size, asphalt content, droplet size distribution, environmental conditions, and composition of the aqueous phase. The setting or breaking rate can be increased with the increase of asphalt cement content and by reducing the size of the asphalt droplets. Smaller aggregates will break the asphalt emulsion faster than the large aggregates. The rate of breaking can be increased by using the porous and rough textured aggregates. High temperatures and low humidity will increase the breaking and curing rate. The setting rate can be increased or decreased with additives.

According to the Asphalt Emulsion Manual (Asphalt Institute 1999), some of the factors affecting breaking and curing rates of asphalt emulsions include:

- Water Absorption: a rough-textured, porous aggregate speeds up the setting time by absorbing water from the emulsion.
- Aggregate Moisture Content: while marginally wet aggregate may facilitate coating, excessive wetness tends to slow curing process by increasing the amount of time needed for evaporation.
- Weather Conditions: temperature, humidity, and wind velocity all have a bearing on the water evaporation rate, emulsifier migration and water release characteristics. While breaking usually occurs more quickly at warmer temperatures, it is not always the case. Hot weather can cause skin formation on seal coats, trapping water and delaying curing. Some chemical formulations have also recently been developed to expedite breaking at lower temperatures.
- Mechanical Forces: roller tire contact pressure and, to a limited extent slow moving traffic, forces the water from the emulsified asphalt and help attain mix cohesion, curing and stability.
- Aggregate Surface Area: greater the surface area, particularly due to excessive fines or dirty aggregate, quickens the breaking of emulsion.
- Surface Chemistry: intensity of the aggregate surface charge, in combination with the intensity of emulsifier charge, can impact setting rate, particularly for cationic emulsions. Calcium and magnesium ions on the aggregate surface can react with, and destabilize, certain anionic emulsifiers, thus accelerating setting.
- Emulsion and Aggregate Temperature: breaking is retarded when emulsion and aggregate temperatures are too low.
- Type and Amount of Emulsifier: the surfactant used in the manufacture of emulsion determines the breaking characteristics of seal coat and mixing grade emulsions.

## Zeta Potential

Zeta potential is a parameter defined generally to express the mobility of the electrically charged colloidal particles in the emulsion when it is placed in an electrical field. It is used to identify the repellent force between the emulsion particles and evaluate the stability of colloid system.

Figure 2.13 illustrates the concept of Zeta potential in an emulsion. A particle with negative surface charge will attract positive ions and form a tightly bonded layer called a *Stern* layer. With increasing distance from the surface of the negatively-charged particle, both negative and positive ions will be diffused more freely around the particle. This layer of diffused ions is called the *diffuse* layer. However there is a nominal boundary somewhere within the diffuse layer which is called the *Hydrodynamic Plane of Shear*, also known as the *Slipping Plane*. The Slipping plane separates the ions inside from those outside the plane. The plane indicates that ions outside who are too far away from the particle will not move with the particle as a single entity. The ions within will move with the particles as a single entity.



Figure 2.13 Schematic of Zeta Potential Concept

A Potential Energy Curve can be constructed for this scenario which shows several representative electrical potentials. First it has a surface potential, which is the potential of the particle surface. As one moves away from the particle surface, the *Stern* potential

will be applicable. It tells the edge potential of the most tightly bonded ion layer. Further away from the charged particle surface, upon reaching the slipping plane, the potential is referred to as the Zeta potential. This zeta potential value is used to identify the electrical energy level needed for ions to escape from the attractions of the charged particle.

The charge density at the interface between the asphalt particle and the emulsified water medium, as measured by the *Zeta potential*, determines the stability of the emulsion. The thickness of double layer at the interface is determined by the molecular size of the emulsifier, and its ability to dissolve in the water phase (Hooleran, 1999).

The stability of an emulsion is generally interpreted as the ability of that emulsion's particles to hold against coagulation or flocculation with adjacent similar charged particles in the dispersion. For particles that are small enough, a high zeta potential will bring stability and the dispersion will resist aggregation. However, when the potential is low, the attraction force exceeds the repulsion force and the dispersion will break and flocculate. A Zeta potential value can be expressed as "High" both in the positive and negative sides of the scale and Potentials beyond  $\pm 30$ mV would be considered as high zeta potentials.

Zeta potential is highly pH dependent. A Zeta potential versus pH curve will be positive at low pH and lower or negative at high pH. There may be a point where the plot passes through zero Zeta potential value. This point is called the isoelectric point and it indicates where the colloidal system is least stable.

Several different methods and equipments are available to determine the Zeta potential of charged particles. One of the most commonly used principles for this purpose uses the electrophoresis phenomena. When the dispersed charged asphalt emulsion particles are placed in the electrical field, the particles with a certain Zeta potential will migrate towards the electrode of opposite charge with a velocity proportional to the magnitude of the Zeta potential. The zeta potential is calculated using Henry's equation given below.

$$U_E = \frac{2\varepsilon z f(\kappa \alpha)}{3\eta}$$

 $U_E$  = Electrophoretic mobility;

Z = Zeta Potential;

 $\boldsymbol{\varepsilon}$  = Dielectric constant;

 $\eta =$ Viscosity, and

f ( $\kappa \alpha$ ) = Henry's function, usually 1.0 or 1.5 depending on the situation.

It is known that cationic emulsions have been better aggregate compatibility and build stronger interfacial bonds than anionic emulsions. This statement is generally true for

most of the aggregate types, and as a result, it is the more commonly used asphalt emulsion.

Gorman (1998) published Zeta potential values for aggregates used in Australia. Dybaiski (1993) reported Zeta potential values for 87 naturally occurring aggregates obtained from North and South America, Asia, Europe and Africa. Of these 87 aggregates, 31 were limestone and dolomite and 53 were varying siliceous types. For each aggregate, a negative surface charge, with Zeta potentials ranging from +11.91mV to -88.1mV were obtained. Sherwood also published surface charge values for highway aggregates using the streaming potential method.

Paez et al (1999) reported that Zeta potential of bitumen particles decreases with increasing pH and presents a different isoelectric point for each emulsion. They also observed that at higher pH, the Zeta potential is negative as a consequence of negative surface charge density. Their results indicated that the surfactants used to manufacture cationic slow setting (CSS) emulsions have a stronger basic character than those used in cationic rapid setting (CRS) emulsions. In other words, it keeps positive charge at higher pH. The negative surface charge density at high pH is due to the acid groups present in bitumen from natural oxidation processes. Siliceous aggregates have acidic character. The *Zeta potential* of siliceous filler suspended in pure water is negative and is close to -10mV. Decreasing the pH, the potential decreases slightly but remains negative across the whole pH range. In fact, this decrease could be attributed to the increase of ionic strength that the decrease of pH leads to, and not to any specific adsorption. It may be concluded that pH changes do not affect significantly the surface properties of siliceous aggregates (Paez et al., 1999).

The adsorption of surfactants leads to a neutralization of initial negative charge and, after that, adsorption continues up to a Zeta potential close to 50 mV. In spite of the sharper increase on *Zeta potential* with slow setting (SS) surfactants, the saturation potential is the same for both. These results indicate that cationic surfactant adsorption on aggregate surface is not only led by electrostatic interaction between opposite charges, but also by thermodynamically favored adsorption on a positively charged surface. The formation of insoluble salts between siliceous and amino groups agrees with the electrokinetic behavior (Paez et al., 1999).

Paez et al. (1999) also reported that the desorption rate of surfactant used to manufacture CRS bituminous emulsions is higher than the one used to manufacture CSS emulsions. The adsorption of RS-type surfactants on pen 110-130 bitumen particles led to higher Zeta potentials than the SS-type surfactants. They observed that the Zeta potential of bituminous emulsions decreases with pH and that the isoelectric point of CSS emulsion is higher than that of the CRS emulsion. The surface charge of bitumen particles at high pH is negative due to the natural acid groups in the bitumen. The adsorption of cationic

surfactants on siliceous aggregates changes the surface charge from low negative potentials to positive potentials as high as 50 mV (Paez et al., 1999).

Deneuvillers (1999) also conducted research on this subject and reported that adsorption of cationic surfactants at the mineral aqueous solution interface modifies the wetting behavior of mineral surfaces. It was observed that one of the key steps of mineral coating by emulsified asphalt is the transformation of mineral surfaces from hydrophilic to hydrophobic, through the addition of surfactants which results in the hetero-coalescence of bitumen droplets upon the mineral. This operation is based on the selective adsorption of surfactants at the mineral aqueous solution interface, the nature of which will govern adsorption mechanisms. In mineral-cationic surfactant systems, the adsorption phenomena are resulting from physical mechanisms such as electrostatic attraction and hydrocarbon chain association. Electrostatic forces are created by attraction of the positively charged polar groups of the surfactant ion by a negatively charged site of the mineral surface. When the adsorption density of the surfactant ions exceeds a critical concentration (which depends on mineral nature and pH), the hydrocarbon chains of adsorbed surfactants begin to associate themselves at the interface. This association can be in different forms. The electrostatic characteristics of the mineral surface and the nature of cationic surfactant solution and its concentration are two important parameters that will control the adsorption processes of surfactant in the system (Deneuvillers, 1999).

Irrespective of the specific surface of materials, the nature of their surface determines the mechanism of surfactant adsorption. It governs the breaking of emulsion globules when they come in contact with the mineral. In the ideal scenario, when the mineral surface is covered by surfactant molecules and has becomes hydrophobic, the minerals are penetrated by bitumen globules which are also hydrophobic, and the hetero-coalescence begins. For acid type materials, the quantity of surfactant necessary in aqueous phase to make the surface hydrophobic is lower. The coating of this kind of mineral should be done with emulsions that have less surfactant(s). However, it is generally common to use emulsions with high concentrations of surface-active agent, and it assumes that mineral particles are free, and that their surface can be wetted by water. The globule charge is an additional parameter which affects the breakdown mechanism. With basic-type materials, the pH increases rapidly, and the surfactant which is less hydrophilic, will be less soluble in water and will then facilitate hetero-coalescence where the mineral will be almost as hydrophobic as if it was covered by only one layer of surfactant. For maximum effectiveness, the bitumen emulsion formulation should be optimized with regard to the type of mineral to treat while its stability is maintained (Deneuvillers, 1999).

According to Pinilla and Agnusdei (1975), in the case of RS emulsion, the time of setting decreases with increase of the temperature, within the interval 5 °C (41 °F) to 50 °C (122 °F). At low temperatures, siliceous materials result in setting times higher than for

calcareous ones. As temperature increases, differences in setting times decrease for the different aggregates. The time of setting for different aggregates is about the same when the temperature reaches 50 °C (122 °F). The influence of the water vapor pressure that strengthens its influence from temperature could be a reason for this. As the amount of emulsifier in the emulsion is increased, the time of setting also increases within the entire temperature interval considered for any aggregate type. Siliceous aggregates result in the greatest differences. The residual adherence of bitumen on the aggregates is neither influenced by temperature nor by the amount of emulsifier. When SS emulsion is used with fine aggregates similar to that of a slurry seal, the variation of the rate of setting with temperature is more perceptible than in the case of mixtures with the RS emulsion. Using different concentrations of stabilizer agents within the pre-wetting water significantly increases the time of setting when non-pretreated aggregates are used. But, as the temperature increases, the differences decrease to zero at temperatures reaching 30 °C (86 °F) (Pinilla and Agnusdei, 1975).

#### Asphalt Recovery from Emulsion

Button (1994) studied the distillation of polymer-modified asphalt emulsions used in Texas. It has been commonly recognized that the ASTM D244 Standard Test Methods for Emulsified Asphalts alters the rheological and flow properties of the residual binder since the emulsified asphalt will be exposed to temperature as high as 260°C. Button reported that changes to polymer modified emulsions are even more significant. The high temperatures alter the micro-chemical components of the polymer, reduce the original polymer to non-continuous monomers, and thus drastically reduce their viscosity. The researchers set up a plan to test the effects of distillation processes on the base asphalt with and without polymer modification. Viscosity and penetration are measured on the base asphalts and on residues from distillation to determine the influence of high temperature exposure. Button investigated 5 different distillation procedures used with six emulsified asphalts. The residue recovery methods included ASTM D244, Tex-521-C, ISSA, California Test Method 331 and the Rotavapor Procedure. The six emulsified asphalts included 4 polymers modified and 2 non-modified binders. For each polymer modified emulsion, the corresponding polymer-modified base asphalt was tested as a basis for comparison. Emulsions containing no polymer were tested and compared with their respective base asphalts.

Results indicated that the ASTM D244 procedure results in a binder that is softer than the original base asphalt which may indicate that the polymer were broken down or depolymerized during distillation. The smoother texture surface also supported the breakdown of polymer. As the maximum temperature of distillation decreased (ASTM D244 at 500°F, Tex-521-C at 350°F, and ISSA at 280°F), the mean value of the residue/base ratios for these three methods showed that an increasingly harder residue was produced. However, with the ISSA method, the foam on the surface indicated that not all of the water was removed by the process. Except the Rotavapor method, all four

other methods yielded translucent or cloudy distillates. The physical appearance of the ASTM D244 distillate exhibited brown oil floating on top of the cloudy water suggesting that the light oils were distilled from the asphalt. Button (1994) indicated that without performing other tests such as gel permeation chromatography, which gives molecular size distribution, there is no way to tell whether or not the de-polymerization occurred. All five residue recovery methods exhibited wide variability in properties and/or residues with low consistency. This may suggest that neither of five is suitable for recovering polymer modified asphalt from emulsion. It was also indicated that, at temperatures of 280°F and above for 1.5 hours or more, breakdown of the polymer may be triggered.

Kadrmas (2007) described the emulsified asphalt residue recovery procedures in the United States. He outlined two reasons for the extraction of residual asphalt; to know the amount of binder in an emulsion sample, and to characterize the properties of the residual binder. For the second reason, Kadrmas indicated that it is very important to conduct this process in a way that interferes least with the binder's intrinsic properties. Two classes of methods are currently being used to extract the residual asphalt from the emulsion; distillation and evaporation. In distillation methods, emulsion sample is heated to temperatures as high as 260 °C to get rid of the water and the organic volatiles, and such temperatures are never observed in the field. The introduction of polymer modified emulsion has resulted in lowering this temperature to between 177 °C and 204 °C to prevent the polymer from disintegrating, but these temperatures are still quite high. Recent advance in technology has led to the use of vacuum distillation which is conducted at 135 °C which is still higher than what is seen in the field. Evaporation methods also involve heating the emulsion to drive away the aqueous fraction and the high temperatures can still alter and degrade the polymers. A study was conducted to compare four different methods (Vacuum distillation, Distillation at 177 °C, Distillation at 260 °C, and Oven Evaporation). Results of penetration test and DSR ( $G^*/\sin \delta$ ) were compared. It was observed that in most cases, the standard deviation was lower for higher temperatures. Other than that, the results from this study were inconclusive. The researchers also observed that field comparisons of applications to each of these recovery techniques may lead to the appropriate selection of the proper recovery technique.

Hazlett (2007) reported a study conducted by the Texas Department of Transportation (TxDOT) to compare six emulsion residue recovery techniques on the basis of the following criteria:

- Complete removal of the water
- Produces a residue that is representative of the field
- Cost of equipment
- Total procedure time
- Size of the sample produced so that it can be used for further testing
- Effort on the part of the technician
- Interference by outside sources that may affect the outcome

The methods studied were Distillation, Evaporation, Weathering Rack, Thermostatically controlled hot-plate, Dehydrator and SAFT (Stirred Air Flow Test) with nitrogen. The study used 7 different emulsion specimens from two different plant locations. Three replicates were done for each of the different procedures. The comparisons between the different procedures were done on the basis of the DSR results (G\*/sin  $\delta$ ) with a minimum requirement of 0.650 kPa at the test temperature. To compare the different methods, DSR tests were run with a continuous grading approach in the Superpave performance-graded binder specification.

The results showed that the SAFT produced the lowest grade almost in every case whereas the dehydrator produced the highest grade. In terms of the phase angle, the SAFT produced the lowest phase angle while distillation produced the highest in case of unmodified binders. In case of polymer modified binders, the phase angles were significantly lower than any of the rest which is clearly indicative of the fact that there is some polymer damage during the extraction process. Since the dehydrator invariably produced the highest grades, further investigation was undertaken using the FTIR Spectroscopy that revealed a substantial increase in the carbonyl group for the dehydrator which clearly indicated oxidative aging. The study concluded the SAFT to be the most efficient extraction procedure. Researchers also observed that if suitability of the test method shall be based on how closely it mimics the field conditions, then the weathering racks are the most efficient mechanism. This study also made the following recommendations.

- Usage of silicone containers and silicone sheets to modify the dehydrator procedure (Using lower temperature and thinner films will help in avoiding oxidation and also speeding up the process)
- Automation of the SAFT
- Testing more samples

### **Recent Developments in Emulsified Asphalt Research**

Bahia et al. (2008) investigated the performance grading of bituminous emulsions by looking at both distresses in sprayed seals (bleeding, raveling, fatigue and thermal cracking) as well as constructability considerations (storage stability, spray-ability and drain-out, breaking rate, setting rate and wetting of aggregates). The authors highlighted climate, aggregate mineralogy and coating of the emulsion as important when investigating its setting. The researchers conducted two tests on emulsions to investigate the stiffness development in emulsion over time as well as the aggregate-emulsion compatibility. The first was a DSR-based strain sweep test conducted on emulsion samples, first placed on an aggregate substrate, at different stages of curing. This test was based on the work done by Kucharek (2006) on emulsion samples placed on a metal plate. The other is the new ASTM D7000 Sweep test. The researchers showed the merits of both these test methods to provide measurable parameters that could help the industry evaluate setting and curing of emulsions as well as the emulsion-aggregate compatibility.

The Frosted Marble Test (FMT) is another test that investigates the aggregate-emulsion bond development in the early stages. Its origins date back to the work done in Mississippi and variations of the original tests have been developed since it was first proposed. In this test, a foot is used to apply torsion to a frosted (acid-etched) marble and the torsion required to move the marble is used to evaluate the degree of curing of the emulsified asphalt. Howard (2009) indicated that the FMT shows value in the assessment of emulsion binder curing, film development around the aggregate and excessive stiffening of the binder during early service. Some of the shortcomings in the original FMT have been addressed by later versions of this equipment. These shortcomings included the difficulty in developing an absolute scale for curing data, and the variability in test results. The shaft not being vertical during the torsion process and the marble hitting the tray and causing artificially high readings contributed to such variability.

Guiles (1995) used the Modified ISSA Frosted Marble Cohesion Tester to evaluate setting and curing characteristics of polymer-modified emulsified asphalts. There is ample evidence to indicate that polymers enhance the bonding between emulsified asphalt and aggregate. However, field evidence has also indicated that some polymers may alter emulsion set times. In this paper, the author presented results for a CRS-2 emulsified asphalt which had a proven track record of excellent field performance. The study revealed that polymer modified emulsions set slower than the standard unmodified emulsion and that polymer modification did not increase the set time of the emulsion or the chip retention in the first 6 hours. It also showed that polymer modification decreased the emulsion set time and stiffness at 2 hours, compared to the unmodified binder. The polymer modification was shown to significantly improve chip retention strength at 16 hours.

Kim and Lee (2006) studied the influence of various factors on chip seal performance, particularly aggregate retention and bleeding. The evaluations were made using the MMLS3 and the skid resistance test. They pointed out that other currently available laboratory test methods (Aggregate Retention Test Tex-216-F; Vacuum Test, Sweep Test ASTM D7000; Penn Aggregate Retention Test and Vialit Test) all apply a different form of mechanical energy to assess the aggregate-binder bond interaction instead of applying a mechanical energy force that simulates traffic wheels which MMLS3 can provide. They also indicated that these other tests tend to predict the bond rather conservatively. One of the factors they investigated that is relevant to this study was the time delay between aggregate spreading and rolling. They found that aggregate retention is influenced by both the moisture content in the aggregate as well as the rolling delay. They found that aggregate loss decreased with rolling delay up to a certain optimum value and then increased again with increasing roller delay. The optimal rolling times they reported are between 3.5 minutes and 7.5 minutes for dry aggregate, and between 5 minutes to 9 minutes for wet aggregate with a 2% water content.

Asphalt emulsion is just a way to obtain a film of asphalt in highway applications. Therefore, the way in which emulsified asphalt is transformed into an asphalt film is of great significance to the pavement engineer. Field observations have indicated that during the curing (i.e. drying) process, a "skin" is formed on top of the asphalt film that is exposed to the atmosphere. It is reasonable to think that this skin may act as a hindrance to the continued curing of the emulsified asphalt. Lesueur et al. (2001) conducted a study that investigated the skin formation of a cationic slow-setting emulsion under laboratory conditions. They were able to observe skin formation under a variety of conditions, and to devise a way to separate the skin from the emulsified asphalt beneath it by draining the emulsion through a hole drilled at the base of the plate on which the emulsion was spread. They found that during the early phase of skin formation, the coalescence of asphalt droplets in the skin has not yet taken place and therefore can be re-dispersed when put in an acidic medium. Lesueur et al. (2001) believed that a skin is formed as a "...consequence of the competition between the advancing drying front that tends to accumulate particles at the surface and diffusive forces tending to level off this increase in concentration." The skin was found to have a higher bitumen volume fraction that the emulsified asphalt (75% to 65%). They also observed that faster drying rates greatly favored skin formation and that emulsion asphalt content slows down the skin formation. The authors used three different mineral fillers to mix with the emulsion and found that aggregate that release significant quantities of cations in acidic environments (e.g. carbonates) can speed up the skin formation and increase the asphalt content within the skin.

# **Chapter 3. Laboratory Evaluation of Binder Curing**

## 3.1 Introduction and Background

Asphalt products for road construction include asphalt cements, cutback asphalts, and emulsified asphalts. The basic purpose of producing emulsified or cutback asphalt is to reduce the temperature at which it can be applied. From an environmental perspective, reduced temperatures and the omission of volatile fractions other than water are beneficial.

An asphalt emulsion is composed of three ingredients – asphalt cement, water, and an emulsifying agent. Asphalt emulsions that include an anionic surfactant base are categorized as anionic emulsions, while those with a cationic surfactant base are classified as cationic emulsions. Anionic emulsifiers are commonly fatty acid derivatives that are saponified with sodium or potassium hydroxide. Similarly, cationic emulsifiers are derived through salification of fatty amines. Special emulsion grades like "High Float" emulsions use higher quantities of emulsifiers, which gives a gel character to the residual binder after the emulsion breaks down.

The use of polymers to produce modified asphalt binders has created a whole new family of asphalt emulsions types. Letters P (Polymer-modified) or L (Latex-modified) are suffixed to the name of base emulsion to designate emulsions that utilize polymer modified binders. Some agencies have an additional cationic sand-mixing grade (CMS-2S) that contains more solvent than usual (Asphalt Institute, 2008). The suitability of an emulsion for a particular project is dependent on factors such as climate, type of job and type of aggregates, among many others.

Emulsion breaking signifies the phenomena in which a great majority of the droplets of the emulsion undergo an irreversible process that results in the formation of a continuous macroscopic bituminous phase (SFERB, 2008). The emulsions are formulated to have a sufficient level of potential repulsion between the adjacent particles to prevent premature coagulation. The particles coalesce only if modification occurs, at least locally within the surfactant films (SFERB, 2008). This can be due to a variety of reasons including:

- A change in the hydrophilic and lipophilic balance of the emulsifier
- A change in acidity or basicity (pH)
- An increase in the ionic force of the medium
- Adsorption of the surfactant by the mineral aggregates

Emulsion curing signifies the combination of phenomena which result in the removal of the water from the bituminous material, after the breaking phase is completed or is at least sufficiently initiated. This improves the mechanical properties of the product prepared with the emulsion (SFERB, 2008). The statement above therefore implies that the development of stiffness as well as its ability to hold the aggregates back will be governed by the rate of moisture removal from the system. Early placement of aggregates can lead to tire pickup by rollers while delayed placement of stones will require more

compaction effort, or in some cases improper embedment of stones which are dislodged within the first few weeks of opening to traffic (King, 2010).

Rejection of water from the aggregate surface is both a thermodynamic and kinetic effect. The thermodynamics relate to the energy differences between the emulsifier and the aggregate charge, the kinetic to the diffusion controlled loss of water through the coalescing binder (Holleran and Motina, 2006). The end result of the curing phase is a continuous cohesive film that holds the aggregate in place with a strong adhesive bond. Water evaporation can be fairly fast under favorable weather conditions, but high humidity, low temperatures, or rainfall will retard the curing process (Asphalt Institute, 2009).

Various techniques are used to cure emulsions in the lab including distillation at different temperature with or without the aid of vacuum, evaporation, stirred air flow using nitrogen and many others. A number of emulsion residue recovery techniques were evaluated as part of a study by the Texas Department of Transportation (TxDOT) (Hazlett, 2007). The study concluded (based on limited data) that the weathering racks can be considered as the gold standard for recovering the emulsion residues as it mimics field conditions very closely. This study investigated the curing of emulsions on weathering racks as well as in forced draft ovens at moderate temperatures.

# 3.2 Objective

One of the major constructability issues related to the use of emulsions in the field is loss of aggregates. Loss of aggregates may be triggered by a number of factors such as improper timing of placement of emulsified asphalt or cover aggregates, insufficient binder to cement the cover aggregates to the existing surface, and allowing fast traffic before proper adhesion is developed. However, the underlying reason for loss of aggregates is due to inadequate development of the adhesive bond between the emulsified binder and the cover aggregate. If the aggregates are placed too late, the emulsion's viscosity would have increased due to partial setting, resulting in improper or inadequate coating of the aggregate. Thus, it is necessary to estimate the rate of moisture lost due to evaporation as it will govern the evolution of the mechanical properties of the binder and determine the optimal timing for placement of the aggregate after spraying the emulsion. The main objective of this study is to develop a predictive model to quantify the amount of water lost as a function of time and changing weather conditions.

# 3.3 Modeling the Amount of Evaporative Water Loss

Surface treatment binders generally have an asphalt content ranging between 60 to 70%. Consequently, the emulsion is sufficiently viscous at ambient temperatures which prevent them from flowing when applied on pavements that have elevation differences due to their geometry. However, the preferred application temperature is kept above 131°F. The higher temperature allows the emulsion to spread faster and cool more rapidly when it comes in contact with the pavement surface and thus prevents drain-off. Therefore, the amount of water lost to evaporation before the aggregates are spread occurs in two stages: first while the emulsion cools from the application temperature to ambient conditions, followed by normal evaporation due to convectional heat transfer from the ambient air.

The total amount of evaporative water loss can be modeled as follows:

$$M_{tot} = M_{cool} + M_{con}$$
 Eqn. 3.1

Where,

 $M_{tot}$  = Total amount of moisture lost

- $M_{cool}$  = Amount of moisture lost while the emulsion comes to a thermal equilibrium with its surroundings
- $M_{con}$  = Amount of moisture lost due to absorption of latent heat of vaporization by the emulsion from its surroundings

The following section discusses an empirical approach that can be adopted towards modeling these two different modes of moisture loss from an asphalt emulsion.

#### 3.3.1 Loss of Moisture Due to Cooling of the Emulsion

Emulsions are generally applied at temperatures around 140°F. In most cases, the applied emulsion will exchange heat with the surrounding environment and during this process the water in the emulsion will absorb the latent heat of vaporization from the internal energy of the system, thus lowering its temperature further. The kinetics of the cooling process will depend upon certain physical properties of the air and the temperature gradient between the emulsion and its surroundings. The total amount of water vapor that can be generated will be limited by the amount of water that the emulsion had as part of its original formulation. It has been pointed out in the foregoing discussion that the evaporation of water from the emulsion starts to break and from a continuous phase. The diffusion barrier will continue to evolve with time as the emulsion. Thus the rate of water vapor will gradually slow down. The total amount of moisture lost while the emulsion is cooling can be modeled as a series of incremental time intervals where each of the intervals can be characterized by:

- The temperature specific to the time interval, which will be lower than its previous time interval due to the cooling process, and
- The amount of water present in the emulsion, which will also evolve with time due to continuous removal of water from the system due to evaporation.

According to the framework of the problem described above, the temperature during the first time interval will be equal to the application temperature for the emulsion and the amount of water in the system will be equal to the water present in the original formulation of the emulsion. Given that the convective heat transfer coefficient of air (k) is known, the amount of evaporative water loss can be determined using Newton's law of cooling (Winterton, 1999). It should be noted in this context that "k" is dependent on the type of media, gas or liquid, the flow properties such as velocity, viscosity and other flow and temperature dependent properties and therefore needs to be determined for asphalt emulsions from laboratory tests.

#### 3.3.2 Loss of Moisture Due to Vapor Pressure Deficit

Evaporation is the transformation of liquid water into a gaseous state and its diffusion into the atmosphere (Davie, 2008). The evaporation cycle requires two basic elements: the availability of water and the availability of energy. The main sources of energy for evaporation come from the sun in the form of incident radiation and heat waves re-radiated from other surfaces. In addition, the atmosphere should be dry enough to receive any water vapor produced. Dalton (Ward et al., 2004) was the first to propose a model connecting wind speed and dryness of ambient air to the evaporation rate.

According to Boyle's law, the total amount of water vapor that a definite volume of air can carry is temperature and pressure dependent. This implies that for a given temperature and air pressure, it is possible to determine the maximum amount of water vapor or saturation vapor pressure that a given volume of air can carry. Mathematically, the saturation vapor pressure can be calculated as:

$$e_s = 0.6108e^{\frac{17.27T}{T+237.3}}$$
 Eqn.3.2

Where,

#### $e_S$ = saturation vapor pressure in kPa, and

T = ambient temperature in °C.

By quantifying the ambient and saturation vapor pressures, one can estimate how much more water vapor the air can take until it reaches saturation. The difference between the ambient and saturation vapor pressure is the vapor pressure deficit (VPD). The amount of water vapor that can escape from an evaporative surface has a direct relationship with the vapor pressure deficit. Therefore, the higher the vapor pressure deficit, the higher the potential loss of water due to evaporation. Mathematically, the vapor pressure deficit can be expressed as follows:

$$VPD = e_s \left( 1 - \frac{RH}{100} \right)$$
 Eqn. 3.3

Where,

VPD = vapor pressure deficit, and

RH = relative humidity in percentage.

The other component of the evaporative relation is the rate of atmospheric mixing, which indicates how rapidly the given volume of air can diffuse into the surrounding atmosphere. "*The best indicator of atmospheric mixing is the wind speed at different heights above an evaporating surface*" (Davie, 2008). If there is no turbulence in the air above the evaporative surface, sooner or later the air will get saturated with water vapor, which will stall the evaporation process. On the contrary, higher wind speeds promote atmospheric mixing and result in drier air replacing the moist air blanket over the evaporation surface. Meyer's empirical formula, which is based on Dalton's law, is used to estimate the evaporation from open water surfaces (Ward et al., 2004). Meyer's

formula accounts for the atmospheric mixing component of the evaporation equation through the following expression:

$$E = C \times VPD\left(1 + \frac{u_{25}}{10}\right)$$
 Eqn. 3.4

Where,

E = Amount of moisture loss due to evaporation,

*VPD* = vapor pressure deficit in inches of Mercury,

C = empirical constant that depends on the nature of the water surface, and  $u_{25}$  = average wind speed in mph at a height of 25 feet above the evaporative surface. Meyer's equation includes a term to estimate the atmospheric turbulence and rate of diffusion of the ambient air. The equation integrates all the weather parameters into one single expression that could be used to predict the evaporation rate. However, the focus of this study is to model the amount of evaporative water loss as a function of time and weather conditions until the chips are spread. The amount of water that can evaporate from the emulsion sample is therefore limited to the total amount of available water in the system. In addition, water in the emulsion has to diffuse through the emulsion film to the surface in order to evaporate. It should also be noted in this context that as the water leaves the system, the emulsion undergoes a phase change from a liquid to a solid state (the residual asphalt binder) and therefore the phase front is dynamic in nature which evolves with time. Thus the problem in hand is a classic example of the Stefan problem which involves studying problems related to phase transformation of the matter (Budak et al., 1965). However, the wait time for placement of aggregates in the field is in order of 5-15 minutes. Thus, the relatively small time frame that is of interest allows one to ignore the effect of phase transformation in asphalt emulsions and simplify the problem as a case of evaporation of water.

## 3.4 Experimental Data

To evaluate the rate of evaporation as the emulsion cools, it is important to develop an experiment that can help quantify this rate. A mass loss experiment using the Thermo-Gravimetric functionality on an SDT Q600 DSC/TGA from TA instruments was conducted under isothermal conditions. The goal was to determine the rate of vaporization of water under different temperature regimes and therefore the test was conducted at seven different temperatures ranging from 122°F to 176°F. Figure 3.1 shows the rate of moisture loss from a 30 gram CRS-1P emulsion sample at each of these seven temperatures.



Figure 3.1 Evaporation vs. Time for a CRS-1P Emulsion at Different Temperature Profiles

The sample size was confined between 3.2 and 3.5 grams in order to keep the measurements consistent. To quantify the amount of moisture lost after the emulsion reaches a state of thermal equilibrium with the surroundings, a different approach was adopted. Weathering racks were used as the reference standard to cure asphalt emulsions. The racks, almost 4 feet above the ground, were exposed to external weathering agents. A sample size of 30 grams with a film thickness of 2 mm was used for this part of the study. The application rate was decided after considering the typical application rate for emulsions for seal coat jobs (0.45 gallons per square yard for Grade 4 aggregates) in Texas. Samples of emulsion where placed in PAV pans and placed on the weathering racks. The evaporative water loss was typically measured three to four times during the first 6 hours of the emulsion's exposure to the atmospheric conditions. It will be worthwhile to mention that the sole purpose of this study was to determine the optimal time to put down the aggregates in the field and therefore the experiment was conducted without bringing any mineral surface (aggregates) in contact with the emulsion. In addition, it has generally been observed that the time to put down the aggregates typically range between 3 to 15 minutes and therefore monitoring the rate of evaporation for the first 6 hours will help generate sufficient data to precisely determine how the weather parameters affect the rate of evaporation. The specific emulsions included as part of this study to determine the rate of curing are as given below.

- CRS-2P
- CRS-2
- HFRS-2
- HFRS-2P
- CRS-1P

Where, "C" stands for Cationic, "RS" stands for Rapid Setting, "2" stands for High Viscosity, "1" stands for Low Viscosity and "P" stands for Polymer Modified.

Figure 3.2 shows the mass loss data for a CRS-2 emulsion sample recorded on the weathering racks over a period of one week.



Figure 3.2 Mass Loss Measurements for a CRS-2 Emulsion Sample

A weather station was installed for meteorological monitoring at the testing location. Currently, the weather station is configured to report the following weather parameters at 5 minute intervals:

- Temperature data in °F
- Relative humidity in percentage
- Solar radiation in kW
- Wind speed in mph
- Precipitation in inches

The temperature, humidity and wind speed data as recorded by the weather station over a typical 10-day period is given in Figures 3.3 through 3.5, respectively.



Figure 3.3 Temperature Data from 05/13/2009 to 05/23/2009



Figure 3.4 Relative Humidity Data from 05/13/2009 to 05/23/2009.



Figure 3.5 Wind Speed Data from 05/13/2009 to 05/23/2009

## 3.5 Statistical Modeling of Evaporative Water Loss

Experimental data on the rate of evaporation of water from emulsion shows evidence that the rate of mass loss starts to drop with time. This drop in the rate of evaporation can be attributed to phase transformation as well as the decrease in the amount of moisture available in the system. The rate of evaporation can therefore be modeled using a time dependent relation as given below.

$$\frac{dm}{dt} = k_T \times m^\beta$$

Where,

dm/dt = Instantaneous rate of loss of water with respect to time,

 $k_T$  = Evaporation rate at temperature T (non-dimensional)

m = Amount of water present in the system with respect to original water content of the emulsion

Eqn. 3.5

 $\beta$  = Proportionality constant

Ideally one would normally expect, the coefficient of evaporation  $(k_T)$  to increase with increasing temperature as the energy input to the system is being raised. As the measurement of evaporation rates is being conducted under 7 different temperature profiles, it will therefore be possible to construct a relationship between evaporation rate and the temperature by fitting a function to these 7 different points. Thus a relationship,

 $k_T = f(T) Eqn. 3.6$ 

can be developed. The function f(t) can then be utilized to determine the evaporation rate for any given temperature. Once the proportionality constant and the evaporation rate

are known, it will then become possible to determine the amount of evaporation from the emulsion at any temperature profile, given that a continuous measurement of the temperature and the type of the emulsion are known. It should be noted that different emulsions will react differently when exposed to the same condition and therefore the evaporation rate should be determined separately for each of these emulsions. The total amount of evaporation of water while the emulsion cools down from its application temperature can now be represented using the expression given below.

$$M_{cool} = m_0{}^\beta k_{T_0} + m_1{}^\beta k_{T_1} + \cdots$$
 Eqn. 3.7

or, 
$$M_{cool} = \sum_{t=T_i}^{t=T_f} m_t k_{T_t}$$
 Eqn. 3.8

Where,

 $T_i$  = Initial temperature of the system (application temperature)

 $T_f$  = Final temperature of the system (ambient temperature)

However, it has to be remembered that a higher energy input would accelerate the process of evaporation at early stages but it might at the same time inhibit the evaporation rate due to a faster skin formation due to the phase change phenomenon discussed previously. For example, a higher temperature will accelerate the loss of water from the surface layer than what it would have at a lower temperature. On the other hand, a slower evaporation rate will retard the rate of transformation of the liquid emulsion to the solid asphalt binder which will actively influence the rate of diffusion. Diffusion is faster in liquids than solids due to their higher degrees of freedom (Dickinson et al., 1999). Thus the transformation of liquid to solid phase will retard the rate of evaporation from the sub-surface layers of the emulsion. Together these two forces (the faster rate of evaporation and slower diffusion rates with increasing temperature or vice versa) will compete against one another to define the final rate of evaporation in the emulsion and as a result of that we fail to see a clearly increasing or decreasing trend in the rate of evaporation with increasing temperature.

Experimental data also provides evidence to support this hypothesis. The model parameters were estimated using linear regression and the results are summarized in Table 3.1.

<b>k</b> <sub>T</sub> <b>@</b>	CRS-2P	CRS-1P	HFRS-2	HFRS-2P	CRS-2
122°F	0.0127	0.0155	0.0125	0.0147	0.0147
131°F	0.0132	0.0212	0.0117	0.0108	0.0165
140°F	0.0135	0.0161	0.0128	0.0078	0.0294
149°F	0.0158	0.0239	0.0133	0.0108	0.0398
158°F	0.0126	0.0213	0.0115	0.0082	0.0353
167°F	0.0113	0.0286	0.0136	0.0063	0.0413
176°F	0.0104	0.0301	0.0211	0.0062	0.0523
Average	0.0128	0.0224	0.0138	0.0093	0.0328
β	2.13	2.08	2.44	2.14	1.92

 Table 3.1 Estimation of the Evaporation Rate

It can be seen from the results presented in Table 3.1 that the rate of evaporation varies proportionally as the square of the amount of water present in the system. On the contrary, the coefficient of evaporation  $(k_T)$  has increasing as well as decreasing trends as the temperature varies, depending on the type of emulsion. These model parameters were estimated based on 18 to 20 data points and therefore it can be concluded that the trend in the data is not mere coincidence or the effect of any kind of interference by random errors. Due to lack of any systematic trends in the data, an average value of  $k_T$  was computed and its temperature dependency was ignored.

Once the emulsion has cooled down and has reached a state of thermal equilibrium with its surroundings, the rate of evaporation will now be governed by the deficit in the vapor pressure (VPD) in the surrounding air. The whole process can therefore be divided into a finite number of infinitesimally small time intervals where the amount of water lost during each of these time intervals will be a function of the VPD. By definition, VPD will be a dynamic factor that will evolve with changing weather conditions and so will the amount of water that will evaporate during each of these time intervals. As already mentioned, the mass loss measurements were recorded at finite time intervals that ranged between 1 and 3 hrs, it can therefore be reasoned that the total amount of water vapor lost from the system will actually be a summation of the water vapor lost during each of these infinitesimally small time intervals. This implies that in order to model the amount of evaporative water loss, it is essential that the integral of any time-dependent parameter is calculated in the model.

It is important to choose a suitable empirical form for the model such that its values are constrained within reasonable physical maximum and minimum bounds. The empirical form of the model chosen for this study is as given below.

$$M_{con} = 100(1 - e^{-(\beta_0 + \beta_1 E)})$$
 Eqn. 3.9

Where,

 $M_{\text{con}}$  = Total amount of water evaporated as a result of vapor pressure deficit in the ambient air

 $\beta_0, \beta_1 = \text{Regression Coefficients}$ 

E = Product of Vapor Pressure Deficit (in kPa) and Wind Speed (in mph)

The regression coefficients are given in Table 3.2.

f-statistic	18.63		
Adjusted R-Squa	0.64		
Number of Observa	53		
Effect	Coefficients	t-statistic	p-value
Intercept ( $\beta_0$ )	0.89	3.6	0.002
E: Product of VPD & Wind Speed ( $\beta_1$ )	0.23	4.3	0.000

**Table 3.2 Model Statistics and Regression Coefficients** 

The vapor pressure deficit and the wind speed data were obtained from the weather station that was deployed for the entire duration of this study. The temperature, relative humidity and the wind speed data was monitored at 5 minute intervals which were then integrated over the duration of the experiment and used for statistical modeling of the curing rates. The analysis of the results reveals that the curing rate of the emulsion is positively correlated to the product of the vapor pressure deficit and wind speed. This is indicated by the high t-statistic in Table 3.2, i.e. 4.3. A higher vapor pressure deficit implies a higher potential for the ambient air to absorb more water vapor while a higher wind speed translates to more vigorous atmospheric diffusion of the water vapor in the surrounding mass of air.

# **3.6 Field Evaluation of Emulsion Surface Treatment Projects**

The TechMRT research team first undertook a field evaluation of emulsion surface treatment projects constructed by TxDOT in-house forces as well as contractors. The objective of these field evaluations was to first study the TxDOT practices, and then to identify parameters and parameter values for a laboratory experiment program undertaken at TechMRT. For the most part, field projects were the sources of emulsions and aggregates that were used in the laboratory. During the calendar years of 2009 and 2010, six different projects were visited by the TechMRT team (Table 3.3). At each project, weather data was collected using a weather station and data was also collected on the construction process including materials used, timing of material applications and pavement temperatures.

# **3.7 Conclusions**

A methodology for the determination of the waiting period before the aggregates can be placed in a chip seal construction job was proposed as part of this task. The procedure involves using an empirical approach to model the rate of moisture loss from the emulsion which is tied to the time to place the aggregates in a chip seal construction job. The methodology breaks down the problem into two steps – determining the amount of water lost while the emulsion cools down, and determining the amount of water lost to evaporation due to the vapor pressure deficit in the ambient air. The amount of water lost while the emulsion cools was modeled as a time dependent parameter that relates to the amount of water present in the system and a temperature specific evaporation coefficient for any given emulsion. The evaporation coefficient is a relative index of the emulsion's response to thermal stimulus in terms of rate of moisture loss while the time dependent parameter captures the evolutionary trend of the rate of moisture loss from the system. However, results showed that the evaporation coefficient is only dependent on the nature of the emulsion and not the temperature of the system. It was also shown that the rate of moisture loss drops as the square of the residual water in the emulsion.

The second half of the equation accounts for the moisture loss that can be attributed to the vapor pressure deficit in the ambient air. A statistical model based on Meyer's equation was developed to predict the amount of water loss under ambient conditions due to three different aspects of the prevailing weather conditions: temperature, relative humidity, and wind speed.

No.	Location	Date	Aggregate	Emulsion
1	Breckenridge, FM701/FM1287	6/23/2009	Limestone	CRS-2
2	Paris, (FM895/FM2949	7/2/2009	Sandstone	CRS-2P
3	Plainview, FM2301	8/17/2009	Quart	CRS-2P
4	Floydada, FM651	11/4/2009	Quart	CRS-1P
5	Giddings, US77	6/14/2010	Lightweight	HFRS-2P
6	Comanche, SH36	7/23/2010	Limestone	CRS-2H

Table 3.3 List of Field Test Projects

# **3.8 Field Test Construction Projects**

## 3.8.1 Weather Station Data

The Davis Vantage Pro2<sup>TM</sup> weather station was used to monitor and record the field ambient weather parameters including but not limited to ambient air temperature, relative humidity, wind speed and heat index. These parameters were plotted to study the weather change pattern for specific project and assists in the modeling of the problem.

The length of time for which weather data was collected range from a couple of hours to 12+ hours. Due to a malfunctioning of the weather datalogger, the data for the field projects in the Paris districts were not recorded. The ambient weather plots are presented in Figures 3.6 through 3.10.



Figure 3.6 Weather Profile (Breckenridge-TX, June 23-June 24, 2009)



Figure 3.7 Weather Profile (Plainview-TX, August 17, 2009)



Figure 3.8. Weather Profile (Floydada-TX, November 4, 2009)



Figure 3.9 Weather Profile (Giddings-TX, June 14, 2010)



Figure 3.10 Weather Profile (Comanche-TX, July 23, 2010)

### 3.8. 2 Seal Coat Construction Data

9 min

11 min

13 min

15 min

21 min

30 min

35 min

45 min

60 min

Aggregate

**Spread Delay** 

Temperature

Before

Aggregate

Spread

Temperature

Soon After

Aggregate

Spread

For each field project listed in Table 3.3, basic project inventory, construction and pavement surface temperature data were monitored and recorded. Data are presented in Tables 3.4 through 3.9.

		1		<b>o</b> /		
District	Brownwood	Dood	FM701	Data	06-23-09	
County	Stephens	Koau	FM1287	Date	06-24-09	
City	Draalsonridge	Aggregate	Limestone	Binder Grade	CRS-2	
City	Dieckennuge	Type/Rate	100-130 sy/cy	and Rate	0.42gal/sy	
Weather Sta	tion on Duty	12:50 PM	Weather Sta	tion on Duty	08:53 AM	
	FM701		FM1287			
Before Spray	13	35	Before Spray	98		
Immediately	11	0	Immediately	100		
After Spray	11	9	After Spray	108		
15 sec	11	1	15 sec	103		
30 sec	109		30 sec	100		
1 min	103		1 min	98		
4 min	102		4 min	93		

9 min

11 min

13 min

15 min

21 min

30 min

35 min

45 min

60 min

Aggregate

Spread Delay

Temperature

Before

Aggregate

Spread

Temperature

Soon After

Aggregate

Spread

108

115

114

124

118

117

112

109

112

15 min

124 °F

106 °F

Table 3.4 Field (	Construction	Temn	erature	Record	for	Brecke	nridge.	TX (	in °F	)
Table 3.7 Field	Construction	runp	vi atui t	<b>I</b> UUUU U	101	DICCAC	mnuge,	1 1 1	111 1	,

88

89

88

86

87

90 91

95 98

9 min 30 sec

90 °F

87 °F

District	Paris	Dood	EM805	Data	07 02 00
County	Delta	Koau	111095	Date	07-02-09
City	Dorio	Aggregate	Sandatana	<b>Binder Grade</b>	CRS-2P
City	Palls	Type/Rate	Sandstone	and Rate	0.35gal/sy
Weather Sta	tion on Duty	09:49 AM			
		Maintenance work	k started at 09:55		
		FM	895		
Before Spray	1(	)8	10 min	9′	7
Immediately	17	12	12 min	0	-
After Spray	12	25	12 min	9.	5
15 sec	11	12	14 min	90	6
30 sec	11	10	16 min	9:	5
45 sec	1(	)8	18 min	90	6
1 min	1(	)6	20 min	90	6
1.5 min	1(	)5	25 min	96	
2 min	8	7	30 min	98	
2.5 min	9	5	40 min	102	
3 min	9	5	50 min	107	
4 min	9	8	60 min	11	1
5 min	9	7			
6 min	9	6			
7 min	9	8			
8 min	10	00			
9 min	9	6			
Aggregate			1' 55"		
Spread Delay			1 55		
Temperature					
Before	104 °F				
Aggregate			101 1		
Spread					
Temperature					
Soon After	87 °F				
Aggregate			<i></i>		
Spread					

 Table 3.5 Field Construction Temperature Record in Paris, TX (in °F)

District	Paris	Road	FM2040	Data	07-02-09
County	Delta	Kuau	11112.949	Date	07-02-09
City	Paris	Aggregate Type/Rate	Sandstone	Binder Grade and Rate	CRS-2P
Wea	ather Station on <b>I</b>	Dutv	Data N	N/A (Around 02:00	PM) <sup>1</sup>
		L.		· · · · · · · · · · · · · · · · · · ·	,
		FM	2949		
	Patch Area			Seal Area	
Before Spray	12	27	Before Spray	12	28
Immediately	17	0	Immediately	10	00
After Spray	12	29	After Spray	12	.9
15 sec	12	24	15 sec	12	26
30 sec	12	22	30 sec	12	25
45 sec	12	22	45 sec	12	24
1 min	12	23	1 min	125	
1.5 min	121		1.5 min	124	
2 min	12	21	2 min	126	
2.5 min	12	23	2.5 min	125	
3 min	12	23	3 min	125	
4 min	12	24	4 min	128	
5 min	12	27	5 min	129	
6 min			6 min		
7 min	11	16	7 min	116	
9 min	11	17	9 min	118	
10 min	11	17	10 min	119	
15 min	11	18	15 min	121	
20 min	11	18	20 min	121	
Aggregate Spread Delay	5' 3	35"	Aggregate Spread Delay	5' 3	35"
Temperature Before Aggregate Spread	129	) °F	Temperature Before Aggregate Spread	130 °F	
Temperature Soon After Aggregate Spread	110 °F		Temperature Soon After Aggregate Spread	113 °F	

Table 3.5 Continued. Field Construction Temperature Record in Paris, TX (in °F)

District	Lubbock	Dood	EM2201	Data	08-17-09	
County	Floyd	Koau	FIV12301	Date		
City	Plainview	Aggregate Type/Rate	Quartzite	Binder Grade and Rate	CRS-2P	
Weather Sta	tion on Duty	09:30 AM				
	09:35 AM			02:47 PM		
F	M 2301 Southbour	nd	F	M 2301 Northbour	ıd	
Before Spray	9	2	Before Spray	123		
				·		
1 min	8	8	25 sec	120		
3 min	8	5	2 min	104		
5 min	8	4	3 min	108		
Aggregate 7'15"		Aggregate Spread Delay	3'30"			
Temperature			Temperature			
Soon After	Soon After		Soon After	83	٥E	
Aggregate 05 F		Aggregate	83 F			
Spread			Spread			
Rolling Delay 15' 40"		<b>Rolling Delay</b>	4' 4	40"		

 Table 3.6 Field Construction Temperature Record in Plainview, TX (in °F)

County	Floyd	Road	FM651	Date	11-04-09
City	Floydada	Aggregate Type/Rate	Quartzite 110 sy/cy	Binder Grade and Rate	CRS-1P 0.48 gal/sy
Weather Sta	tion on Duty		15:10 PM (Work	started at 15:22)	
		FM	1651		
		1 1/1	Temperatures F	Before and After As	gregate Spread
Before Spray	9(	)			
Immediately	11	0			
After Spray	11	0			
15 sec	10	2			
30 sec	98	3			
1 min	91	[			
1.5 min	87	7			
2 min	83	5			
2.5 min	83	3			
3 min	80	)			
4 min	79	)			
5 min	77	7			
6 min	77	7			
/ IIIII 8 min	77	7			
0 min	75	2			
10 min	75	2			
13 min	70	) )			
15 min	71	, 	13.5 min	6	8
20 min	71	5	14.5 min	0	0
20 min 25 min	70	7	14.3 min	7	2
2.5 min 30 min	75	2	16.5 min	7	2
40 min	78	<u>,</u> R	17.5 min	7	3
50 min	76	5 5	18.5 min	7	5
60 min	80	)	19.5 min	7	7
Aggregate Spread Delay			13' 30"		
Temperature					
Before			<b>5</b> 0.05		
Aggregate			78 °F		
Spread					
Temperature					
Soon After			68 °F		
Aggregate			00 1		
Spread			1.52.002		
<b>Koller Delay</b>			15 00"		

Table 3.7 Field Construction Temperature Record in Floydada, TX (in °F)DistrictLubbockRoadFM651Date11-04-09

District	Austin	Dead	LIC 77	Data	06 14 10	
County	Lee	Koau	03-77	Date	06-14-10	
City	Giddings	Aggregate Type/Rate	Lightweight (TXI) 125 sy/cy	Binder Grade and Rate	HFRS-2P (Ergon- Austin) 0.44 gal/sy	

<b>Table 3.8 Field Construction Temp</b>	perature Record in Giddings, TX (in '	°F)
--	---------------------------------------	-----

Weather Stat	Weather Station on Duty 08:40 (Maintenance work started at 09:30)								
TX-77 Northbound (inside lane)									
Before Spray	102	10 min	93						
0.25 min	110	11 min	90						
0.50 min	102	12 min	93						
0.75 min	101	13 min	91						
1 min	98	14 min	88						
1.25 min	99	15 min	90						
1.50 min	99	20 min	91						
1.75 min	98	25 min	93						
2 min	98	30 min	99						
3 min	93	40 min	100						
4 min	87	50 min	90						
5 min	95	60 min	103						
7 min	96								
Aggregate Spread Delay	3' 26"								
Temperature Soon After Aggregate Spread		84 °F							
<b>Roller Delay</b>	4' 40''								
District	Brownwood	Pood	TV 36	Data	07 23 10				
----------	-----------	------------------------	--	--------------------------	---------------------------------------	--			
County	Comanche	Noau	17-30	Date	07-23-10				
City	Comanche	Aggregate Type/Rate	Limestone (Vulcan, Brownwood) 120 sy/cy	Binder Grade and Rate	CRS-2H (Ergon-Waco) 0.36 gal/sy				

<b>Table 3.9 Field Construction Terr</b>	perature Record in Comanche, TX (in °F)
--	---

Weather Sta	Weather Station on Duty07:50 AM (Maintenance work started at 08:00 AM)					
	TX-36 S	outheast bound				
Before Spray	89	10 min	83			
0.25 min	109	15 min	82			
0.50 min	102	20 min	81			
0.75 min	98	25 min	81			
1 min	96	30 min	80			
1.25 min	94	40 min	81			
1.50 min	92	50 min	83			
1.75 min	91	60 min	85			
2 min	90					
3 min	87					
4 min	88					
5 min	87					
6 min	86					
7 min	86					
8 min	83					
9 min	84					
Aggregate		7' 20"				
Spread Delay		7 20				
Temperature						
Soon After		83 °F				
Aggregate		00 1				
Spread						
Roller Delay	8' 00''					

District	Brownwood	Road	TX-36	Time	07-23-10	
County	Comanche					
City	Comanche	Aggregate Type/Rate	Limestone (Vulcan, Brownwood) 120 sy/cy	Binder Grade and Rate	CRS-2H (Ergon-Waco) 0.36 gal/sy	

1 adie 3.9 Continued Field Construction Temperature Record in Comanche, TA (in <sup>5</sup> F
---

Weather Sta	tion on Duty	07:50 AM (Main	tenance work star	ted at 09:40 AM)	
		TX-36 Nort	thwest bound		
Before Spray	9	8	10 min	87	
0.25 min	102		15 min	90	
0.50 min	9	9	20 min	87	
0.75 min	9	7	25 min	94	
1 min	9	3	30 min	98	
1.25 min	9	3	40 min	100	
1.50 min	9	0	50 min	95	
1.75 min	9	92		98	
2 min	9	2			
3 min	9	0			
4 min	8	7			
5 min	8	8			
7 min	8	5			
8 min	8	7			
9 min	8	5			
Aggregate			6' 50"		
Spread Delay			0 30		
Temperature					
Soon After	92 °E				
Aggregate			05 1		
Spread					
<b>Roller Delay</b>	9' 20"				

### 3.8.3 Selection of Laboratory Experiment Parameters and Values

Based on the observations made by experts on seal coats, the aggregate delay (the time between emulsion spray and aggregate spread), and the rolling delay (time between aggregate spread and rolling) are two factors that can significantly influence the durability of bond between the emulsion and the aggregate. The general consensus of scientists and engineers is that aggregates must be spread as soon as practically possible after emulsion is spread. Reduced aggregate delay is expected to provide the best opportunity for the charged asphalt particles in the emulsion to adhere to aggregate surfaces under electrostatic attraction. Table 3.10 lists the aggregate delay and roller delay values used in field projects. Those results show no consistent practices for the timing of material application. The aggregate delay ranged from 1 minute 55 seconds to as much as 15 minutes. In the Plainview project, the aggregate delay for the section sealed in the afternoon at 14:47 hours was almost half of that used in the section sealed in the morning at 09:35 hours. The aggregate delay used in the Breckenridge project was much longer (at least twice that of other summer projects).

As far as the rolling delay is concerned, rolling was typically completed within a few minutes after the aggregate was spread, except for the section in Plainview and two other projects for which information was not available.

Based on past experience from successful projects, values were selected for these two time delay parameters to be used in laboratory experiments. It was considered appropriate to select aggregate delays of two and minutes and ten minutes, and two and ten minute delays for rolling as well for the ASTM D7000 Sweep Test and the Modified Aggregate Retention Test.

					·	
No.	Location	Date	Aggregate	Emulsion	Aggregate Delay	<b>Rolling Delay</b>
1	Breckenridge, TX	6/23/2009	Limestone	CRS-2	15 min 9 min 30 sec	N/A
2	Paris, TX	7/2/2009	Sandstone	CRS-2P	1 min 55 sec 5 min 35 sec	N/A
3	Plainview, TX	8/17/2009	Quart	CRS-2P	7 min 15 sec 3 min 30 sec	8 min 35 sec 1 min 10 sec
4	Floydada, TX	11/4/2009	Quart	CRS-1P	13 min 30 sec	1 min 30 sec
5	Giddings, TX	6/14/2010	Lightweight	HFRS-2P	3 min 26 sec	1 min 14 sec
6	Comanche, TX	7/23/2010	Limestone	CRS-2H	7 min 20 sec 6 min 50 sec	0 min 40 sec 2 min 30 sec

Table 3.10 Aggregate Delay and Rolling Delay Data for Projects

# **3.9 Laboratory Investigation of the Influence from Aggregate and Climate on Stiffness Development in Seal Coat Asphalt Emulsions**

## 3.9.1 Introduction and Background

Emulsified asphalt is widely used in many highway maintenance and construction applications, and their effectiveness is greatly influenced by the construction process and climatic conditions at the time of application and soon thereafter. This research studied the rate of residue formation of emulsified asphalts over time, with particular emphasis on two key construction-related factors; the aggregate it is in contact with, and the climatic conditions. Emulsified asphalts were sampled both from a plant location as well as three construction sites. Test specimens were prepared by pouring asphalt emulsion directly on aggregate substrates and then cured under different climate regimes in an environmental chamber. These climate regimes were selected to simulate field conditions and anticipated laboratory test environments. The stiffness of the asphalt was measured at specified time intervals by a Dynamic Shear Rheometer (DSR) using a strain-sweep test protocol.

Asphalt is a by-product of the oil refining process. Once all of the usable material and fuel has been extracted from crude oil the substance that is remains is asphalt. Depending on the crude oil source there is between 1% and 60% asphalt remaining after the refining process. The United States uses about 36 million tons of asphalt per year with roughly 31 million tons used for roadway applications of which 2 to 3 million tons are asphalt emulsions (*1*). Emulsified asphalts are commonly used in spray seal applications. An emulsion is any colloidal suspension of one liquid in another, where typically the two liquids do not mix together, as is the case with emulsified asphalts. An asphalt emulsion contains asphalt particles suspended in a water medium. When an emulsified asphalt is applied on a roadway surface, it goes through a time-dependent transformation. Therefore, it is important to study both the changes and the rates of changes of properties in the material over time in order to optimize construction processes such as aggregate spreading, rolling and brooming. See Figure 3.11 for a basic construction flow chart for an emulsified asphalt sprayed seal.



### Figure 3.11 Typical Construction Process Flow Chart for Emulsified Asphalt Sprayed Seals

It is imperative to study the interactions between asphalt and aggregate with respect to different ambient construction conditions in order to fully understand the changes that take place within the asphalt emulsion after application. The best approach to evaluate the changes that take place in the material with time is by using rheology, which is the study of the deformation and flow of a material. A standard test method exists to study the flow properties of emulsified asphalt using the Dynamic Shear Rheometer (DSR). The test is governed by the standard specification AASHTO T 315 which specifies that the material is to be heated and then poured into a silicone rubber mold where it is cooled to room temperature before being tested. Instead of using this standard test method, for the purposes of this research, the material was heated to the desired application temperature and then poured into O-rings resting directly on an aggregate surface. Another change from the standard specification was that the material at different strains applied by the DSR equipment. Based on the strain-sweep test results, the material response can be divided into two different regions - linear and non-linear (Figure 3.12).



Figure 3.12 Strain-Sweep Used to Determine the Linear Region of the Material (2)

Data from the Strain-Sweep Test can be used to evaluate the interactions between different binder-aggregate combinations as well as the influence of climactic curing conditions on the binder-aggregate interaction. This is useful in the initial planning and design phases of a project. In addition to evaluating the interaction between the binder and aggregate, another objective of this research was to provide information to practitioners about the optimum sequence of construction operations. An asphalt residue after application on the road should be stiff so as not to deform too much, elastic to recover some of the unavoidable deformations, and not be brittle at low temperatures to avoid cracking or loss of bond between the asphalt and aggregate (King et al, 2010).

Asphalt emulsions were first developed in the early 1900s but were not commonly used until the start of the 1920s. The use of asphalt emulsions increased dramatically following World War II with the significant increase in personal vehicles and the desire to freely move from one place to another. The use of emulsions continued to increase as time progressed because emulsions produce less atmospheric emissions than other suitable asphalt products and also because emulsions require significantly less energy to produce and apply (Asphalt Institute, 2008). Some typical applications of asphalt emulsions include: chip-sealing, fog-sealing, slurry sealing, tack coats, and soils stabilization.

Asphalt emulsions contain three basic parts; asphalt, water, and emulsifier. Slight changes in any of the three basic components can lead to large changes in the properties of the final emulsions mixture. It is basically thought that an emulsion is a mixture of asphaltenes in

maltenes and is dispersed through peptization via emulsifiers. The higher the amount of peptization the more stable an emulsion will be in terms of storage and workability, with the particle size distribution of the asphaltenes being perhaps one of the most important factors to the amount of peptization. It has been found that one of the largest factors to be controlled is the particle size distribution. The particle size distribution can affect the breaking index, storage stability, and adhesion to aggregate (Eckman, et al ). The optimum emulsion would have a particle size distribution of asphaltenes of 28% less than one micron in diameter, 57% at one to five microns in diameter, and 15% at five to ten microns in diameter (Dybaiski, 1976). In addition to the particle size distribution of the asphaltenes, the water that is added to the mixture can also have an effect on the end product. Water can contain ions of calcium, magnesium, sodium, potassium, carbonate, or bicarbonate, to name a few. The presence of any of these ions can change the behavior of the material being produced. For instance, calcium and magnesium are sometimes added to the mixture when making a cationic emulsion, but if they are added when making an anionic emulsion the anionic emulsion will clump as opposed to form a homogenous liquid. The same is true with the addition of carbonate or bicarbonate to an anionic formulation but would cause deleterious effects when added to a cationic emulsion. The water quality is extremely important because even a low amount of any of these kinds of ions, such as in normal tap water, can cause an emulsion to not form properly. Particulate matter within the water, regardless of whether it has a charge, can also hinder the emulsion's manufacturing process by changing the amount of surface area available for the emulsifiers to react with the asphalt particle, which is discussed later in this paper.

There are two kinds of emulsions: cationic and anionic, based on the emulsifier added to the mixture during formulation. An anionic emulsion has asphalt particles that have a negative surface charge, and cationic emulsions contain asphalt particles that have a positive surface charge (TxDOT, 2006). Emulsions are made from mixing hot bitumen with water containing emulsifying agents and mechanical energy to break up the hot bitumen into the desired size droplets (James, 2006). The surface charges of both the aggregate and asphalt particles will affect how quickly the emulsion breaks after application. The majority of aggregates used in Texas have a negative surface charge. When an anionic emulsion is applied to a negatively charged aggregate, the asphalt particles within the emulsion and the aggregate particles repel each other; therefore, the only means for the mixture to break is through water evaporation. A cationic emulsion. The cationic emulsion breaks faster because of the attraction of opposite charges that enables the water to evaporate much more quickly.

A typical asphalt emulsion pavement contains 40% to 75% bitumen, 0.1% to 2.5% emulsifier 25% to 60% water, and other minor components by weight (James, 1999). Emulsifiers change the amount of energy needed to emulsify an asphalt through a colloid mill (James, 2008). There are three types of emulsifiers used to make an asphalt emulsion: anionic, cationic, and nonionic. An emulsifier or surfactant, regardless of the chemical makeup, is a water soluble compound which contains a lipophilic (oil-loving) hydrocarbon tail and a hydrophilic (water-loving) hydrocarbon head. The hydrophilic portion aligns itself towards the more polar phase of the mixture, whereas the lipophilic portion aligns itself with the less polar or non-polar phase of the compound is ionized. The opposite is true for a cationic formulation, where the hydrophilic

portion of the emulsifier contains a positive charge after ionization (Dybaiski, 1976). Cationic emulsions are typically considered to be acidic whereas anionic emulsions are considered alkaline (James, 1999)). In addition to changing the surface charge of the asphalt particles within an emulsion, the choice of emulsifier can also have an effect on the viscosity of the final emulsion. One of the main specification criteria for asphalt emulsions is the viscosity of the final emulsion, as it can dictate adhesion to aggregate, storage stability, and run-off from the road once applied. Changing the type of emulsifier used in an emulsion formulation can be a significantly cheaper way to achieve the desired viscosity than changing the amount of residue, which is the classic way the viscosity was controlled. Changing the emulsifier can also be effective because it does not change the particle size distribution of the emulsion, whereas changing the amount of residue can also change the particle size distribution. There is inherently some amount of trapped water within a certain proportion of asphalt particles within an emulsion due to the high strains applied during the formulation through the colloid mill. An emulsifier can directly change the amount of trapped water within asphalt particles, which directly translates into a different viscosity because viscosity is essentially just a measure of the amount of trapped water within the emulsion (James, 1997). The choice of emulsifier is extremely important in the formulation of an emulsion because it can directly affect the performance of the material during manufacturing, storage, transport, and placement.

Asphalt can be modified by the addition of other products such as polymer to improve some of the properties of the material. A polymer is made of many (poly) small particles, monomers. They can be added to asphalt to improve certain material properties such as resistance to permanent deformation at high temperatures, an increase in flexibility at low temperatures, better fatigue resistance, better tensile strength and an overall reduced temperature susceptibility (Baughman, XX). There are many different types of polymers available for use in asphalt emulsions but they all typically improve the characteristics of the mixture in the same general way—they form a polymer network within the asphalt. The formation of the polymer network adds strength and changes the overall properties of the laid pavement. Polymers can typically be classified into two categories; elastomers and plastomers. Elastomers can be stretched and regain their shape almost immediately when the load is removed; Styrene Butadienes (SBS) or Styrene Butadiene Rubber (SBR) aretypically used. Plastomers, typically Ethylene Vinyl Acetate (EVA) polyethylene/polypropylene or polyolefins (Baughman, XX), give a significantly higher strength to the material by forming a rigid three-dimensional network within the asphalt material. Polymers used for asphalts are typically solid and need to be heated to dissolve into the solution, but are safe to handle at room temperature. Polymers can be an easy way to improve the properties of an asphalt emulsions but can also add a significant cost to the end product.

In order for the asphalt emulsion to act as cement on the road, the material must first form a continuous asphalt film. For the continuous film of asphalt to form, two different processes within the material must take place; the first is flocculation (setting) and the second is coalescence (curing). The surface charges on the asphalt droplets provide an electrostatic barrier which must be overcome for the asphalt particles to flocculate; as the barrier is overcome the asphalt particles are pulled closer together due to the surface tension of the water droplets separating them (James, 2006). The flocculation stage of an asphalt emulsion can be reversed by agitation, dilution, or addition of more emulsifier. After flocculation begins, as more water leaves the mixture, coalescence between asphalt particles can start. Coalescence is irreversible.

Generally, flocculation occurs within the system quickly while coalescence takes more time to begin, dependant on ambient conditions. Less viscous asphalts coalesce faster than more viscous asphalts and lead to curing times varying from a few hours, in the case of a less viscous emulsion, to several weeks, for a dense-mix high viscosity emulsion. Factors which effect both flocculation (setting) and coalescence (curing) include: absorption of both water and emulsifier into the aggregate, mineralogy of the aggregate, evaporation of water from the system, mechanical action and ambient environmental conditions.

Asphalt emulsions are divided into grades which indicate specific properties of the material so that the appropriate grade of asphalt emulsion can be chosen based on the specific application. The first basic designation, as explained above, is either cationic or anionic relating to the surface charge of the asphalt particles within the mixture. The next subdivision within emulsion classification is the rate at which the material is designed to set. A rapid setting (RS) emulsion sets quickly and is typically used with un-reactive aggregates for chip seal applications. A medium setting (MS) emulsion sets less quickly than the rapid setting materials and is typically used with low surface area aggregates of open-graded mixes. The last division related to the curing properties of the material is a slow setting designation, SS. The slow setting emulsions are usually considered un-reactive and can be used effectively with reactive aggregates of high surface area (James, 2006). A cationic emulsion would have a C in front of the name whereas an anionic emulsion has no prefix before the rate of setting designation. For example, a cationic rapid setting emulsion would be a CRS, whereas an anionic rapid setting emulsion would just be an RS. Asphalt emulsions are also graded based on their consistency using a viscosity measure. A lower number in the grade indicates a thinner viscosity and a higher number indicates a thicker viscosity; the number follows directly after the setting characterization. When a dense graded aggregate is used for the roadway, an emulsion with a thinner viscosity is required, whereas an emulsion with a thicker viscosity is typically used for an open-graded application in order to control the adhesion between asphalt and aggregate as well as to prevent run-off when applied to the roadway (Kadrmas, 2006). After the number indicating the viscosity of the material there could also be a letter. An "h" after the viscosity number indicates that a harder base asphalt was used to make the emulsion, for example a PG64-22 may have been used instead of a PG58-22 to achieve the h criteria (Utah DOT, XX). Instead of an h, an emulsion may have a P after the viscosity number indicating whether the emulsion is a polymer-modified material. The emulsion used for a specific application needs to be chosen based on the desired outcome of the project, but the main properties desired are the ability to coat aggregate, form a uniform film, and stability for application purposes.

There are three broad categories that asphalt emulsion work typically falls under: construction applications, maintenance applications, and preventative maintenance applications (Simpson, 2006). Construction applications include tack coats and prime coats. Maintenance applications include chip seals, scrub seals, slurry seals, micro-surfacing, and ultrathin bonded wearing courses, though typically fog seals are used for preventative maintenance applications. Tack coats are used to bond different layers of a pavement system to one another. A tack coat can use either an anionic or cationic emulsion designed to break quickly and is applied at a very low application rate. A prime coat is designed to treat a base course in order to ensure good bonding between the base and the upper layers of a pavement system. A prime coat can also aid in dust abatement during construction. Specially designed slow setting asphalt emulsions are used for

this application, typically asphalt emulsion prime (AEP) or penetrating emulsion prime (PEP). Chip seals are an effective tool to rehabilitate the driving surface of a road. A layer of either cationic or anionic emulsion is applied, in a relatively thin film only a couple millimeters thick, and then rock "chips" are laid down shortly after asphalt application. Scrub seals function by sealing small cracks within an existing pavement. An emulsion is applied and scrubbed into the existing pavement using large scrub brooms. Slurry seals are designed for low-speed low-traffic volume roads. A slurry seal produces a smooth, even driving surface similar to a hot mix asphalt but much thinner. Micro-surfacing is similar to a slurry seal but it is design for heavy traffic areas. The last common maintenance application is an ultra-thin bonded wearing course. For this application an emulsion is sprayed on the road and then immediately paved over with hot mix asphalt concrete. Lastly, fog seals are a thin layer of asphalt emulsion applied to any existing asphalt surface. It is the most cost-effective way to perform preventative maintenance but cannot be used on roads beyond a certain threshold of disrepair.

Standard material test methods have been developed over the last few decades to characterize the material properties of asphalt emulsions. There are tests to determine viscosity, ductility, demulsibility etc. Each of these tests provides information about material properties, but they only provide information for standard test conditions. The material properties of emulsified asphalt change with time which makes it important to understand how the material is changing with time in order to optimize the construction process. To fully understand how the material is changing over time, different, non-standard test methods can be useful. One such emerging material testing area is rheology. Rheological data can be obtained at different times after sample preparation but data can also be obtained at different strains or stresses applied to the material sample at different curing durations. This laboratory experimental program used rheological testing to understand aggregate-emulsion interactions under different climatic conditioning regimes.

The Strategic Highway Research Program (SHRP) developed standard rheological test methods to characterize asphalt in the early 1990s (Peterson et al, 1994) (Anderson et al, 1994). From this research the AASHTO specification for rheological testing was derived and this procedure was used as a way to select appropriate materials for highway construction and maintenance. This standard test method, typically used to test asphalt cement or the asphalt residue obtained from an emulsion, makes no allowances for different residue recovery techniques as related to asphalt emulsion testing. Furthermore, when a sample is tested using the standard test method it only yields data for a specific temperature, strain, and loading frequency of the rheometer. The value given by the rheometer is then compared to standard material specifications and the material is determined to be either passed or failed based on whether the data was above or below the standard value listed for that grade of asphalt. Typical data obtained from a dynamic shear rheometer, dependant on the software being used, include: binder temperature, angular frequency (rads/sec), oscillation stress, oscillation strain, phase angle (delta), storage modulus (G'), loss modulus (G"), and the total elapsed time. The complex modulus (G\*) can then be calculated as  $(G'^2 + G''^2)^{(1/2)}$ . As part of the same research, SHRP also looked briefly at different kinds of rheological testing that could potentially be used to characterize material properties of asphalt in the future. SHRP conducted strain-sweep tests, frequency-sweep tests, and temperature-sweep tests. From these tests, isochronal plots of viscoelastic function versus temperature were created, and it was found that a change in testing temperature of 1°C can change the modulus of the

material by as much as 25%, showing the temperature dependence of the stiffness of asphalt material. SHRP concluded that the upper limit of the linear-viscoelastic region of the material was when the complex modulus decreased to 95% of its initial value. The testing conducted was on asphalt cement, either aged or unaged, and the research was essentially limited to the linear-viscoelastic region of the material. The report suggests that more research is required in order to fully understand the rheological properties of asphalt and how those properties change with time, temperature, and strain/stress rates.

The SHRP guidelines that were developed to qualitatively quantify the rheological properties of asphalt materials still need to be refined. They should be used as guidelines and not as defined rules of material behavior as they can only analyze a small sample of material at one time. From the temperature sweeps performed for the initial research project it was found that a change as small as 1°C changes the modulus of the material by as much as 25%, this change in modulus values directly changes the limits of the linear viscoelastic region domain of the sample. Research performed at NYNAS Petroleum Group found that the stiffness derived through rheological testing of a bituminous material is dependent on the magnitude and rate of the stress or strain applied to the sample as well as the temperature. The testing temperature needs to match the expected in-service temperature of the binder in order to be able to accurately predict the service properties of the material, otherwise lab tests do not necessarily correlate to the behavior of the material on the roadway. The same research project also proposed that trying to keep rheological testing within the liner viscoelastic region may not be the best approach to characterize the behavior of the material. As a vehicles' wheels pass over a particular place of a pavement there is a load distribution which is transferred through the entire thickness of the roadway. The stress or strain applied over the entire thickness of the pavement may be sufficiently small so as to be able to conclude that the asphalt particles are staying within their linear viscoelastic domain, but when a finite element analysis of the structure is analyzed there are localized areas which encounter extremely large stresses. Therefore, dependent on the stress distribution over the entire structure some of the asphalt particles may always be operating within their non-linear range throughout the entire life of the pavement (Eckman, XX). That is not to say that valuable information cannot be gained from analyzing the linear viscoelastic region of a bituminous material but that one cannot stop with the linear region and must try to understand the behavior of the material in the non-linear region as well.

Rheological properties of asphalt emulsions cannot be tested directly on the emulsion itself, but can be tested on the residue of an asphalt emulsion. Obtaining the residue can be difficult and variable depending on the test method being used. Research by Epps et al. looked at five different residue recovery techniques for emulsified asphalts (Epps et al, 2001). Studying residues obtained from: the hot oven method, rotavap method, hot plate method, stirred can method and the standard distillation method. The hot oven method used was similar to that of ASTM D 244-97C, except that in this case nitrogen was flowing over the material to prevent oxidation, using 50 grams of material in a beaker and placing it in an oven, equipped with nitrogen gas, at 163°C for 2 hours. The sample was then stirred, and cured for another hour. The rotavap method was similar to the modified ASTM D 5404-97. Here, 16 grams of emulsion were evaporated for 30 minutes in a water bath of 100° C, then another 70 minutes in a water bath of 163° C. Again nitrogen was used to prevent oxidation. The hot plate method was developed by the construction division of TxDOT. 20 grams of emulsion is poured into a tin and

then placed on a hot plate set to 180° C for one hour, stirring periodically. The standard test method for residue recovery of emulsified asphalt is distillation, ASTM 244-97C. Two hundred grams of emulsion is poured into a still and heated. The material is distilled at 215° C for 45-60 minutes, and then it is distilled at 260° C for another 15 minutes. Lastly, the stirred can method was evaluated. 1250 grams of emulsion was placed in a gallon can, with heating tape, and stirred continuously with an impellor while nitrogen gas was bubbled through the specimen to prevent aging due to oxidation. The researchers concluded that the best residue recovery method tested was the stirred can method. The stirred can method took the least amount of time and yielded the most weight of residue material. This research did not specifically analyze the effects of the asphalt-aggregate interaction, nor did it consider the effects of temperature and humidity cycling on the material properties with time.

The Texas Department of Transportation (TxDOT) has also looked at different residue recovery techniques and the effect the recovery techniques have on the residue (Hazlett, 2006). In addition to some of the methods discussed above this research also looked at the residue recovery from the evaporation method, the weather rack method, and the dehydrator method. The evaporation method was based on ASTM D 6934 which is typically used to find the amount of residue recovered but not to run further tests on the residue after recovery. A 50g sample of emulsion is heated for 2 hours at 163°C, then stirred and heated for another hour. This method produces a large quantity of residue in a short amount of time for very little cost and without any environmental effects on the material. The heating temperature still raised questions about the changes within the material and if it was really representative of an actual in place asphalt emulsion. A weather rack was also studied in accordance with ASTM G 7. A 50g sample of emulsion was poured in thin film oven pans and placed on a rack outside in ambient environmental conditions. Samples were weighed periodically until a constant weight was achieved, typically two to seven days after exposure. This procedure produces large quantities of residues that can be used for further testing. The main disadvantage of this kind of residue recovery is that it is based on one specific environmental conditioning which may or may not be representative of the actual location of the construction site. The dehydrator method used a commercially available food dehydrator with stackable trays. A 10g sample of emulsion was poured into thin film oven pans, placed in a dehydrator, and weighed periodically until consistent weights were achieved indicating that all of the water had left the system. All three of the techniques studied yielded similar residue recovery values but it was found that the dehydration method prematurely ages the material and therefore should not be used for residue testing.

Another research endeavor performed at the Colas Campus Scientifique et Technique looked at some international standards for residue recovery techniques including the Belgian procedure (08-34), the Spanish method (NLT 147), and the ethanol precipitation method (Gueit et al, 2007). The Belgian procedure (08-34) takes 1 Liter of aqueous asphalt emulsion and heats it with a Bunsen burner, stirring continuously, until a constant temperature of 163°C is reached. The sample is then placed in an oven at 163°C for half an hour to eliminate all remaining water; the residue can then be tested in the desired manner. The Spanish method consists of heating 50g of emulsion for 2 hours at 163°C, after heating only the asphalt residue remains in the beaker. The last method evaluated was the ethanol precipitation method. Here, the separation of water and asphalt is not performed through heating but through a chemical reaction. It was found that since

the residue was recovered through a chemical reaction and not through heating that it was more representative of the base bituminous material than any other residue recovery technique.

Bahia et. al., presented a new testing protocol to evaluate emulsion setting behavior using a DSR (Miller et al, 2009)). According to this publication, the time required for the asphalt to gain adequate strength and adhesion to the aggregate is dependent on the compatibility between the emulsion and aggregate; environmental conditions such as temperature and humidity; and mechanical action. Sufficient strength must be gained within the material before the road can be opened to traffic, though this threshold at the moment is subjective. Two sequential processes must occur in order for the asphalt to retain the aggregate chips. First, the emulsion must break and secondly the asphalt particles must coalesce. Both of these processes must occur in order for the asphalt to retain the aggregate chips. The researchers prepared asphalt emulsion samples directly on two different aggregate substrates. The samples were cured at constant temperature and humidity and tested using a DSR at 2, 6 and 24 hours after preparation. The Testing temperature was 25° C with a frequency of 10 rads/sec using a 25 mm parallel plate geometry, with a 1 mm gap, between 1 and 50 percent strain. They found that the following parameters were statistically significant: curing temperature, curing humidity, curing time, and aggregate mineralogy. Samples cured on the limestone substrate, after 2 hours, gained 75% of the strength value found after 24 hours of curing. Samples cured on a granite substrate, after 2 hours, gained less than half of the total strength value found after 24 hours of curing. They concluded that testing using a dynamic shear rheometer can be used to evaluate the effects of curing temperature, aggregate mineralogy, and curing time on the stiffness of the material. The testing conducted as part of this study only begins to scratch the surface of understanding how asphalt emulsions change their material properties with time and ambient conditions. More research is needed to fully understand the effects of temperature and humidity cycling on the stiffness of the material.

### 3.9.2 Materials and Methods

In this research, different emulsion-aggregate combinations were tested using different climate regimes to study the effect of both aggregate type and climate conditions on stiffness development in the emulsified asphalt binder. To accurately simulate field conditions of a construction site, the temperature and humidity needed to be cycled over time. A BEMCO model FLW-30/65C-340 environmental chamber was utilized to accurately control the temperature and humidity. The rheological testing was performed using a TA Instruments TA550 Asphalt Rheometer.

The DSR used for this research was a controlled stress machine which used a water bath for temperature control. A controlled stress DSR machine applies a known stress to the upper geometry and then calculates the resulting strain within the material. The software associated with the DSR allows the user to specify the type of test performed. In this case, all testing was performed using a strain-sweep from 2 to 100 percent strains and the DSR was allowed five attempts to reach each target strain before moving to the next strain level. Testing times were limited by the water bath associated with the DSR. If the material was tested too soon after preparation, the water bath caused the sample to re-emulsify resulting in unusable data

Boulders were collected from four sources, representing the most commonly used seal coat aggregates in Texas. The boulders were cut into thin slabs for the purposes of this testing. Each slab was then washed to ensure removal of all particulate matter from the surface, and blown dry. The washing process was repeated until the slabs were free from all loose particles. The slabs were then stored at ambient lab conditions until needed. Before using the aggregate slabs for emulsion application, they were placed in the environmental chamber for a given climactic condition for a minimum of 12 hours. This ensured proper conditioning of the aggregate substrate to the ambient surface temperature or the climate regime before the application of the binder.

The specific absorption of each type of aggregate was also measured. The large slabs could not be accurately used to measure the specific absorption so a small piece, approximately 2 inches square, was cut from one slab of each type of aggregate. To ensure uniformity, each side of the smaller rock square had to be a saw-cut side. The squares were then washed in the same manner as the larger aggregate slabs to remove all of the particulate matter and then inserted into a forced draft oven at 300°F for 24 hours. After the 24 hour drying period, the specimens were weighed. Next the rock squares were soaked in a cup of water for 24 hours, wiped with a damp cloth to ensure a saturated surface dry condition, and reweighed. This yielded data about the specific absorption characteristics for each of the four kinds of aggregates tested.

Nine different climactic regimes were used to study the effects of climate cycling on the asphalt emulsion material. Two cycles kept constant temperature and humidity, though different temperature and humidity between the two constant cycles. Two other cycles were formulated to study the specific effects of both temperature and humidity, separately, by keeping one of the variables constant and cycling the other. Weather data was collected from www.weatherunderground.com for two locations in Texas; Lubbock and Beaumont, representing a hot-dry and hot-humid climactic area of the state. Data was collected from May 1 to August 31 for years 2007, 2008 and 2009. The high and low temperature and humidity was averaged, separately, for the two different locations over the three summer period. From this data the environmental cycles were determined by using the average summer conditions for each location. The last three cycles were all based on data collected from field locations. A portable weather station was used to collect temperature and humidity data during three different construction projects located in Giddings TX, Comanche TX, and Floyd County TX. The data was then used to create environmental profiles in the lab to test material also collected at those sites. All other variables, such as specimen preparation and testing temperature, stayed the same regardless of the environmental cycling.

One warm weather binder was tested on four aggregates: two different limestones, a granite, and a sandstone. The testing was conducted using four different temperature and humidity cycles, as well as one of constant temperature and humidity in order to establish a baseline for the binder-aggregate combination. Field samples of HFRS-2P, CRS-2H, and CRS-1P were tested under the three profiles based on field observations and prepared on different aggregates. The specific test combinations are shown in Table 3.11. Testing times were 4, 8, 12 and 24 hours after submersion within the environmental chamber except for the CRS-1P samples which were tested at 12, 16, 20, and 24 hours after submersion within the environmental chamber. The temperatures and associated humidity were on a 24-hour cycle. Samples were inserted into the

environmental chamber for conditioning at time t= 0 and the last test was performed at time t= 24:00 hours. All the environmental profiles are represented graphically in Figures 3.13 through 3.21.

Profile Name	Temperature (°F)	Humidity (% RH)	Limestone 1	Granite	Sandstone	Limestone 2
СТСН	T = 85 Test at 77 (	H = 20	CRS-2	CRS-2	CRS-2	CRS-2
	T1 = 90	H1 = 30				
H&D	T2 = 64	H2 = 80	CRS-2	CRS-2	CRS-2	CRS-2
	Test at 77 (25C)					
	T3 = 95	H3 = 60				
Н&Н	T4 = 72	H4 = 90	CRS-2	CRS-2	CRS-2	CRS-2
	Test at 77 (	25C)				
	T5 = 90	H5 = 60		CRS-2	CRS-2	CRS-2
VT	T6 = 70	H6 = 60	CRS-2			
	Test at 77 (25C)					
	T7 = 78	H7 = 40		CRS-2	CRS-2	CRS-2
VH	T8 = 78	H8 = 80	CRS-2			
	Test at 77 (25C)					
CTCU	T9 = 10	H9 = 50				
2	T10 = 70	H10 = 50	HFRS-2P			HFRS-2P
	Test at 77 (25C)					
	T10 = 76	H11 = 85	-			
H&H2	T11 = 101	H12 = 40	HFRS-2P			HFRS-2P
	Test at 77 (25C)					
	T12 = 77	H13 = 80				
H&H3	T13 = 91	H12 = 68	CRS-2H	CRS-2H	CRS-2H	CRS-2H
	Test at 77 (25C)					
	T14 = 41	H14 = 68				
C&D	T15 = 76	H15 = 23	CRS-1P	CRS-1P		
	Test at 77 (25C)					

 Table 3.11 Experimental Factorial Table



Figure 3.13 Constant Temperature and Constant Humidity 1



Figure 3.14 Hot and Dry Environmental Profile



Figure 3.15 Hot and Humid Environmental Profile



Figure 3.16 Varying Temperature Environmental Profile



Figure 3.17 Varying Humidity Environmental Profile



Figure 3.18 Constant Temperature and Constant Humidity 2 Environmental Profile



Figure 3.19 Hot and Humid Environmental Profile 2



Figure 3.20 Hot and Humid Environmental Profile 3



Figure 3.21 Cool and Dry Environmental Profile

Each specimen was environmentally cycled and tested independently, except for the CTCH cycles. This ensured each specimen was immersed within the environmental chamber for the same amount of time and at the same time during the temperature and humidity cycling. All of the CRS-2 material was sampled daily from a TxDOT certified tank at a plant location to ensure uniformity of the material and to minimize the storage effects of the container on the asphalt emulsion. The material was transported from the plant to the lab using a standard HDPE quart container. The average storage time of the asphalt emulsion in the can before specimen preparation was 18 hours. Field samples were taken directly from the nozzle of a distributor at a construction site and transported back to the lab in a gallon HDPE container to be tested the next morning.

To contain the emulsion on the rock substrate, O-rings, 3 mm thick and 20 mm internal diameter (ID) were used. The asphalt emulsion was heated to the desired field application temperature, 150 °F (65.5 °C), before being poured to the brim of each O-ring on the rock surface. Once the emulsion was poured on the aggregate substrate, the entire aggregate slab, along with the emulsion, was placed in the environmental chamber to start its climate regime. Each sample was tested at 4, 8, 12, and 24 hours after placement in the environmental chamber. The CRS-1P samples were tested at 12, 16, 20 and 24 hours after insertion within the environmental chamber. Samples had to be removed from the aggregate substrate before testing in the DSR. The O-ring was cut using a razor blade and then carefully removed from the periphery of the asphalt disk. The disk was then scraped off of the rock substrate by using a razor blade and affixed to the top geometry of the DSR. Heating the geometry on a hot plate or similar device helped the asphalt

adhere to the geometry. If the plates were heated to too high a temperature though, the samples would no longer be usable because the heat would cause the remaining water to instantly evaporate, changing the characteristics of the material. If the plate was too hot to touch with the hand, it was too hot to load a sample on and had to be cooled slightly before use. The sample was then loaded into the DSR and a strain-sweep test was performed.

### 3.9.3 Results and Analysis

The specific water absorption for each kind of aggregate tested in this experiment is shown in Table 3.12. Since an emulsion must evaporate water out of the mixture in order to gain stiffness, both the climate regime and the water absorption by the aggregate affect the rate at which water can leave the asphalt emulsion mixture. The aggregate with the lowest percent absorption of water is granite, which was an immediate indicator that the samples poured on the granite aggregate should take the longest amount of time to achieve the same level of stiffness as a sample poured on a different aggregate with a higher absorption subjected to the same climate regime. The opposite conclusion is also true for the aggregate which had the highest percent absorption of water, Sandstone. The more water that can be absorbed into the aggregate, the quicker the material can cure and thus gain stiffness. These conclusions can be verified when analyzing the data for the different aggregates and different environmental profiles.

	I doite et		, or p crom	
	Dried (g)	SSD (g)	% increase	
Limestone 1	56.2	56.8	1.10%	
Granite	103.2	103.4	0.20%	
Sandstone	70.1	71.3	1.68%	
Limestone 2	69.8	70.4	0.90%	

Table 3.12 Aggregate Absorption

Strain-sweep test results for all of the aggregates and environmental profiles can be found in Figures 3.21 through 3.35. The Y-axis provides the name of the environmental profile, testing time, and individual percent-strains for the given testing time. The X-axis is the G\* value in Pascals.



Figure 3.22 Asphalt Stiffness for CRS-2 and Limestone 1



Figure 3.23 Asphalt Stiffness for CRS-2 and Limestone 2



Figure 3.24 Asphalt Stiffness for CRS-2 and Sandstone



Figure 3.25 Asphalt Stiffness for CRS-2 and Granite



Figure 3.26 Asphalt Stiffness for CRS-2 and All Aggregates for 4 and 8 Hour Tests



Figure 3.27 Asphalt Stiffness for CRS-2 and all Aggregates for 12 and 24 Hour Tests



Figure 3.28 Asphalt Stiffness for HFRS-2P and Limestone 1



Figure 3.29 Asphalt Stiffness for HFRS-2P and Limestone 2



Figure 3.30 Asphalt Stiffness for HFRS-2P and all Aggregates



Figure 3.31 Asphalt Stiffness for CRS-2H and Limestone 1



Figure 3.32 Asphalt Stiffness for CRS-2H and Limestone 2



Figure 3.33 Asphalt Stiffness for CRS-2H and all Aggregates



Figure 3.34 Asphalt Stiffness for CRS-1P and Limestone 1



Figure 3.34 Asphalt Stiffness for CRS-1P and Granite



Figure 3.36 Asphalt Stiffness for CRS-1P and all Aggregates
Aggregate and Curing			CRS	8-2		HFRS-2P			CRS-2H				
Regi	me	4	8	12	24	4	8	12	24	4	8	12	24
	СТСН				9								
	G&D				10								
	G&G			13	9								
	VH				12								
Limestone 1	VT				75								
	CTCH2												
	H&H2												
	Н&Н3												3
	C&D												
	СТСН	11	9	16	8								
	G&D	9	8	8	8								
	G&G		24	8	8								
	VH		9	10	7								
Limestone 2	VT												
	CTCH2							12	12				
	H&H2						49	15	13				
	Н&Н3									4	4	4	4
	C&D												
	CTCH		10	8	7								
	G&D		12	10	8								
	G&G		24	8	8								
	VH			8	12								
Sandstone	VT				8								
	CTCH2												
	H&H2												
	Н&Н3									4			4
	C&D												
	CTCH				10								
	G&D												
	G&G												
	VH				12								
Granite	VT												
	CTCH2												
	H&H2												
	Н&Н3												3
	C&D												

Table 3.13. Percent Strain when Max. Machine Stress Limit (16.3 kPa) is Reached

Limestone 1 had the greatest stiffness gain after eight hours for the H&D and H&H climate regimes. The VH and CTCH environmental profile yielded similar results to each other after eight hours, though the ultimate strength gain was lower than it was for H&D and H&H. The H&D and H&H climate regimes seemed to have a better effect on the samples than the CTCH profile, in terms of stiffness gains with time. After 24 hours, all the strength values were relatively close to each other except for the VT profile which produced the lowest stiffness results of all of the environmental profiles tested.

Granite had the lowest percent absorption of water of all of the aggregates tested for this experiment, which appears to be the greatest contributing factor to the four-hour test under the H&D profile not being performed. After four hours the sample was too friable to be safely removed from the aggregate substrate. As soon as the O-ring was cut and removed, the top half of the specimen started to crumble and fall from the bottom half. It was also noted that the top half of the specimen was black while the bottom half was still brown, indicating that water was only leaving the mixture through evaporation and not through absorption into the aggregate. Once some of the material was able to be loaded into the DSR it was unable to be trimmed. The material would not "cut" away from the rest of the sample but instead pulled material from the middle of the disk between the plates. The 24-hour test for the H&D profile also experienced problems adhering to the plates of the DSR machine, which is why the data for the 12-hour test appears to be more realistic than for the 24-hour test. The highest stiffness after 24 hours was achieved through the CTCH profile. The H&H climate regime produced the second lowest results, and lower than any other aggregate tested at the same time interval. The VT profile still yielded the lowest stiffness gain within the material and was the lowest overall for the granite aggregate.

Sandstone had a much higher rate of stiffness gain compared to Limestone 1 or Granite. The ultimate stiffness of the materials tested on the Sandstone was two to four times greater than Limestone 1 or Granite. Again the VT profile had the lowest overall stiffness gain of any of the other climate regimes tested. For the CTCH, H&H and VH profiles, the 12-hour stiffness was very nearly equal to the 24-hour stiffness; indicating that the strength gain after 12 hours is not nearly as much as the strength gain during the first twelve hours of curing.

Limestone 2 produced similar results as that of the Sandstone aggregate, though the values were slightly lower. The lowest stiffness gain for all time intervals was again the VT profile. The CTCH profile produced slightly skewed results because the 12 hour test had poor adhesion to the plates of the DSR, which is why the data for the 12 hour test is lower than for the 8 hour test for the CTCH regime. All other tests produced comparable results regardless of the climate regime.

The CRS-2H binder that was tested yielded the greatest stiffness gain for all of the aggregates and environmental profiles tested. This is due to both the extremely thick viscosity of the material as well as the harder base asphalt that was used to make the emulsion. The granite aggregate did finally reach the same ultimate stiffness as the other aggregates but it took 24 hours before it was able to reach that stiffness. The HFRS-2P was taken from a field site and was the only anionic emulsion that was tested for this research.

Many different factors contribute to the stiffness gain within an emulsion. No stiffness can be achieved until water starts to evaporate from the mixture first causing flocculation and then coalescence of the oil mixture. The evaporation rate is directly affected by temperature, humidity, binder-aggregate combination, surface charge, pH of the aggregate surface, and chemical additives used to make the emulsion at the plant. Chemical additives can immediately cause oil particles to begin to flocculate upon contact with an aggregate surface causing separation from the water phase of the mixture. This pushes the water to the surface and aids in evaporation.

## **3.10 Conclusions and Recommendations**

The VT environmental profile yielded the lowest stiffness values for all time intervals and all aggregates tested. The ambient humidity level had a larger impact on the stiffness of the emulsion over time than temperature, for the profiles tested. If the ambient humidity is rising, or static, then the evaporation of water from the asphalt sample is slower than if the ambient humidity is dropping. This is directly related to the stiffness of the material because stiffness can only be gained as water leaves the mixture due to the coalescence and flocculation of the material causing the asphalt particles to form a continuous phase. Before the asphalt is able to eject the water it acts like a liquid and not a solid.

The type of aggregate being combined with a given emulsion, as well as the environmental factors such as temperature and humidity, will change the necessary waiting time between construction operations, mostly brooming and opening to traffic. For instance, the Limestone 2 or Sandstone, with a CRS-2, could be broomed 12 hours after application, as opposed to waiting the standard 24 hours. If Granite were being used though, depending on the environmental factors, longer than 24 hours may be required before the material has gained enough stiffness to broom or open to traffic. The CRS-2H on the other hand could be broomed 4 hours after application on the road due to the high rate of stiffness gain. Performing laboratory testing for the binder-aggregate combination of interest, under appropriate climatic conditions, could save time not only for construction crews but also for commuters by being able to open the road to traffic sooner.

The strain-sweep test, using the aggregate substrate and environmental conditioning, could be implemented into the planning portion of any emulsion project. After the alternative designs for the road have been developed, the specific options for aggregate-binder combinations could be evaluated using a strain-sweep test. This data would provide information about the stiffness gain in the binder with time, given the specific geographic and seasonal environmental conditions. Based on the strain-sweep data for a specific location and the alternative binder-aggregate combinations, a more accurate estimation of the necessary time between application and opening for traffic could be made. Conclusions about the durability of the road can also be developed based on the rate of stiffness gain within the material as well as the ultimate stiffness value of the material. For instance, if binder-aggregate combination 1 has a higher stiffness value than the binder-aggregate combination 2, then the better choice would be the binder-aggregate combination 1 because it will last longer than the binder aggregate combination 2. An economic analysis could then be performed in order to justify the binder-aggregate combination no matter which is chosen. This type of performance based analysis would justify possible additional costs

from using more expensive materials by optimizing both the durability expectations of the road as well as the closure time for construction.

#### ASTM D7000 Sweep Test

ASTM D7000 Sweep Test is a test procedure developed in earlier research (Cornet, 1999; Barnet, McCune and Vopat, 2001) to predict chip loss in emulsion chip seals. This test has the capability to predict surface treatment performance in the formative stage using constructionrelated parameters. This performance test is intended to evaluate the potential curing characteristics of a binder-aggregate combination to ensure that the surface treatment is sufficiently cured before opening the chip seal surface to public traffic.

The sweep test samples are prepared inside a circular area defined by a steel template with a central hole of 300mm over the center of asphalt felt paper disk. Emulsion-aggregate samples prepared on the asphalt felt paper are tested by brushing the sample with a specified nylon brush when the sample is mounted on a platform in the Hobart<sup>TM</sup> Mixer apparatus (Figure 32). The mixing platform is lifted up to make contact with the brush and a shear sweep is applied on the aggregate in the sample using the rotating nylon brush. Aggregate loss due to brushing is used to evaluate the effectiveness of the emulsion-aggregate bond.



Figure 3.37 ASTM D7000 Sweep Test Apparatus and Test Specimen

#### Test Procedure and Experimental Design

The asphalt felt disk is weighed and recorded as the asphalt sample disk weight. The strike-off template is placed over the felt disk, centering the hole of the template over the felt disk. At least 150% amount of the emulsion required to fill the template hole is poured along the top arc of the exposed felt disk. Excess bituminous emulsion is removed with the strike-off rod side to side in 3 seconds. The standard ASTM D7000 specification requires immediate application of the pre-weighed aggregate sample onto the bituminous emulsion using an even back-and-forth motion.

The aggregate is compacted using the specified sweep compacter by rolling it three times in one direction and three half cycles in a perpendicular direction.

The standardized test specification does allow the user to adopt suitable curing conditions and testing parameters to evaluate the performance of bond between the emulsion and aggregate under required conditions. In this testing program, aggregate delay and rolling (compaction) were included as experimental parameters. In the first set of tests for Plainview project materials, 2 min/5 min aggregate delay and 2 min/6 min rolling delay were used. Based on the results from that first series of tests, the parameter values were changed for subsequent tests to 2 min/10 min aggregate delay and 2 min/10 min rolling delay in the hope of better representing the range of field conditions observed.

Three different levels of curing, 12-hours, 24-hours and 72-hours were used for each material combination. A constant temperature and humidity profile was used. At the end of the stipulated curing duration, the sample is taken out of the curing chamber and is turned vertically to remove any loose aggregate by gently hand-brushing the fingers back and forth across the sample. The sample is then weighed and recorded as the initial specimen weight.

The specimen is then attached to the platform in the Hobart mixer using clamps for 180 seconds. During this equilibration time, the brush is secured into the head. The brush is lowered into contact with the seal specimen disk. The Hobart mixer is turned to setting #1 (0.83 gyrations per second) for 1 min. After the brush has come to a complete stop, the brushing platform is lowered and the specimen is unclamped. Any loose or dislodged aggregate, even just hanging on a thread, are removed from the surface. The weight of the specimen is recorded as final specimen weight. The percent mass loss is then determined by

% mass loss = 
$$100 * 1.33 \frac{(Initial specimen weight - final specimen weight)}{(initial specimen weight - asphalt sample dish weight)}$$

The sweep ratio is the ratio of the swept area to the specimen area. The standard specimen sweep ratio is considered 1.33 in the ASTM specification. However, it is recommended that it is determined separately for each test program (Shuler, 2009).

Several iterations of "preliminary" tests were conducted to make early assessment of the ASTM D7000 Sweep Test method. In one of the earliest tests, materials (CRS-1P and a Quartzite aggregate) sampled from a cool weather field test project was tested The standard sweep test specifies several aggregate gradation and size specifications to choose from and all these gradation-size options have particles that are smaller than the sizes typically used in TxDOT projects. Table 12 shows results for this material combination in which an aggregate size larger than those specified in the standard specification was tested. A TxDOT standard gradation 4S was selected to see how the larger particles perform under the standard test conditions. Aggregate delays of 2 and 5 minutes were used along with roller delays of 2 and 6 minutes.

As one might expect, the percent aggregate loss calculated for those conditions turned out to be smaller than typically observed for standard (smaller) aggregate particle sizes. Larger particle sizes generally are more likely to have higher stability due to their larger weight. Specimens

were tested at 12 hours, 24 hours and 72 hours after they were prepared and all were cured under a variable climate profile (in an environmental room) similar to the Lubbock area cool weather profile outlined under the DSR Sweep Test climate regimes. Results from this series of tests showed that as expected, percent aggregate loss decreased as curing time increased from 12 to 72 hours as one might anticipate. Asphalt-aggregate binder takes time to develop and more curing time would allow the bond between aggregate and the asphalt binder strengthen.

On the other hand, the aggregate delay did not show much sensitivity to percent aggregate loss in the range of values selected (i.e. 2 and 5 minutes). It is possible that using a softer binder such as CRS-1P might require more time beyond the six minutes considered in this set of tests to develop a bond. However, increasing the roller delay from two to six minutes resulted in a reduced aggregate loss and appeared to have helped the bond development. This suggests that the observation made by Kim and Lee (200) – that rolling of an emulsion seal too soon might disturb the bonds being developed – has merit. Further testing may be warranted to carefully investigate the influence of roller delay on aggregate retention in emulsion seals.

			· · · · · · · · · · · · · · · · · · ·	-	
Test Conditions	Initial Specimen	Asphalt Sample Disk	Total Weight before Sweep	Total Weight after Sweep	% Aggregate
	Weight	Weight (g)	(g)	(g)	LOSS
2-2-12	515.7	67.4	506.6	468.8	11.4
2-2-24	532.9	67.4	523.3	482.2	12.0
2-2-72	508.3	67.4	497.3	465	10.0
2-6-12	534.3	67.4	523.3	481.7	12.1
2-6-24	517.5	67.4	506.9	483	7.2
2-6-72	521.2	67.4	507.9	483.3	7.4
5-2-12	520.2	67.4	507.5	473.9	10.2
5-2-24	536.4	67.4	521.5	476.8	13.1
5-2-72	529.6	67.4	516.2	483.7	9.6
5-6-12	484.6	67.4	470.1	424.8	15.0
5-6-24	522	67.4	505.7	474.8	9.4
5-6-72	535.5	67.4	513.7	492.8	6.2

Table 3.14 Sweep Test Results for CRS-1P (Holly) and Grade 4S Quartzite Aggregate

Note: Test condition notation is aggregate-delay-rolling delay-curing duration.

Table 3.15 shows results for a CRS-1P from a different asphalt plant (Ergon – Waco) and the same Quartzite aggregate as shown in Table 3.14. The aggregate tested in this case was also a Grade 4 quartzite used in the Texas Panhandle area. In this series of tests, the aggregate delay values of two minutes and ten minutes, along with roller delays of two minutes and ten minutes were used. Specimens were tested at 12 hours, 24 hours and 72 hours after they were prepared and all were cured under a variable climate profile (in an environmental room) similar to the Lubbock area cool weather profile outlined under the DSR Sweep Test climate regimes. It can be seen from results in Table 3.14 that in the case of 2-minute aggregate delay and 2-minute rolling delay (2-2-X specimens) and for 10-2-X specimens, the aggregate-binder bond showed significant improvement from 24 to 72 hours of curing. This is intuitively evident for softer binders such as CRS-1P, which take some time to cure (lose the moisture) and develop the bond.

For 2-10-X and 10-10-X specimens, no such improvements were observed, possibly indicating to an influence from the later rolling (i.e. rolling delay of 10 minutes).

Test Conditions	Specimen Weight after Curing (g)	1st Loss (g)	2nd Loss (g)	Final Specimen Weight (g)	% Aggregate Loss
2-2-12	641.1	38.5	54.3	542.3	25
2-2-24	637.3	43.8	53.5	539.1	25
2-2-72	634.2	11.4	23.4	598.1	12
2-10-12	643.5	26.3	59	559.7	23
2-10-24	640.5	27.2	41.9	573.7	19
2-10-72	632.2	34.5	45.4	550.2	22
10-2-12	639.9	25.6	69.9	541.4	24
10-2-24	638.8	37.1	57.9	545.4	25
10-2-72	631.4	25.7	32.8	571.6	17
10-10-12	631.3	27.9	90.7	507.3	31
10-10-24	636.4	28.6	28.1	579.6	16
10-10-72	634.1	24.2	47.9	560.1	21

Table 3.15. Sweep Test Results for CRS-1P (Ergon-Waco) and Grade 4S Quartzite Aggregate

Note: 1<sup>st</sup> loss is aggregate loss (in grams) when sample is swept lightly using fingers while holding vertically. The 2<sup>nd</sup> loss is the loss occurred on the testing machine.

Table 3.16 shows results for an anionic HFRS-2P emulsion and <sup>1</sup>/<sub>4</sub>-inch lightweight aggregate combination. From this point on, tests were limited to one of the aggregate gradations recommended in the ASTM specification D7000. In this series of tests, percent aggregate loss was determined at curing durations of 12, 24 and 72 hours, and the curing was done in this case in a forced-draft oven at a temperature of 109 °F. The primary observation from the results in Table 3.16 is that aggregate loss did not show sensitivity to two- and ten-minute aggregate delay and 2 and 10 minute rolling delays. Field observations made over many years have shown that the highly porous TXI lightweight aggregate performs well with emulsified asphalt. The high porosity and the ability of the asphalt emulsion to penetrate into the aggregate quite easily compared to other normal weight aggregate might be a reason for the relative insensitivity to experimental conditions investigated.

Test Conditions	Specimen Weight after Curing (g)	1st Loss (g)	2nd Loss (g)	Final Specimen Weight (g)	% Aggregate Loss
2-2-12	348.8	1.1	3.4	343.7	22
2-2-24	352.8	2.6	2.2	348	23
2-2-72	346	1.9	1.5	342.4	23
2-10-12	355	1.1	1.2	353	22
2-10-24	356.1	4	2	350.3	23
2-10-72	344.4	3	0.9	340.7	23
10-2-12	349.8	2.1	1.5	346	18
10-2-24	348.5	3.5	2.5	342.8	22
10-2-72	347.5	1.9	1.7	344.1	23
10-10-12	345.8	7.7	6.2	331.3	26
10-10-24	347.6	5.5	3.6	338.3	25
10-10-72	346.2	2.9	2.7	340.3	23

Table 3.16 Sweep Test Results for HFRS-2P (Ergon – Austin) and Lightweight Aggregate (TXI)

Table 3.17 shows results from the ASTM D7000 Sweep Test for the CRS-2H and Limestone aggregate combination. These materials were sampled from a project in Comanche, Brownwood District. The binder was specifically produced for the requirements stipulated by the district using a high-float emulsion. The district project manager indicated that the binder was specifically requested to reduce drain-down of the binder in the rolling hills during hot summer days. The limestone aggregate for this project was obtained from Vulcan Materials quarry in Brownwood.

In this series of tests percent aggregate loss was determined at curing durations of 12, 24 and 72 hours and the curing was done in this case in a forced-draft oven at a temperature of 109 °F. Several observations can be made from the results shown in Table 15. In general, the results show gradually decreasing aggregate loss (improving bond) except in the case of 10-minute aggregate delay and 2-minute rolling delay in which case the aggregate loss increased with increasing curing duration

One key observation that is evident from these results is the fact that aggregate-emulsion bond appears to be the result of a complex set of interactions between the emulsion binder, aggregate, aggregate delay, rolling delay, and the curing duration.

Research done at Colorado State University by Shuler (2009) studied the ASTM D 7000 Sweep Test. They have suggested some modifications to this test method which will be published in NCHRP Report 680. Montana DOT has developed a field version of this Sweep Test and NCHRP Report 680 is expected to have more details of their test method.

Test Conditions	Specimen Weight after Curing (g)	1st Loss (g)	2nd Loss (g)	Final Specimen Weight (g)	% Aggregate Loss
2-2-12	514.2	7.9	1.5	505.0	11.3
2-2-24	523.2	3.5	2.0	517.8	10.7
2-2-72	516.3	1.9	1.0	513.6	9.3
2-10-12	515.6	3.8	7.7	504.2	11.7
2-10-24	517.5	11.4	5.0	501.2	13.3
2-10-72	521.9	3.5	0.0	518.4	10.2
10-2-12	517.9	3.9	5.5	508.3	9.7
10-2-24	514.8	4.0	3.9	506.9	10.5
10-2-72	518.1	8.5	0.0	509.8	11.0
10-10-12	526.8	9.7	11.3	505.1	13.0
10-10-24	511.5	13.7	6.9	491.0	13.9
10-10-72	522.2	2.3	0.0	519.9	9.3

Table 3.17 Sweep Test Results for CRS-2H (Ergon-Waco) and <sup>1</sup>/<sub>4</sub>-inch Limestone Aggregate (Vulcan Brownwood)

Based on the ASTM D7000 sweep test experiments conducted at TechMRT, the following observations can be made.

- The aggregate size used in the test (1/4-inch nominal maximum size) is too small to effectively evaluate the aggregate-binder bond. This nominal maximum aggregate size is much smaller than the aggregate sizes used in Texas.
- The aggregate quantity recommended in the ASTM D7000 test results in much larger spread coverage of aggregate in the seal than that desired by TxDOT. This also causes the aggregate loss numbers to be artificially inflated because many of the aggregate particles have no room to come into contact with the binder during compaction. Therefore, the aggregate loss does not reflect the true state of the aggregate-binder bond.
- The small aggregate particles cause the bristles of the sweeping brush to come into contact with the asphalt emulsion and the stickiness in the brush that is caused by this can influence the proper functioning of the sweeping mechanism.
- Use of a larger aggregate size that is more representative of TxDOT specifications for surface treatments would require a significant modification of the test including brush specifications and rotating speed.

The research team does not recommend that the ASTM D7000 Sweep Test be used by TxDOT unless a modification to the test method can be developed.

#### Aggregate Pull-Out Test

The pull-out test was designed to examine the bond strength between an asphalt binder and an aggregate. Aggregate was prepared by cutting half inch diameter cores from larger stones collected at quarries commonly used by TxDOT. The long cores were then cut into lengths of half an inch, and weighed, in order to maintain uniformity of the aggregate samples. It was decided to use circular aggregate cores, to avoid any edge stresses, which would be caused by other shapes, thereby causing a uniform stress distribution. After the aggregate was cut into the appropriate size pieces, nails were attached to one side of the aggregate using a two phase epoxy mixture to ensure that the mode of failure would be with the asphalt-aggregate bond and not with the nail-aggregate bond. The epoxy was allowed to set for a minimum of 12 hours before the tests were conducted. Metal plates were custom prepared in order properly fit onto the testing apparatus, also to allow pouring of the sample onto a uniform surface for testing. A layer of asphalt felt paper, to mimic paving conditions on top of an existing asphalt pavement, was adhered to the metal plates using a silicon solution and allowed to dry. Once the asphalt felt paper was adhered to the metal plates a Nitrile Buna Rubber O-ring, (ID 4" and thickness 3/32") was placed on the felt paper in order to contain the asphalt sample when poured. The emulsion film thickness was decided to be 2mm, to mimic the standard protocol in the industry, thereafter; deciding upon the 3/32" thickness for the O-ring. In order to ensure uniform samples the total weight of asphalt, 19.68 grams, was calculated based on a film thickness of 2 mm for the given diameter of the O-ring. The metal plate with the asphalt paper already attached was placed on a digital scale and set to zero. The asphalt emulsion was heated to the desired application temperature, using a water bath and continuously stirred. Once the desired temperature was reached the emulsion was slowly poured onto the center of the metal plate resting on the scale until the desired weight, 19.68 grams, was reached. After the emulsion was prepared, the aggregate was embedded into it after a given time delay (time from pour to aggregate embedment), and the sample with the aggregate was cured at a given *environmental cycle* condition (time from embedment to pull-out) until tested. These time delays are shown in Experimental Factorial (Tables 3.18 – 3.19). Each X in the Tables 3.18 and 3.19 indicate a test replicate for that cell.

Aggregate	Aggregate	Time from	Aggregate Type Used							
Embedment	Delay	Aggregate Embedment to Pull-Out	Lime- stone 1	Sand- stone	Granite	Lime- stone 2				
Forced Settled	5mins	24 hrs	XX	XX	XX	XX				
Gravity Settled	5mins	24 hrs				XX				

Figure 3.37 images depict the different stages of the pull-out test. These pictures show the emulsion on the steel plate contained by the O-Ring, after the aggregate with fixed nail is embedded, and while in the process of the test. For the test two different binders, CRS-2 sampled from a TxDOT approved plant tank and CRS-2H sampled in the field from a construction site, were used. The CRS-2 was tested using Limestone 1, Limestone 2, Sandstone, and Granite. The CRS-2H was tested on Limestone 2 only. The test was conducted using a

500N Quantrol<sup>™</sup> Advanced Force Gauge. This gauge was mounted on a Quantrol<sup>™</sup> mechanical test stand, which could control the rate at which the aggregate was being pulled from the emulsion sample. This rate was set at a constant 1.5 in/min, to simulate possible field conditions. The force gauge was connected to a computer and Data Plot-X<sup>™</sup> data capture software was used to collect data for the tests (see Tables 18-20). The nominal bond strength, in psi, was calculated using the measured force and the cross-sectional area of the cylindrical aggregate specimen.

Aggregate Delay (Minutes)	Temperature of Specimen at Time of Pull-Out (°F)	<b>Time from A</b> ggregate Embedment to Pull-Out	Experiments and Replicates
		15 min	
		60 min	
	32 °F	120 min	
		24 hrs	XX
		15 min	XX
		60 min	XX
5	70 °F	120 min	XX
_		24 hrs	XX + (XX -
			Var. Temp)
		15 min	17
	1.40.05	60 min	
	140 °F	120 min	
		24 hrs	XX
		15 min	
	22 %E	60 min	
	32 °F	120 min	
		24 hrs	
		15 min	
10	70 °F	60 min	
10	70 °F	120 min	
		24 hrs	XX
		15 min	
	140.95	60 min	
	140 F	120 min	
		24 hrs	
		15 min	
	22 °E	60 min	
	32 F	120 min	
		24 hrs	
		15 min	
15	70 °F	60 min	
15	70 F	120 min	
		24 hrs	XX
		15 min	
	140 °F	60 min	
		120 min	
		24 hrs	

Table 3.19 Experimental Factorial for CRS 2H

The first pull-out test conducted was to determine the pull-out bond strength between CRS-2 and different aggregate types commonly used in Texas surface treatments. Through this analysis, it was determined that the Limestone from Eastland (LS-1) outperformed the other three aggregate types (Figure 3.38). The subsequent tests were conducted using that limestone (LS-1). The bond

strength between the LS-1 and CRS-2H emulsion seems to be greatest at moderate temperatures (70°F), with lower time delays. These results also appeared for the 24-hour environmental cycle (Figures 3.39 and 3.40). Next, LS-1 was tested under extremely short duration environmental cycle. As expected, even with a 5 minute time delay the samples tested after 15 minute environmental cycles had very poor results, while the same test conducted at 2 hours had significantly higher bond strength. These results show that an appropriate cure time is 24 hours, since as the cure time decreases down to 2 hours the bond strength decreases by close to 44%. The testing also shows conclusive evidence that the shorter time delays perform very well; therefore, it is recommended to use 5 minute or lower aggregate time delay.



Figure 3.38. Images Depicting Different Stages of the Pull-Out Test

	L	LS-1		SS Replicate		GR		5-2	LS-2 Replicate		
	Rep	olicate			Replicate		Replicate		(Gravity		
									Settled)		
	1	2	1	2	1	2	1	2*	1	2	
Max. Force	18.4	4.02	10.8	10.28	5.24	5.04	4.46	-	2.98	4.88	
(lb)	8										
Aggregate	0.49	0.49	0.50	0.49	0.49	0.49	0.50	0.49	0.50	0.49	
Diameter											
(in)											
Peak Bond	96.7	21.3**	55.7	54.5	27.4	26.7	22.7	-	15.4	25.9	
Stress (psi)											

Table 3.20 Pull-Out Test Results for CRS-2 and Different Aggregate Types

\*Problems associated with the specimen replicate; \*\*Test specimen failed premature



Figure 3.39 Pull-Out Bond Strength Between CRS-2 and Different Aggregate Types at 24 hrs

	Pull-Out (a	a) 15-Min	Pull-Out	@ 1 Hour	Pull-Out @ 2 Hours				
	1	2	1	2	1	2			
Max. Force (lb)	0.5	0.44	2.22	2.18	3.24	4.18			
Aggregate Diameter (in)	0.50	0.50	0.51	0.49	0.49	0.49			
Peak Bond Stress (psi)	2.6	2.3	10.9	11.4	17.2	22.2			
Avg. Peak Stress (psi)	2.4		11	.1	19.7				

Table 3.21Pull-Out Test Results for CRS-2H and Limestone-1 for 5-min Aggregate Delay at<br/>70°F Pull-Out Temperature



Figure 3.40 Variation of Pull-Out Bond Strength with Time for CRS-2H and Limestone-1

14010 5.22 1	un-Out	1 Cot Kes	uns ioi	CR0-21		meston	$C^{-1}$ at $2^{-1}$	+ Hours	)		
	Pul	l-Out	Pull	l-Out	Pull	-Out	Pull	-Out	Pull-	Out	
	a	@ 70 °F		@ 70 °F		@ 70 °F		@ 50 °F		@ 120 °F	
	5-	min	10-	min	15-	min	5-r	nin	5-n	nin	
	Agg	regate	Agg	regate	Aggr	regate	Aggr	regate	Aggr	egate	
	D	elay	De	elay	De	lay	Dela	y; 32	Delay	140 °F	
		-						°F			
	1	2	1	2	1	2	1	2*	1	2	
Max. Force (lb)	8.04	5.4	5.4	5.42	5.5	5.04	4.2	0.66	2.02	2.84	
Aggregate Diameter (in)	0.49	0.49	0.50	0.50	0.50	0.49	0.50	0.50	0.50	0.50	
Peak Bond	42.1	28.3	27.5	28.0	28.0	26.7	21.4	3.4	10.3	14.7	
Stress (psi)											
Avg. Peak Stress (psi)	3	5.2	2'	7.7	27	7.4	12	2.4	12	.5	

Table 3.22 Pull-Out Test Results for CRS-2H and Limestone-1 at 24 Hours



Figure3.41 Variation of Pull-Out Bond Strength with Aggregate Delay and Temperature for CRS-2H and Limestone-1 at 24 hrs

# Chapter 4. Characterization of Binder Breaking Trends

## 4.1 Introduction

An asphalt emulsion is normally a heterogeneous system with two or more liquid phases consisting of a continuous liquid phase and at least a second liquid phase, dispersed in the first phase in the form of fine droplets (ISO 862, 1984). This implies that an emulsion is an imposed mixed state which qualifies as thermodynamically unstable or metastable. Natural forces tend to separate these two phases which will eventually form a stable state but on a time scale this process might last from a few seconds to months (SFERB, 2008). Therefore, in order to reduce the chances of a spontaneous separation of the two phases, surface active agents (aka surfactants/soaps/emulsifiers) are introduced that reduce the surface tension of the droplets of the dispersed phase and stabilize the two phases.

The term surface treatment refers to single, double, or triple application of an asphaltic material, each covered with aggregate and constructed over an existing pavement or prepared base course. Seal coat and surface treatment practitioners encounter a number of construction related problems involving emulsified and cutback asphalt binders. One of the major constructability issues faced in the field is loss of aggregate. Loss of aggregates may be triggered by a number of factors such as improper timing of placement of emulsified asphalt or cover aggregates, insufficient binder to cement the cover aggregates to the existing surface, and allowing fast traffic before proper adhesion is developed. However, it is believed that the underlying reason for loss of aggregates is due to inadequate development of the adhesive bond between the emulsified binder and the cover aggregates due to inadequate curing may be sheared by the moving loads. Thus it is necessary to estimate the curing rate of the emulsion as it breaks and hardens and optimize the time to open the road to traffic at highway speeds. Knowing the rate of curing (and breaking) of the emulsion is also important to predict the rate at which it gains strength.

Epps et al. (2001) reported that loss of aggregates or raveling at high and low temperatures were the primary failure mechanisms that were controlled, at least in part, by the binder properties. At high temperatures they hypothesized that loss of aggregates was triggered by the high shear stresses generated on the road surface under the action of wheel loads. When these shear stresses exceed the shear strength of the asphalt binder, the wheel loads knock the aggregates off from the road surface. The PG binder specification uses the term G\*/sin  $\delta$  as the high temperature parameter under the assumption that binders with lower amount of work dissipated in a load cycle (or higher G\*/sin  $\delta$ ) would be more resistant to permanent shear deformation or rutting (Roberts et al., 1996). In the context of surface treatment binders, higher values of G\*/sin  $\delta$ would reflect higher resistance to shear deformation under the action of traffic loads. Recent studies have pointed out that the thin film of asphalt residue left behind from a fully cured emulsion is subjected to high levels of shear strains as soon as the surface treatment is opened to traffic (Redelius et al., 2006). Thus it is essential that the binder has developed adequate strain tolerance or stiffness to prevent aggregate loss due to moving wheel loads. Testing the emulsion residue in a dynamic shear rheometer (DSR) can simulate this particular form of distress provided that the test is conducted at high strain levels (Hanz et al., 2009). In a recent study, select parameters from a strain sweep test on a variety of emulsions were shown to have a strong correlation with the results from the ASTM D7000 sweep test (Kucharek et al., 2007) in which a surface seal is brushed repeatedly to evaluate aggregate retention.

Research studies that have looked into the effect of surfactants on asphalt emulsion stability have reported that the interfacial tension between the asphalt and water molecules actively contributes to the rate of setting in surface treatment applications. However, other factors like rheological properties, interfacial viscosity and electro-kinetic properties of the emulsion can be key factors and will therefore determine its stability, curing and breaking characteristics. However, details like these are not published and therefore a mechanistic approach to model breaking and setting of emulsion is an infeasible option. It was therefore more practical to follow an empirical approach where the exact similar conditions in terms of material and their typical application rates in the field are replicated in a controlled environment within the laboratory which can then be analyzed using statistical techniques to derive meaningful conclusions from observed data.

# 4.2 Objective

Early loss of aggregates continues to be one of the major problems with chip seal applications. Such failures are often attributed to incompatibility between aggregates and the binder. Most early failures in chip seals are either due to construction related issues or premature opening of the road to traffic. A common rule of thumb that is popular in the chip seal industry is traffic should be restricted to a maximum speed of 25 mph until the emulsion sets, rolling has been completed and the first brooming is done.

The purpose of this study is to quantify what is the optimal time to open to traffic for a newly constructed chip seal based on a scientific approach that captures the several dimensions which are understood to be instrumental in determining the setting characteristics of the emulsion in the field. The study uses statistical modeling tools to derive a model that can predict the curing trends of the emulsion as function of time, changing weather conditions, nature of emulsion and type of aggregates. It has been also pointed out in past research that the stiffness of the binder plays a key role in determining the resistance to raveling under shearing action of traffic loads. Therefore the statistical model that was developed as part of this study has been also linked to the binder's stiffness in order to focus on performance related properties of the chip seal. Finally, field measurements were conducted to determine the most optimal time for a road to be opened to traffic which were used as thresholds that define the boundary that separates superior performing chip seals to those that show early signs of distress.

# 4.3 Experimental Design

As already discussed, raveling is one of the most prominent forms of distresses seen with chip seals. While raveling may be due to a variety of reasons, the focus area for this study is investigation of the optimal timing of traffic on the performance of the chip seal. In order to achieve the objectives, a carefully designed experimental plan was devised for the study which included running a simple mass loss test parallel to shear testing on the rheometer. While an ordinary mass loss test has the potential to capture the curing and setting characteristics of the

emulsion, shear testing on the rheometer provides the opportunity to monitor the evolution of the rheological properties of the binder as a function of time. The advantage offered by running these two tests lies in the ability to correlate the results of one with the other as well as using a simple mass loss test as a surrogate for shear testing. To put things in perspective, the shear testing has the potential to throw light on the performance properties of the binder but might need a more detailed setup and a controlled environment which confines it within the perimeters of the laboratory. On the contrary, a mass loss test does not provide information on the performance characteristics of the binder. However its strength lies in its simplicity, portability, ease of execution and the ability to provide a measure of the amount of curing.

The experimental variables that were investigated as part of this study are as given below:

- Sampling Time: 2 hours, 6 hours, 24 hours and 48 hours
- Curing Temperature: 25°C (77°F) and 50°C (122°F)
- Type of Aggregates: Limestone Grade 4, Quartzite Grade 4 & Light Weight Grade 4
- Type of Emulsion: CHFRS-2P, CMS-1P, CRS-2, CRS-2P, HFRS-2 & HFRS-2P

#### 4.3.1 Methodology

Sample preparation is one of the most crucial aspects of an experimental study as repeatability and consistency of measurements depend on the steps followed during this step. As for this study is concerned, the process involved the following steps:

- The emulsion is heated to 60°C (140°F) in a closed container to minimize evaporative loss of water following which a sample weighing around 30 grams is poured on a flat pan with a diameter of 140 mm which gives a film thickness of approximately 2 mm. The application rate for the emulsion was determined after carefully analyzing the typical application for a chip seal job in Texas, which was around 0.03 litres/sq. cm.
- The next step in the procedure involved adding aggregates to the emulsion which was typically done 5 minutes from the time the emulsion was applied on the flat pan to let the emulsion break when brought in contact with a mineral surface. The aggregates were prewashed and oven dried before they were added to the emulsion. Typical stone application rates for limestone and quartzite aggregates were chosen as 4 kg/sq. m. and that for light weight aggregates it was about 3 kg/sq. m.
- Curing rates of emulsions were measured at 2 different temperature-humidity profiles. The curing temperature of 42.5°C with 16% relative humidity was achieved in a thermostatically controlled oven while the 25°C with 57% relative humidity conditioning was attained under normal room temperature conditions in the laboratory.
- The weights of the samples were measured at the specified time intervals of 2, 6, 24 and 48 hours to determine the amount of evaporative water loss. As for the shear testing is concerned, sample preparation normally involves reheating the samples and molding according to the size of the plates that are used for the testing procedure. However, the binder in question involves a partially cured emulsion sample and reheating the sample will render the test useless as all the water will be lost in the process. Testing a reheated sample will therefore will be equivalent to testing the base binder in the emulsion. It is because of this reason that the sample for shear testing was taken directly from the flat pans in which the emulsions were being cured and placed on the lower plate of the rheometer. The shear tests on the emulsion samples were performed at 63°C to capture the in-field pavement temperatures typically seen in Texas. Adopting a temperature as

high as 63°C also allows for significant reduction in the torque capacity of the instrument. The shear testing on the rheometer was performed with 25 mm parallel plate geometry in a controlled stress mode on a Bohlin DSR-II Rheometer.

#### 4.4 Results

Figure 4.1 presents the mass loss measurements that were recorded in the laboratory for a CHFRS-2P emulsion sample. The symbols ■, ◆ represent the amount of moisture loss from the emulsion when exposed to the low and high temperature profiles while the color codes Blue, Green, Red and Black represents the Limestone, Quartzite, Light Weight and the control experiment respectively.



CHFRS-2P

Figure 4.1 Rate of Evaporative Loss of Water for different Emulsion-Aggregate-Temperature Combinations

As can be seen from Figure 4.1, the rate at which the emulsion sets depends on the type of exposure in terms of temperature as well as the nature of aggregate that is in contact with the emulsion. It should be noted in this context that the amount of water lost has been expressed as a percentage of the total water present in the system as part of the original formulation. The trend in the data suggests that temperature has a significant influence on the rate at which water evaporates. It is also evident from Figure 4.1 that the amount of water lost due to evaporation reaches a plateau as it gets closer to 100%, which is complete removal of water from the system. Figure 4.2 presents the stiffness measurements at 63°C and 10 Hz for the emulsion residue recovered from a CHFRS-2P emulsion sample in the laboratory with the legends carrying the same significance as in Figure 4.1.

#### CHFRS-2P



Figure 4.2 Complex Modulus of the Residue for Different Emulsion-Aggregate-Temperature Combinations

As stated, the experiment included measuring the amount of water lost as well as the mechanical properties of the emulsion residue as the emulsion continues to set with the aggregate. Unlike in the previous case where the goal was to measure the amount of evaporative loss of water, the complex modulus of the residue fails to reach a plateau within the time period of the experiment. The stiffness of the residue increases as the emulsion cures and sets. However its stiffness continues to change due to aging of the residual binder and this effect is more pronounced where the residual binder is exposed to a higher temperature.

# 4.5 Statistical Modeling of Setting Rates and Complex Modulus of the Emulsion Residue

One of the focus areas of this study involves looking at the rate of evaporation of water as the emulsion sets. The maximum amount of water that can evaporate in this scenario should therefore be limited to the total amount of available water in the emulsion. However not all of the available water will have access to the evaporation front and thus the water present in the superficial layer will evaporate much faster as opposed to water present in the bulk of the emulsion. Due to effects of skinning (6, 7, 10), water present in the core of the emulsion has to diffuse through the emulsion film to the surface in order to evaporate. This suggests that the type of emulsion will also influence the rate of evaporation. Past studies (8, 10) have pointed out that the mineralogy of the aggregates plays a major role in the breaking of emulsions and therefore

the type of aggregates should also influence the setting characteristics of the emulsion. The amount of water lost to evaporation can be modeled as a set of covariates that represent the weather conditions, binder and aggregate characteristics. It is also important to choose a suitable empirical form for the model such that its values are constrained within reasonable physical maximum and minimum bounds. After careful examination of the data trends, the empirical form of the model that was chosen for this study is given below,

$$y = 100 \left\{ 1 - e^{\left[ \left(\beta_0 + \beta_1 Q + \beta_2 W + \beta_3 VPD + \beta_4 VPD \times \frac{CMS}{HFRSP} + \beta_5 VPD \times CRSP + \beta_6 VPD \times HF \right) x^{\beta_7 VPD + \beta_8 CMS + \beta_9 HFP + \beta_{10} CRS + \beta_{11} CRSP} \right] \right\}}$$
  
Eqn. 4.1

#### Where,

*y* = Response Variable (% of water lost in a given interval of time),  $\beta_0, \beta_1, \beta_2, \beta_3, \beta_4, \beta_5, \beta_6, \beta_7, \beta_8, \beta_9, \beta_{10}, \beta_{11}$  = Regression Coefficients *Q*, *W* = Dummy variables for the three different types of aggregates – Quartz & Light Weight *VPD* = Average hourly Vapor Pressure Deficit *CMS*, *HF*, *CRS*, *HFP*, *CRSP* = Dummy or indicator variables for emulsions CMS-1P, HFRS-2P and CRS-2, HFRS-2P and CRS-2P (either 1 or 0)

It should be noted that the setting rate for the emulsion type CHFRS-2P with no aggregate was considered as the baseline.

Table 4.1 presents the summary of the statistical analysis and the relevant model parameters for predicting the amount of water lost to evaporation.

Effect	Regression Coefficient	t-stat	p-value
β(Intercept)	-0.1833	-18.3	0.00
$\beta$ (Vapor Pressure Deficit)	2.1764	26.4	0.00
β(Quartz)	0.0136	2.6	0.01
β(Light Weight Aggregate)	0.0099	1.9	0.03
β(CMS-1P)	0.0566	1.4	0.08
β(HFRS-2P)	-0.0518	-1.9	0.03
β(CRS-2)	0.0434	1.6	0.06
β(CRS-2P)	0.3129	7.8	0.00
β(Intercept: VPD)	0.0344	4.5	0.00
β(Intercept: VPD×CMS/HFRSP)	0.0226	3.5	0.00
β(Intercept: VPD×CRS-2P)	0.0404	5.2	0.00
β(Intercept: VPD×HFRS-2)	0.0725	3.2	0.00

Table 4.1 Model Parameters for Prediction	ction of the Amount of `	Water Lost to Eva	poration
---	--------------------------	-------------------	----------

It can be seen from the results presented above that some of the variables included in the experimental design are statistically insignificant. It should be remembered that the prediction model that has been presented has been developed on the basis of a limited number of measurements which were conducted over the duration of this study. It is, therefore,

recommended that the predictive model should include these variables until further evidence is found which suggests that these variables do not contribute to the rate of setting of emulsions. Therefore the final predictive model for determination of the amount of water lost due to evaporation from the emulsion is given below,



Figure 4.3 Observed v/s Predicted Water Lost to Evaporation

The experiment included measuring the amount of water lost as well as the mechanical properties of the emulsion residue, as the emulsion continues to set with the aggregate. Unlike in the previous case where the goal was to measure the amount of evaporative loss of water, the complex modulus of the residue fails to reach a plateau within the time period of the experiment. The stiffness of the residue increases as the emulsion cures and sets. However, its stiffness continues to change due to aging and thixotropic effects of the residual binder and this effect is more pronounced where the residual binder is exposed to a higher temperature. The standard error of prediction was found to be 6.1%, which is low considering the differences that exist between the materials and exposure conditions applied.

Studies have suggested that the resistance to raveling or aggregate loss during the early phase is governed by the stiffness of the binder (1, 4) and its resistance to shear deformation. In an

attempt to determine the optimal time to open a newly constructed chip seal to traffic, it is therefore important that the film of asphalt residue develops sufficient stiffness to resist high levels of deformation due to the shearing action of traffic loads which provides the inspiration behind modeling the stiffness of the residue as function of changing weather conditions, time and material properties. Initial analysis of the data suggested that the stiffness of the residual binder increases as the emulsion sets due to gradual loss of water and aging due to oxidation. Aging of binder is a never ending process though the kinetics of such a reaction will be affected by factors like temperature and pressure. Because of the contribution due to aging, the stiffness of the residue fails to reach an asymptote which makes using an exponential model inappropriate for modeling the stiffness of the binder. Therefore a power law was considered to be suitable for modeling the complex modulus of the residue obtained from curing of emulsions. The empirical form of the model is:

 $y = (\beta_0 + \beta_1 L + \beta_2 Q + \beta_3 W + \beta_4 VPD + \beta_5 (VPD \times CHFP) + \beta_6 (VPD \times CRS) + \beta_7 CRS + \beta_8 (VPD \times CRSP)) x^{\beta_9 + \beta_{10}(VPD \times CHFP/CRSP) + \beta_{11}CRS + \beta_{12}(VPD \times CRS) + \beta_{13}CHFP/CRSP}$ 

Eqn. 4.3

Where,

*Y* = Response Variable (Stiffness of the residue in Pa),  $\beta_0, \beta_1, \beta_2, \beta_3, \beta_4, \beta_5, \beta_6, \beta_7, \beta_8, \beta_9, \beta_{10}, \beta_{11}, \beta_{12}, \beta_{13}$  = Regression Coefficients *L*, *Q*, *W* = Dummy variables for the three different types of aggregates – Limestone, Quartz & Light Weight *VPD* = Average hourly Vapor Pressure Deficit *CMS, CHFP, CRS, CRSP* = Dummy variables for emulsions CMS-1P, CHFRS-2P, CRS-2 and CRS-2P

It should be noted that the setting rate for the emulsion type HFRS-2P with no aggregate was considered as the baseline and therefore the regression coefficients capture the incremental change with respect to a residue obtained from curing of a HFRS-2P emulsion.

The model parameters for Equation 4.3 were estimated using nonlinear regression which is presented in a tabular format in Table 3.2.

Effect	Regression Coefficient	t-statistic	p-value
β(Intercept [Baseline])	904.3	5.5	0.00
$\beta$ (Intercept: CRS-2)	1694.6	1.6	0.05
$\beta$ (Intercept: VPD)	319.6	3.7	0.00
$\beta$ (Intercept: VPD×CHFRS-2P)	2664.0	10.1	0.00
$\beta$ (Intercept: VPD×CRS-2)	-1272.1	-2.5	0.01
$\beta$ (Intercept: VPD×CRS-2P)	848.0	4.7	0.00
β(Limestone)	-291.9	-3.0	0.00
β(Quartzite)	-225.0	-2.5	0.01
$\beta$ (Light Weight Aggregate)	-94.9	-1.2	0.12
β(Slope [Baseline])	0.484	9.8	0.00
$\beta$ (Slope: CHFRS-2P, CRS-2P)	0.067	1.7	0.04
β(Slope: CRS-2)	-0.545	-3.0	0.00
β(Slope: VPD×CHFRS2P/CRS2P)	-0.093	-6.3	0.00
β(Slope: VPD×CRS-2)	0.395	3.8	0.00

 Table 4.2 Model Parameters for Prediction of Stiffness of the Emulsion Residue

The standard error of prediction was found to be 2.5kPa which is low after taking into consideration the differences that exist between the materials and exposure conditions. The final predictive model for determination of the stiffness of the residue is as given below,

```
y = (904 + 1695CRS + 320VPD + (2664VPD \times CHFP) - (1272VPD \times CRS) 
+ (848VPD \times CRSP) - 292L - 225Q 
- 95W)x^{0.48-(0.09VPD \times CHF/CRSP)+(0.4VPD \times CRS)+0.07CHFP/CRSP-0.55CRS}
```

Eqn. 4.4





Figure 4.4 Observed v/s Predicted Stiffness of the Emulsion Residue

In their current form, the predictions from the stiffness model are biased towards the initial time periods, due to the fact that the models were developed based on laboratory data collected at intervals of 2 hours, 6 hours, 24 hrs and 48 hours. It is the data corresponding to the 48<sup>th</sup> hour measurement that affects the intercept as well as the slope of the predictive model. The model in its current form provides an excellent fit against observed data for measurements recorded between 2 and 24 hours. However, one would not expect to wait longer than 24 hours to open a newly constructed chip seal in most cases and therefore for most practical purposes the statistical models discussed in this paper give reliable estimates for opening to traffic.

## 4.6 Conclusions

This chapter summarizes the experimental results and focuses towards proposing a statistical model for predicting the rate of moisture loss as well as the gain in stiffness as a newly-constructed chip seal sets under ambient conditions in the field. The objective of the research was to determine the minimum time to open a newly constructed chip seal to traffic such that the thin film of asphalt residue left from partial curing of the emulsion offers sufficient resistance to raveling so as to minimize early loss of aggregates. The research work focused on data generated from controlled experiments conducted in a laboratory setup to simulate the most typical materials used for chip seals under typical field conditions. Based on statistical modeling of this data, empirical models were proposed that can predict the amount of water lost to evaporation as well as the gain in the stiffness of the residue as function of time. Thus this chapter lays the ground work for proposing an algorithm that can be used for determining the time to open the road to traffic. In the subsequent chapters, the application of the model will be discussed in more

detail so as to facilitate its use as a practical tool for making a decision on the time to open the road to traffic.

# Chapter 5. Developing a Field Test to Assess Emulsion Quality

# **5.1 Introduction**

Early failures in chip seals are sometimes attributed to bad construction materials or poor construction practices. While the latter can be addressed through regular inspection when a chip seal is being paved, problems related to poor quality materials can be rectified by adhering to specifications and guidelines established by the state highway agencies. To this effect, it is necessary to develop suitable test protocols that will enable the field inspector to differentiate between superior performing materials and those which have a history of poor performance.

The first step in this direction starts with choosing a test that can accurately assess the suitability of the material for a seal coat or chip seal paving job. One example of such a test is the tack test that is commonly used in the adhesive industry. A tack test essentially provides quantitative information on the cohesive strength of an adhesive. This is achieved by using a standard probe stuck to the adhesive surface (using controlled pressure) and then detached to determine the magnitude of pull-off force required (at a controlled rate) for the probe to debond. Several portable models for such tack testers are available. Other researchers have used tackiness of asphalt binder to determine its cohesive properties. In principle, the test looks promising and has the potential to deliver the results considered necessary for the success of this project, but in reality certain practical constraints deemed this test unsuitable for the scope of this research project. The research team visited a field job in the San Antonio district in April 2009 to gauge the practicality of this test. The researchers found that the adhesive force working at the binderaggregate interface was negligible and therefore very difficult to measure accurately in the field. The researchers also observed that on this project there was very little improvement in the adhesive force over a period of 15 days, which led to the obvious conclusion that the test was probably not well suited for a field environment.

The use of portable spectroscopic techniques to determine the quality and consistency of the emulsions being supplied to the field was also evaluated as part of this research study. For example, the characteristic infra-red spectra of the emulsion that was used in the laboratory for design and quality control can be determined (Figure 5.1).



Figure 5.1 Evolution of an HFRS-2 Emulsion Sample on a Weathering Rack

The characteristic spectra may then be used in the field to compare the quality and consistency of the material that was supplied. If found feasible, results from the spectroscopic tests can be used to determine the level of dilution among different batches as well as any significant changes in the composition of the binder. However, it was realized that cleaning the instrument would be a major hurdle in a field setup. In addition, the cost of a portable FTIR is in excess of \$10,000, which would involve a significant investment on the part of the DOT to procure one for every few of districts. Therefore, in consultation with the PD of this project, it was decided to dismiss the idea of using a portable spectrometer from the list of field tests that were to be proposed as part of the final toolkit.

Another relatively simple test could be the stick test, which is often used extensively by field inspectors and involves using a wooden stick to cut a notch on the thin film of emulsion after it has partially cured and record the time taken by the emulsion to close that groove. The idea is to determine the flow characteristics of the emulsion as it loses water to evaporation. The test provides a quick and easy way to determine the time to put down the aggregates. Both early and late placement of chips can lead to excessive loss of aggregates, often termed as raveling due to improper adhesive bonding between the rocks and the binder. Figure 5.2 demonstrates the "Stick Test" procedure as it is carried out in the field.



Figure 5.2 Demonstration of the "Stick Test" by the Field Inspector in Comanche, TX

The viscosity of the asphalt emulsion is one of the key determinants to judge the consistency and quality of the plant emulsion sample. TxDOT Item 300 provides detailed guidelines and specifications for asphalt emulsions used for chip seal and seal coat paving jobs in the State of Texas. However, it has been observed that a suitable test that can measure the viscosity of the binder in the field is lacking. The use of viscosity cups, namely Zahn and Shell cups, can provide the breakthrough in this area.

A Shell cup can provide an easy and quick way to measure the viscosity of the asphalt in the field. Extensive viscosity testing of emulsions in the field using Shell cups was done as part of the study. Viscosity is dependent on temperature; therefore, the use of a water bath in conjunction with the Shell cups to control the temperature of the emulsion tested is recommended to reduce the variability in the data.

The consistency or the viscosity of the asphalt emulsion used for paving is not the only problematic area. Often it has been found that additional water is added to the emulsion before it is shot to reduce its viscosity, especially for fog seal and priming of bases. This leads in an asphalt film which is thinner than the desired thickness due to a higher dilution ratio of the asphalt emulsion. It is, therefore, well understood that developing a mass loss test that can be

used to determine the water percentage of the emulsion as used in the field can be helpful in these situations. Knowing the dilution ratio will enable the field inspector to determine the application rates prior to addition of water to attain the desired film thickness after complete curing of the asphalt emulsion.

# **5.2 Evaluation of the Proposed Field Tests**

As already stated, the research team shortlisted the following tests for quick and easy evaluation of emulsified asphalt products in the field:

- Mass loss test using a hot plate/gas oven to determine the dilution ratio of the emulsion
- Shell cups for determination of the viscosity of the emulsified asphalt

During the summer 2010 chip seal season these two tests were evaluated extensively at each of the field sites that were visited by the research team. The results from each of these two tests had been very consistent at each of these project locations.



Figure 5.3 The Water Bath Used for Maintaining Steady Temperature of Emulsion Sample



Figure 5.4 Infrared Temperature Gun to Record Temperature of Emulsion



Figure 5.5 (a) Time Required for Emulsion to Drain from Shell Cup



Figure 5.6 (b) Time Required for Emulsion to Drain from Shell Cup

Table 5.1 and Figure 5.7 shows the dilution ratio and viscosity measurements recorded in the field using the proposed tests at the project location in Jasper.

	_
Sample #	Binder Residue (%)
Repeat 1	69.60
Repeat 2	69.23
Repeat 3	71.61
	$\mu = 70.15 \ (>65), CoV = 1.83\%$

T 11 <b>F</b> 4		<b>.</b>			CDC AD	<b>F</b> 1 ·	4 <b>T</b>		<b>x</b> 7
Table 5.1	Dilution <b>F</b>	katios r	neasured	on the	CRS-2P	Emulsion	at Jas	per, T	Х



Figure 5.7 Viscosity Measurements using the #6 Shell Cup for CRS-2P at Jasper, TX

The results presented in Table 5.1 show that the dilution ratios determined using the mass loss were very consistent with a low coefficient of variability. However, the results from the viscosity measurements using the Shell cup (Figure 5.7) were more variable with respect to the mass loss measurements (Table 5.1). The reason for that can be due to a temperature gradient in the sample, or there might be cooling of the emulsion as it drains through the Shell cup. The use of a water bath was, therefore, considered necessary for all future measurements because of the inherent shortcoming of this procedure.

Table 5.2 and Figure 5.8 show the dilution ratio and viscosity measurements determined in the field using the proposed tests at a project location in Giddings, Texas.

Sample #	Binder Residue (%)
Repeat 1	69.17
Repeat 2	68.46
Repeat 3	68.31
	$\mu = 68.65 \ (>65), CoV = 0.67\%$

Table 5.2 Dilution Ratios measured on the HFRS-2P Emulsion at Giddings, TX



Figure 5.8 Viscosity Measurements using the #5 Shell Cup for HFRS-2P at Giddings, TX



Figure 5.9 (a) Mass Loss Tests Carried Out in Field on Gas Oven


Figure 5.10 (b) Mass Loss Tests carried out in the Field on a Gas Oven



Figure 5.11 Negative Impact of Applying Excessive Heat While Recovering Residual Binder from Emulsion

Table 5.2 shows that the mass loss measurements using the hot plate were consistent as before. The important point to note here was the repeatability of the viscosity measurements using the Shell cups also improved (Figure 5.8). This can be attributed to the use of water bath to maintain the temperature of the asphalt emulsion.

Tables 5.3 and 5.4 and Figures 5.12 show the dilution ratios and viscosity measurements determined in the field using the proposed tests at project locations in Mason and Comanche, respectively.

Sample #	Binder Residue (%)
Repeat 1	62.77
Repeat 2	66.16
Repeat 3	67.06
	$\mu = 65.33 \ (>65), CoV = 3.46\%$

T 11 <b>F 3</b>	<b>D11</b> /	<b>D</b> /		41	CDC AD	<b>F</b> 1 •		
Table 5.3	Dilution	Katios	measured	on the	CRS-2P	Emulsion	at Mason,	TΧ



Figure 5.12 Viscosity Measurements using the #5 Shell Cup for CRS-2P at Mason, TX

Sample #	Binder Residue (%)
Repeat 1	67.18
Repeat 2	64.39
	$\mu = 65.78$ , CoV = 3.00%

 Table 5.4 Dilution Ratios measured on the CRS-2H Emulsion at Comanche, TX

The viscosity of the CRS-2H emulsion could not be measured using the Shell cup #6 due to extremely high viscosity of the emulsion. It was also observed that the flow through the orifice was not steady. An alternative to this unusual problem was also tried in the field; researchers raised the temperature of the asphalt emulsion to reduce its viscosity. This alternative did not solve the problem, because raising the temperature of the water bath resulted in evaporation of the water from the emulsion. Water condensed on the cap of the closed container. It is possible that eventually the water vapor would be enough to saturate the air inside the container, but this was not observed in the field. Researchers concluded that using the Shell cup to measure extremely high viscosities in the field may not work because of these reasons.

In addition, it should be also noted that the emulsion sample was very viscous in nature, which caused sputtering of the asphalt when the water was being boiled out of the emulsion. A lower temperature on the gas oven helped in averting the problem. It is recommended that problems related to sputtering of the residual binder while boiling the water out can be effectively controlled by opting for a lower procedure temperature. It should be also noted in this context that a cylindrical container that is sufficiently deep helps reduce loss of asphalt if the emulsion happens to foam and come out of the container. For the sake of convenience, the use of a gas oven is recommended instead of a hot plate, as the latter produces a lower heat transfer rate

which also increases the procedure time. Other factors like strong wind gusts may interfere with the duration of the test, as they tend to produce a cooling effect.

# 5.3 Repeatability and Comparison of Results with Specifications According to Item 300.2

The dilution ratios as measured using the mass loss test were found to be repeatable and consistent when compared against the Item 300.2 specifications for emulsified asphalt products. According to the specification, the minimum residue after distillation of the emulsion should be at least greater or equal to 65%. The results as measured at each of the field sites were in line with these specifications for the two different batches of CRS-2P that were used at Jasper and Mason, the HFRS-2P emulsion used at Giddings and the CRS-2H emulsion that was used at Comanche. It can be also observed from the results presented in Tables 4.1 through 4.4 that the measurements are very repeatable with coefficients of variability in the range of 0.5 to 3.5%.

As for the viscosity measurements are concerned, the repeatability of the measurements improved significantly due to the addition of a water bath for maintaining the temperature of the asphalt emulsion. The Shell Cup #3.5 was also evaluated in the laboratory with a CRS-1P emulsion sample to test the repeatability of the instrument. Figure 1.13 shows the repeatability of the test based on measurements at three different temperatures with four repeats at each temperature.



Figure 5.13 Viscosity Measurements using the #3.5 Shell Cup for CRS-1P under Laboratory Conditions

Therefore it can be clearly seen from Figure 1.13 that the viscosity measurements using the Shell cups are very repeatable under the right conditions. The same fact has also been highlighted in Figures 1.8 and 1.12. However, there seems to be a slight shift in the viscosity measurements as recorded with the Shell cups when comparing against the Item 300.2 specifications for emulsified asphalt products. According to the specifications, the normal range of viscosity for a CRS-2P emulsion should be between 150 and 400 Saybolt Furol Seconds (SFS) when measured

at 122°F. However for none of the CRS-2P samples tested either at the Jasper or the Mason field site, the viscosity of the emulsion (determined on the basis of the manufacturer's conversion charts) was found to be within this range for temperatures lower than 122°F. Thus applying a temperature correction to the measured viscosities will further lower the reading recorded with the Shell cups. This has proved to be one of the major shortcomings of this test procedure. However, there is an easy way to go around this problem and that is by concurrently measuring the viscosity of each of the binders that are commonly used in chip seal applications using the Shell cups as well as the Saybolt Furol viscometer. This will help in establishing a relationship between the measurements recorded using each of the two test procedure which will then help us to develop a correction factor that can be applied to field measured viscosities using the Shell cups to relate to specifications as listed in the Item 300.2 of the TxDOT Standards.

#### 5.4 Conclusions and Recommendations

The primary objective of Task 4 was to develop a simple yet robust test procedure for judging the quality of the emulsion in the field. As it can be seen from the foregoing discussion that the two tests that satisfied both these criteria were:

• Mass loss test that can be used effectively to determine the dilution ratio and the residual binder of the asphalt emulsion

• Measurement of viscosity using Shell cups to determine the consistency of the emulsion The data presented as part of Tables 4.1 through 4.4 bears evidence to repeatability of the mass loss measurements. It has also been pointed out that the dilution ratios as determined using this test is very much within the range of water content that one would normally expect for the emulsions that were tested.

As for the viscosity measurements are concerned, the results presented as part of Figures 4.7, 4.8 and 4.12 shows that the readings are indeed repeatable. However, the use of a water bath is recommended so that one can obtain consistent measurements in a field setup. It has been witnessed that the effect of atmospheric cooling can significantly affect the viscosity measurements and thus adds random noise in the measured data. Figure 4.13 shows that in a laboratory environment the measurement of viscosity using Shell cups can produce repeatable results. This underscores the importance of trying to minimize the effect of unwanted factors in the field. In addition, it was also witnessed that the viscosity measurements recorded using the Shell cups were lower than the TxDOT Standards for emulsified asphalt products. It is therefore recommended that viscosity measurements using the Saybolt Furol viscometer and the Shell cups should be recorded concurrently and based on the results certain corrective terms should be developed which will enable us in correlating field measured viscosities using the Shell cups to the TxDOT Item 300.2 Standards.

## Chapter 6. Evaluation of Emulsion Sampling/Handling Processes

The objective of Task 5 of this research was the evaluation of sampling processes and containers to ensure representative results.

Any pavement design and construction process considers that materials used for a project are of acceptable quality. This assumption can only be valid if sound quality management practices are in place for materials such as asphalt binders. TxDOT research project 0-4681 (*Further Developments of Binder Quality Assurance Program*) revealed that no binder material is more sensitive to sampling and sample handling/transport procedures than emulsified asphalts. Therefore, it was decided to assess the influence of properties of selected surface treatment binders to the material sampling and transport processes. The experimental factors investigated in this task include sample container type, container size, and the method and duration of transport. Sampled materials were subjected to both standard TxDOT tests as well as other tests designed to simulate actual handling and transportation processes. In order to minimize the effect of between-laboratory errors, it was decided in consultation with the PMC that all standard tests will be conducted at the TxDOT asphalt laboratory in Cedar Park.

Two types of tests were conducted under this Task.

- 1. Standard TxDOT specified tests on emulsified asphalt samples taken at field projects and emulsified asphalt plants; these tests were conducted at the TxDOT Cedar Park asphalt laboratory.
- 2. Tests conducted at the TechMRT asphalt laboratory on emulsified asphalt samples that were subjected to laboratory simulation of emulsion transportation process.

#### 6.1 Standard TxDOT Tests on Emulsified Asphalt Samples

For these tests, container type, container size, emulsion type, and grade were considered as experimental factors. Three types of can materials, tin-coated steel, high density polyethylene (HDPE), and fluorinated plastic cans were selected to be included in the experimental program. TxDOT currently uses the tin-coated steel cans. Two types of plastic cans were added to the experimental plans because they were likely to maintain the quality of emulsified asphalt over a longer period of time. The fluorinated plastic is considered even better than the normal HDPE can, and it was included for evaluation as well.

Two types of can shapes, F-Type and Cylindrical, were identified to be included in the test program. However, after discussing with the PMC, it was decided to drop the cylindrical can because it was not likely that TxDOT would use that type in the future. Based on the number of tests performed by TxDOT on emulsion samples and the material requirements for each test, a decision was made to include a quart-sized and gallon-sized can in the study. Pictorial images of these cans are shown in Figure 6.1. The regular HDPE plastic and fluorinated plastic cans used in the study looked very similar except for the material, and they are shown in Figure 6.2. The

cylindrical 1-gallon plastic can considered for the study is shown in Figure 6.3. This can was not used because it was unlikely to be used by TxDOT in its sampling work.





Figure 6.1 1-Gallon and 1-Quart F-Type Tin-Coated Steel and HDPE Plastic Cans





Figure 6.2 F-Type HDPE Plastic and Fluorinated Plastic (Marked "F") Cans used in Study



Figure 6.3 Cylindrical 1-Gallon Plastic Can Considered (but not used) for Study

Based on the requirements stipulated in TxDOT standard specification ITEM 300 for bituminous binders, a test factorial was prepared for each binder tested. Table 6.1 shows a sample test factorial prepared, in this case, for CRS-2.

			CRS-2							
Material	Shape	Capacity	Tests on Emulsion			Tests on Residue				e
			SFV	Demul	BI	Pen	Duct	Visc	Dist	Float
Tin-Plated Steel		3207(10t)		X				XX	Χ	
	F Style	5202 (1 Qt)								
	I-Style	12807 (1 Gal)	XXX	X				XX	Χ	
		12802 (1 Gal)								
	Round	32oz								
	F-Style	32oz (1 Qt)		Х				XX	Х	
		128oz (1 Gal)	XXX	Х				XX	Х	
HDPE	Dound	$32_{07}$ (1 Ot)								
	Koulia	5202 (1 Qt)								
	E Stulo Elucrinoto d	32oz (1 Qt)		X				XX	Х	
	r-Style Fluorinated	128oz (1 Gal)	XXX	Х				XX	Х	

 Table 6.1 Sample Test Factorial Prepared for CRS-2 for Tests Conducted at TxDOT Cedar

 Park

Samples were obtained from the field sites under the supervision of the TxDOT inspector to ensure that standard TxDOT procedures were followed during the sampling process. In some cases, samples were also obtained from TxDOT-approved tanks in emulsion plants. In such situations, a supplier company-authorized technician obtained the samples. Once the samples were obtained, they were shipped to TxDOT Cedar Park lab by bus, which is the way samples are typically transported from field sites. In the few cases where sampling was done from supplier plants, a TechMRT researcher transported such samples to the TxDOT Cedar Park lab by vehicle.

Results from tests conducted at the TxDOT Cedar Park lab are presented in Figures 6.4 through 6.9. The legend in each chart indicates the type and size of container from which the test sample came. This material was sampled from the field in the morning and then shipped to Cedar Park by bus the evening of the day of sampling. The tests were conducted two days later. The blue

shaded areas indicate the specification requirement regions. The results for the samples from different containers were very close to one another and met specifications except for the material in the 1-quart metal and 1-quart HDPE cans, for which the Saybolt-Furol Viscosity came out to be significantly lower than the lower specification limit of 150. It was noted by the testing personnel that the containers were not completely filled in some of the cans and it is possible that some of the material was affected by the resulting influences.



Figure 6.4 Test Results for CRS-2 Sampled from BWD (Source: Ergon Waco)

As shown in Figure 6.5, the CRS-2P material sampled from an in-house sealing project in the Paris District showed no significant difference between materials sampled from the different cans. All materials tested met the specification criteria as well. Figure 6.6 shows the absolute viscosity test results conducted on the residue of the same material and those material samples showed the same tendencies of no appreciable difference between cans and meeting the specification criterion.



Figure 6.5 Test Results for CRS-2P Sampled from PAR (Source: Ergon Mt. Pleasant)



Figure 6.6 Absolute Viscosity Test Results (in Poise) for CRS-2P Sampled from PAR (Source: Ergon Mt. Pleasant)

Figure 6.7 shows results from a CRS-2 emulsion sampled from the Ergon Asphalt Waco plant. In this case, all material except the 1-gallon HDPE and fluorinated plastic cans met specification requirements. For those two 1-gallon plastic can types, the elastic recovery (ER) test criteria were not met (i.e. the two samples had ER values somewhat lower than the specification minimum of 55 %.) In addition, the Saybolt-Furol Viscosity values for the three 1-gallon cans showed lower values than for the three quart cans. However, they all met the specification criteria. Figure 6.8 shows the absolute viscosity values for the same CRS-2P material and the samples met the specification criteria except the 1-gallon HDPE plastic can material, which out had a viscosity value of approximately 1250 Poise at 140 °F, compared to the specification lower

limit of 1300 Poise. Also for absolute viscosity, the two 1-gallon fluorinated plastic can material had a much lower viscosity. It, however, met the specification criteria.



Figure 6.7 Test Results for CRS-2P Sampled from Ergon Waco Plant



Figure 6.8 Absolute Viscosity Results (in Poise) for CRS-2P Sampled from Ergon Waco Plant

The results shown in Figure 6.9 are for the only anionic emulsion available for testing in this study and this was sampled from a project in the Giddings area of Austin District. The TxDOT

testing personnel reported that this binder had already gone through the breaking process by the time the material cans were opened at the lab. Ironically, this material sample set also happened to be the set that was transported to Cedar Park lab the quickest. As can be seen in Figure 6.9, results for material from the two 1-gallon plastic cans are not presented because material in those two cans were transformed significantly enough that testing them was found to be of no practical use. The testing personnel also reported that those cans could not be regurgitated with the utensils they normally use. As for the specifications, it is clear that this material did not meet the Saybolt-Furol Viscosity criteria by a wide margin. They also did not meet the demulsibility criteria for any of the samples. The material did, however, meet specifications for the distillation residue and penetration criteria on the residue. There is no published ER specification criterion for HFRS-2P.



Figure 6.9 Test Results for HFRS-2P Sampled from AUS (Source: Ergon Austin)

One additional material was sampled from a project in the Brownwood district that used a CRS-2H emulsion from Ergon Waco plant. This material could not be tested in a timely manner due to the fact that it was sampled on a Friday and the material did not arrive at the TxDOT lab until several days later; by that time it was too late to test.

#### 6.2 Tests on Emulsified Asphalt Subjected to Simulated Transport

The purpose of this test was to simulate the variability of vibrations and movement during the transportation of asphalt samples to and from the Texas Department of Transportation laboratories in Austin, TX. Many times the samples are shipped in various types of containers, usually in civilian buses in the cargo hold.

To simulate the vibrations caused by the bus, an *L.E.D. orbit shaker* manufactured by *Lab-line instrument Inc.* was used as shown in Figure 6.10. Three container types (F-Type metal, HDPE

and fluorinated plastic) were used in this study because those size containers could be attached to the shaker device. The containers were filled to the top, leaving very little airspace, with CRS-1P at 170°F collected from Holly Asphalt in Lubbock. The containers were securely fastened, along with two replicates, using standard masking tape by looping the tape twice around the Erlenmeyer flask holder prongs, also shown in Figure 6.10. The three containers (F-fluorinated, N-non-fluorinated and M- metal) were run for 8 hours on the shaker table, while three more marked-identical containers were run for 3 hours at the beginning, with the rest of the containers (total of 6 containers), then the three marked containers were removed for 2 hours (to simulate a stop). Finally, the containers were replaced and run till the end of the cycle when all the containers were removed at the same time. The shaker table was run at 240 rpm. Once the shaker table part was complete, all samples were run through the Saybolt-Furol Viscometer test (standard protocol was used). Results from this test are presented in Figure 6.11. For the CRS-1P material, samples from all three container types met the SFV specification requirement. However, it can be seen from Figure 6.11 that the laboratory simulation of the container transportation caused the SFV to drop significantly when compared with the control specimen left in containers that were not taken through the shaking process. A similar test procedure was used for a CSS-1P sampled from Ergon Asphalt Waco plant. Saybolt-Furol Viscosity results for that binder are presented in Figure 6.12. The CSS-1P material did not meet the SFV specification criteria under simulated and control conditions, except for the fluorinated plastic can that underwent the 2-hour rest between 3-hour shaking sessions.



Figure 6.10 One-Pint Cans of Emulsion Mounted on Shaker Table to Simulate Transportation



Figure 6.11 Results for CRS-1P (Holly Asphalt) for SFV Tests Conducted at TechMRT Lab



Figure 6.12 Results for CSS-1P (Ergon Asphalt Waco Plant) for SFV Tests Conducted at TechMRT Lab

#### **6.3** Conclusions

There appears to be no difference between different types and sizes of containers when emulsified asphalt is transported. In some cases, the material showed differences in results, either in the form of discrepancies for different container types or the container size. However, in all cases, those differences could be traced back to a problem associated with the original material quality or handling. TxDOT currently uses the 1-quart metal cans to transport materials and these cans did not show any problem associated with test results. The larger 1-gallon cans – particularly the two plastic types – showed inconsistencies and many of the inconsistencies could be traced back to the inability to stir the larger cans effectively.

# **Chapter 7. Field Monitoring Program**

#### 7.1 Recommendations

Early failures in chip seals are often attributed to inferior construction materials or poor construction practices. It is therefore understood that executing a chip seal paving job requires making the right decision at the right time. Major milestones in the paving process include placing the aggregates, applying the compaction and opening the road to traffic. The timing of these events is critical to the successful application of the roadway. The focus of this TxDOT funded study has been primarily towards developing guidelines for successful execution of these events and hence minimizing the likelihood of early failure due to poor construction practices. As part of this task, the research team primarily focused on proposing thresholds for the models that were developed as part of Chapters 2 and 3 based on the field monitoring program. These thresholds are used to determine appropriate time limits to 1) place aggregates after emulsion application and 2) open the newly paved section to traffic. In the following subsections, details of the procedure are discussed.

#### 7.2 Determination of Thresholds for Placing the Aggregates

To properly develop a protocol for determining the waiting period before aggregates can be placed in a chip sealing job, it is essential to know the optimal amount of curing an emulsion must go through to minimize aggregate loss. The amount of moisture lost in the field was not directly measured due to the relatively short time from the instant the emulsion was shot till the aggregates were placed. The alternative was to estimate the amount of water loss that the emulsion would have until the aggregates were placed. To satisfy the problem, researchers must collect ambient weather conditions including temperature, humidity, and wind speed; this is sufficient to determine the amount of moisture lost due to the vapor pressure deficit and the atmospheric turbulence in the surrounding air. However, the rate of curing while the emulsion cools from the spray temperature is faster, because of the higher vapor pressure deficit of the surrounding air from the heat exchange happening between the emulsion and the environment. It is, therefore, necessary to gather information on the rate of thermal cooling in the asphalt emulsion from the moment it is sprayed along with simultaneous measurements of the meteorological parameters. Given that these two pieces of information are available, one can use Newton's law of cooling to determine the time before the emulsion reaches a state of thermal equilibrium with the surrounding environment. Table 7.1 summarizes project related information that was collected during the field validation phase of the study. The purpose of the field evaluation study was to look at the typical waiting period before the aggregates are placed at construction job sites under varying conditions. Type of emulsion, geographical region, and the season were some of the variables that were focused on as part of this study. For example, field sites like Floydada and Plainview are located in North Texas which is relatively drier and cooler compared to sites like Giddings or Comanche in Central Texas. The construction job at Flovdada was executed during the later part of the fall season, while those in Giddings and Comanche were constructed during the hot summer months.

Kates from Field Flogects					
Location	Brackenridge	Plainview	Floydada	Giddings	Comanche
Type of Emulsion	CRS-2	CRS-2P	CRS-1P	HFRS- 2P	CRS-2H
Spray Temperature (°F)	140	140	130	135	133
Mean Air Temperature (°F)	96.6	77.6	75.4	83.3	78.3
Time to reach Thermal Equilibrium	4	5	5	4	3
Time to put Aggregates (mins)	15:00	07:15	13:30	3:30	7:30
Convective Heat Transfer Coefficient (k)	0.0242	0.0288	0.0241	0.0285	0.0191

 Table 7.1 Site-Specific Measurements Related to Measurement of Curing

 Rates from Field Projects

Table 7.1 provides information on the type of emulsion used at each of these project locations along with the temperature at which it was sprayed. The mean air temperature and the time to put down the aggregates were also reported from each of the paving jobs. The time to reach thermal equilibrium refers to the time it took for the emulsion to cool down to the ambient conditions. Information on the rate of cooling rate was measured and used for estimation of the convective heat transfer coefficient.

Having measured the rate of temperature drop in the emulsion as a function of time, Newton's law of cooling can be applied to determine the rate of cooling and the time to reach thermal equilibrium. Newton's law of cooling states that the rate of change of the temperature of an object is proportional to the temperature differential between the object and the surrounding environment. Mathematically the process is represented by the following differential equation.

 $\frac{\mathrm{d}T}{\mathrm{d}t} = -\mathrm{k}(\mathrm{T_o} - \mathrm{T_a})$ Eqn. 7.1 Where. dT/dt = Rate of Change of Temperature w.r.t Time $T_0$  = Temperature of the object  $T_a =$  Temperature of the ambient air Declaring a new variable,  $y(t) = T(t) - T_a$ Therefore,  $\mathbf{y}(0) = \mathbf{T}_0 - \mathbf{T}_a$ Differentiating w.r.t time,  $\frac{\mathrm{d}y}{\mathrm{d}t} = \frac{\mathrm{d}T}{\mathrm{d}t} = -\mathrm{k}(\mathrm{T} - \mathrm{T}_{\mathrm{a}}) = -\mathrm{k}y$ Solving the differential equation-kt =  $\ln\left(\frac{y}{y_0}\right)$ or,  $y = y_0 e^{-kt}$ or,  $T - T_a = (T_0 - T_a)e^{-kt}$ or,  $T = T_a + (T_0 - T_a)e^{-kt}$ Eqn. 7.2 The convective heat transfer coefficient was calculated using a least squares approach. The

ambient air temperature ( $T_a$ ), the spray temperature ( $T_0$ ) and the instantaneous temperature T of the emulsion was recorded during the field trials. Knowing all the parameters in Equation 7.1,

the observed data was fitted with the model using a least squares approach and the convective heat transfer coefficient was estimated.

Given that the ambient temperature, the initial temperature of the emulsion, and the instantaneous change in the temperature are known, the equation can be solved for the convective heat transfer coefficient to determine the rate of cooling. The heat transfer coefficient was determined from the project data that was collected. However, there was some variability observed between the heat transfer coefficients determined for each of the field projects, which led to the decision to use an average value of 0.0249. Having determined the heat transfer coefficient, one can calculate the time taken by the emulsion to cool and reach a state of thermal equilibrium, as well as the amount of water lost during this time interval. Once the emulsion cools to the ambient air temperature, the amount of water that will be lost henceforth will be determined by the vapor pressure deficit of the ambient air, which can be determined by the statistical model presented in Task 2. The amount of water lost before the aggregates were placed was calculated for each of the field projects and the results are summarized in Table 7.2.

Location	Brackenridge	Plainview	Floydada	Comanche	Giddings
% Water Lost before Emulsion Cools down	14.2	8.1	10.2	14.2	3.0
% Water Lost after Emulsion Cools down	14.2	3.3	11.7	3.5	0.0
Total Amt of Water Lost before Aggregates are placed (%)	28.4	11.4	21.9	17.7	3.0
Average (%)	19.8			3.0	

Table 7.2 Determination of Percentage of Water Lost Before Aggregates Were Placed

The results in Table 7.2 vary considerably from one project to the other. However, if the results are segregated into two groups based on the type of emulsifier, the cationic emulsions follow a generic trend. On the contrary, the anionic emulsion exhibited a lower percentage of water loss when the aggregates were placed. The anomaly can be explained by the fact that the cationic surfactants have a relatively lower hydrophilic character. This eventually results in faster breaking rates for cationic emulsions and a higher rate of moisture loss as compared to anionic emulsions.

Under the assumption that the time when the aggregates were placed at each of these projects was the most optimal as no early failures were witnessed, it can be concluded that the amount of water lost was the required amount of moisture loss for providing maximum resistance to tire pickup during rolling. In addition, it also needs to be appreciated that determining the threshold will require developing an experimental design where early failures should be witnessed. However, no highway agency will want to participate in a study where they will know a priori that some of the chip seals constructed will fail prematurely. Keeping in mind all these possible constraints, it was decided that the average of the percent water that was lost until the aggregates were placed is the threshold for placement of stones. Thus, the threshold for placing aggregates was proposed as 19.8% and 3.0% for cationic and anionic emulsions respectively. There may be arguments that the threshold for anionic emulsions is based on observations from a single job

site, which is definitely not sufficient. However, considering the decreasing popularity of anionic emulsions, it has been difficult to get projects that met the requirements and thus it is recommended that further effort in this direction is required to have more confidence in the proposed thresholds.

## 7.3 Determination of Thresholds for Opening to Traffic

#### 7.3.1 Procedure for Determination

As stated, the other focus area of this task was to propose thresholds for determining the time to open a newly constructed chip seal to traffic. It is of utmost importance to determine the amount of water lost before the newly constructed chip seal is opened to traffic. In order to meet this requirement, a field evaluation program was necessary to monitor the rate of setting of emulsions under field conditions.

Steel plates (as shown in Figure 7.1.a) measuring 210 mm × 210 mm were placed on the road surface to be paved before the paving operation was initiated. To keep the experiment consistent with laboratory results (presented in Chapter 3), the area of coverage on the steel plates was kept equal to that of the Pressure Aging Vessel (PAV) pans ( $\emptyset = 140$  mm), a square with 124 mm edges. These steel plates were put at three stations 15 meters apart, with one plate on each of the wheel paths and one along the centerline of the lane – thus giving 9 plates for each project included as part of the field evaluation study. The outer perimeter of each of these steel plates were removed from the road surface after two applications of the pneumatic rollers to ensure that the aggregates were properly seated in the emulsion and the masking paper was removed (Figure 7.1.b). The sample of chip seal collected on the steel plate was allowed to set under ambient conditions, which gave the closest possible replica of the newly paved chip seal. Simulating the field conditions was important as that would allow quantification of the amount of evaporation of water that is simultaneously occurring in the chip seal.



Figure 7.1 (a) Masked steel plates measuring 124mm × 124 mm (b) Final chip seal sample collected on the steel plate after 2 compaction cycles

Weight measurements were taken at regular intervals of 30 minutes for the first 3 hours and at 1 hour intervals thereafter, until the rate of water loss reached an asymptote. The reason is because

the rate of moisture loss gradually slows down with time. However, it should be noted here that the rate of curing cannot be calculated until and unless the amount of emulsion that was sprayed on each of these plates are known. To meet this requirement, the steel plates were stored in a dust-free environment for a period of 14 to 21 days to ensure that the sample reached constant weight, which implies that the combined weight of the sample is the sum total of the asphalt residue from the emulsion along with the aggregates. The samples were then exposed to high temperature in an ignition oven to determine the binder content for each chip seal sample. Dilution ratios for the emulsions were calculated using high temperature evaporative techniques which, when combined with the results from the ignition oven, can be utilized in back calculating the application rate and the rate of evaporation of water as the emulsion sets (Figure 7.2).

Although the amount of water lost to evaporation can be easily quantified using simple field tests, as such it does not give any information on the performance characteristics of the binder residue left from partial or complete curing of the emulsion. Because of this deficiency, the research team felt the need to perform additional testing in a laboratory environment to correlate the amount of curing to the stiffness of the emulsion residue. Weight loss and the shear modulus of the binder residue were measured in a laboratory environment at time intervals of 2, 6, 24, and 48 hours with the same emulsion and aggregate sampled from the project location. This allowed the researchers to construct a smooth curve that relates the amount of evaporative water loss to the stiffness of the residue (Figure 7.3).

The time taken to open the newly paved chip seal to traffic was also recorded for each of the projects which were included as part of the field monitoring program and the details are included in Table 7.3.

The purpose of the field evaluation program was to develop the thresholds for the predictive models to determine the optimal timing for opening the chip seal to traffic. In principle, the ideal situation would be to monitor a certain number of projects where both early failures and successful cases are witnessed. Unfortunately, early failures were not witnessed on any of the projects included as part of the field monitoring program. The lack of data limits the scope of this research to projects that have performed successfully and thus the thresholds that will be presented henceforth are definitely a conservative estimate. It was assumed that the time to open the chip seal to traffic was the most optimal choice under the given weather conditions and materials chosen for the project. However, this obviously leaves room for further adjustments to the thresholds to determine the optimal timing for opening the road to traffic, if condition surveys are performed on these projects in the future.



Figure 7.2 In-Field Measurement of Evaporation Rate for Chip Seal Paving Job in Mason, TX



Figure 7.3 Plot of Evaporative Water Loss vs. Complex Shear Modulus

Therefore, the optimal time to open the road to traffic should be the time interval that is just enough to reach a minimum amount of water lost to evaporation based on the setting rate (presented in Chapter 4). In order to determine the required amount of water that should be lost to evaporation, an average of the water lost at the time of opening to traffic was computed for the different projects. The threshold was thus calculated at 75.8% for the prediction model based on the rate of setting in emulsions.

On the contrary, it has been witnessed that for the same amount of relative loss in water, different aggregate-emulsion combinations gain in stiffness at different rates and therefore using an average of the stiffness from the different projects is not the ideal approach. It should be noted in this context that selecting the minimum stiffness as the threshold value would be a better choice as it will ensure that aggregate-binder combinations that gain in stiffness faster will lead to shorter time delays to open the road to traffic. However, a marked difference was observed in the rate at which stiffness gains over time for cationic versus anionic emulsions. It has been reported in literature that cationic surfactants have a relatively lower hydrophilic character. This eventually results in faster breaking rates for cationic emulsions and a higher rate of moisture loss as compared to anionic emulsions and this might explain the differences that we see in the stiffness measures for cationic and anionic emulsions. It was decided to propose a cutoff value of 2.2 kPa and 4.3 kPa for anionic and cationic emulsions respectively to determine the optimal time to open a road to traffic.

Project Location	Material	Time to open Road to Traffic	% Water Lost @ time of opening to Traffic	G* @ time of opening to Traffic
Giddings, TX	HFRS-2P + Light Wt Agg	8 hrs	79.12	2.2 kPa
Mason, TX	CRS-2P + Dolomite	4 hrs	70.19	4.3 kPa
Comanche, TX	CRS-2H + Limestone	1hrs	78.00	4.4 kPa

Table 7.3 Water Lost to Evaporation and Complex Shear Modulus at the time of openingto Traffic

#### 7.3.2 Application

As stated in the objective, the fundamental goal of this study was to be able to determine the time to open to traffic while the models that were presented as part of Task 3 will help one to determine the amount of setting that the emulsion will undergo and the complex shear modulus of the residue as a function of time, weather conditions and material characteristics. Thus the time to open to traffic will be the time interval that ensures a 75.8% reduction in the moisture has been achieved and a complex shear modulus of 2.2/4.3 kPa has been achieved depending on the emulsion we are dealing with. The equation below represents the setting model that has been presented in Task 3 and is now being solved for the conditions stated below to back calculate the time before it can be opened to traffic.

$$75 = 100 \left\{ 1 - e^{\left[ (-0.2386 - 0.015L + 0.0365Q + 0.0291W) x^{1.0045VPD + 0.1202CMS + 0.0456HF + 0.3606CRS} \right]} \right\}$$

or,  $\left\{1 - e^{\left[(-0.2386 - 0.015L + 0.0365Q + 0.0291W\right]x^{1.0045VPD + 0.1202CMS + 0.0456HF + 0.3606CRS]}\right\} = 0.75$ Considering a hypothetical scenario with an average daytime temperature of 95°F with 55% relative humidity and wind speeds of 10 mph, the time to open to traffic for a chip seal application that uses a CHFRS-2P emulsion with limestone aggregates should be,  $\left\{1 - e^{(-0.2386 - 0.015)x^{(1.0045 \times 0.963)}}\right\} = 0.75$ 

or, 
$$x = 5.033 = 5$$
 hrs 2 mins

It should be noted in this context that the time to open to traffic as calculated above is based on the amount of water lost to evaporation so that it develops enough strain tolerance when the newly constructed road is opened to traffic. However, it does not highlight on the rheological properties of the residue which should be the goal as it has a bigger impact on the performance of the chip seal. In fact, past studies conducted at the University of Wisconsin have proposed that the stiffness of the residue determines the resistance of the chip seal to raveling. It is out of this need that the current research focused on investigating the evolution of the rheological properties as the emulsion continues to lose water as a function of time and weather parameters. The statistical models presented in Task 3 offer the advantage of being able to predict the complex shear modulus of the partially cured emulsion residue. In this case for the conditions stated above, the emulsion residue offers a stiffness value of 5.5 kPa (calculation given below) at the time when it is opened to traffic.

 $y = (3041 - 335.6L - 286.2Q - 141.9W)x^{0.0182 + 0.0703VPD + 0.0662CMS + 0.3564CHF - 0.0446CRS}$ or, y = (3041 - 335.6) × 5.033^{0.0182 + (0.0703 × 0.963) + 0.3564} or, y = 5534 Pa ≈ 5.5 kPa

As mentioned, the minimum stiffness that the residue should have at the time of opening to traffic is 4.3 kPa while in this case the predicted stiffness is 5.5 kPa, which meets the minimum required stiffness for satisfactory performance. However, if the stiffness of the residue failed to meet the minimum required complex modulus as proposed earlier, it would imply that the time to open to traffic is inadequate to ensure satisfactory performance of the chip seal. In a scenario like that, we would have been required to revise the time for opening the newly constructed chip seal to traffic so as to meet both criteria – the stiffness of the residue as well as the amount of water lost to evaporation. Failure to meet either of these criteria could lead to early problems and increased stone loss due to shearing action of traffic.

#### 7.4 Determination of Application Rates

As part of the field monitoring program, the research team was posed with a problem to determine the in-field rates of evaporation of water from the time the emulsion is applied. To meet this demand, a new methodology was proposed and implemented.

The procedure has been described in section 7.3 and has been graphically shown in Figure 7.1.a and 7.1.b. It is in the author's opinion that the test shows promise in terms of repeatability and also gives precise measures for the application rate for emulsions as well as stones. Figure 7.4 summarizes the emulsion application rate for the chip seal project that the research team visited in Mason County of the Austin district during the summer seal coat season of 2010.



Figure 7.4 In-Situ Application Rates for the Chip Seal Project at Mason, Austin

The repeatability of the test is well documented through Figure 6.4 and the application rates that were determined were in line with the contractor's targeted value. In another project in Comanche County of Brownwood district, the field inspector decided to use an application rate along the centerline which will be different from that of the wheel paths. Figure 6.5 presents the application rate that was determined for the Comanche project location and the accuracy of the measurements were once again verified against the contractor's targeted values.



Figure 7.5 In-Situ Application Rates for the Chip Seal Project at Comanche, Brownwood

To summarize, the authors found the results promising and recommend the test to supplement current TxDOT procedures to determine the application rates for emulsions and stone used in surface treatments. In this regard it is recommended to undertake a pilot study to compare the accuracy and repeatability of the proposed test against current TxDOT procedures.

## 7.5 Conclusions and Recommendations

Sections 7.1 through 7.4 focus on proposing thresholds for putting down the aggregates at a chip seal construction job and determining the time before it can be opened to traffic. There is no doubt that slight miscalculations in estimating these parameters may have serious negative consequences. To this effect, a field monitoring program was undertaken to identify the cutoff value that will differentiate a superior performing chip seal from one that has failed prematurely.

The determination of the optimal timing of aggregates at the construction site was based on the amount of water lost till the aggregates are placed. Field data on the rate of cooling of emulsions were collected along with ambient air temperature, relative humidity and wind speed. Under the assumption that the time to put down the aggregates in each of these field projects provided the optimal scenario, the amount of water lost in that time interval was calculated and proposed as the threshold for bringing in the chip spreader.

Field evaluations of the setting rate of emulsions were conducted under ambient conditions to determine the actual rate of setting for different kinds of emulsions. As part of the field visits, the rate of setting and the time to open a particular project to traffic was noted – thus helping to determine the exact time in the evaporation curve when the road was opened to traffic. The amount of water lost to evaporation was determined for that time interval which was set as the threshold for the predictive model that estimates the setting of emulsion as a function of material

properties, weather conditions and time. The amount of water loss as observed in the field monitoring program was correlated with laboratory results to determine the minimum required stiffness for opening a newly-constructed chip seal to traffic. Results suggest that a stiffness of 2.2 kPa and 4.3 kPa for the residue obtained from partial curing of an anionic and cationic emulsion can be considered as the limiting value for satisfactory resistance to raveling for newly constructed chip seals.

## 7.6 Final Monitoring of Test Construction Projects

Each test project identified for detailed investigation in Chapter 3 of this report was visited at the end of the study to observe the performance after one year of performance. Table 7.4 summarizes the basic findings from each project and pictures from selected projects are presented in Figures 7.6 to 7.12.

No.	Location	Construction	Aggregate	Emulsion	Condition in
1.00	Location	Date	198109400	Linuision	February 2011
1	Breckenridge, FM701/FM1287	6/23/2009	Limestone	CRS-2	Some minor flushing visible
2	Paris, (FM895/FM2949	7/2/2009	Sandstone	CRS-2P	FM895 in good condition; FM2949 section overlaid due to roughness
3	Plainview, FM2301	8/17/2009	Quart	CRS-2P	Some minor flushing visible
4	Floydada, FM651	11/4/2009	Quart	CRS-1P	Some minor flushing and raveling visible
5	Giddings, US77	6/14/2010	Lightweight	HFRS-2P	Significant extent of medium flushing
6	Comanche, SH36	7/23/2010	Limestone	CRS-2H	Minor flushing along wheelpaths in some areas
7	Brady, US87/US377	8/10/2010	Limestone	CRS-2H	Extensive and significant raveling

**Table 7.4 Performance Observations from Field Test Projects** 

The two test projects that showed the most noticeable distress were US87 in Giddings and US77/387 in Brady. The US 77 section in Giddings used the only anionic emulsion studied from a test project and the material showed breaking when it was tested at the TxDOT Cedar Park lab within two hours of sampling. This HFRS-2P binder displayed quick breaking characteristics compared to the other binders. On the other hand, the US 87/377 project in Brady used the CRS-2H binder specially formulated for the hot summer days of August. The section was located south of Brady in the rolling hills northwest of Austin, and even with the high stiffness of this binder at early age (as displayed in the DSR tests described in Chapter 3), it seems to have been subjected to drain down. It may also be possible that the high early stiffness of the binder may have affected the aggregate bonding. However, the same binder-aggregate combination was used in the SH 36 project in Comanche, which did not display similar raveling. In fact, no raveling was observed on SH36.



Figure 7.6 FM 701 Monitoring



Figure 7.7 FM 895 Monitoring



Figure 7.8 FM 2949 Monitoring



Figure 7.9 FM 1287 Monitoring



Figure 7.10 SH 36 Monitoring



Figure 7.11 US 87/377 Monitoring



Figure 7.12 US 77 Monitoring

## **Chapter 8. Training Workshops**

Four regional training workshops were conducted under this task to communicate the research findings to TxDOT practitioners. The details of the regional workshops are summarized in Table 8.1. The training materials used in the workshop were submitted as Research Product P2 and are included in Appendix B of this report.

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Region	Workshop Location	Date	# of Registrants
East	Bryan District	Dec 6, 2011	17
South	Corpus Christi District	Feb 2, 2011	35
West	Lubbock District	Feb 9, 2011	15
North	Fort Worth District	Feb 16, 2011	9

Table 8.1	Summarv	of Information	on Regional	Workshops	Conducted
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Each regional workshop was conducted according to the agenda presented in Table 8.2. The presentation of the emulsion construction toolkit included video clips of actual construction projects, as well as a demonstration of the field tests developed in Task 4 of this research. The emulsion construction algorithm included a Microsoft Excel spreadsheet that would allow TxDOT personnel to determine the best time to apply the aggregates and open the roadway to traffic. The workshops were held at district training centers, and allowed participants to receive hands-on training of this computer program.

rubie 6.2 rigenau for Regional Workshops				
10:00am-12:00p	10:00am-12:00pm: TechMRT Presentation			
10:00-10:30am	Research Project Overview			
10:30-10:50am	Field Projects			
10:50-11:00am	Break			
11:00-11:50pm	Laboratory Test Program & Results			
1:00-3:30pm: CTR Presentation				
1:00-1:50pm	Research Outline and Findings			
1:50-2:00pm	Break			
2:00-2:40pm	Emulsion Construction Toolkit			
2:40-2:50pm	Break			
2:50-3:20pm	Emulsion Constructability Algorithm			
3:20-3:30pm: Workshop Evaluation				

**Table 8.2 Agenda for Regional Workshops**
# **Chapter 9. Summary and Recommendations**

## 9.1 Summary

#### 9.1.1 Binder Breaking/Curing and Field Tests for Emulsion

This study focused on several aspects associated with construction related problems in a typical chip seal construction job. The major findings from this study are summarized as below:

- A curing model based on the amount of water lost to evaporation has been proposed. The model focuses on proposing two separate equations, each of these accounting for two distinctively different mechanisms associated with loss of water from the time the emulsion is shot. The rate of evaporation of water is different during the initial few minutes when the emulsion is cooling down from its spray temperature until it reaches ambient conditions when compared against the rate of evaporation under ambient conditions. Experimental data was collected in the laboratory under constant temperature regimes for a suite of different temperature ranges. The test results were analyzed and a statistical model was proposed such that one can predict the amount of evaporation provided the other parameters of the equation are known. Certain generic trends like the unmodified emulsion products had a faster rate of moisture loss as compared to modified emulsions irrespective of the emulsifier that was used. In order to model the rate of evaporative loss in moisture under ambient conditions when the emulsion is in thermal equilibrium with the surrounding environment, mass loss measurements on the TxDOT racks were conducted. The results were analyzed and a statistical model was proposed. The vapor pressure deficit (VPD), which is an indicator that represents the ability of the surrounding atmosphere to carry additional moisture, had a significant effect on the rate of curing. The statistical model that was presented not only indicated that the VPD had a positive effect on the rate of evaporation but also helps somebody to quantify its effect on the rate of evaporation. Certain other trends like emulsions with harder asphalt bases as well as cationic emulsions were observed to cure faster.
- Secondly, extensive experimental work was conducted with the intent to model the rate of setting in emulsions along with the aggregates. The emulsions CRS-2, CRS-2P, HFRS-2, HFRS-2P, CMS-1P and CHFRS-2P were included in the experimental design. Each of these six emulsions was allowed to break and set with three different aggregates limestone, quartzite and light weight aggregates under two different temperature-humidity profiles. The rate of moisture loss and the evolution of the rheological properties of the residue from the emulsion were measured over time at definite time intervals. The experimental data collected in the lab was used for statistical modeling of the rate of setting in chip seal applications with independent variables like weather parameters and the type of the binder and aggregates used for a particular construction job.
- Thirdly, two field tests were developed and evaluated extensively to assess the quality of the emulsion that is supplied by the manufacturer to the job site. The first field test includes running a mass loss test to determine the dilution ratio in asphalt emulsions. The test involves a simple yet robust setup that can be easily taken to the field to determine the amount of moisture in a particular emulsion specimen and thus determine the dilution ratio. Field evaluation of the test was performed and the repeatability of the test was

commendable. In addition, the dilution ratio obtained for the materials that were tested in the field were in accordance with the TxDOT Item 300 specifications for asphalt emulsion products which also provides the flexibility of using it for QC/QA testing of the material in the field. The second test that was evaluated and thus proposed as part of the binder evaluation toolkit was the use of Shell cups for determination of the Saybolt Furol viscosity of the emulsion. The test was evaluated at each of the field locations that were visited as part of the field monitoring program. The repeatability of the test improved drastically when a water bath was introduced to control the temperature of the specimen.

- Finally, a total of six project sites were visited to conduct a field evaluation program for the tests that were proposed and also to determine the thresholds for the statistical models that were developed. For each of the projects that were visited, information on the meteorological parameters like temperature, humidity and wind speed data were collected along with the time interval between the emulsion was shot and the aggregates were placed. This information help the research team to establish the minimum required moisture that must be lost from the emulsion when the aggregates are placed and the reasoning was that too early placement can dislodge the aggregates from the road while delaying it too long can require higher compaction effort. In addition, field measurement of the rate of moisture loss from the newly paved chip seal was recorded. These measurements were used for establishing the thresholds for opening a newly paved chip seal to traffic based on the setting rate and the shear stiffness of the emulsion residue.
- Sampling and handling

#### 9.1.2 Binder Stiffness Development

The VT environmental profile yielded the lowest stiffness values for all time intervals and all aggregates tested. The ambient humidity level had a larger impact on the stiffness of the emulsion over time than temperature, for the profiles tested. If the ambient humidity is rising, or static, then the evaporation of water from the asphalt sample is slower than if the ambient humidity is dropping. This is directly related to the stiffness of the material because stiffness can only be gained as water leaves the mixture due to the coalescence and flocculation of the material causing the asphalt particles to form a continuous phase. Before the asphalt is able to eject the water it acts like a liquid and not a solid.

The type of aggregate being combined with a given emulsion, as well as the environmental factors such as temperature and humidity, will change the necessary waiting time between construction operations, mostly brooming and opening to traffic. For instance, the Limestone 2 or Sandstone, with a CRS-2, could be broomed 12 hours after application, as opposed to waiting the standard 24 hours. If Granite were being used though, depending on the environmental factors, longer than 24 hours may be required before the material has gained enough stiffness to broom or open to traffic. The CRS-2H on the other hand could be broomed four hours after application on the road due to the high rate of stiffness gain. Performing laboratory testing for the binder-aggregate combination of interest, under appropriate climatic conditions, could save time not only for construction crews but also for commuters by being able to open the road to traffic sooner.

### 9.1.3 ASTM D7000 Sweep Test

Based on the ASTM D7000 sweep test experiments conducted at TechMRT, the following observations can be made.

- The aggregate size used in the test (1/4-inch nominal maximum size) is too small to effectively evaluate the aggregate-binder bond. This nominal maximum aggregate size is much smaller than the aggregate sizes used in Texas.
- The aggregate quantity recommended in the ASTM D7000 test results in much larger spread coverage of aggregate in the seal than that desired by TxDOT. This also causes the aggregate loss numbers to be artificially inflated because many of the aggregate particles have no room to come into contact with the binder during compaction. Therefore, the aggregate loss does not reflect the true state of the aggregate-binder bond.
- The small aggregate particles cause the bristles of the sweeping brush to come into contact with the asphalt emulsion and the stickiness in the brush that is caused by this can influence the proper functioning of the sweeping mechanism.
- Use of a larger aggregate size that is more representative of TxDOT specifications for surface treatments would require a significant modification of the test including brush specifications and rotating speed.

## 9.2 Recommendations

### 9.2.1 Binder Breaking/Curing and Field Tests for Emulsion

This research study looked into some of the key aspects involved with construction of chip seals. An algorithm was developed to aid field inspectors and design engineers to aid in critical decisions like when to place the aggregates as well as the waiting period before it can be opened to traffic. However, the research team feels that there are certain aspects that need further effort in order to improve on the reliability of the models and the thresholds that have been proposed. After carefully analyzing what has been achieved as part of this project, the team decided to emphasize the following recommendations:

- Firstly, the statistical models proposed for predicting the rate of setting and the shear stiffness of the residue as a function of time, weather conditions, and the choice of material was based on test results from six different emulsions with replicate measurements for CHFRS-2P, CRS-2P and HFRS-2P. The team realizes the importance of further testing and thus recommends that at least two replicate measurements should be conducted on all six different emulsions, and the results should be analyzed to improve the predictions from the model. In addition, CRS-1P, which is a popular choice for winter season chip seal paving jobs, was not included for development of the model due to unavailability during the testing phase of the project. In its current form, the algorithm presented as part of the toolkit recognizes CRS-1P with breaking and setting characteristics similar to that of CMS-1P, and the predictions will be similar for these two materials. The authors strongly recommend that extensive testing be carried out on this material with the aim to incorporate the test results in the predictions from the model.
- Secondly, it was observed during the course of this project that although the results from the Shell cups are repeatable, they miss the laboratory measured Saybolt Furol viscosity

systematically. In fact, they are generally lower than what is measured in the laboratory with the Saybolt Furol viscometer. In addition, it was also observed that the measured viscosities do not meet the Item 300 specifications but less than the minimum requirement. It is thus suggested that an effort be made towards developing a relationship between the Saybolt Furol viscosity measured with the Shell cups and that on the Saybolt Furol viscometer under laboratory conditions so that field viscosity measurements for the plant sample can be validated against the TxDOT specifications.

• Finally, the field evaluation program that was undertaken for the development of thresholds included six different project sites, of which only one used an anionic emulsion. Therefore the cutoff values that were proposed in Chapter 6 for opening a newly constructed chip seal to traffic, or to place the stones where an anionic emulsion is being used, were developed on the basis of this single data point. It is not known how stable this observation is and thus a few more field visits to projects that are also using anionic emulsions will definitely help improve the confidence in the cutoff values for anionic emulsions.

#### 9.2.2 Binder Stiffness Development

The strain-sweep test, using the aggregate substrate and environmental conditioning, could be implemented into the planning portion of any emulsion project. After the alternative designs for the road have been developed, the specific options for aggregate-binder combinations could be evaluated using a strain-sweep test. This data would provide information about the stiffness gain in the binder with time, given the specific geographic and seasonal environmental conditions. Based on the strain-sweep data for a specific location and the alternative binder-aggregate combinations, a more accurate estimation of the necessary time between application and opening for traffic could be made. Conclusions about the durability of the road can also be developed based on the rate of stiffness gain within the material as well as the ultimate stiffness value of the material. For instance, if binder-aggregate combination 1 has a higher stiffness value than the binder-aggregate combination 2, then the better choice would be the binder-aggregate combination 1 because it will last longer than the binder aggregate combination 2. An economic analysis could then be performed in order to justify the binder-aggregate combination no matter which is chosen. This type of performance based analysis would justify possible additional costs from using more expensive materials by optimizing both the durability expectations of the road as well as the closure time for construction

#### 9.2.3 ASTM D7000 Sweep Test

The research team does not recommend that the ASTM D7000 sweep test be used by TxDOT unless a modification to the test method can be developed.

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# **APPENDIX** A

# SURFACE TREATMENT BINDER CONSTRUCTION TOOLKIT

# **Surface Treatment Binder Construction Toolkit**

Ambarish Banerjee Andre Smit Amit Bhasin Jorge Prozzi Center for Transportation Research University of Texas at Austin

Sanjaya Senadheera Center for Multidisciplinary Research in Transportation Texas Tech University

Research Project 0-5893 Research Product 0-5893-P1

Conducted for the Texas Department of Transportation

# RECOMMENDED TEST PROTOCOL FOR DETERMINING THE DILUTION RATIO OF THE PLANT EMULSION AT THE CONSTRUCTION SITE

#### SCOPE

• Use this test method to evaluate the dilution ratio of asphalt emulsions at the job location for the plant sample.

#### APPARATUS

- A heating device in the form of a hot plate or a gas-oven with the option to control the temperature or heat flow.
- A measuring balance capable of measuring up to 500 grams with an accuracy of up to 0.1 gram.

#### MATERIALS

• Deep labeled slip cover tin cans as shown in Figure 1, one can per test.



Figure 1: 16 oz. deep labeled slip cover tins (Available at http://www.houseofcans.com/deep-style-p-525-l-en.html)

#### PROCEDURE

1. Remove lid of deep slip cover tin can and weigh on the measuring balance. Note the weight (A).

**2.** Weigh approximately 30 grams of the emulsion from the transport truck into one of the deep slip cover tins. Note the combined weight of the tin can with the emulsion inside the can (B).

**3.** Note the type of emulsion being tested. Dilution ratio will differ considerably depending upon the type of emulsion.

**4.** Heat the can to a temperature between 140°C and 180°C (284° - 356°F) to boil the water off the emulsion.

Temperature should be maintained in this range to avoid vigorous agitation of the emulsion due to the removal of water through boiling. This can cause the emulsion to spill out of the can and distort the final results.

**5.** Turn off the heat when a grayish smoke appears, which indicates the complete removal of water from the emulsion. Make sure the agitation due to boiling of water has also ceased, as there should no water left in the emulsion.

**6.** Remove the can from the heating device when the weight reaches a constant level and let it cool to the ambient temperature.

**7.** Weigh the can again to record the combined weight of the can and the residual asphalt left after complete removal of water (C).

8. Calculate the dilution ratio for the emulsion according to the guidelines provided below.

**9.** Conduct four replicate tests on the emulsion sample for each truckload of the material delivered to the job site.

10. Report the average and the coefficient of variation for each of the four sets of measurements.

**11.** Compare the average dilution ratio against Item 300 specifications according to the "Standard Specifications for Construction and Maintenance of Highways, Streets, and Bridges." Report whether the material meets the requirements.

#### CALCULATIONS

Calculate the dilution ratio for the emulsion using the relation given below:

Dilution Ratio, R (%) = 
$$100 \times \frac{B-C}{B-A}$$

Where,

A = Weight of the empty deep slip cover tin can (in grams)

B = Combined weight of the slip cover tin can and the emulsion (in grams)

C = Combined weight of the empty slip cover tin can and the residue from the emulsion (in grams)

Note 1: Reference Table 1 for maintaining records of the different weights that are being collected over the duration of the test

Table 1: Weight Measurements	and	Calculated	Dilution	Ratio	Values
------------------------------	-----	------------	----------	-------	--------

Sample	Weight of	Combined weight of can and	Combined weight of can and	<b>Dilution Ratio</b>
#	empty can (A), g	the emulsion sample (B), g	the emulsion residue ( <i>C</i> ), g	(R), %
1				
2				
3				
4				

The following three equations can be used to calculate key statistical parameters for dilution ratio based on the four values for replicate samples tested.

Average Dilution Ratio, 
$$R_{avg} = \frac{R_1 + R_2 + R_3 + R_4}{4}$$

Standard Deviation,  $sd = \sqrt{\frac{(R_1 - R_{avg})^2 + (R_2 - R_{avg})^2 + (R_3 - R_{avg})^2 + (R_4 - R_{avg})^2}{3}}$ 

Coefficient of Variation, CoV =  $\frac{sd}{R_{avg}}$ 

#### **ARCHIVED VERSIONS**

None

# RECOMMENDED TEST PROTOCOL FOR DETERMINING THE VISCOSITY OF THE PLANT EMULSION AT THE CONSTRUCTION SITE USING SHELL CUPS

#### SCOPE

• Use this test method to evaluate the viscosity of asphalt emulsions at the job location for the plant sample.

#### **APPARATUS AND MATERIALS**

- Shell cup sizes 3, 3.5, 5 and 6 from Norcross Corporation with at least 3 cups for each of the four different sizes. Figure 2 shows a Shell cup #3.
- Temperature control unit in the form of a water bath or an insulating material, capable of keeping the temperature of the sample steady (within ±9°F) while conducting the experiment in the material.
- Stopwatch to record time taken for the emulsion to drain through the orifice in the Shell cups.
- Temperature gun or thermocouple to record the temperature at which the viscosity of the emulsion sample will be measured.



Figure 2: Shell Cup #3 (Available at http://www.viscosity.com/p\_ec\_sc.html)

#### PROCEDURE

1. Fill a quart can with the emulsion to be tested from the transport truck.

2. Place the quart can in an insulated material or in a temperature control unit to reach a steady state temperature as quickly as possible.

3. Monitor the temperature of the sample at 5 minute

intervals to ensure the sample has reached a steady state temperature. Record the final temperature.

4. Submerge the Shell cup in the fluid for approximately 30 seconds to allow the cup to come to sample temperature. The sample must be representative of the actual material to be tested, i.e. thoroughly stirred and at the normal operating temperature.

5. Lift the cup vertically out of the fluid, starting the stopwatch as the cup breaks the surface.

6. Record the time required for the cup to empty, stopping the watch when the stream breaks.

7. Read the viscosity from the appropriate conversion charts provided in Figure 3.

8. Repeat steps 1 through 7 to run three replicate tests on the emulsion sample. It is advised in this context that multiple numbers of different Shell cups should be included in the toolbox in order to avoid the cumbersome process of cleaning the Shell cups to run replicate tests in the field. Unavailability of proper setup and solvents like Trichloroethylene makes cleaning of Shell cups impractical in a field setup.

9. Report the results according to Table 2.

#### CALCULATIONS

The viscosity of the emulsion sample at the measured temperature can be obtained from the chart provided as part of Figure 3, provided that the time and the Shell cup size numbers are available.

The following three equations can be used to calculate key statistical parameters for viscosity (SFS) in centipoises based on the four values for replicate samples tested.

Average Viscosity, 
$$SFS_{avg} = \frac{SFS_1 + SFS_2 + SFS_3 + SFS_4}{4}$$
  
Standard Deviation,  $sd = \sqrt{\frac{(SFS_1 - SFS_{avg})^2 + (SFS_2 - SFS_{avg})^2 + (SFS_3 - SFS_{avg})^2 + (SFS_4 - SFS_{avg})^2}{3}}$ 

Coefficient of Variation, CoV =  $\frac{sd}{SFS_{avg}}$ 

#### **ARCHIVED VERSIONS**

None

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CENTIPOISE 1.00 2.00 3.00 5.00 7.50 10.0 15.0 20.0 25.0 30.0 40.0 50.0 60.0 70.0 a0.0 90.0 100 125 150 175 200 225 250 275 300 325 350 375 400 500 600 700 a00 900 1000 1250 1500 1750 2000 2500 3000 3500 4000 450 5000 6000 7000 a000 9000 10K References 'Ultra Low' Cup 53 68 84 Nete

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#### Figure 3: Shell cup conversion chart

## Table 2: Viscosity of the Emulsion as Measured with the Shell Cups

Sample #	Temperature (°F)	Time Reading (seconds)	Viscosity (SFS)
1			
2			
3			
4			

#### SPREADSHEET ALGORITHM

#### PURPOSE

The spreadsheet provides a guideline for making key decisions related to chip seal construction at the job site. These guidelines include time to place aggregates as well as time to open to traffic.

These guidelines relate to a conservative scenario where chances of early failure are minimized. The final output from the spreadsheet will definitely result in conservative estimates for time for placement of aggregates or opening to traffic. It is therefore highly recommended that proper engineering judgment should be invoked in addition to the guidelines provided by the spreadsheet to make key decisions at the field.

#### COMPATIBILITY

The spreadsheet was developed on Microsoft Excel 2001 and therefore the program can be executed on Microsoft Excel installations including Office 97, 2000, XP, 2003, 2007 and 2010.

The spreadsheet does not contain Visual Basic (VB) macros and therefore the security setting on the computer should not interfere with the execution of the program.

#### INPUTS

The spreadsheet requires 6 input parameters, highlighted in green in the spreadsheet. Figure 4 shows the input parameters required.

Type of Emulsion	HFRS-2P
Type of Aggregate	Limestone
Emulsion Spray Temperature (in °F)	155
Temperature Forecast (in °F)	83.3
Relative Humidity Forecast (in %)	56
Wind Speed (in mph)	5

#### Figure 4: Input Parameters in the Spreadsheet

- 1. The first two input parameters relate to the materials used at the construction site.
  - a. The spreadsheet provides a list of 7 different emulsion types (CRS-2, CRS-2P, HFRS-2, HFRS-2P, CMS-1P, CHFRS-2P and CRS-1P) as probable choices. The models included in the spreadsheet were developed with laboratory testing that involved these 7 emulsion types. However, in the unlikely event the engineer/contractor decides to use a different emulsion,

it is recommended that the user select a material that closely resembles the one being used for that job.

- b. The spreadsheet provides a selection of 3 aggregates limestone, quartzite and light weight. The models were developed based on laboratory results obtained from testing these three aggregates; thus, the predictions will be most accurate for these aggregate choices. However, sound engineering judgment should be used for deviations from these aggregate choices for estimating the time to place stones as well as for the time to open to traffic.
- 2. Enter the spray temperature for the emulsion. The spray temperature will vary based on the type of emulsion and the location of the job site.
- 3. Enter forecasted temperature, relative humidity and wind speed. However, since meteorological factors will fluctuate throughout the day, an average of the weather conditions during the paving period will provide the most accurate estimates of the time to place stones as well as the time to open to traffic.

The spreadsheet also provides three different sets of estimated time for placing stones and opening to traffic. Although each of these estimates refers to three different scenarios, namely "Most Expected Design," "Conservative Design," and "Maximum Risk," they essentially employ the same algorithm. However, this added feature was provided based on recommendations from the Project Monitoring Committee (PMC) to provide field inspectors and engineers the functionality to run simultaneous estimates for varying weather conditions.

#### OUTPUTS

The spreadsheet provides guidelines on the time to place the aggregates from the time the emulsion is shot until the newly constructed chip seal can be opened to traffic. In addition, it provides an estimate of the complex shear stiffness (G\*) at the time of opening to traffic. The output parameters are highlighted in blue. Figure 5 illustrates the output parameters.

Optimal Time to put down Aggregates (mm:ss)	12:56
Optimal Time to Open to Traffic (hh:mm:ss) *	8:12:00
Binder Stiffness @ Time of Opening to Traffic	2200 Pa

## \* - If cell is red, please revise your design

#### Figure 5: Output Parameters in the Spreadsheet

In its present configuration, if the time interval before opening to traffic is more than 8 hours, the spreadsheet will flag the design and request the user to revise the design. However, the cutoff value of 8

hours was decided based on the assumption that the paving crew should be able to open the road to traffic before the end of day, so that nighttime traffic control is not required.

#### CHARTS

Charts showing the rate of setting and the evolution of the shear stiffness of the emulsion residue provide a visual interface for the contractors and field inspectors. These charts will help them to not only judge when to open to traffic, but also be aware of likely risks of early failures if the road is opened before the recommended guidelines. Figure 6 illustrates the graphical section included in the spreadsheet.



Figure 6: Graphical Charts in the Spreadsheet

The spreadsheet also includes a graphical plot of the rate of cooling and the time to reach ambient conditions in the field as a function of time from the instant the emulsion is shot.

# **APPENDIX B**

# TRAINING WORKSHOP ON EMULSIFIED ASPHALT SURFACE TREATMENT CONSTRUCTION TOOLKIT









	Research Tasks	
Task	Description	
1	Literature Search and State-of-Practice Survey	
2	Field/Lab Evaluation of Binder Curing/Breaking	
3	Characterization of Binder Breaking/Curing Trends	
4	Develop a Field Test to Assess Emulsion Quality	
5	Evaluation of Emulsion Sampling/Handling Processes	
6	Field Evaluation Program	
7	Develop and Deliver a Training Course	
8	Reporting	
A Damaser of Damaserian	Research Project 0-5893 Training Workshop	6













#### Interaction of Cationic Emulsion with Aggregate • The cations has closer affinity to aggregate than water. When the breaking starts, emulsifier is attracted to the aggregate and asphalt particles will be fixed on the surface of aggregate. EXECT: The cations has closer affinity to aggregate than water. When the breaking starts, emulsifier is attracted to the aggregate and asphalt particles will be fixed on the surface of aggregate. 21

#### Interaction of Emulsion with Aggregate

•This could be influenced by aggregate mineralogy, ambient temperature and other factors.



Tx	DOT Emulsio 12/01/2007-12	n Shipmeı 2/01/2008	nts	
	Emulsion Grade	Shipments		
	CRS-2P	954		
	CRS-2	936		
	CSS-1H	824		
	SS-1	602		
	CSS-1P	509		
	HFRS-2	316		
	HFRS-2P	212		
	CRS-1P	203		
	CHFRS-2P	140		
Parameter Parameter Parameter	Research Project 0-5893 Training \	Vorkshop		15





































	Temperature (F)	Humidity (9/ DH)		E	Binder	
	remperature (F)	Humidity (% RH)	Limestone 1	Granite	Sandstone	Limestone 2
CTCH	T = 85	H = 20	CBS 2	CDC 2	CDS 3	CDE 3
CICH	Test at 7	77 (25C)	un3-2	ChS-2	un3-2	uno-2
	T1 = 90	H1 = 30				
H&D	T2 = 64	H2 = 80	CRS-2	CRS-2	CRS-2	CRS-2
	Test at 7	77 (25C)				
	T3 = 95	H3 = 60				
H&H	T4 = 72	H4 = 90	CRS-2	CRS-2	CRS-2	CRS-2
	Test at 7	77 (25C)				
	T5 = 90	H5 = 60				
VT	T6 = 70	H6 = 60	CRS-2	CRS-2	CRS-2	CRS-2
	Test at 7	77 (25C)				
	T7 = 78	H7 = 40				
VH	T8 = 78	H8 = 80	CRS-2	CRS-2	CRS-2	CRS-2
	Test at 7	77 (25C)				
	T9 = 10	H9 = 50				
CTCH2	T10 = 70	H10 = 50	HFRS-2P			HFRS-2P
	Test at 7	77 (25C)				
	T10 = 76	H11 = 85				
H&H2	T11 = 101	H12 = 40	HFRS-2P			HFRS-2P
	Test at 7	77 (25C)				
	T12 = 77	H13 = 80				
H&H3	T13 = 91	H12 = 68	CRS-2H			CRS-2H
	Test at 7	77 (25C)				
	T14 = 41	H14 = 68				
C&D	L T15 = 76	H15 = 23	CRS-1P	CRS-1P		
	Test at 1	77 (25C)				
Temperatu	re and humidity cycles	will be set on 12 hour i within envi	amps. Testing tim ronmental chambe	ies at 12, 16, er.	20 and 24 hours	after submgrsion







































### Observations from DSR Strain Sweep Test Results

•Provides consistent results on emulsion setting and curing with time.

•Shows the sensitivity of binder stiffness to time of curing, binder-aggregate interaction and climate.

•Can be used to identify good (and poor) material combinations that work well together locally.

Research Project 0-5893 Training Workshop



























## Outline

- Curing Rates
- · Setting Rate and Development of Stiffness in Residue
- Field Tests (QC/QA)
- · Determination of Threshold for Opening to
- Conclusions
- · Demo of spreadsheet



- · Realize the factors influencing curing
- · Understand the influence of weather
- · Understand the influence of materials
- · Know when to place aggregates
- · Know when to open to traffic
- · Test and realize the quality of emulsions







Type of Emulsion	HFRS-2P
Type of Aggregate	Limestone
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Temperature Forecast (in °F)	83.3
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Mind Cound (in much)	5

Outputs from the Alg	orithm
Optimal Time to put down Aggregates (mm:ss)	12:56
Optimal Time to Open to Traffic (hh:mm:ss) *	8:12:00
Binder Stiffness @ Time of Opening to Traffic	2200 Pa
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## Measurement of Curing Rates

- k<sup>T</sup> is an evaporation constant that depends on the air temperature; for tap water it is proportional to the air temperature
- For emulsions, k<sup>T</sup> values were not consistent due to the dynamic nature of phase transformation boundary





# Measurement of Curing Rates






## Measurement of Curing Rates Determination of the duration of cooling: Depends on the differential between the ambient conditions and spray temperature Higher differential will imply longer duration of cooling

## Measurement of Curing Rates

Type of emulsion	k
CRS-2P	0.0175
CRS-2	0.0327
HFRS-2P	0.0092
HFRS-2	0.0138
CRS-1P	0.0224
<ul> <li>Field data collected sites to determine the Used for determinate end of the cooling p</li> </ul>	from construction ne cooling rate tion of time to reach rocess





## Measurement of Curing Rates

- Weather station deployed on site for 24/7 recording of atmospheric conditions
- Emulsion samples placed on PAV pans
- Film thickness = 2mm
- Decided on the basis of typical application rates (~0.30 gal/yd<sup>2</sup>)
- Sample weight ≈ 30 grams



# Measurement of Curing Rates Water evaporation depends on: Vapor pressure deficit Turbulence in the ambient air. Material properties like type/dosage of the emulsifier, etc.



## Measurement of Curing Rates

Model Statistics Adjusted R-So	nuare		).7	
Standard Er	rror	0.51		
f-statistic	:	0.00		
Number of Obse	rvations		53	
Effect	Coefficient	t-stat	p-value	
Intercept	0.520	3.0	0.01	
VPD × Wind Speed	0.230	9.2	0.00	
Medium Setting = Yes	-0.598	-3.5	0.00	
Cationic = Yes	0.434	2.9	0.01	
Hard Base = Yes	0.438	2.2	0.03	
Anas Countered Orangeotation			V <sub>CT</sub>	

## Measurement of Curing Rates

- Higher VPD translates to faster curing rate.
- Cationic emulsions cure faster than anionic emulsions.
- Emulsions with harder asphalt bases cure faster.







Measurement of Curing Rates								
	Brackenridge	Plainview	Floydada	Comanche	Giddings			
Binder	CRS-2	CRS-2P	CRS-1P	CRS-2H	HFRS-2P			
Spray Temperature (°F)	140	140	130	133	135			
Mean Air Temperature (°F)	96.6	77.6	75.4	78.3	83.3			
Time to reach Thermal Equilibrium	4	5	5	3	4			
Time to chip placement (mins)	15	07:15	13:30	07:30	03:30			
% Water Lost during Cooling	14	8	10	14	3			
% Water Lost due to Evaporation	14	3	12	4	0			
Total Water Loss %	28	11	22	18	3			
Threshold for Chip Placement		3						
Trans Companyor Cl Damportation					CTR			







Prediction Model – Emulsion Setting								
Effect	p-value							
β (Intercept)	-0.1833	-18.3	0.00					
β (Vapor Pressure Deficit)	2.1764	26.4	0.00					
β (Quartz)	0.0136	2.6	0.01					
β (Light Wt)	0.0099	1.9	0.03					
β (CMS-1P)	0.0566	1.4	0.08					
β (HFRS-2P)	-0.0518	-1.9	0.03					
β (CRS-2)	0.0434	1.6	0.06					
β (CRS-2P)	0.3129	7.8	0.00					
β (Intercept based on VPD [Baseline])	0.0344	4.5	0.00					
β (Intercept based on VPD [CMS-1P & HFRS- 2P])	0.0226	3.5	0.00					
β (Intercept based on VPD [CRS-2P])	0.0404	5.2	0.00					
β (Intercept based on VPD [HFRS-2])	0.0725	3.2	0.00	_				
Standard Error								
Department of Demportation			VC1	R				























Effect	Coefficient	t-stat	p-value					
β (Intercept)	904.3	5.5	0.00					
β (Intercept CRS-2)	1694.6	1.6	0.05					
β (Intercept based on VPD [Baseline])	319.6	3.7	0.00					
β (Intercept based on VPD [CHFRS-2P])	2664.0	10.1	0.00					
β (Intercept based on VPD [CRS-2])	-1272.1	-2.5	0.01					
β (Intercept based on VPD [CRS-2P])	848.0	4.7	0.00					
β (Limestone)	-291.9	-3.0	0.00					
β (Quartzite)	-225.0	-2.5	0.01					
β (Light Wt Aggregate)	-94.9	-1.2	0.12					
β (Slope [Baseline])	0.484	9.8	0.00					
β (Slope [CHFRS-2P, CRS-2P])	0.067	1.7	0.04					
β (Slope [CRS-2])	-0.545	-3.0	0.00					
$\beta$ (Slope based on VPD [CHFRS-2P, CRS-2P])	-0.093	-6.3	0.00					
β (Slope based on VPD [CRS-2])	0.395	3.8	0.00					
Standard Error =	= 1.7 kPa							























### Summary – Dilution Ratio Mass Loss Test Results from diff Projects Site Location # of Samples Mean CoV Floydada, Lubbock (CRS-1P) 69.4% 1.8% 4 Jasper, Beaumont (CRS-2P) 3 70.2% 1.8% Giddings, Austin (HFRS-2P) 68.7% 0.7% 3 Mason, Austin (CRS-2P) 3 65.3% 3.5% Comanche, Brownwood (CRS-2 65.8% 2H) CTR











Summary – Viscosity Measures								
Temperature (in °F) Time (seconds) Viscosity (SFS)								
	#1	107	46	135				
Shell Cup #5 (Jasper)	#2	98	39	116				
(ouopoi)	#3	111	38.4	114				
	#1	95.6	31.7	93.1				
Shell Cup #5 (Giddings)	#2	92.3	31.6	92.8				
(olddingo)	#3	92.1	31.9	93.7				
	#1	94.7	37.9	112.2				
Shell Cup #5 (Mason)	#2	95.1	40.7	120.9				
(11123011)	#3	93.6	37.6	111.2				
and The second s				le le				

































Thresholds for Opening to Traffic Thresholds for timing a newly constructed chip seal to be opened to traffic								
	Project Location	Type of Emulsion	Time to Open to Traffic	% Water Lost	Stiffness of Residue in kPa			
	Giddings	HFRS-2P	8 hrs	79	2.2			
	Mason	CRS-2P	4 hrs	70	4.3			
	Comanche	CRS-2H	1 hr	78	4.4			
	Minimum amount of water lost (%) before opening to traffic = 76							
Minimum required stiffness for the emulsion residue for satisfactory performance = 2.2 kPa (Anionic), 4.3 kPa (Cationic)								
						TR		











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