# Chip Sealing Systems: Improving Early Age Chip Retention

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ABSTRACT: A number of solutions aiming at better chip retention have been tried over the years, such as using quick-set polymer-modified emulsions, using smaller and cleaner chips or modifying certain aspects of construction practices. This paper is intended to take an in-depth and more systematic look at various technical aspects that can have an impact on improving early chip retention by an asphalt emulsion. A group of asphalt emulsions have been selected for this laboratory study, containing both anionic and cationic types and having different types of polymer modifications. Curing of the emulsions is studied by assessing the development of film strength in the binder layer by rheological measurements and by the Frosted Marble Cohesion test. Subsequently, chip retention on a variety of stone types is assessed by means of the Sweep Test for Surface Treatments. The outcome indicates that the CRS type emulsions and certain polymer systems show advantages in improving early chip retentions.

KEY WORDS: Chip retention, surface treatments, emulsion curing, asphalt emulsion, chip seal

# 1 INTRODUCTION

The concept of pavement preservation is gaining ground in today's world, as more road agencies start implementing advanced asset management systems. As a way of extending service life, it becomes essential to protect existing asphalt pavements from traffic and environmental distresses. This is done most commonly by applying surface treatments on structurally sound roads. By employing surface treatments, a number of immediate and important benefits are gained for a relatively low cost: restoring surface quality and aspect of the road, waterproofing the existing pavement and extending its life. Chip seals are the most cost-effective surface treatment suitable for high volume roads. In cold climates, a layer of calibrated chips is applied onto a layer of a rapid or medium-set asphalt emulsion (single chip seals). Warmer climates also see the usage of hot-applied bituminous binders for chip seals, with ongoing debates about one or the other system's advantages and benefits (Gransberg, 2003). Our current study will focus exclusively on chip seals using bitumen emulsions.

Some of the most important factors that will determine the success or failure of a chip seal are project selection, aggregate and emulsion application rates, quality of the selected materials and construction practices. Several chip seal design methods are available and performance prediction models have been developed (Roque et al. 1991). A performance-graded binder specification for surface treatments has also been proposed (Barcena et al. 2002). A significant number of aspects

will still be left at the latitude of the engineer, especially when it comes to aggregate and emulsion type selection.

#### 2 OBJECTIVES

The current study is intended to expand the existing information regarding curing patterns of different emulsion types and improvement of early stone retention through increased cohesion development in chip seals. Carefully selected and prepared asphalt emulsions used in chip seal applications are investigated by looking at rheological properties such as strain dependency and viscoelastic modulus values at different curing times. Cohesion development during film curing is measured by the Frosted Marble Cohesion test. Three representative aggregates of different mineralogical nature have been selected for evaluation of curing characteristics of emulsion-aggregate combinations by measuring stone loss using the Sweep Test of Bituminous Surface Treatment Samples (ASTM D 7000-04).

#### 3 EXPERIMENTAL DESIGN

#### 3.1 Materials

Ten bitumen emulsions and three aggregates have been selected for the current study.

The first 8 emulsions are prepared in the laboratory under controlled conditions, from base asphalt of the same grade and source (PG 58-28, penetration ~120). The first four are cationic rapid-sets and are prepared using a commercially available emulsifier for CRS-2 emulsions, at identical dosages. One is non-modified (CRS-2) and 3 are polymer modified, all containing the same polymer level of 3% to the emulsion. The difference between these 3 emulsions lies in the type of polymer modification: one is modified using SBR latex (CRS-2P Latex) while the second is made by modifying the asphalt with SBR prior to emulsification (CRS-2P PMA). The third contains the same 3% SBR level but half is contained in the binder and half is contained as latex (CRS-2P Comb). The reason for this approach was that morphology and structure of the polymer network is believed to have impact on curing properties and on the development of film strength.

The next four emulsions are of anionic HFMS-2 type and have been designed and prepared in the lab using the exact same concept as the cationic rapid-sets: HFMS-2 is non-modified, HFMS-2P Latex has 3% SBR in latex form, HFMS-2P PMA is made from SBR modified asphalt while HFMS-2 Comb contains the SBR polymer in both forms. These emulsions are prepared using a commercially available tall oil fraction as an emulsifier, all having the same dosage.

The last 2 emulsions are plant samples of HFRS type. The structure of these emulsions is different, however, as they are prepared from a pre-gelled asphalt cement and use a tall oil as emulsifier. HFRS-2 is non-modified and HFRS-2P is modified using SBR latex. Although the compositions of these emulsions do not correlate with the first group, we decided to include them in the study. They will not qualify for comparative conclusions but they can illustrate some different curing behaviours. A summary of the emulsions is presented in Table 1.

Three aggregate types have been selected. The first is a limestone, the second is a granite and the third is a meta-gabbro traprock. All three aggregates are from Ontario and all are relatively clean (fines  $\leq 1\%$ ). The total aggregate gradation is not essential, as only a selected fraction is used for the sweep test.

Emulsion	Viscosity,	Demulsibility*,	Dist. Residue,	Res. Penetration,
	50°C, SFS	%	% mass	25°C, dmm
CRS-2P	112	98.1	72.4	101
CRS-2P Latex	83	97.5	71.8	91
CRS-2P PMA	123	66.9	71.5	87
CRS-2P Comb	118	98.2	70.8	97
HFMS-2	201	68.0	64.9	115
HFMS-2P Latex	137	62.5	66.3	88
HFMS-2P PMA	288	62.9	65.9	83
HFMS-2P Comb	250	65.6	67.5	86
HFRS-2	82	97.4	65.1	157
HFRS-2P	85	94.2	63.8	140

Table 1: Summary of Emulsion Properties

\* - Following solution quantities have been used for demulsibility test:

- 35 ml Dioctyl Sodium Sulfosuccinate 0.8 % for CRS

- 35 ml CaCl<sub>2</sub> 0.02N for HFRS

- 50 ml CaCl<sub>2</sub> 0.02N for HFMS

# 3.2 Testing Protocol

# 3.2.1 Frosted Marble Cohesion Test

The current test was developed for measuring the chip retention abilities of an emulsion (Benedict 1990, Guiles 1995). It uses the modified ISSA cohesion tester (the same used for measuring cohesion for microsurfacing and slurry seal). The 1 1/8" original tester foot is replaced with a special 50 mm hooked foot. Each specimen is prepared in a trough plate with 3 rows. In each row 9 grams of emulsion are poured and 5 frosted marbles are placed in the emulsion using a template, within 5 minutes after pouring the emulsion (heated to  $60^{\circ}$ C). Curing times for testing have been selected at 2, 4, 6 and 24 hours, as our focus is on stone retention during the very early stages of a chip seal application (the first few hours especially). Curing of the specimens has been done on the lab bench, at ambient temperature (22 - 25°C) and no heat lamps or forced draft ovens have been used. This environment simulates the curing of a chip seal on a cloudy summer day (no direct exposure to sunlight) in a cold climate area. At each of the specified curing intervals, the frosted marbles are each torqued out by use of the cohesion tester and the average cohesion value is recorded.

## 3.2.2 Rheological Tests

A SmartPave dynamic shear rheometer by Anton Paar has been used for performing the rheological tests, using parallel plate geometry (25 mm diameter). It is essential to use a temperature control chamber in air, as the water bath conditioning chambers can't be used for incompletely cured emulsions.

Immediately after performing the frosted marble cohesion test, a sample of the residue at the specified curing time has been loaded into the DSR (at 2, 4, 6 and 24 hours curing). After a very short period of gentle heating to ensure good adhesion to the plates (45°C) strain sweeps were

performed at 25 (1-20% strain) and 60°C (2-200% strain). The test frequency was set at 10 rad/s. Each sample was allowed to equilibrate for 10 minutes at each test temperature before testing. The DSR testing protocol of choice has to address specific failure mechanisms and at the same time has to have a relatively short duration, in order to capture the material properties at the desired curing stage. A long testing protocol would be inappropriate due to changes in properties of the material with curing.

For comparison purposes, the same strain sweep test was also performed on the distillation residues of the bituminous emulsions.

#### 3.2.3 Sweep Test of Surface Treatment Samples

A number of tests have been developed over time, aimed to measure the capability of binders to retain the stone. The most established are the Vialit test, the ACTE test (Adhesion-Cohesion Test Esso) (Serfass et al, 1997), the Nynas Spin Test (Redelius & Stewart, 1992) and the ASTM D 7000 Sweep Test. The last one has been chosen for our study.

Specimen preparation consists of spreading a precise amount of emulsion on an asphalt felt disk, then uniformly spreading an quantity of an aggregate fraction, collected between the 9.5 and 4.75 mm sieves. The spray and spread rates are defined by the test protocol and have no direct relation with field application rates. The exact aggregate amount is calculated by a formula and is based on the bulk specific gravity of the stone and on the % fractions between the 9.5 and 6.3mm sieves and between the 6.3 and 4.75 mm respectively (ASTM D 7000, 2004). Specimens are compacted using a specially designed compactor and then cured. At the desired curing time, the specimens are brushed off with the fingertips to remove loose stone and then they are swept for 1 minute in a Hobart mixer, using a specially designed nylon brush. Weights are recorded prior to and after sweeping and the percent mass loss during sweeping is calculated.

Specimens of each emulsion with each of the aggregates have been prepared and cured on the lab bench, as previously described. Sweep tests have been performed at 2, 4, 6 and 24 hours curing time. Sweep tests loss, as well as total loss have been recorded (cumulates the aggregate lost during the sweep test and during the "loose aggregate removal" fingertip brush-off).

## 5 RESULTS AND DISCUSSION

#### 5.1 Analysis of the Frosted Marble Cohesion Test Data

The cohesion values obtained for the 10 emulsions at each of the curing times are listed in Table 2 and the curing trends for each emulsion are shown in Figure 1.

The curing rate (represented by the slope of the data trend) is steeper for the cationic emulsions. The CRS-2 and CRS-2 PMA have consistently higher cohesion values than all the anionic emulsions. The two cationic emulsions containing latex have lower initial cohesion values compared to the other cationics but they are the fastest cohesion gainers. At 24 hours, all the cationics show significantly higher cohesion values than all other types. The difference observed between the latex and the PMA cationic emulsions lies with the difference in polymer morphology. The PMA emulsion contains the SBR polymer in the binder therefore, with breaking of the emulsion (rapid by design), the cohesion buildup can take advantage instantly of the polymer presence. This seems to be especially effective during the first 2-3 hours of its curing time.

Cohesion (kg.cm)	2 Hours	4 Hours	6 Hours	24 Hours
CRS-2P	9	10.83	12.23	21.92
CRS-2P Latex	4.4	7.8	11.6	22.42
CRS-2P PMA	11.2	13	16.4	23.8
CRS-2P Comb	6.6	18.6	19.6	23.8
HFMS-2	7.2	9.8	13	17.26
HFMS-2P Latex	8.2	10.6	11.6	17.36
HFMS-2P PMA	7.4	10.8	12.8	15
HFMS-2P Comb	7	9.6	13.2	15.87
HFRS-2	4.2	5.6	6.6	8.1
HFRS-2P	4.6	5.2	6.6	9.7

Table 2: Emulsion Cohesion Values by the Frosted Marble Test



Figure 1: Emulsion Curing Trends by Frosted Marble Cohesion

With the emulsions containing latex, the breaking and setting processes are well described in literature (Takamura, 2000). The SBR particles are creating a honeycomb structure with curing of the residue. This morphology is very effective but it needs almost complete curing of the binder before it can deliver its full mechanical benefit. The combination CRS-2P shows relatively low cohesion at two hours but has the highest cohesion of all emulsions at 4 and 6 hours. All 4 cationic emulsions display similar cohesion values after 24 hours.

All 4 emulsions belonging to the HFMS group show a close behaviour pattern. Their cohesion values are lower than those of the cationics and their rate of cohesion gain is also lower. At 24 hours they have developed about 70% of the strength of the cationics. No significant differences are visible between the different types of modification systems.

The two HFRS members have consistently lower cohesion values and also slower cohesion build-up. However, these two emulsions are the most "rapid setting" of the whole group, as they have the highest demulsibility values. This emphasizes that speed of breaking on the one side and curing characteristics on the other side are not directly related. Just because it is rapid setting does not guarantee that an emulsion will work well in chip seals.

#### 5.2 Analysis of the Rheological Test Data

Experience shows that the majority of aggregates that get dislodged from a chip seal application at early stages suffer cohesive failure. If the design, aggregate selection and key construction steps are done correctly, the number of chips that are not properly wetted and embedded in the binder should be minimal. If loose stones fly around, the key word here is "loose" and that becomes a construction problem and not necessarily a chip retention issue.

In developing the performance-graded specification for surface treatments, Barcena proposes a failure mechanism at intermediate and high temperatures that is based on the upper bound theorem and that is based on ductile yielding (Barcena et al. 2002). Such a mechanism will account for very high strain levels in the binder adjacent to the aggregate-bitumen interface. Testing for strain tolerance of the binders becomes very important, as failure does occur outside the linear viscoelastic region. Their strain dependency varies widely with binder and modification type and is a strong function of temperature and frequency (Bahia et al. 1999).

Figures 2 to 11 show the complex shear modulus G\* versus strain, tested at 25 and 60°C, at each of the curing times. These tests should illustrate the strain dependency of the binder at a particular curing stage and at two relatively extreme temperature conditions that can occur during chip seal curing: a cool day and a hot summer day. For comparison, strain sweeps have also been performed on the distillation residues.

DSR data shows that the cationic emulsions have considerably higher shear moduli during early curing stages. After just 2 hours, in tests at 25°C, all the cationic emulsions have developed at least 75% of their complex shear modulus value at 24 hours cure, with some as high as 90%. Surprisingly, even the CRS-2P Latex, which has performed poorly in cohesion tests at 2 and 4 hours shows rapid gain in modulus. From the anionic group, the HFMS-2P PMA followed by the HFMS-2P Comb show the quickest gain in modulus, with the non-modified HFMS being the slowest. At early stages moduli for the anionics are significantly lower than those of the cationics and, although the gap diminishes with curing, the moduli never achieve close values with the cationics within the first 24. Complex shear modulus values of the distillation residues are close.



Figure 2: Complex Modulus, 2h cure, 25°C



Figure 3: Complex Modulus, 2h cure, 60°C



Figure 4: Complex Modulus, 4h cure, 25°C



Figure 6: Complex Modulus, 6h cure, 25°C



Figure 8: Complex Modulus, 24h cure, 25°C



Figure 5: Complex Modulus, 4h cure, 60°C



Figure 7: Complex Modulus, 6h cure, 60°C



Figure 9: Complex Modulus, 24h cure, 60°C



Figure 10: Complex Modulus, Residue, 25°C

Figure 11: Complex Modulus, Residue, 60°C

The comparison of the complex shear moduli for the 24 hours curing with those for the distillation residues shows that for all the HFMS emulsions the 24 hour values represent 50% or less of the residue values. Meanwhile, for the CRS type emulsions, the 24 hour and the residue values are in the same range. This indicates that, under the described curing conditions, the studied CRS emulsions gain almost their entire strength within the first 24 hours, while the HFMS emulsions gain about half or less. The data also points out that for the cationic emulsions containing latex, the modulus for the residue is notably lower than the one at 24 hours cure. This confirms that the polymer structures that develop during curing are destroyed with distillation and that the properties of the distilled residue do not reflect the full performance of cured emulsions in the field (Takamura, 2000). It is also worth pointing out that while at 25°C the non-modified CRS-2 has much higher modulus than all the HFMS emulsions, regardless of curing time, in the 60°C test the HFMS-2P PMA and HFMS-2 Comb start overtaking the CRS-2 after 4 hours cure. The contribution of the polymer is evident in the anionics as well, and becomes more obvious at higher test temperatures.

Strain dependency is more pronounced during early stages of curing. Cationic emulsions seem to have more strain tolerance than the rest. Some data shows gradual drop in the modulus with increased strain while other shows abrupt discontinuities. These are most likely caused by catastrophic structural failure of the test sample where the sample integrity is lost (ex. through internal slippage). One shall not forget that we are attempting to capture the "snapshot" of a changing system at one point in time. The uniformity and isotropy of these samples during curing stages cannot be assumed with certainty.

The two HFRS emulsions show lower moduli and slower curing, in agreement with the marble cohesion test results.

#### 5.2 Analysis of the Sweep Test Data

The chip loss as a result of the sweep test can yield unexpected conclusions in some situations, as the ASTM procedure only requires the recording of the loss of aggregate under the direct action of the brush. Our experience with slower emulsions shows that there can be a fairly large aggregate loss during the finger brush-off step of the specimen preparation. As a result, the %

sweep loss recorded by the ASTM method can be small while, in reality, the aggregate loss of one specimen is next to total. This observation has led us to recording three different parameters:

- Sweep Loss (SL), as described by ASTM D 7000, is calculated as % mass loss of the specimen during the sweep test
- Loss Before Sweep (LBF) is the % mass lost during specimen preparation and curing and includes moisture loss by the emulsion while curing, loss of loose un-embedded aggregate and of the loosely embedded aggregate that come off during the hand brushing step
- Total Loss (TL) is the % mass lost by the specimen since preparation and until after completion of the sweep test. It includes the loss of moisture, un-embedded chips, stone lost during hand brushing and stone loss during the sweep test.

It can be assumed that under identical specimen preparation and curing conditions, the mass loss resulting from moisture evaporation and from the excess aggregate is constant. Therefore, the TL parameter is the best to reflect the chip lost due to poor chip retention by the binder, before and during the sweep test. Figures 12-14 show the total loss (TL) for the selected aggregates.

90



Figure 12: Percent Total Loss, Limestone



Figure 13: Percent Total Loss, Granite



Figure 14: Percent Total Loss, Traprock

Based on the previous curing data observed, it is expected that cationic emulsions will have lower chip loss during the early curing stages. The sweep test results confirm this assumption for all aggregate types tested. Chemical compatibility with the aggregate becomes more important once the emulsion residue starts building modulus and the failure mechanism starts shifting from cohesive towards more adhesive mechanisms. At 24 hours, for example, anionic and cationic polymer modified have very close TL results with the limestone chip, while with the granite chip the anionics show considerably higher losses. This can be attributed to a weaker bond between anionic emulsions and silica type aggregates.

# 6 CONCLUSIONS

Ten asphalt emulsions were analyzed with respect to their curing properties during the first 24 hours. Each emulsion was subsequently tested with three different aggregates for assessing stone retention using the sweep test. The following are the main findings of this study:

- The cationic emulsions studied developed cohesion and modulus quicker than the anionic emulsions under similar curing conditions. The distillation residues showed comparable modulus values but the time needed for the curing emulsions to achieve that modulus range was significantly shorter for the cationics. Measuring the cohesion of the curing emulsion by the Frosted Marble test reflected the same trend.
- The type of polymer modification of the emulsion impacts the film strength development in the very early stages of curing. PMA emulsions can benefit of their polymer content within the first 2 hours while emulsions containing latex require more curing time before the polymer becomes of benefit. Within 24 hours under the described curing conditions their performance becomes comparable. This behaviour has been more obvious with cationic emulsions. Anionic emulsions containing PMA and latex have shown fairly similar behaviour.
- Combination PMA-Latex emulsions have performed well but no special benefit has been observed so far from having the SBR polymer both inside and around the asphalt binder. More research is needed to fully characterize such systems.
- The strain tolerance of the emulsion residues increases with curing. As they cured faster, the cationic emulsions in our study have shown less strain dependency during early stages. This should be beneficial for improved early stone retention, as failure in fresh chip seals is predominantly cohesive in nature.
- The current study allows no direct comparison between distillation residues and cured emulsions, as during the first 24 hours at room temperature complete curing of the emulsions is not achieved. However, the properties of distillation residues seem to poorly reflect the properties of cured cationic latex modified emulsion residues in particular, confirming earlier literature observations.
- The cationic emulsions have performed consistently better in sweep tests with all the aggregate studied. They have also shown less sensitivity towards the different chemical composition of the stone than the anionic emulsions.

The result of this research underlines the many factors that affect early stone retention following the construction of a chip seal. It only emphasizes the need for the engineer to thoroughly assess all the aspects affecting the final chip seal design. Good material evaluation, selection and understanding beyond basic specifications is critical of achieving best possible results.

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