DEVELOPMENT OF ADHESIVE AND COHESIVE PROPERTIES OF RAPID-SETTING BITUMEN EMULSIONS DURING CHIP SEAL CONSTRUCTION

Anton S. Kucharek*, J. Keith Davidson* and Jean-Martin Croteau**

*McAsphalt Industries Limited, 8800 Sheppard Ave. E, Toronto, Ontario, M1B 5R4
**Miller Paving Limited, 287 Ram Forest Road, Gormley, Ontario, L3R 9R8

ABSTRACT

Quick and strong chip retention by the asphalt emulsion is extremely important, especially when used on high-speed highways where flying chips can present a hazard. The risk of stone loss is increased during the first few hours after chip seal construction. The current paper is taking a more in-depth look at various cohesive and adhesive factors that can have an impact on improving early chip retention by an asphalt emulsion. A group of quick-set asphalt emulsions have been selected for this study, both anionic and cationic, with and without polymer modification. The curing of the emulsions is studied at different time intervals by measuring rheological parameters and cohesion values. These values are an indication of how fast film strength develops in the binder layer during emulsion coalescence, subsequent to the chip seal construction. Stone retention on a variety of aggregate types is then measured, by means of the Sweep Test for Surface Treatments and by the Vialit test. The outcome indicates that the CRS type emulsions and certain polymer systems build cohesion quicker and have rheologiocal properties during curing that are less susceptible to failure. Cationic emulsions are less aggregate sensitive when it comes to adhesion but the capability of penetrating dust films on the stone also plays an important role.

RESUME

1   INTRODUCTION AND OBJECTIVES

The current lab 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 complex shear 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)(1). Mechanical adhesion aggregate-binder at 24 hours is then evaluated by the means of the Vialit Plate Shock Test method (12272-3:2003)(2).

2   EXPERIMENTAL DESIGN

2.1   Materials.
The current study involves the usage of 10 different bitumen emulsions and 3 different aggregate, each having a different mineralogy. 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, all using using equal amounts of a commercially available tall oil fraction as an emulsifier. Polymer modification is identical in nature and dosage as described for the CRS emulsions.

The last 2 emulsions are plant samples of HFRS type, prepared from a pre-gelled asphalt cement. A summary of the emulsion properties is presented in Table 1.

The first of the aggregates is a limestone, the second is a granite and the third is a meta-gabbro traprock. All three aggregates are relatively clean (fines <= 1%). The limestone has the highest dust content (just under 1%), with a visible dust film deposit on the chips.

Table 1: Summary of Emulsion Properties

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

Solutions used for demulsibility:

[a] - 35ml DOSS 0.8%
[b] - 50ml $CaCl_2$ 0.02N
[c] - 35ml $CaCl_2$ 0.02N

2.2   Testing Protocol
2.2.1   <u>Frosted Marble Cohesion Test</u>. The current test was developed for measuring the chip retention abilities of an emulsion (3, 4). The 1 1/8" original foot of an ISSA cohesion tester 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°C). Curing times for testing have been selected at 2, 4, 6 and 24 hours. Curing of the specimens has been done on the lab bench, at ambient temperature (22 - 25°C). 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.
2.2.2   <u>Rheological Tests</u>. A SmartPave DSR by Anton Paar has been used for performing the rheological tests, using parallel plate geometry (25 mm diameter) and a temperature control chamber in air.

A sample of the residue 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), at 10 rad/s. 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.
2.2.3   <u>Sweep Test of Surface Treatment Samples (ASTM D-7000)</u>. Several other test methods for determining chip retentions exist today (5, 6). For the sweep test, the sample preparation and test method is described by the ASTM D-7000 procedure. 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 has been recorded.
3.2.4   <u>Vialit Plate Shock Test (12272-3:2003)</u>. Each of the 10 emulsions has been tested for mechanical adhesion with all three aggregate, at 24 hours curing time, in a lab environment similar as described for the previous tests.

**3   RESULTS AND DISCUSSION**

3.1   Analysis of the Frosted Marble Cohesion Test Data
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.
With the emulsions containing latex, the breaking and setting processes are well described in literature (7). 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.

Table 2; Fig 1: Cohesion by Frosted Marble Test

Cohesion (kg.cm)	2 Hrs	4 Hrs	6 Hrs	24 Hrs
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 Com	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
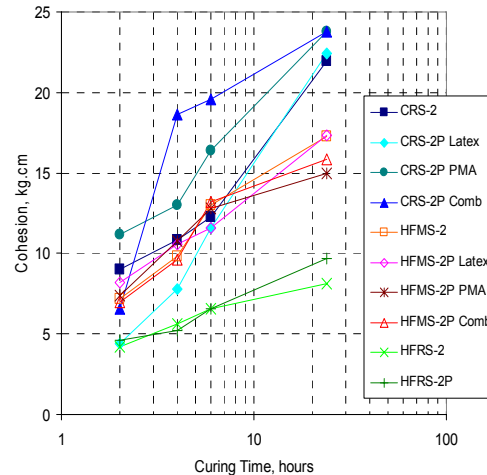
All 4 emulsions belonging to the HFMS group display cohesion values that are lower than those of the cationics. Their rate of cohesion gain is also lower. At 24 hours they have developed about 70% of the strength of the cationics. No significant differences are visible between the different types of 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.
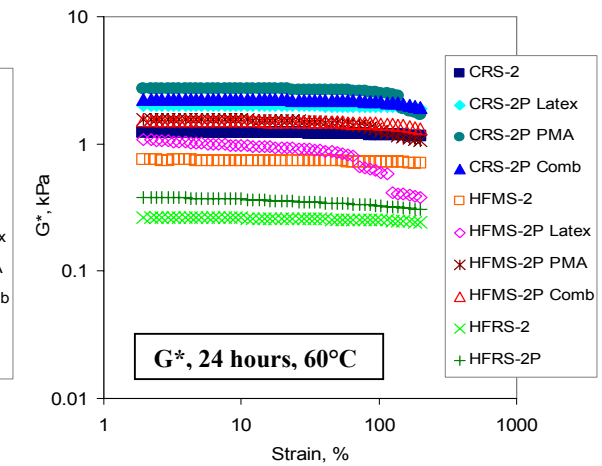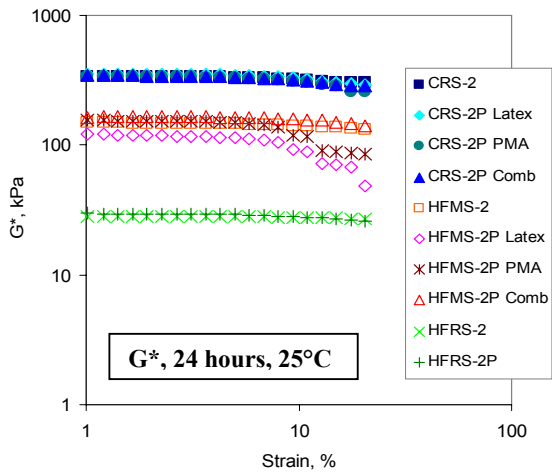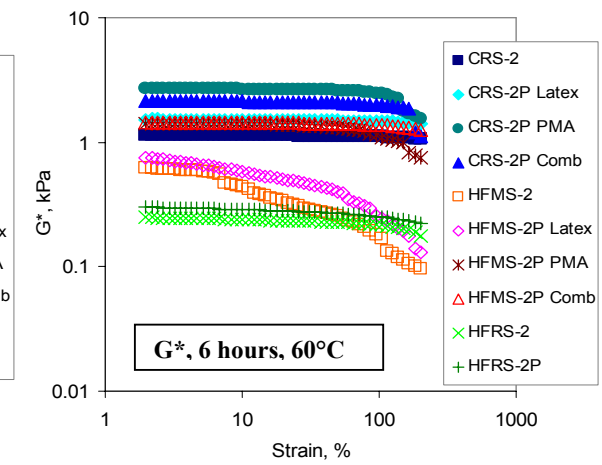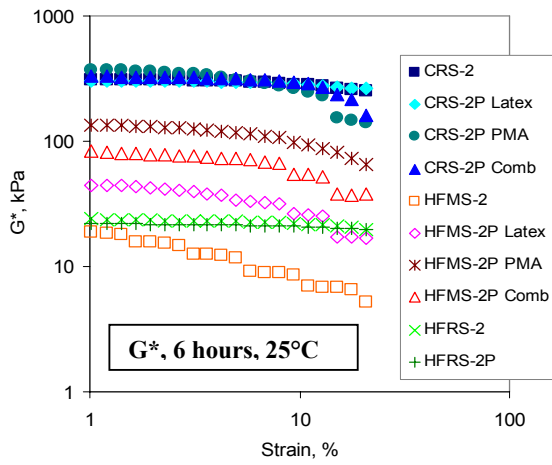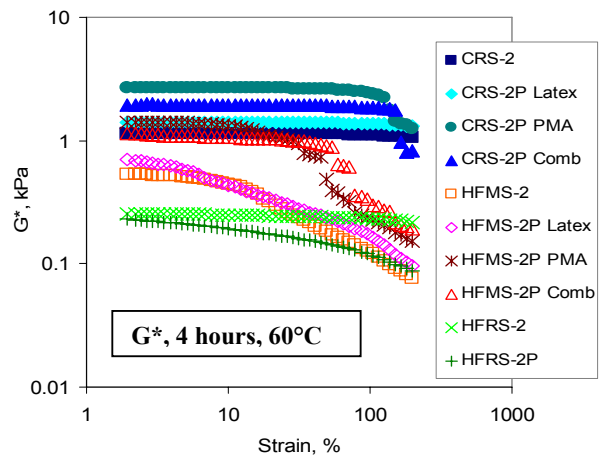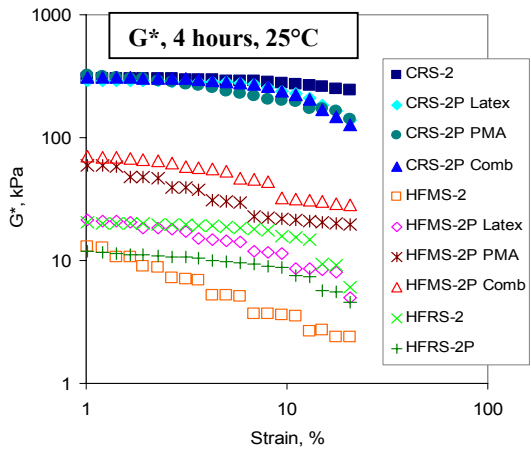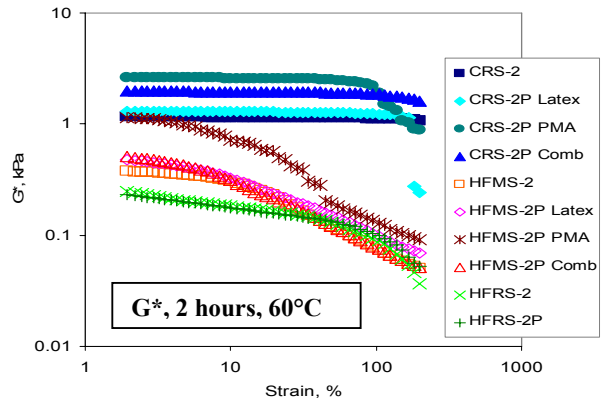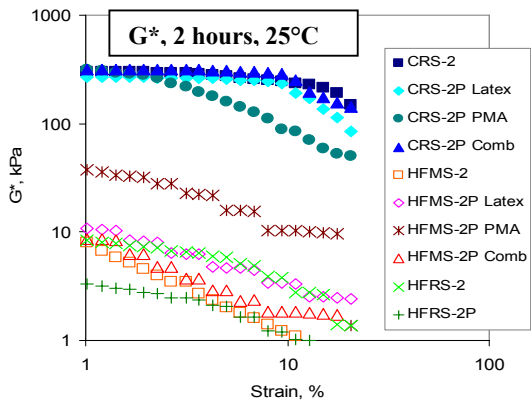
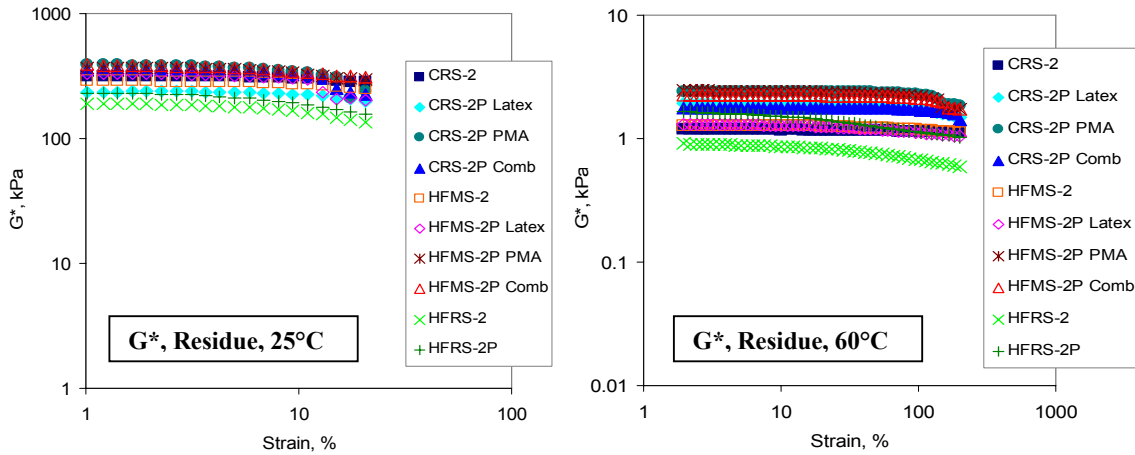### 3.2   Analysis of the Rheological Test Data

Experience shows that the majority of aggregates that get dislodged from a chip seal application at early stages suffer cohesive failure. 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 (8). 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 (9).

Figures 2 to 11 show the complex shear modulus G* versus strain, tested at 25 and 60°C, at each of the curing times, plus those of the distillation residues. 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. 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%. During the first 24 hours, moduli for the anionics are significantly lower.

Under the described curing conditions, the studied CRS emulsions gain almost their entire modulus value within the first 24 hours, while the HFMS emulsions gain about half or less. 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.

Figure 2-11: Strain sweep data, different curing times, 25 & 60°C test temperature

G*, 2 hours, 25°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 2 hours, 60°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 4 hours, 25°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 4 hours, 60°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 6 hours, 25°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 6 hours, 60°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 24 hours, 25°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

G*, 24 hours, 60°C

Legend: CRS-2, CRS-2P Latex, CRS-2P PMA, CRS-2P Comb, HFMS-2, HFMS-2P Latex, HFMS-2P PMA, HFMS-2P Comb, HFRS-2, HFRS-2P

G*, kPa — Strain, %

Strain dependency is more pronounced during early stages of curing and the cationic emulsions seem to have more strain tolerance than the others. Some data shows gradual drop in the modulus with increased strain while other shows abrupt discontinuities. These drops are most likely caused by catastrophic structural failure of the test sample.

3.3   Analysis of the Sweep Test Data

Our experience with sweep tests on slower set 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 as 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 two parameters:

Sweep Loss (SL), as described by ASTM D 7000, is calculated as % mass loss of the specimen during the sweep test

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.
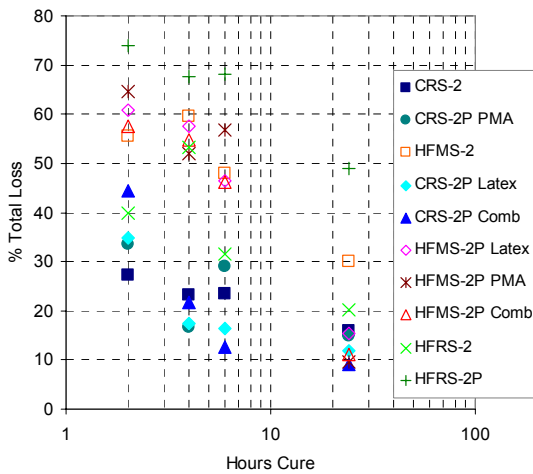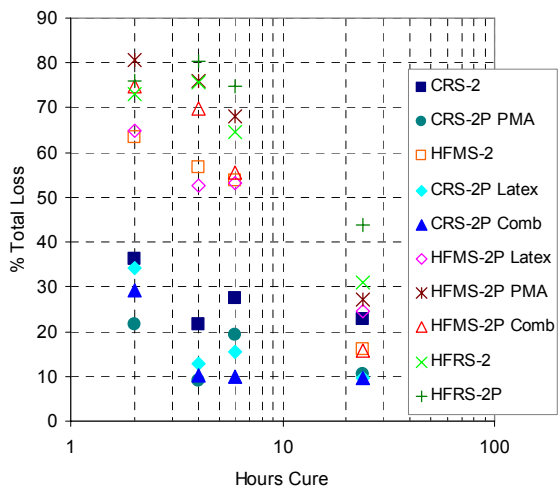

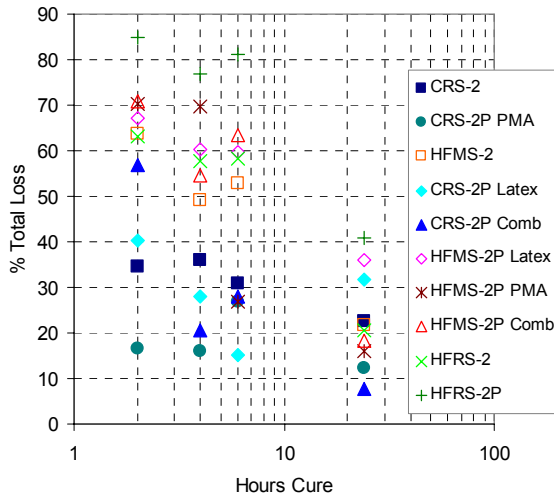Figure 12: Percent Total Loss, Limestone

Figure 13: Percent Total Loss, Granite

Figure 14: Percent Total Loss, Traprock

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.

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. 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, 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 aggregates.

3.4 Analysis of the Mechanical Adhesion by Vialit Test Data
Table 3. Adhesivity at 24 hours cure by Vialit test

Adhesivity, %	Limestone	Granite	Traprock
CRS-2	100	100	100
CRS-2P Latex	100	100	100
CRS-2P PMA	93	100	100
CRS-2P Comb	100	100	100
HFMS-2	100	100	100
HFMS-2P Latex	100	100	100
HFMS-2P PMA	100	100	100
HFMS-2P Comb	100	100	100
HFRS-2	95	100	100
HFRS-2P	94	100	100

All 10 emulsions showed 100% adhesivity to the granite and the traprock chips, at 24 hours. With the limestone chip, the two HFRS and the CRS-2P PMA show some degree of failure. This is most likely as a result of the dust film on the chips, which some emulsions can't penetrate entirely. The HFMS emulsions contain 1% solvent, which helps wet the dust. Also, the latex modified emulsions have shown better wetting capability for dusty aggregate.

4 CONCLUSIONS

The cationic emulsions studied developed cohesion and modulus quicker than the anionic emulsions under similar curing conditions.

The type of polymer modification of the emulsion impacts the film strength development in the very early stages of curing.

The strain tolerance of the emulsion residues increases with curing. The cationic emulsions in our study have shown less strain dependency during early stages.

The cationic emulsions have performed consistently better in sweep tests and they have also shown less sensitivity towards different aggregate chemistry.

Heavy dust layers on chips' surface impact mechanical adhesion binder-aggregate to a higher degree than chemical compatibility emulsion-aggregate. Emulsions containing solvents or latex will penetrate dust films easier while those having no solvents or are produced from PMA only might have difficulty wetting dusty chips.

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