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Refining Particle Size Specification for Asphalt Emulsion

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Refining Particle Size Specification for Asphalt Emulsion

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ABSTRACT

Pavements begin to deteriorate as soon as they are constructed, and traffic is released. The rate at which a pavement deteriorates depends on the material quality, the environment, and the loads carried. To properly preserve pavements and extend their life, various pavement preservation treatments are used. For flexible pavements, a considerable number of these treatments use asphalt emulsion (which are refer so simply emulsions from here on). Emulsions are classified based on their particle charge and reactivity. Emulsions used in chip seals are usually of rapid set reactivity and are classified based on tests over fifty years old.

It has been shown that particle size has an influence in rheology and stability of emulsions. Preliminary particle size specifications have been developed based on only Cationic Slow Set (CSS) and Cationic Medium Set (CMS) emulsions. Therefore, there is a need for a particle size standard specification for asphalt emulsions that encompass Cationic Rapid Set (CRS) with additives such as solid polymer and latex polymer.

This research links particle size metrics such as mean, D10 (particle size at which 10% is smaller than), D50, D90 and span, to an existing performance test – the sweep test- for chip seals (CRS-2, CRS-2P and CRS-2L asphalt emulsions). In addition to this performance test, it has been shown that the particle size distribution correlates to viscosity, which is one of the few rheological tests currently run on asphalt emulsions. In this research, the moderate correlation between storage time, viscosity and particle size led to develop a specification for CRS-2, CRS-2L and CRS-2P. Based on this analysis, a mean particle size of 1.9 μm for CRS-2, 3.0 μm for CRS-2L and 10.7 μm for CRS-2P are recommended.

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I dedicate all my work to my dad, Jose Luis Diaz Romero (†). Thank you for your love and support, you will always be my motivation.

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INTRODUCTION

Asphalt emulsions have been around for almost 100 years. In fact, in 1922 the first patent filed by Hugh Alan Mackay is regarded as the start of emulsion technology (MacKay 1922), during this time only anionic emulsions were produced. In 1951, cationic emulsions became available in the market and by 1980 cationic emulsifiers dominated in asphalt emulsions because of their breaking behavior. This led into much shorter return to traffic as well as better performance (Redelius and Walter 2005). In the mid-1980s, after the development of polymer modified binders, different processes of emulsions became available. The residues of these modified asphalt emulsions had superior properties compared to unmodified asphalt binder (Hunter et al 2015). Polymer modified emulsions improved aggregate retention and advanced bleeding and rutting resistance (Kim 2009). By 2018, up to 3 million tons of emulsions were produced in the United States which accounted for 5% to 10% of the total asphalt binder consumption (Buss 2018).

Asphalt emulsions are used in different pavement preservation and rehabilitation treatments. Surface application treatments are, by far, the largest use of asphalt emulsions. With pavement preservation, highway agencies gain the ability to improve pavement conditions and extend pavement life without increasing expenditures (Galehouse 2003). There are many treatments for pavement preservation, each with different applications and performance. Asphalt emulsions are used in chip seals as a spraying application. Chip seals are the most common surface treatment for pavement preservation, they cover more than 139,700 miles of road surface in the Unites States. If the best practices are followed, chip seals can extend the life of a pavement by 7 years on average (Gransberg 2005). Chip seals are implemented to renew aged pavements, improve skid resistance, seal the pavement, offer a quick return to traffic and provide lane demarcation. Current research moves towards the development of performance related material specification for emulsified asphalt binder use in chip seals.

BACKGROUND

Currently, there are a variety of tests for asphalt emulsion quality control/quality assurance (QC/QA) and for research purposes. They all fall into four categories: handling properties, classification, residue properties and performance tests with aggregate (Braham 2020). There are many fields for improvement when it comes to quality control tests for asphalt emulsions. One of them is finding improved ways of measuring viscosity, stability, breaking and adhesion all which relate to surface dressing requirements (Hunter 2005). Research has shown that there is an interrelationship between particle size and the manufacturing temperature, the amount of emulsifier present, the mill gap and rotor speed, the thixotropic agents, the aqueous phase composition, asphalt binder content and asphalt binder type . Therefore, the metrics from a particle size analysis are variables to consider. Particle size can be controlled during formulation, as well as with quality of materials and equipment used for the manufacturing of asphalt emulsion.

Generally, there are five types of asphalt emulsion stability: chemical, storage, freezing, mechanical and mixing (Redelius and Walter 2005). Storage stability is affected by the asphalt density, the asphalt binder content, freezing and particle size. Sedimentation occurs during storage because of the smaller particles being able to agglomerate the large particles under the effect of Brownian motion (Liu 2017), therefore; particle size can also give an indicator about the emulsion stability. Because of sedimentation, over time the particles of asphalt emulsion will increase making them easier to coalesce and cause emulsion to break (Buss 2018, Ronald and Luis 2016).

Having an increase in the particle size would deteriorate the stability of the emulsion and interfere with the spray application as the asphalt emulsion passes through the spray nozzles (Kiihnl 2021). On mix applications, an unstable asphalt emulsion would decrease the workability

of the mix. A less workable mix would indicate higher energy consumption during construction and a poor performance of the material over time.

Much of the research done has shown the correlation between particle size to rheology, in particular to viscosity. Many of these studies include a recommended value for particle size. For instance; a Cationic Slow Set Low Viscosity (CSS-1) and a Cationic Medium Set High Viscosity (CMS-2) asphalt emulsions were recommended to have a median size of 4.4 μm and 6.9 μm respectively (Kiinhl 2020). In another study, a mean particle size of 11μm for CRS-2 and two mean sizes for CRS-2P of 3 μm and 11 μm were analyzed (Buss 2018). Moreover, in another study a CRS-2L was recommended to have a median size of 9.3 μm for CRS-2L and 5.27 μm for Cationic Quick-Setting Polymer Modified Low Viscosity (CQS-1HP) (Ragheb 2019). This study correlates particle size metrics to rheology while inducing performance with aggregate to particle size metrics of cationic rapid set asphalt emulsions that are widely used in chip seals.

Research Objectives

Quality control and quality assurance are activities and techniques that ensure the quality objectives are being met. These activities are essential for the optimization and improvement on the performance of the materials used. The objectives of this research are:

- Link quality control tests and performance tests for asphalt emulsion to particle size analysis.
- Revise a draft specification for particle size analysis of asphalt emulsions using a laser diffraction technique.

MATERIALS AND METHODS

In this study, three cationic rapid set asphalt emulsions were collected from two asphalt emulsion plants located in Oklahoma and Arkansas. It was intended to collect asphalt emulsions that are widely used in surface treatments, specifically those used in chip seals. Two emulsions were first collected at the beginning of this study: a Cationic Rapid Set with High Viscosity (CRS-2) and Cationic Rapid Set High Viscosity with Latex Polymer (CRS-2L). Then, a Cationic Rapid Set High Viscosity with Solid Polymer (CRS-2P) was collected. These types of asphalt emulsions were selected based on the availability and supply of the asphalt emulsion manufacturing facilities near the Northwest Arkansas region. After collection, all three emulsions were stored in a forced draft oven for 180 days at 122°F, or 50°C. Testing on all three emulsions began the day after emulsions were collected, this is referred to as Day 1.

The three emulsions were evaluated on Day 2, Day 5, Day 7, Day 14, Day 21 and approximately every 21 days until the 180th day mark was reached. Due to unforeseen circumstances, CRS-2P testing lasted until Day 189. Four asphalt emulsion tests and one performance test with aggregate were performed.

A typical testing day started with the oversized particles test commonly known as the “sieve test” following ASTM D6933. Since the asphalt emulsions were previously stored in a forced draft oven at 50°C, there was no need to condition the emulsion to the recommended temperature. The container was not vented prior to testing because of the risk of the emulsion breaking with the surface area being exposed to heat specially if rapid set type emulsions are being tested. Before testing, proper homogeneity was accomplished by stirring the container with asphalt emulsion using a glass rod. Because of the amount of asphalt emulsion needed for the sieve test (around 800 grams of emulsion per sample), the sieve test was only conducted on 10 testing days, on intervals of 18 days ± 5 days. Three replicates were performed on each testing day.

On the days that the sieve test was conducted, the emulsion poured through the No 20 sieve was used to measure viscosity following ASTM D7226 using a Cannon Digital Paddle Viscometer. This allows for an effective use on the amount of material needed for such tests. Because of how effective and easy to test viscosity using a rotational paddle viscometer is, the viscosity was measured on the proposed intervals during the proposed 18 testing days. Approximately 50 grams of emulsions is poured in a cup at a testing temperature of 50°C. Three replicates per testing day were measured.

The third emulsion test was the residue test. This test evaluates the amount of asphalt binder present in the emulsion. The residue test was only conducted 4 times through the 180 days testing period for CRS-2 and CRS-2L and three times for CRS-2P. The hypothesis that there would be no change on the amount of the residue present in the emulsion was met; therefore, this test was not performed on every testing day. The residue on asphalt emulsions should not change over time unless an unrepresentative sample was collected during sampling. Results for the residue test are shown in Table 1. The residue met the standard value on ASTM D2397 for CRS-2 and AASHTO M 316 for CRS-2L and CRS-2P.

Table 1. Residue by Evaporation

	CRS-2		CRS-2L		CRS-2P		
Day	Avg	Std	Avg	Std	Day	Avg	Std
1	71.5	3.2	67.2	1.0	1	66.9	2.9
33	70.3	1.3	68.7	0.2	34	65.9	2.5
69	72.1	0.9	68.5	0.5	111	65.6	2.4
145	71.7	1.1	68.0	0.9			
Avg	71.4		68.1		Avg	66.1	
Std	1.6		0.7		Std	2.6	

The fourth emulsion test performed was a particle size analysis using a Horiba LA 350 AP laser diffraction analyzer. Currently there is no a known standard for a particle size analysis

(PSA) on asphalt emulsion, however; analysis and recommendation from experts in the field and literature explored on PSA for asphalt emulsions were followed. No sample preparation was needed for the PSA because of the emulsions being already in liquid form. Approximately a droplet of asphalt emulsion was poured in the machine using a 1.5 ml pipet, samples were taken approximately an inch from the surface of the container. Distilled water was used as a dispersion medium, a refractive index of 1.63 was used for all three emulsions and a dispersion index of 1.33 was used for distilled water. Potential circulation speed on the equipment ranged from 1-15, for this study and because the asphalt particles are subject to energy being induced because of the circulation speed, circulation was kept to a level of 2. Ultrasound could be used to disperse particle to a single particle state prior measurement. Because this induced another variable to our research, and the length of time and intensity for the use of ultrasound was not clear, ultrasound was not used during measurement. Before each measurement, the laser was aligned to maximize signal to noise and a blank sample was acquired to reduce noise. The system flushed out the dispersion medium every time a measurement was completed, 3 runs per sample were taken and 3 samples were collected on each testing day for a total of nine sets of data points. For a draft specification for particle size analysis using a laser diffraction analyzer refer to the appendix of this thesis.

Finally, ASTM D7000 commonly known as the Sweep Test, is a laboratory method for evaluation of the performance of a chip seal in terms of aggregate loss and was suggested by the NCHRP report 680 (Kutay et al 2017). This test evaluates differences in adhesive properties of different asphalt emulsions with a single aggregate type. The sweep test was run on the proposed intervals and proposed testing period. The aggregate used for this test was a No 4 limestone from a quarry located in Springdale, AR with a specific gravity of 2.563. About 98% of the aggregate was retained in a 6.3 mm (1/4”) sieve and 69.6% was retained in the 4.75 mm (No 4) sieve, resulting in 750 grams of aggregate to be used for each sample. 300 mm diameter

roofing paper disks were cut and placed directly above a scale so that the proper amount of emulsion is poured. The testing temperature was 50°C and 88 grams of emulsion were poured. Following the application of aggregate, samples were manually compacted with a metal compactor with a curved surface radius of 500 mm and a weight of 7700 grams. After sample fabrication, samples cured in a forced draft oven for 2 hours at 35°C. Following the curing stage, the sample was turned vertically so that any loose aggregate is removed. The specimen was placed in a Hobart mixer and the brooming application began using a nylon strip brush attached to the mixer for one minute. The retention performance is studied by measuring the weight of the sample before and after the test.

RESULTS & DISCUSSION

The goal of this research is to develop a specification for particle size of CRS-2, CRS-2L and CRS-2P asphalt emulsions. The tests conducted aimed to explore the relation between a particle size analysis to quality and performance.

Results for CRS-2 are shown in Table 2 and the linear Pearson R values are shown in Table 3. There are several interesting trends that are apparent in Table 2, such as an increase in viscosity and in particle size, a decrease of the adhesion of the aggregate to the emulsions over time, and a disconnect between the sieve test and the particle size analyzer results.

While there is a correlation between the change in particle size and the increase in viscosity, such behavior could relate to a change in the level of internal water rather than fluctuations in particle size as presented by Furlong and James (1998). During sedimentation, particles start to flocculate due to the Oswald ripening that consumes the smallest particles which coalesce into large particles (Redelius and Walter 2005). This leads to an increase in volume of water trapped between particles. Therefore, an increase in the level of internal water and a decrease in the level of external water could be reason why there is a tremendous increase in viscosity but not a significant increase in particle size. This is not to say that particle

size does not influence viscosity. In fact, there is a moderate correlation between particle size and viscosity, as shown in Table 3. However, the significant increase in viscosity could be explained by the changes in internal water, especially when a higher binder content (70% for CRS-2) is present in the emulsion.

On one hand, there is a moderate correlation between the mass retained from the sweep test and the storage time. This led to believe that the sweep test could be a test to be performed for quality control especially if emulsions are meant to be stored for longer periods of time. On the other hand, the decrease in the adhesion of the aggregate to the emulsion could be related to the mobility of the particles towards the surface of the aggregate, smaller particles tend to move faster than larger particles (Engman et al. 1998).

Another takeaway from Table 2 is the disconnection between the oversized particle test and the particle size. This is because of the emulsion breaking as the test is run. In Section 7.3 of ASTM D6933, the test dictates to use distilled water through the sieve until clear water washes through. This provokes the breaking of the emulsion and agglomeration of the asphalt particles as cooled water is being poured on top the emulsion and through the sieve. Unfortunately, the standard does not imply water to be heated to a specific degree, so such a coalescence can happen due to a change in temperature. The breaking of the emulsion during the procedure could also be related to some sort of reaction between the sieve fabric material - in this case metal - and the emulsion, especially if rapid set emulsions are being tested.

This test is meant to quantify the volume of particles that are larger than the No 20 sieve (850 µm), which is a size that the particle size analyzer is rated to capture. We do not see a strong correlation between the particle size and the oversized particle test because of the properties of the emulsion during such tests. The oversized particle test is representing particles that have coalesced while particles during a particle size analysis may or may not experience coalescence. It is also believed that during the PSA, there is some sort of kinetic energy being

induced in the sample as it circulates through the machine. This prevents the agglomeration of particles during the particle size analysis and the reason why a low circulation speed is recommended (Horiba 2019). Many specifications around the nation specify a content of oversized particles greater than 150 or 800 microns determine by screening (Engnam 1998). Unfortunately, this test does not represent the suspended particles on the aqueous phase of the emulsion, which in theory, should be the driving force for quality and performance.

Table 2. Summary Results for CRS-2.

	Viscosity (cP)		Sweep Test Mass Retained (%)		Sieve Test (%)		Median/D50 (µm)		Mean (µm)		Mode (µm)		D10 (µm)		D90 (µm)		Span	
Day	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std
1	516.7	20.0	72.25	4.0	0.20%	0.02%	1.65	0.0	1.76	0.0	1.61	0.0	0.82	0.0	2.53	0.1	0.84	0.0
3*	664.2	11.4	75.36	6.8	-	-	1.25	0.3	1.33	0.4	1.25	0.3	0.78	0.4	1.96	0.4	0.99	0.2
6*	568.3	52.2	71.06	3.6	-	-	1.33	0.3	1.42	0.3	1.31	0.2	0.81	0.4	2.11	0.3	1.01	0.3
8*	625.7	36.9	75.50	4.2	-	-	3.10	0.3	5.44	2.3	2.70	0.5	1.64	0.2	6.78	0.4	1.68	0.3
10	654.0	41.9	70.11	1.9	0.19%	0.07%	1.83	1.1	2.18	1.4	1.64	0.7	0.85	0.6	3.94	2.7	1.66	0.2
14*	1022.0	7.8	72.02	8.3	-	-	1.96	0.2	1.96	2.1	1.86	0.2	1.26	0.1	3.50	0.5	1.14	0.2
17*	859.1	16.0	66.62	4.4	-	-	1.80	0.1	1.98	0.2	1.76	0.1	1.19	0.0	2.99	0.5	0.99	0.2
21*	875.7	14.3	70.62	4.4	-	-	1.95	0.4	2.10	0.4	1.81	0.4	1.29	0.2	3.12	0.7	0.93	0.1
29	1234.0	45.0	71.11	3.2	0.19%	0.06%	1.77	0.1	3.24	0.1	1.76	0.1	1.19	0.1	2.90	0.4	0.96	0.1
35*	1469.7	103.4	72.61	9.3	-	-	1.77	0.1	1.94	0.1	1.63	0.0	1.17	0.0	2.92	0.3	0.99	0.1
42	1298.7	69.3	61.28	7.2	0.25%	0.13%	1.62	0.1	1.75	0.2	1.49	0.1	1.09	0.1	2.57	0.3	0.91	0.1
56*	1234.0	84.9	66.74	2.9	-	-	1.87	0.3	2.04	0.4	1.78	0.3	1.24	0.2	3.05	0.7	0.96	0.1
69	1000.4	57.6	73.03	1.2	0.25%	0.06%	1.47	0.4	1.59	0.4	1.44	0.3	0.90	0.4	2.42	0.6	1.06	0.1
84	1023.3	25.6	67.49	2.9	0.23%	0.01%	1.56	0.4	1.68	0.5	1.63	0.4	0.93	0.5	2.57	0.5	1.11	0.3
104	1115.2	169.4	70.44	0.8	0.24%	0.10%	1.80	0.0	2.06	0.1	1.62	0.0	1.13	0.0	3.32	0.2	1.22	0.1
126	1214.3	76.2	69.20	2.4	0.26%	0.02%	1.57	0.2	1.72	0.2	1.54	0.1	1.00	0.2	2.64	0.2	1.06	0.2
147	1422.0	105.9	63.95	6.2	0.19%	0.10%	2.58	0.5	2.88	0.5	2.47	0.5	1.54	0.3	4.64	0.8	1.21	0.0
180	1902.0	361.6	63.79	5.0	0.19%	0.01%	2.57	0.2	3.11	0.2	2.23	0.3	1.42	0.1	5.56	0.1	1.62	0.2

* Note: Due to the emulsion amount needed for the sieve test, the test was only conducted on 10 testing days.

Table 3. Pearson Linear Correlation Values (R) for CRS-2

	Viscosity	Storage Time	Sweep Test Mass Retained	Sieve Test	Median	Mean	Mode	D10	D90	Span
Viscosity	1.0	0.764	-0.567	-0.111	0.659	0.624	0.614	0.642	0.626	0.298
Storage Time	0.764	1.0	-0.542	0.020	0.608	0.472	0.595	0.467	0.632	0.464

The second emulsion explored in this research is a CRS-2L - another asphalt emulsion widely used in surface applications. Results for CRS-2L are shown in Table 4 and the linear Pearson R correlation values are shown in Table 5.

In practice, most of the emulsions produced for chip seals in North America are SB and SBS block copolymers and SBR latex (Lubber et al. 2007). SBR latex is typically batched with the asphalt emulsion soap. Latex in asphalt emulsion is uniformly dispersed but when the emulsion breaks, droplets containing latex coalesce along the surface of asphalt binder particles, forming the so-called honeycombed polymer network. This network welds the asphalt binder particle resulting in a higher tensile strength, stone retention for chip seals and resistance to cracking (Takamura 2003).

Interesting trends are apparent in Table 4. First, there is a poor-to-moderate correlation between particle size and viscosity, indicating that the viscosity does increase as well as particle size over time. Second, a much better but not significant adhesion is experienced with CRS-2L than CRS-2. Third, just like with CRS-2, an apparent disconnection is seen between the oversized particle test and the particles measured with a particle size analyzer.

The poor-to-moderate correlation between the particle size and viscosity as seen in Table 5, could be explained by the work done by Greenwood et al (1997). Their research showed that the ratio of the diameter between the small particles and large particles of latex has an effect on viscosity. The particle packing theory is very simple: the small particles just fit into the interstitial volume between the larger ones, thus occupying a maximum volume while offering a minimum amount of surface contact between the particles consequently slowly increasing the viscosity of the emulsion. This is one of the advantages that latex has on typical industrial products, the macroscopy viscosity remains low for solid contents up to 55% (Guyot et al 2002). Studies not related to asphalt emulsions show that a concentration of solids - around

44% - and the ratio between the diameter of small and large particles of latex help achieving a minimum viscosity to pass specifications (Kemmere et al, 1998).

As seen in Table 5, there is a good correlation between the mass retained from the sweep test and storage time. The adhesion of the aggregate to the emulsion decreases as storage time increases showing promise that the sweep test could be implemented for quality control especially if storage stability is a variable to consider.

Scholten (2006) showed that the mean particle size of SBR latex is around 60 nm, to put this in context, the diameter of latex particle is as much as 1000 times smaller than asphalt binder droplets. In the case of latex, latex particles surround asphalt binder particles, this could be the reason why the particle size is larger than CRS-2 particles but due to the latex particles being nanoparticles the difference in particle size is not significant.

Important to note and as seen later in Figure 4, there is an even distribution on particles indicating a good compatability between latex and asphalt binder. This shows that the particle size analysis can also be a tool to analyze the compatability between latex and asphalt binder.

As seen with CRS-2, there is a disconnection between the oversized particle test and the particle size analysis. The sieve percentage is lower for CRS-2L than CRS-2. As mentioned before, the viscosity plays a significant role in the procedure of this test. Because of the viscosity being lower than the viscosity for CRS-2, the emulsion is capable of flowing through the sieve with ease and the testing time is a lot quicker not allowing the emulsion to cool and break. Important to note that after taking the sieves of the oven a thick film of latex is present on the sieve. As emulsion breaks, the latex leaves a latex lattice behind, these are the so-called honeycombed polymer bonds. This reaction is meant to happen. However, as mentioned before, this reaction only takes place once the emulsion breaks; therefore, the particles in the sieve are particles that have reacted with water and followed evaporation after the completion of

the test. Unfortunately, this is not considered on the limits for the oversized particles test, the latex film formed due to the breaking of the emulsion contributes to the weight on the sieve.

Table 4. Summary Results for CRS-2L

	Viscosity (cP)		Sweep Test Mass Retained (%)		Sieve Test (%)		Median/D50 (µm)		Mean (µm)		Mode (µm)		D10 (µm)		D90 (µm)		Span	
Day	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std
1	516.8	20.2	76.05	3.9	0.07%	0.02%	2.13	0.4	2.31	0.5	1.99	0.2	1.36	0.1	3.51	1.1	1.06	0.2
3*	618.2	2.7	77.82	3.1	-	-	1.93	1.5	2.10	1.7	2.06	1.6	1.02	0.9	3.38	2.7	1.22	0.0
6*	565.7	21.2	71.25	2.4	-	-	3.23	0.0	3.60	0.0	3.64	0.0	1.63	0.0	5.14	1.7	1.09	0.5
8*	604.2	25.0	72.41	2.8	-	-	4.13	0.4	4.54	0.4	4.58	0.4	2.01	0.1	7.60	0.8	1.35	0.0
10	614.2	15.1	71.50	4.9	0.11%	0.03%	4.45	0.4	6.83	3.6	5.01	0.4	2.06	0.2	8.31	0.8	1.41	0.1
14*	677.5	44.1	70.74	2.3	-	-	3.14	0.8	3.14	1.4	3.02	1.0	1.75	0.4	5.60	1.1	1.24	0.2
17*	650.8	11.4	66.05	3.5	-	-	2.86	0.2	3.17	0.2	2.80	0.4	1.59	0.0	5.18	0.5	1.25	0.1
21*	675.5	36.1	73.26	3.6	-	-	2.64	0.5	2.64	0.5	2.58	0.6	1.52	0.1	4.69	0.9	1.19	0.1
29	822.6	5.4	66.80	3.2	0.11%	0.01%	2.43	0.2	2.43	0.2	2.32	0.2	1.46	0.1	4.24	0.4	1.14	0.1
35*	333.7	5.3	72.64	5.5	-	-	2.78	0.2	3.08	0.3	2.68	0.4	1.61	0.1	4.96	0.5	1.20	0.0
42	370.0	27.7	71.06	3.2	0.17%	0.02%	2.64	0.1	2.91	0.1	2.55	0.2	1.58	0.1	4.60	0.2	1.14	0.0
56*	349.8	50.8	67.97	6.2	-	-	3.22	0.6	3.51	0.6	3.38	0.7	1.76	0.3	5.65	0.8	1.21	0.0
69	407.0	19.1	72.10	4.4	0.14%	0.08%	2.61	0.2	3.36	0.3	3.41	0.3	1.56	0.4	5.35	0.5	1.48	0.4
84	345.3	8.3	67.80	3.8	0.09%	0.04%	2.45	0.1	2.72	0.1	2.33	0.2	1.50	0.0	4.30	0.2	1.14	0.1
104	442.0	33.9	66.62	2.8	0.13%	0.10%	2.56	0.1	2.83	0.1	2.43	0.0	1.55	0.0	4.46	0.1	1.14	0.0
126	554.4	16.9	63.06	3.5	0.13%	0.06%	3.28	0.1	3.60	0.1	3.50	0.3	1.70	0.0	5.94	0.2	1.29	0.0
147	640.1	53.2	53.92	5.7	0.15%	0.00%	4.33	0.7	4.72	0.7	5.03	0.7	2.04	0.2	7.85	1.2	1.34	0.0
180	899.9	78.3	47.77	1.7	0.16%	0.05%	5.02	0.3	5.48	0.3	5.74	0.4	2.23	0.1	9.29	0.6	1.41	0.0

* Note: Due to the emulsion amount needed for the sieve test, the test was only conducted on 10 testing days.

Table 5. Pearson Linear Correlation Values (R) for CRS-2L

	Viscosity	Storage Time	Sweep Test Mass Retained	Sieve Test	Median	Mean	Mode	D10	D90	Span
Viscosity	1.0	0.136	-0.452	0.608	0.429	0.331	0.402	0.296	0.418	0.247
Storage Time	0.136	1.0	-0.876	0.546	0.598	0.624	0.607	0.592	0.629	0.473

Chip seals may also be constructed with emulsions that contain polymer modified asphalt binder. In the state of Arkansas, styrene butadiene styrene (SBS) is the most widely used co-polymer in asphalt binder (Hossain et al. 2017). Results for CRS-2P are shown in Table 6 and the linear Pearson R correlation values are shown in Table 7.

SBS is typically added to the unmodified asphalt binder before emulsification. In this process, the polymer disperses in the bitumen and swelling of the particles occurs. In a good compatible blend, the asphalt binder contains enough oil fraction to dissolve and expand the polymer (Zielinski et al 1998). Here, the binder contains discrete smaller particles of polymer, areas of swollen polymer/maltenes phase and dark areas of asphaltenes aggregation (Forbes et al 2001). This is called a monophasic system where a single phase of polymer modified asphalt binder is present and an even distribution of asphalt droplets is present (Becker et al 2001).

There are several points of interest in Table 6. First, there is almost no correlation between particle size and viscosity. Second, a moderate correlation is shown between storage time and particle size but still very poor in comparison to CRS-2 and CRS-2L. Third, the best aggregate retention is experienced with CRS-2P when compared to CRS-2 and CRS-2L.

In an unmodified asphalt emulsion, the maltenes tend to agglomerate to the asphaltenes during sedimentation. This is because of the higher molecular weight that characterize asphaltenes over resins, aromatics and saturates. However, as seen in Table 7, the migration of smaller particles into larger particles is not apparent with CRS-2P. This could be the reason why there is a poor correlation between viscosity and storage time, the interaction at the molecular level can not be captured by the particle size analyzer. An optimal CRS-2P emulsion with thorough and homogenous dispersal of the polymer phases within the bitumen leads to improved mixture stability increasing storage life (Forbes et al. 2001), this could be the reason why the viscosity does not increase over time.

The best performance is seen with CRS-2P, and this is due to the characteristics of SBS. The elasticity and strength benefits imparted by SBS modifiers are attributable to the molecule's rubbery polybutadiene (PB) "mid-blocks" capped at either end by polystyrene end blocks which provide strength and rigidity (Airey 2004). Studies show that CRS-2P emulsions, even under different climatic conditions, perform better compared to CRS-2L and CRS-2 (Adams et al. 2018). The comparison of the performance between all three emulsions in this research can help towards the development of a performance-based standard rather than a material-based standard.

While the performance of a CRS-2P is better than CRS-2 and CRS-2L, the particle size at all metrics is larger. This implies that not always a narrower distribution and smaller particle size indicates a better performance specially is some polymer is present.

Table 6. Summary Results for CRS-2P

	Viscosity (cP)		Sweep Test Mass Retained (%)		Sieve Test (%)		Median/D50 (μm)		Mean (μm)		Mode (μm)		D10 (μm)		D90 (μm)		Span	
Day	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std	Avg	Std
1	635.8	64.6	85.13	2.1	0.04%	0.02%	6.8	0.6	9.9	1.0	8.3	1.1	2.0	0.1	21.1	2.4	2.8	0.1
4*	627.3	13.1	85.13	0.8	-	-	7.8	0.4	11.4	0.8	10.8	0.0	1.9	0.2	25.6	1.6	3.0	0.1
6*	637.4	18.3	84.66	1.6	-	-	8.3	0.2	12.2	0.6	10.3	0.8	2.1	0.2	27.1	1.1	3.0	0.1
8*	748.9	7.2	83.69	3.3	-	-	8.3	0.3	12.4	0.7	9.9	0.8	2.1	0.2	27.7	1.5	3.1	0.1
13	683.6	48.9	83.75	1.9	0.07%	0.03%	8.0	0.5	12.3	0.4	9.0	0.7	2.2	0.0	26.7	1.7	3.1	0.0
18*	605.9	29.4	84.02	2.7	-	-	7.9	0.1	7.9	0.4	9.9	0.8	2.0	0.0	27.2	1.0	3.2	0.1
22	619.1	11.3	83.74	1.8	0.04%	0.01%	7.9	0.5	11.9	1.2	10.8	0.0	1.9	0.1	27.0	2.8	3.2	0.2
27*	666.5	16.6	85.58	1.6	-	-	7.6	0.8	7.6	0.8	9.4	0.0	1.9	0.2	30.2	8.8	3.7	0.8
34*	982.9	112.5	82.03	2.8	-	-	8.0	0.2	8.0	0.2	9.9	0.8	2.1	0.2	25.8	2.6	3.0	0.3
35*	560.6	53.1	86.36	1.2	-	-	7.5	0.8	11.0	3.8	10.1	1.0	1.9	0.1	22.8	8.4	2.8	0.8
42	976.6	48.5	84.74	1.9	0.11%	0.07%	7.7	1.1	11.0	2.9	9.0	0.7	2.1	0.1	23.9	7.3	2.8	0.6
56*	562.9	42.2	82.85	1.8	-	-	8.0	0.6	11.1	1.8	9.5	1.3	2.3	0.0	23.6	4.5	2.6	0.4
70	658.9	25.8	82.09	2.1	0.06%	0.01%	8.0	0.6	11.2	1.8	9.5	1.3	2.3	0.0	23.7	4.5	2.7	0.4
84	760.9	13.3	80.90	2.4	0.04%	0.01%	8.2	0.1	11.3	0.4	8.2	0.0	2.5	0.1	25.0	1.5	2.8	0.2
105	890.0	57.9	78.53	3.7	0.05%	0.05%	8.1	0.3	12.7	0.2	9.9	0.8	1.9	0.1	29.7	0.6	3.5	0.1
133	832.9	37.5	80.93	3.0	0.08%	0.07%	6.7	1.2	9.2	2.8	8.7	1.3	1.9	0.1	19.7	7.2	2.6	0.7
162	621.5	29.7	83.12	0.3	0.03%	0.02%	6.9	1.7	10.8	5.1	7.9	1.3	2.2	0.4	20.7	10.2	2.6	0.8
183	896.5	79.4	81.45	1.9	0.08%	0.06%	8.1	1.3	11.2	3.2	9.0	0.7	2.4	0.2	24.2	8.6	2.6	0.7

* Note: Due to the emulsion amount needed for the sieve test, the test was only conducted on 10 testing days.

Table 7. Pearson Linear Correlation Values (R) for CRS-2P

	Viscosity	Storage Time	Sweep Test Mass Retained	Sieve Test	Median	Mean	Mode	D10	D90	Span
Viscosity	1.0	0.352	-0.533	0.456	0.109	-0.068	-0.178	0.116	0.065	-0.014
Storage Time	0.352	1.0	-0.644	0.537	-0.279	0.044	-0.525	0.387	-0.408	-0.460

Developing a Particle Size Specification for Cationic Rapid Set Asphalt Emulsions. During a chip seal job, viscosity is critical. It should be low enough to avoid problems with spraying applications but high enough to prevent run-off once sprayed. A typical V slot tach nozzle size diameter is about 1/16” or 1.58 mm (Etnyre 2021). If particles agglomerate during pumping and transportation, then they will interfere with the nozzles resulting in a poor performance of the material.

Because of this, viscosity is taken as the starting point to recommend particle size values for all three asphalt emulsions shown in this research. The viscosity limits found in AASHTO M 208 and ASTM D2397 are 200 cP as the lower limit and 800 cP as the upper limit. In this research, the recommended particle size is based on the upper limit of the viscosity. This is because some specifications that provide a value for particle size are expressed in the format NMT, not to be more than.

While testing was done with extreme care, it is unclear why there is a jump on particle size on Day 8 for CRS-2 and CRS-2L in Tables 2 and 4. The CRS-2 and CRS-2L emulsions were tested on the same day, so the error and jump on particle size in Day 8 could be a representation of the emulsions themselves or an error during testing. Table 8 shows the linear Pearson R values for median particle size and storage time with and without Day 8 data, for this section Day 8 is disregarded.

Table 8. Day 8 for CRS-2 and CRS-2L

CRS-2		CRS-2L	
With Day 8	Without Day 8	With Day 8	Without Day 8
0.283	0.659	0.598	0.741

As stated before, viscosity is selected as the starting point for the following analysis. Using a linear regression between storage time and rotational paddle viscosity as shown in

Figure 1, a day at which the viscosity reaches 800 (cP) was found. In the case of CRS-2, this time is equal to 25.34 days.

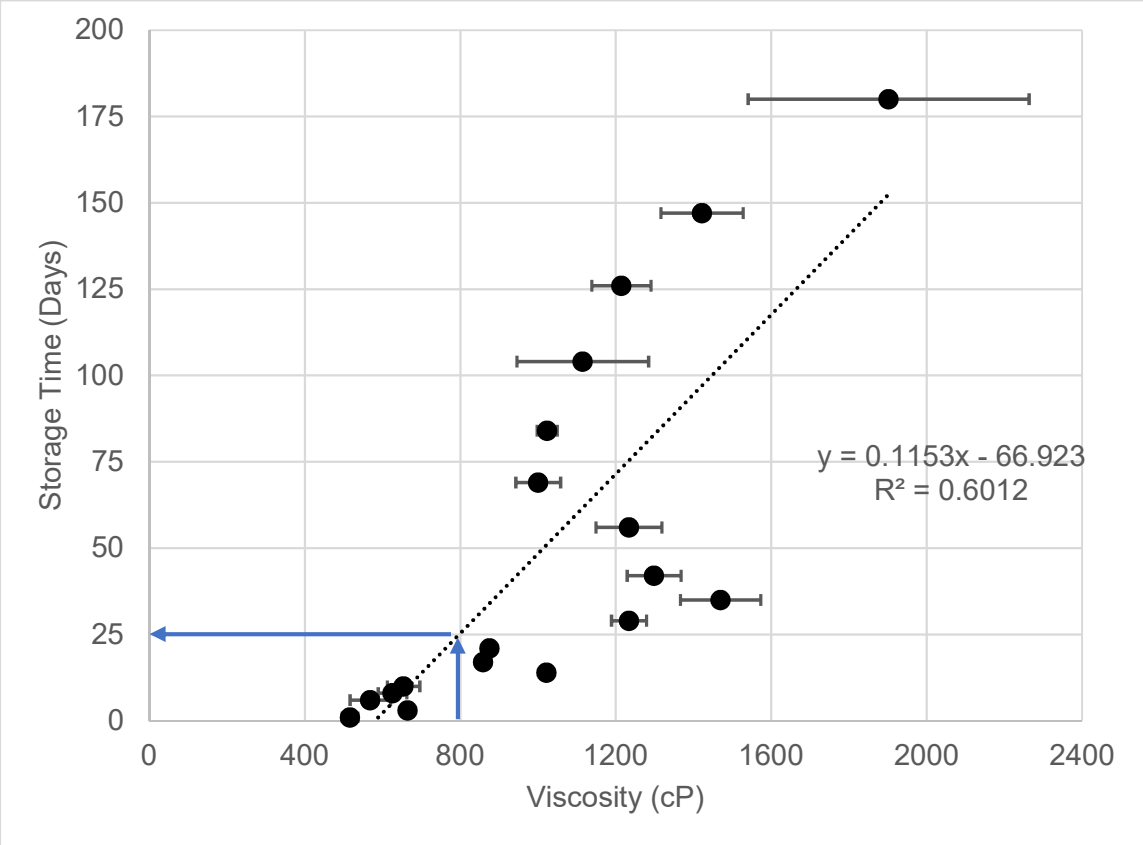

Figure 1. Storage time vs Viscosity for CRS-2

As seen before, there is a strong correlation between the storage time and the metrics from the particle size analysis. Using a linear regression analysis between storage time and each particle size metric, a value for each metric that equates 25.34 days - in the case of a CRS-2 - was found. This is the same approach that was taken for the analysis of CRS-2L and CRS-2P. The storage time found for CRS-2L is 62.2 days and 67.1 days for CRS-2P.

As seen in Figure 2, with storage time data is possible to find a value for particle size that correlates to the day where the viscosity is on the upper limit of the specifications. In the case of CRS-2, the storage time is 25.34 days and the median particle size of 1.7 μm is found. For CRS-2L, with storage time being 62.2 days the median particle size is 3.0 μm. Finally, CRS-

2P shows a storage time equal to 67.1 days and a recommended median particle size of 7.7 µm. A similar approach was conducted with the mean, mode, D10, D50, D90, span and as well as mass retained to estimate performance and are shown in Table 9.

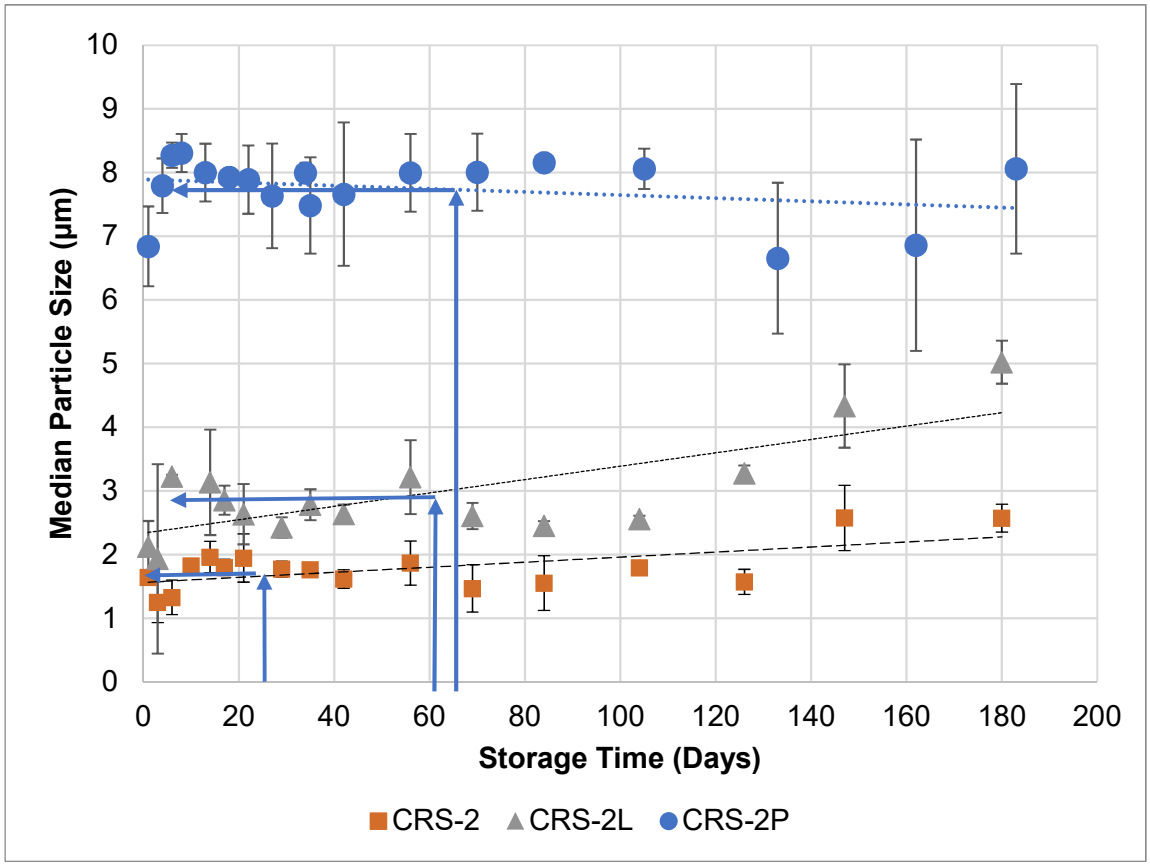

Figure 2. Median Particle Size vs Storage Time

Recommending particle size for CRS-2, CRS-2L and CRS-2P

A couple of assumptions were made for this analysis. First, it assumes that changes in viscosity are proportional to changes in storage time. Second, changes in storage time are proportional to changes in particle size. Third and most important, it is assumed that the behavior between storage time, particle size and viscosity is linear.

For a particle size specification, it is suggested to include metrics regarding the size of the particles in order to describe the width of the distribution (Horiba 2019). The D10, D50 and D90 and the particle size distribution for CRS-2, CRS-2L and CRS-2P are shown below.

In Figure 3, it is apparent the change in distribution for CRS-2. The statement that smaller particles flocculate into larger particles holds true, the D90 shows a considerable change in size, thus impacting the distribution as a whole. In Figure 4, similar trends are experienced with CRS-2L. Again, an apparent change in distribution width over time and a reduction in the volume of small particles. Just like CRS-2, small particles flocculate into larger particles. In Figure 5, no major changes are apparent for CRS-2P in terms of the distribution. However, a bimodal distribution is shown with CRS-2P. In bimodal asphalt emulsions, the small droplets tend to fit in the voids of the matrix provided by the large particles (Querol 2017). This movement lowers the sedimentation rate, this phenomenon could be the reason why there is not a significant change over time or the rheology and particle size for CRS-2P.

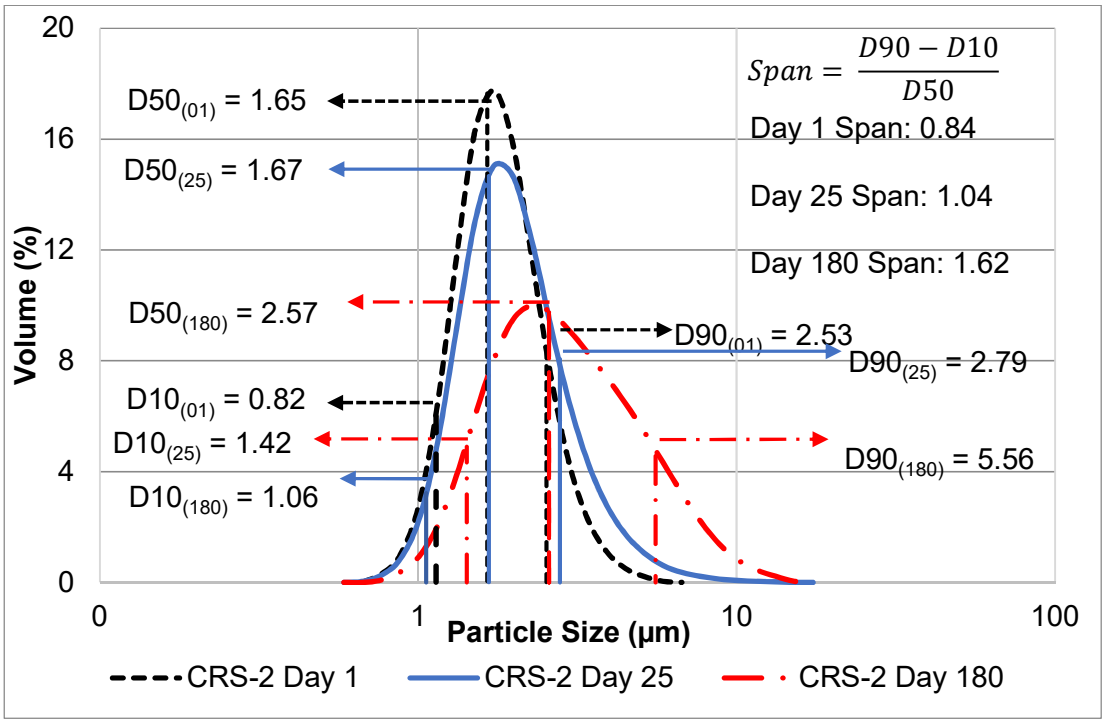

Figure 3. Particle Size Distribution for CRS-2

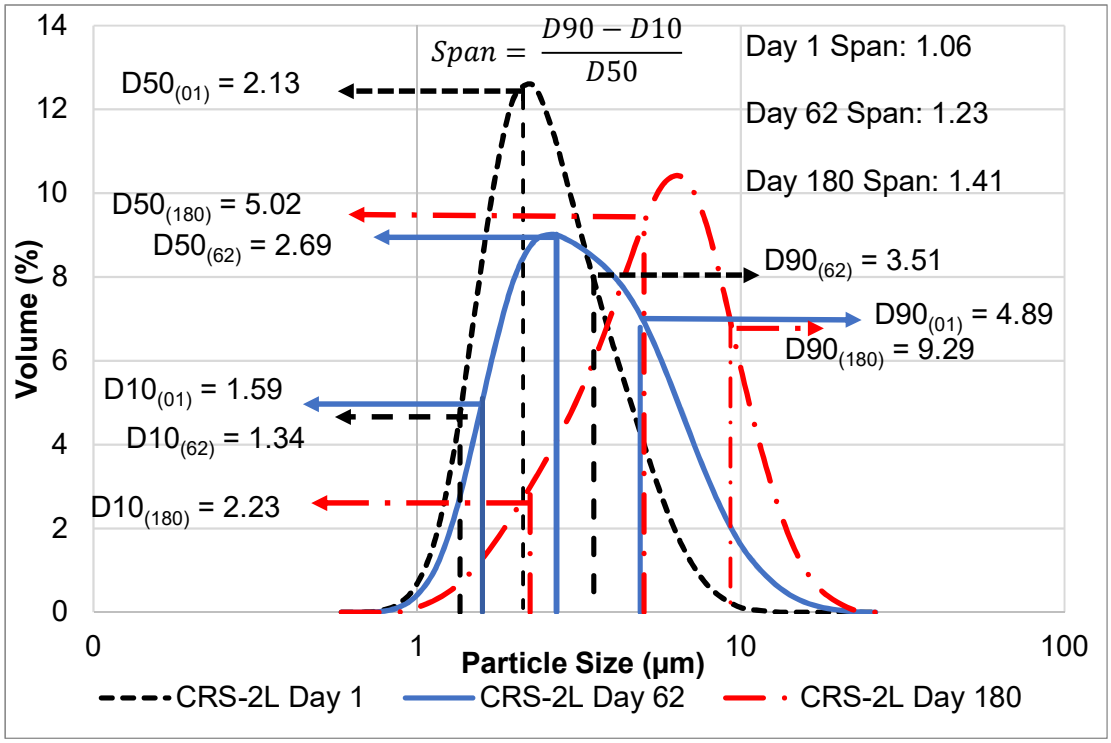

Figure 4. Particle Size Distribution for CRS-2L

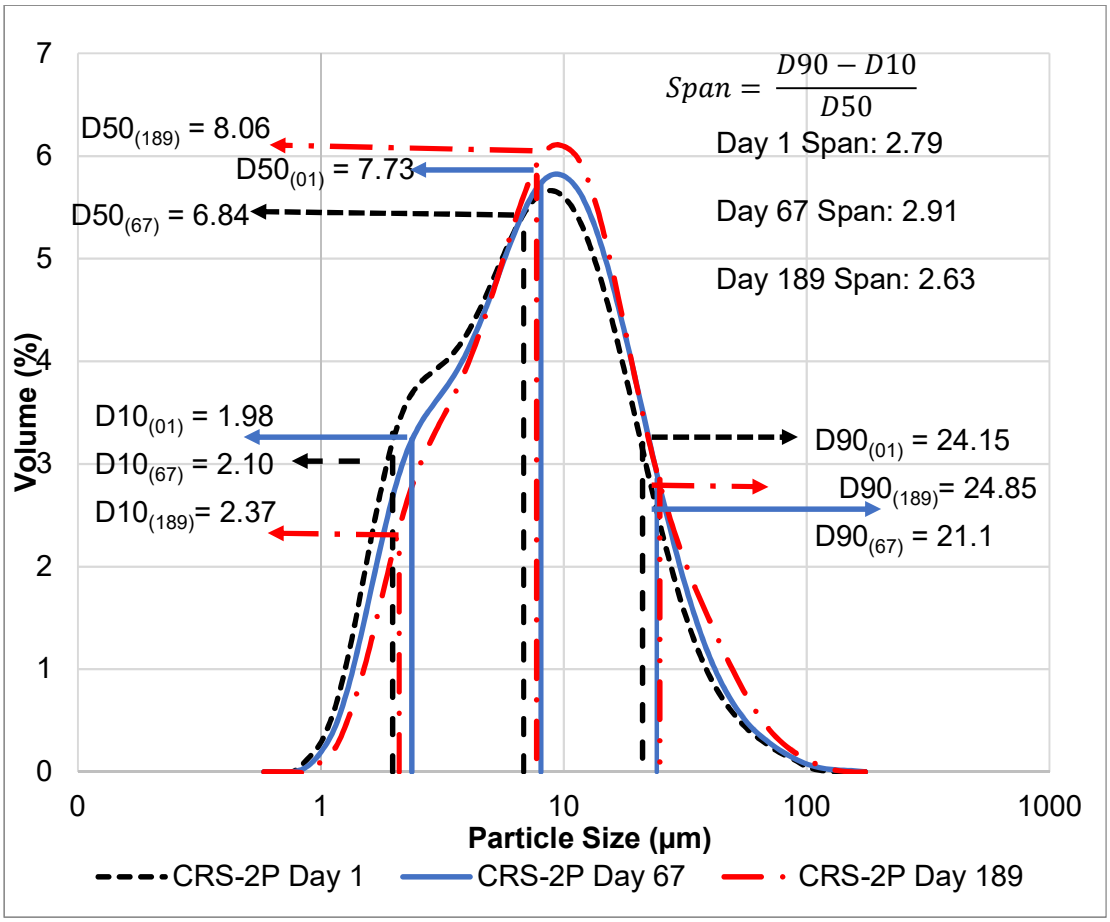

Figure 5. Particle Size Distribution for CRS-2P

Table 9. Recommended Particle Size and Mass Retention

Emulsion	CRS-2	CRS-2L	CRS-2P
Median (µm)	1.7	3.0	7.7
Mean (µm)	1.9	3.0	10.7
Mode (µm)	1.6	2.9	9.4
D10 (µm)	1.1	1.6	2.1
D50 (µm)	1.7	2.7	7.7
D90 (µm)	2.8	4.9	24.9
Span	1.0	1.2	2.9
Sweep Test Mass Retained (%)	70	67	83
Storage Time (Days)	25	62	67

Particle Size Analysis: Gaps of Knowledge

In this section, three challenges and gaps found while conducting a particle size analysis on asphalt emulsions are discussed. The first challenge faced deals with the concept of multiple scattering. The algorithm used for converting scattered light to particle size distribution assumes that light interacts with a single particle and then scatters onto the detectors. Multiple scattering occurs when light scatters off one particle and then interacts again with one or more particles before reaching the detectors. This phenomenon occurs when a high concentration of the sample being tested is poured in the machine. Out of the literature explored, no recommendations were found as to what is an appropriate concentration or if the sample of emulsion should be diluted before testing.

The second gap of knowledge faced has to do with the software used in this research. While innovative techniques are most effective and less time consuming, the software used for particle size analysis smooths the data almost immediately after the sample has been added to the machine. This creates great uncertainty due to the user not being able to distinguish the actual distribution (unsmooth) of the sample being tested. Although metrics such as the mean should not change with a different distribution basis, the distribution width may vary. This could be the reason why there is some disconnection between tests such as the oversized particle test or why we are unable to distinguish between polymer particles and asphalt particles, more research in regard to data acquisition should be explored.

The third gap of knowledge explored in this research has to do with sample preparation. Many operators tend to use a surfactant solution to overcome the problem of dispersion and agglomeration of the particles during the test. This does not only alternate the microstructure of asphalt emulsions, but it also makes it difficult to correlate the particle size to performance. The addition of surfactant will disperse the emulsion particles during the particle size analysis. Therefore, any particles that may or may not flocculate in the field would not correlate to the

asphalt emulsion being tested in the particle size analyzer. In addition, the amount of asphalt emulsions used in this test may or may not be a representative sample. The test results are based on a single drop. In this research, a surfactant was not used because it was intended to mimic the interactions of the particles over time.

CONCLUSION

The particle size of asphalt emulsions is as important as gradation is for aggregate. It does not only influence the rheological properties of asphalt emulsion, but it also drives stability. In this study, metrics from a particle size analysis were compared to quality control tests and one performance test. Three emulsions were collected from manufacturing facilities, these emulsions are widely used in surface applications, particularly chip seals.

The best correlation among all three emulsions regarding particle size and viscosity is shown with CRS-2, an unmodified asphalt emulsion. For modified emulsions with latex and solid polymer, a poor to moderate correlation between particle size and viscosity were found. Research has shown that polymers in asphalt emulsion improve the storage life and rheological parameters of asphalt emulsions.

Moreover, the sweep test shows promise to be conducted for quality control especially if storage stability is a variable to consider. Although the particle size is larger and a wider distribution is shown, the best aggregate retention is experienced with CRS-2P. Indicating that not always a small particle size and a narrower distribution is better to achieve quality and high performance on asphalt emulsions.

Furthermore, a disconnection between the oversized particle test and the particle size was experienced with all three emulsions. During the oversized particle test, all three emulsions showed particles that have coalesced during and after the test. However, based on the interrelation between particle size, viscosity, and storage time the analysis of this research

recommends a mean particle size of 1.9 μm for CRS-2, 3.0 μm for CRS-2L and 10.7 μm for CRS-2P.

During the process of this research, gaps of knowledge in terms of sample preparation and data acquisition for particle size analysis came across. Future research should focus on adequately prepare the sample without alternating their microstructures and on data acquisition.

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APPENDIX

Draft Standard Test Method for Particle Size Analysis of Asphalt Emulsion using a Laser Diffraction Analyzer.

1. Scope

    1.1. This test method performs a particle size analysis on asphalt emulsion though the analysis a light scattering properties[1].

    1.2. A precision and bias statement for this standard has not been developed at this time. Therefore, this standard should not be used for acceptance or rejection of a material or purchasing purposes.

    1.3. The values stated in SI units are to be regarded as standard.

    1.4. The text of this standard references notes and footnotes which provide explanatory material. These notes and foot notes shall not be considered as requirements of the standard.

    1.5. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, heal, and environmental practices and determine the applicability of regulatory limitations prior use.

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[1]The specification was developed using a Horiba LA 350 AP, other equipment that use a laser diffraction technique may vary in their processes.

2. Reference Documents

    2.1. ASTM Standards:

        D140/D140M: Standard Practice for Sampling Asphalt Materials

        D2397: Standard Specification for Cationic Emulsified Asphalt

2.2 AASHTO References

M 208-18 – Cationic Emulsified Asphalt

M 316-18 – Polymer-Modified Emulsified Asphalt

2.3 ISO References

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3. Terminology

3.1. Diffraction: Scattering of light around the contour of a particle, observed at a substantial distance.

3.2. Multiple scattering: Consecutive scattering of light by more than one particle, causing a scattering pattern that is no longer the sum of the patterns from all individual particles.

3.3. Particle size: Equivalent spherical diameter reported from a distribution of spherical that creates a scattering pattern that matches the light scattering

3.4 Particle size distribution: Statistical distribution of particles of different sizes.

3.5 Sample: A physical sample used for testing

3.6 Sample Bath: Feeds the sample and dispersion medium

3.7 Dispersion medium: Optically transparent liquid of known refractive index

3.8 Debubble: Removes any trapped air bubbles in the cell

3.9 Alignment: Ensures the focal point of the detector coincides with that of the lens

3.10 Blank: Scattering light of the dispersion medium

3.11 Run: One repetition of a test

4. Summary of Test Method

4.1 An emulsified asphalt sample is tested in a particle size analyzer that uses a laser diffraction technique. This test is capable of measuring metrics such as D10, D50, D90, mean, mode median and the width of the distribution.

5. Significant and Use

5.1 Current research shows a moderate correlation between the particle size and particle size distribution of asphalt emulsion to rheology, stability and performance. Conducting a particle size analysis can help understand the quality and predict stability and performance of asphalt emulsions.

6. Apparatus

6.1 Laser Diffraction Analyzer: This standard utilized a Horiba LA-350 capable of measuring particle size distribution from 0.1 μm to 1000 μm, along with the software and algorithm provided by the manufacturer.

6.2 Mechanical pipette, 1.5ml: An adjustable volume mechanical pipette capable of transferring at least 1.5 ml of liquid.

6.3 Polypropylene container, 86 oz: A polypropylene container capable of storing 2000 grams of asphalt emulsion.

7. Reagents and Materials

7.1 Emulsified Asphalt – Emulsified asphalt sampled in accordance with ASTM and AASHTO Standards.

7.2 Distilled Water – Distilled water free of contaminants, chemical and bacteria with a pH of at least 5.

7.3 Xylol Xylene – A solvent to be used for the removal of asphalt residue trapped in the apparatus. Other solvents capable of dissolving asphalt binder may be used.

8. Conditioning

8.1 Enter the sample information. Information should include: sample name, material, source, lot number and test number.

8.2 Select the desired refractive index. In the case of asphalt emulsions, a refractive index of 1.63 may be used.

8.3 Select the desired refractive index for the dispersion medium. In the case of distilled water, a value of 1.33 may be used.

9. Procedure

9.1 Feed the sampling bath with the dispersion medium. A liquid level of medium is suggested

9.2 Start the circulation motor to circulate the dispersion medium through the unit

9.3 Begin debubbling so that any trapped air is removed from the system. Transmittance level should be close to 100%

9.4 Begin alignment so that the optical axis is completed.

9.5 Perform a blank measurement, scattering data is recorded as the blank value. Transmittance level becomes 100%.

9.6 Mix the container containing the emulsified asphalt thoroughly, a glass rod may be used.

9.7 Using a pippete, transfer about 0.3 grams (a droplet) of asphalt emulsion to the sampling bath

9.8 If necessary, an ultrasound may be used at this time.

9.9 Check the transmittance level so that multiple scattering is avoided.

9.10 Click measure and the particle size measurement begins. The measurement is displayed in the measurement screen

9.11 Click measure again until three runs are measured.

9.12 Begin rinsing the equipment with the dispersion medium. Additional dispersion medium is fed to the machine circulating it inside the unit, and draining is automatically performed.

10. Cleaning Procedure

10.1 After all measurements have been completed and once the unit has emptied the dispersion medium after rinsing. Add approximately 300 ml of Xylol Xylene to the sampling bath. The addition must be done manually and no pumping is required.

10.2 Begin circulation for approximately 15 minutes. A circulation speed of 5 may be used. At this point the dispersion medium (Xylol Xylene) should have a dark color. Ignore transmittance level

10.3 Click drain so that the cleaning agent is removed from the system.

10.4 Feed the sampling bath with distilled water and circulate distilled water through the machine at a high circulation speed.

10.5 Click rinse until clear water flushes out the system.

11. Precision and Bias

11.1 Since a precision estimate for this standard has not been developed, this test method is to be used for research or informational purposes only. Therefore, this standard should not be used for acceptance or rejection of a material for purchasing purposes.

12. Report

12.1 Report particle size metrics using the volume basis of measurement.

13 Keywords

13.1 Particle size analysis, laser diffraction technique, asphalt emulsion.