EFFECT OF CRM PARTICLE SIZE ON STORAGE STABILITY OF RUBBERIZED BINDERS

By

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FAIR USE AND AUTHOR'S PERMISSION STATEMENT

DEDICATION

I would like to dedicate this dissertation to My Family, Teachers, and God.

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ABSTRACT

The purpose of this study is to evaluate the impact of CRM (Crumb Rubber Modifier) particle size on storage stability and the tests required for its characterization. In this experimental analysis, PG64-22 containing three rubber crumb particle sizes (≤0.5 mm, ≤1 mm, and 1-2mm) was blended individually using a low shear mixer at the same proportion of 10% by weight of the original binder. Based on the analytical results (1), the Superpave test method was selected as the ideal test method and the G*/Sin δ and % Recovery tests performed on the Dynamic Shear Rheometer (DSR) were used to investigate the Separation Index (SI). (2) particle size after conditioning showed a direct effect on SI; (3) SI index values were found to be higher at the bottom than at the top and middle of the sample parts.

Keywords: CRM particle size, Super pave test method, Separation index.

1. INTRODUCTION

Background

The recycling of tires has always been challenging due to the materials it is made up of as it can become more hazardous when it is changed from one form to another as it can lead to toxic reaction when it is burnt due to its non-biodegradable nature (Karger-Kocsis et al., 2013; Uriarte-Miranda et al., 2018). Usually, because of the above problem, most end-of-life tires end up in landfills (Asaro et al., 2018; Sienkiewicz et al., 2012; Wang et al., 2018). But it is seen in recent years that governments have produced many projects to recycle end-of-life tires as they are composed of many good materials which have the reinforcement capability to improve flexible pavements (Association, 2019; Karakurt, 2015; Markl & Lackner, 2020). So, one of the best ways to recycle and impart this property of the tire in the HMA (Hot Mix Asphalt) pavement is by three methods namely dry process, terminal blend process and wet process. For this research, the primary consideration was to blend crumb rubber to PG64-22 binder through the process called the "wet process" as it is found as one of the remedial methods. This method enhances the performances of the mix which leads to improvements in the resilience, rutting, fatigue, cracking, and the improved life cycle of the pavement(Abdelaziz & Karim, 2003; Bockstal et al., 2019; Brasileiro et al., 2019; Bressi et al., 2019; Chen et al., 2019; Fontes et al., 2010; Katman et al., 2009; Liu et al., 2009; Mashaan et al., 2014; Picado-Santos et al., 2020; Presti, 2013; Presti et al., 2012; Thodesen et al., 2009; Wong & Wong, 2007). Despite these advantages CRM (Crumb rubber modifier) is known for its poor storage stability which is the profound focus of this research(Akisetty et al., 2009; Polacco et al., 2015; Yu et al., 2013; Zheng et al., 2021).

Objective of study

The purpose of this study is to determine the effective particle size of crumb rubber, among the following sizes, to improve the storage stability of asphalt binders by studying the viscosity, viscoelasticity, and MSCR (Multiple Stress Creep Recovery) properties. There are many tests for examining storage stability, but the “cigar tube test method” is considered the most common and effective method (Wang et al., 2020).

2. LITERATURE REVIEW

CRM Particle Size

The particle size of the rubber granules plays an important role in phase separation. The rubber granules (≤0.5mm, ≤1mm and 1-2mm) used in this study are produced by mechanical milling at ambient temperature, and the pieces of rubber produced by this process have wavy textures and are often asymmetrical (Putman, 2005).

Crumb rubber modified asphalt binders

Crumb rubber with effective sizes from ≤0.5mm, ≤1mm and 1-2mm is mixed with pure binder PG64-22 by wet process as this method has proven to be one of the effective methods and oldest (McDonald, 1966). Previous research has strongly demonstrated the ability to improve pavement life and increase stiffness performance at low temperatures (Bahia & Davies, 1994; Billiter et al., 1997). Although it has many advantages, previous studies have demonstrated that it has low storage stability and high separation index, especially at temperatures above 140℃ (Zhang et al., 2009). At this high temperature, the crumb rubber initially tends to swell as it begins to absorb the aromatic oils present in the virgin binder, and the viscosity tends to increase and follow a non-Newtonian fluid character. As the process continues, the inflated crumb rubber granules tend to decompose with increasing viscosity and then show a form of deposition at the bottom of the container due to the increased density, which means why CRMA (Crumb Rubber Modified Asphalt) has a low storage stability (Airey et al., 2003; Chehovits et al., 1982; Huffman, 1980; Liang et al., 2015). The mixing time was kept constant at 30 min throughout the experiment as it was found that increasing the mixing time was not significant (Mashaan et al., 2011).

Storage stability

The main objective of this research is to figure out the particle size of crumb rubber which would be effective in conducting further research for separation index% using percentage recovery data found through performing MSCR test on the sample as it is known that CRMB is very weak in storage stability (Airey et al., 2003; Billiter et al., 1997; Chehovits et al., 1982; Huffman, 1980; Zhang et al., 2009). The main aim of the study is to find out the difference in percentage increase of separation index as the particle size of crumb rubber increase. As the particle size increases the segregation and specific gravity of the mixture increases which tends to increase settling of particle due to gravitational force as suggested (Mashaan et al., 2011). The cigar tube test method is still popular and is implemented in this research as it has become one of the standardized tests for storage stability in many countries (Lu et al., 1999). It consists of an aluminum tube of diameter 32mm and height 160mm and the modified binder is poured into that aluminum tube 50±0.5 grams and placed in the oven with their tops sealed at 165±5 ℃ for 48 hours for conditioning and later shifted to freezer for minimum 4 hours at 10±10 ℃ (Freezing temperature to be maintained). Then the samples are cut into three equal halves namely top, middle, bottom and tested for its properties (Isacsson & Lu, 1999; Kim & Lee, 2013).

3. EXPERIMENTAL DESIGN

The experimental design model is explained using process flow chart followed by elaborated flow chart model as shown below in the form as Figure.1

Abbreviation and terms

PG: Performance Grade.

64-22: Maximum high temperature property of binder is 64 ℃ and minimum temperature property is -22 ℃.

CRM: Crumb rubber modified.

MSCR: Multiple shear creep test.

G*: Complex shear modulus

δ: Phase angle.

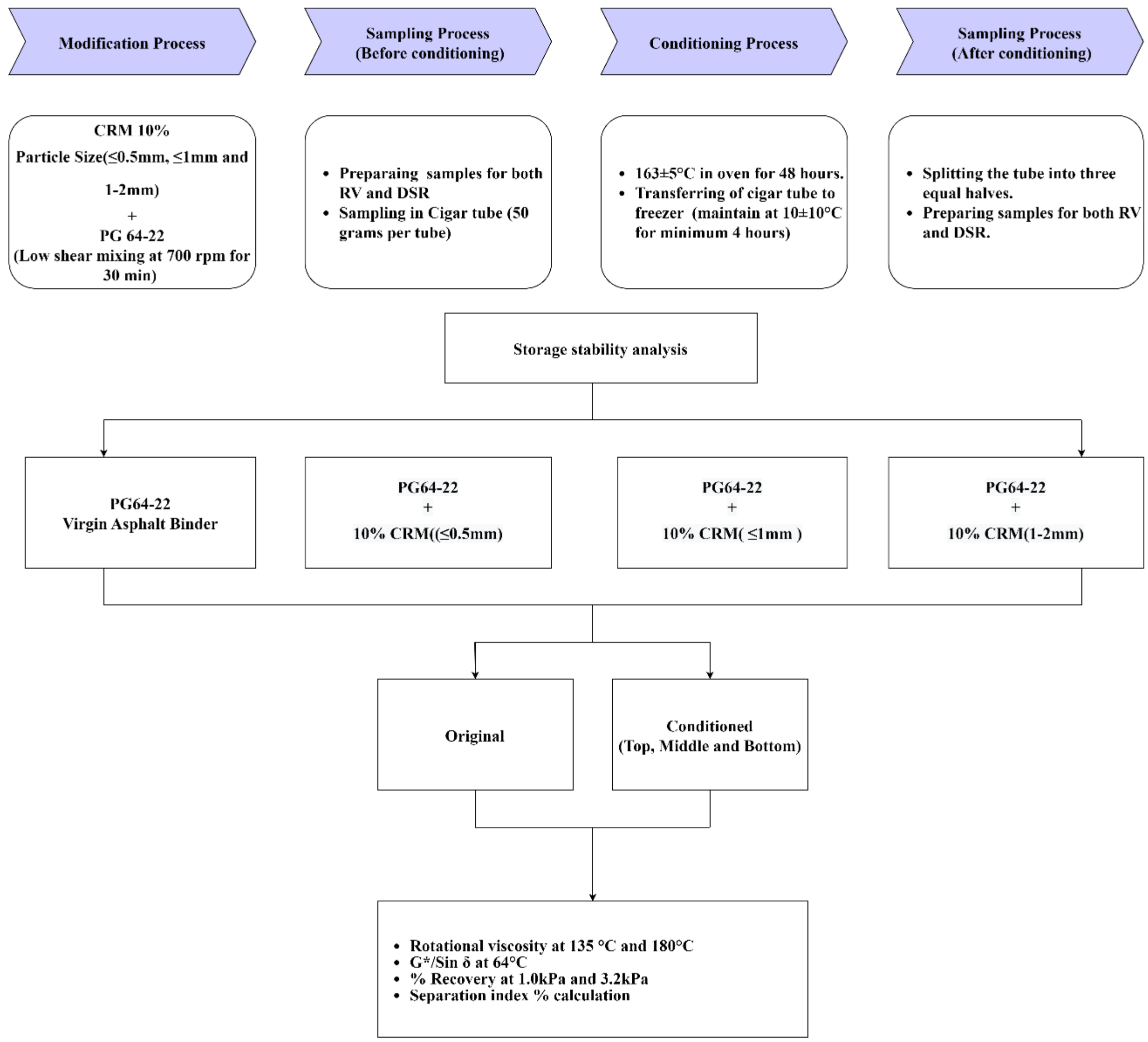

Figure.1 Experimental design model

Material used for modification

Virgin binder PG64-22 was used in this study along with various sizes of crumb rubber from ≤0.5mm, ≤1mm, 1mm to 2mm, and its weight percentage was kept constant at 10% of the original binder weight. Table 1 shows the properties of the base asphalt binder before modification, and Table 2 shows the sieve test results of the crumb rubber used in this study.

Table1. Properties of virgin asphalt binder PG 64-22 (Base asphalt binder)

Aging states	Test properties	Test result
Unaged binder	Viscosity @ 135 ℃ (cP)	540
	$G^*/\sin\delta$ @ 64 ℃ (kPa)	1.39
RTFO aged residual	$G^*/\sin\delta$ @ 64 ℃ (kPa)	3.83
RTFO+PAV aged residual	$G^*\sin\delta$ @ 25 ℃ (kPa)	4405
	Stiffness @ -12 ℃ (MPa)	207
	m-value @ -12 ℃	0.325

Table2. Sieve test results of crumb rubber

Sieve Number(μm)	% Cumulative Passed size ≤0.5mm	% Cumulative Passed size ≤1mm	% Cumulative Passed size 1mm-2mm
4	100	100	100
8	100	100	100
30	100	39.4	0.6
50	57.7	12.2	0
100	14.2	3.7	0
200	1.8	0.1	0

Modifying process of crumb rubber asphalt binders

Crumb rubbers were modified with base binder PG64-22 at a temperature of 178 ± 2°C for 30 min using a low shear mixer at a constant mixing speed of 700 rpm (Lee et al., 2006; Lee et al., 2008). The percentage of colloidal particles was kept constant at 10% of the original binder weight, but the particle size of colloidal particles varied in increasing order in each phase.

Test specimen preparation

The samples were prepared individually. After modifying the binder for 30 minutes, RV (rotational viscosity) samples were taken (minimum 3 samples), followed by samples for DSR (Dynamic Shear Rheometer) testing (minimum 3 samples), and finally poured into cigar test tubes (aluminum tubes) (min. 3 samples) maintained at 50 ± 0.5 grams according to ASTM D7173. Before taking each sample, the binder was stirred manually and poured into the aluminum tube. The aluminum tubes are then placed in the drying oven for conditioning for 48 hours (approx. 2 days). After 48 hours of conditioning in the oven (the oven temperature was maintained at 163±5 ℃), the aluminum tubes were transferred to the refrigerator for at least 4 hours. (The refrigerator temperature is kept at 10 ± 10 ℃). Finally, the cigar test tube was cut into three equal halves (top, middle and bottom) and placed in an oven set at 163±5 ℃). The sample must be held in the oven until melted and transferred to a hot plate where the sample is continuously stirred with a contaminant free stainless-steel spatula and the samples taken are similar to those before the conditioning process.

Rotational viscosity (ASTMD4402)

The Brookfield rotary viscometer is used to evaluate the viscosity properties of the sample. Samples were measured at two different temperatures (135℃ and 180℃). Spindle SP-27 was used for this test and spindle rotation was maintained at 20 RPM. The weights of the samples used for the tests were maintained between 10 and 11 grams and a total of 20 readings were taken at 1-minute intervals for each reading and the values were measured in cp.

Viscoelasticity and MSCR

The DSR machine was used to measure viscoelasticity and MSCR with the temperature held constant at 64℃ and then the following was measured: G*/sin δ, percent recovery at 1.0 kPa and 3.2 kPa. A 25mm geometry (both spindle and parallel plate) was used to test the samples.

Separation Index

This is an alternative method to test softening point and penetration. The calculation formula is shown in below equations 1 and 2. The separation index indicates the compatibility of the mixture, i.e. as the SI percentage decreases, the compatibility of the mixture increases(ASTM; Bahia & Zhai, 2000; Hallmark-Haack et al., 2019; Hosseinnezhad et al., 2019; Kabir et al., 2020; Kim & Lee, 2013; Li et al., 2019; Nasr & Pakshir, 2019; Shatanawi et al., 2012; Shen et al., 2006; Xu et al., 2017; Yu et al., 2018)

$$\text{Separation index}= \frac{(G*/Sin\delta)max-(G*/Sin\delta)avg}{((G*/Sin\delta)avg)} \text{-------------------------------------}1$$

$$\text{Separation index}= \frac{(\%recovery)max-(\%recovery)avg}{(\%recovery)avg} \text{------------------------------}2$$

Statistical analysis methodology

Static analyzes using SPSS was performed within the effective particle size of the crumb rubber using the one-way ANOVA method to examine whether there was a difference in mean values at the significance level $\alpha = 0.05$ i.e., 95% confidence interval. Post-hoc analysis of the data was performed using the LSD (Least Significant Difference) method. LSD compares sample pairs and declares the detected population mean to be statistically significant if the detected difference between two samples is greater than or equal to $\alpha = 0.05$.

4. RESULTS

Rotational viscosity

Rotational viscosity is one of the important tests used to evaluate the performance of binders. One of the major factors affecting the optimum compaction in the field is the viscosity of the mixture. Two temperatures of 135℃ and 180℃ were considered for this study and one-way ANOVA and graphical methods were used to interpret the results. The results obtained from the graph clearly show the tendency to decrease viscosity as the size of the rubber crumb particles increases. In the lower part of the sample, the trend is up, which is defined in the results as having a significant change from the initial condition. The important result clearly demonstrates that the shredded rubber particles have a tendency to settle to the bottom of the tube due to its increased density compared to the pure binder and can be clearly seen in separation index analysis, when the size efficiency of crumb rubber increased, SI % increased. The following analysis results are interpreted through the SPSS output and graph as shown in the table and figure below.

Table.3 SPSS statistical analysis report of effective particle size of CRM binder for viscosity at 135 ℃ as function of top, middle and bottom parts for (α=0.05)

Viscosity at 135 ℃													
Crumb Rubber Size(mm)		Original			Top			Middle			Bottom		
		≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2
Original	≤0.5	-	N	N	S	S	S	S	S	S	S	S	S
	≤1	-	-	N	S	S	S	S	S	S	S	S	S
	1-2	-	-	-	N	N	N	S	S	S	S	S	S
Top	≤0.5	-	-	-	-	N	S	S	S	S	S	S	S
	≤1	-	-	-	-	-	N	S	S	S	S	S	S
	1-2	-	-	-	-	-	-	S	S	S	S	S	S
Middle	≤0.5	-	-	-	-	-	-	-	N	S	S	S	S
	≤1	-	-	-	-	-	-	-	-	S	S	S	S
	1-2	-	-	-	-	-	-	-	-	-	S	S	S
Bottom	≤0.5	-	-	-	-	-	-	-	-	-	-	S	S
	≤1	-	-	-	-	-	-	-	-	-	-	-	S
	1-2	-	-	-	-	-	-	-	-	-	-	-	-

S- Significant
N- Non-significant

Table.4 SPSS statistical analysis report of effective particle size of CRM binder for viscosity at 180℃ as function of top, middle and bottom parts for (α=0.05)

Viscosity at 180 ℃													
Crumb Rubber Size (mm)		Original			Top			Middle			Bottom		
		0.5	1	1-2	0.5	1	1-2	0.5	1	1-2	0.5	1	1-2
Original	≤0.5	-	N	N	S	N	N	S	S	S	S	S	S
	≤1	-	-	N	S	S	S	S	S	S	S	S	S
	1-2	-	-	-	S	S	S	S	S	S	S	S	S
Top	≤0.5	-	-	-	-	N	N	S	S	S	S	S	S
	≤1	-	-	-	-	-	N	S	S	S	S	S	S
	1-2	-	-	-	-	-	-	S	S	S	S	S	S
Middle	≤0.5	-	-	-	-	-	-	-	N	N	S	S	S
	≤1	-	-	-	-	-	-	-	-	N	S	S	S
	1-2	-	-	-	-	-	-	-	-	-	S	S	S
Bottom	≤0.5	-	-	-	-	-	-	-	-	-	-	S	S
	≤1	-	-	-	-	-	-	-	-	-	-	-	S
	1-2	-	-	-	-	-	-	-	-	-	-	-	-

S- Significant
N- Non-significant

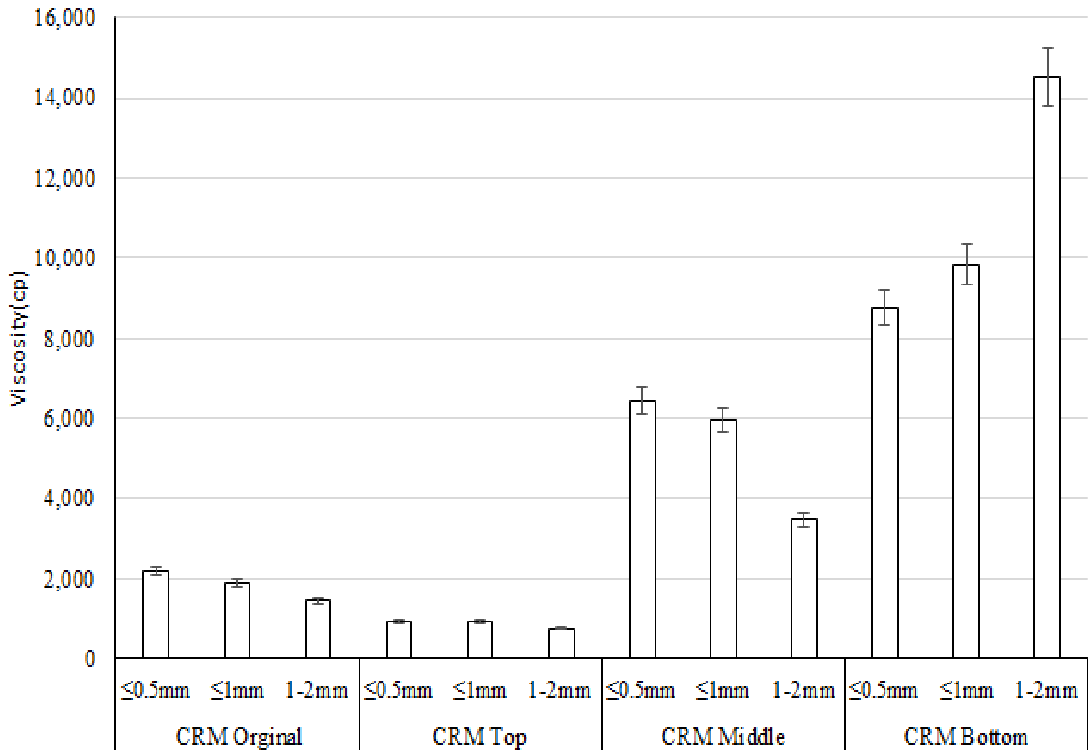

Figure.2 Viscosity at 135℃ of effective particle size of crumb rubber for orginal sample and top, middle,bottom part (Conditioned)

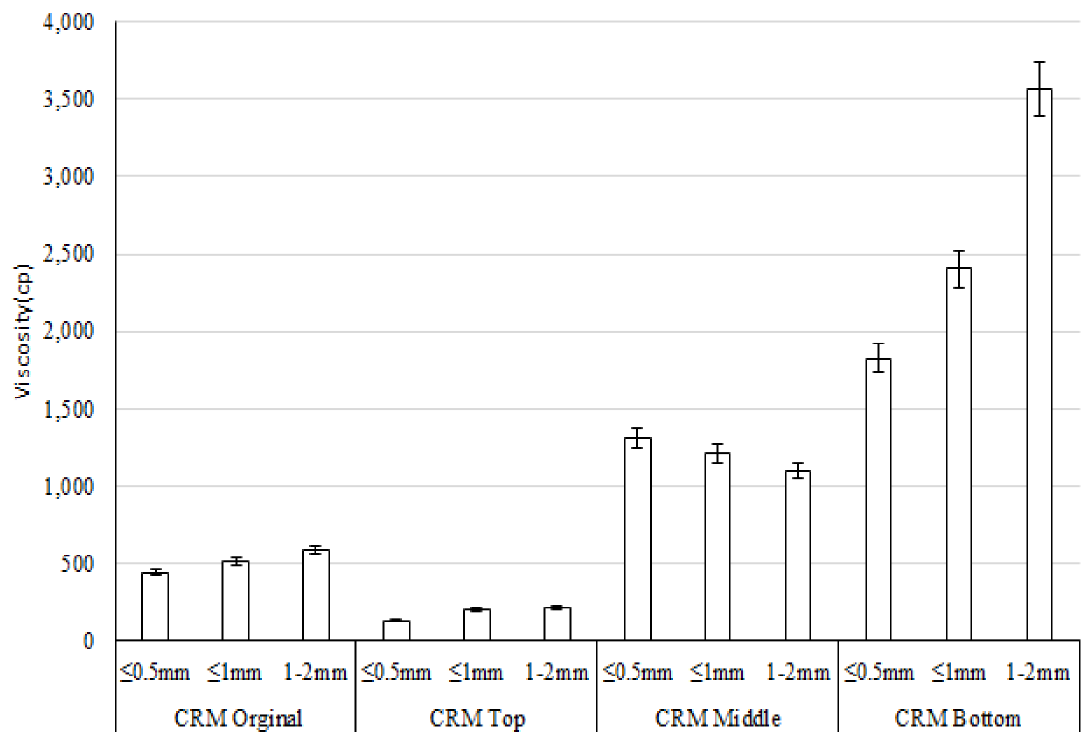

Figure.3 Viscosity at 180℃ of effective particle size of crumb rubber for orginal sample and top, middle,bottom part (Conditioned)

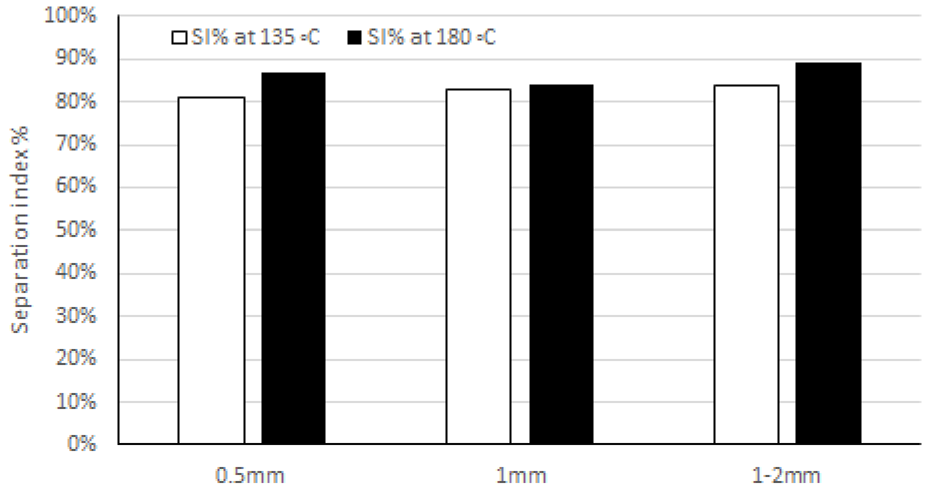

Figure.4 Separation index % for viscosity for effective particle size at 135℃ and 180℃ considering only top and bottom part (Conditioned)

Viscoelasticity (G*/sin δ) property of modified binders

The viscoelastic nature of the effective particle size crumb rubber modified binder was evaluated using a DSR machine. 64 ℃ turned out to be the ideal temperature for this study. During the analysis, it was found that as the particle size increased, the viscoelastic nature decreased significantly as the crumb size increased. But it is found that there is a significant change when examining the lower part of the sample as the values continue to increase as the grain size increases identified in the table below as significant and the graph below also shows the same change. On the other hand, SI% tends to increase as the particle size increases. There appears to be an average 20% increase in the percent separation index for each of ≤1mm increase in particle size.

Table.5 SPSS statistical analysis report of effective particle size of CRM binder for viscoelasticity at 64℃ as function of top, middle, and bottom parts for (α=0.05)

Viscoelasticity (G*/Sin δ) at 64℃														
Crumb rubber size (mm)		Original			Top			Middle			Bottom			
		≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2
Original	≤0.5	-	N	S	S	S	S	N	S	S	S	S	S
	≤1	-	-	S	S	S	N	N	S	S	S	S	S
	1-2	-	-	-	N	S	S	S	S	S	S	S	S
Top	≤0.5	-	-	-	-	N	S	N	S	S	S	S	S
	≤1	-	-	-	-	-	N	S	S	S	S	S	S
	1-2	-	-	-	-	-	-	S	S	S	S	S	S
Middle	≤0.5	-	-	-	-	-	-	-	S	S	S	S	S
	≤1	-	-	-	-	-	-	-	-	S	N	S	S
	1-2	-	-	-	-	-	-	-	-	-	N	S	S
Bottom	≤0.5	-	-	-	-	-	-	-	-	-	-	S	S
	≤1	-	-	-	-	-	-	-	-	-	-	-	S
	1-2	-	-	-	-	-	-	-	-	-	-	-	-

S- Significant
N- Non-significant

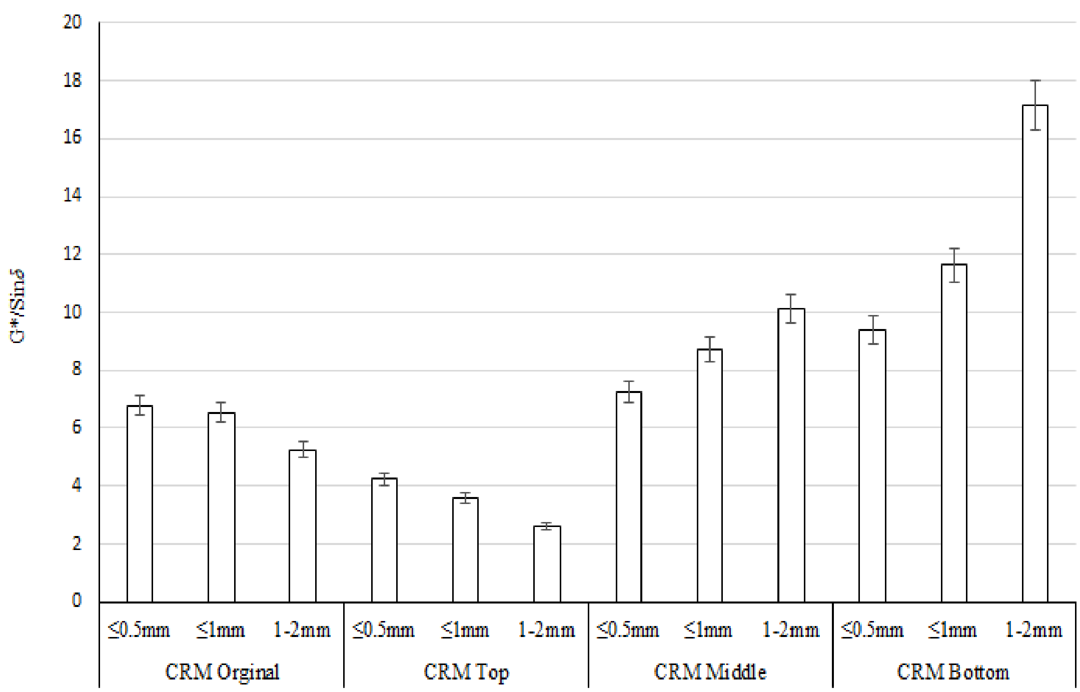

Figure.5 Viscoelasticity(G*/sin δ) at 64℃ of effective particle size of crumb rubber for orginal sample and top, middle,bottom part(Conditioned)

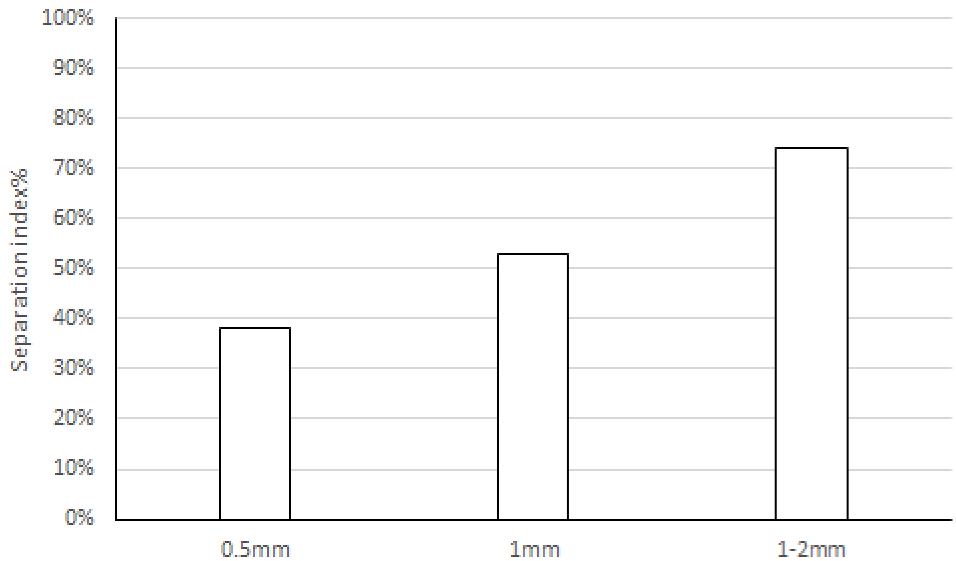

Figure.6 Separation index % of (G*/sin δ) for effective particle size at 135℃ and 180℃ considering only top and bottom part (Conditioned)

Multiple-stress creep recovery property of modified asphalt binders

This test was performed by the same equipment DSR and was used to find out the stress creep percentage recovery for both 0.1 kPa and 3.2 kPa at constant temperature of 64℃ as per AASHTO TP 70. For this research, the percentage recovery at 0.1 kPa and 3.2 kPa is just taken into consideration. The analysis shows the percentage recovery is significantly decreased when particle size is increased and then later shows an increasing pattern which clearly shows a significant change in the pattern during 0.1 kPa load is applied on the original and top part of the sample. But when tested the middle and bottom part of the sample percentage recovery tends to be increasing which is insignificant from current course which is shown in the table below and the graph results too acknowledges the results found on the analysis. But when tested for 3.2 kPa the percentage recovery significantly follows a decreasing trend for both the original and top part of the sample and shows a non-significant pattern when the value starts increasing on middle and bottom part of the sample which clearly shows the effect of particle size. On the other hand, the SI% is found to be similar for both 0.5mm and 1mm particle size for 0.1 kPa which indicates there is not much variance but for 3.2 kPa the separation index percentage found increasing which is portrayed in the graph below.

Table.6 SPSS statistical analysis report of effective particle size of CRM binder for %recovery at 64℃ for 0.1 kPa as function of top, middle and bottom parts for (α=0.05)

% Recovery for 0.1 kPa at 64℃													
Crumb rubber Size (mm)		Original			Top			Middle			Bottom		
		≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2
Original	≤0.5	-	N	S	S	S	S	S	S	S	S	S	S
	≤1	-	-	S	S	S	S	S	S	S	S	S	S
	1-2	-	-	-	S	S	S	S	S	S	S	S	S
Top	≤0.5	-	-	-	-	N	N	S	S	S	S	S	S
	≤1	-	-	-	-	-	N	S	S	S	S	S	S
	1-2	-	-	-	-	-	-	S	S	S	S	S	S
Middle	≤0.5	-	-	-	-	-	-	-	S	S	S	S	S
	≤1	-	-	-	-	-	-	-	-	S	S	S	S
	1-2	-	-	-	-	-	-	-	-	-	S	N	N
Bottom	≤0.5	-	-	-	-	-	-	-	-	-	-	S	S
	≤1	-	-	-	-	-	-	-	-	-	-	-	S
	1-2	-	-	-	-	-	-	-	-	-	-	-	-

S-Significant
N-Non-significant

Table.7 SPSS statistical analysis report of effective particle size of CRM binder for %recovery at 64°C for 3.2 kPa as function of top, middle and bottom parts for (α=0.05)

% Recovery for 3.2 kPa at 64°C

Crumb rubber Size (mm)		Original			Top			Middle			Bottom		
		≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2	≤0.5	≤1	1-2
Original	≤0.5	-	N	N	S	S	S	S	S	S	S	S	S
	≤1	-	-	N	S	S	S	S	S	S	S	S	S
	1-2	-	-	-	S	S	S	S	S	S	S	S	S
Top	≤0.5	-	-	-	-	N	S	S	S	S	S	S	S
	≤1	-	-	-	-	-	N	S	S	S	S	S	S
	1-2	-	-	-	-	-	-	S	S	S	S	S	S
Middle	≤0.5	-	-	-	-	-	-	-	S	S	S	S	S
	≤1	-	-	-	-	-	-	-	-	S	N	S	S
	1-2	-	-	-	-	-	-	-	-	-	S	N	S
Bottom	≤0.5	-	-	-	-	-	-	-	-	-	-	S	S
	≤1	-	-	-	-	-	-	-	-	-	-	-	S
	1-2	-	-	-	-	-	-	-	-	-	-	-	-

S- Significant
N- Non-significant

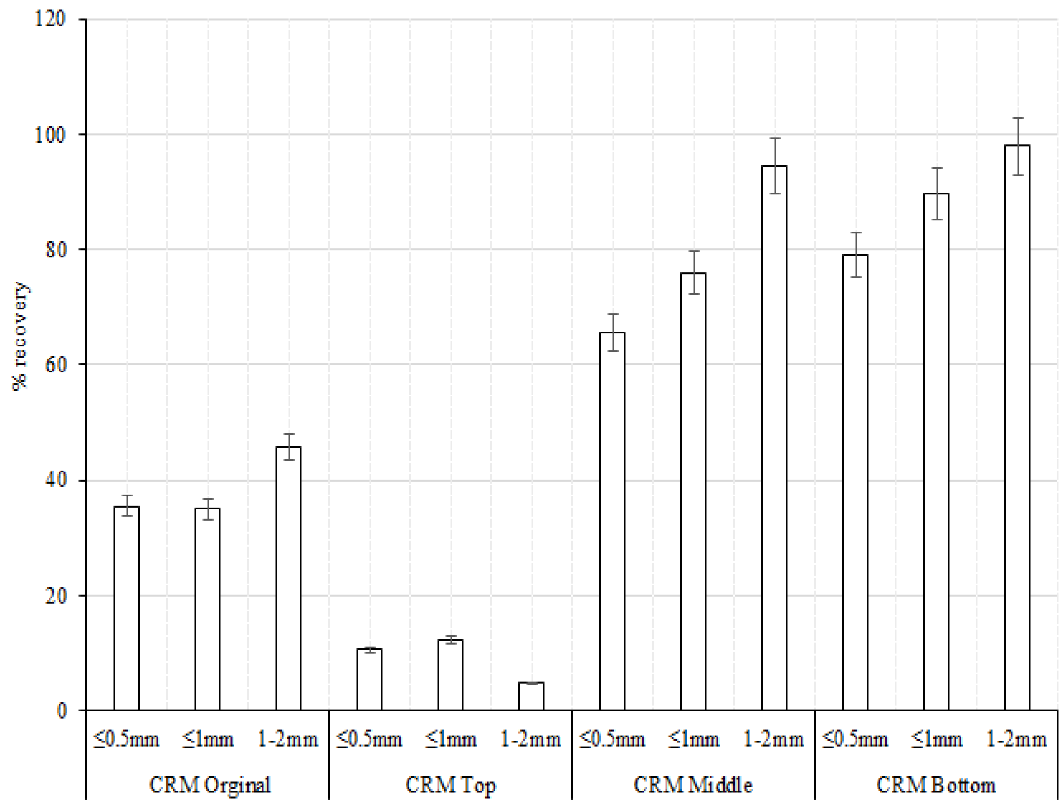

Figure.7 % recovery at 64°C of effective particle size of crumb rubber at 0.1 kPa

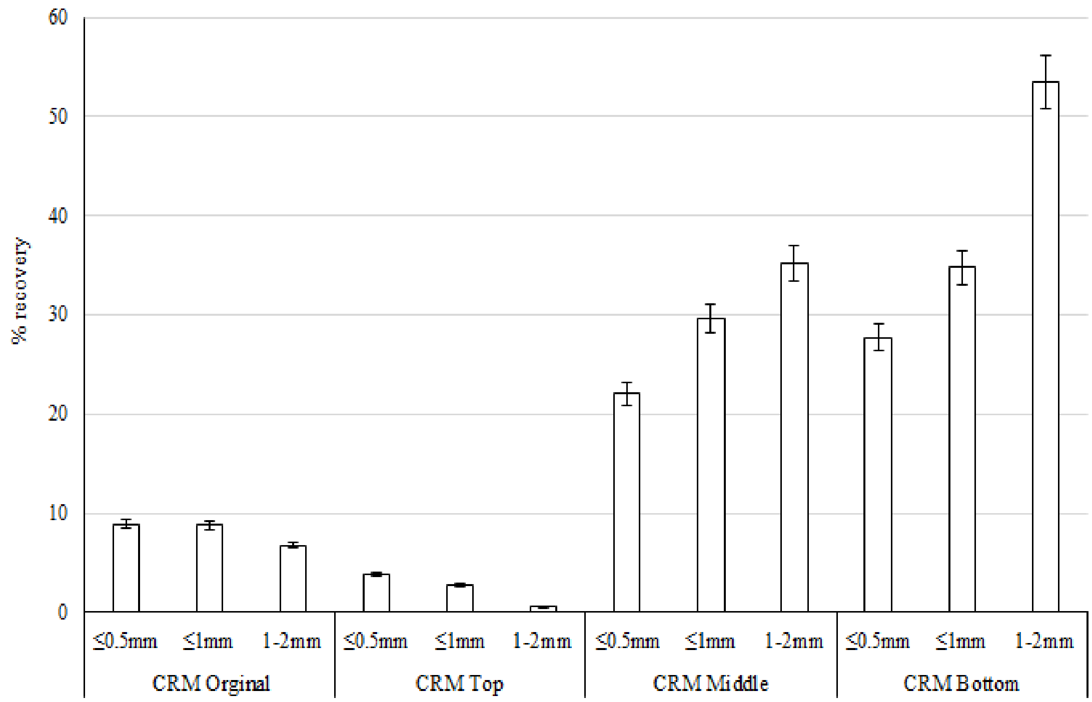

Figure.8 % recovery at 64°C of effective particle size of crumb rubber at 3.2 kPa

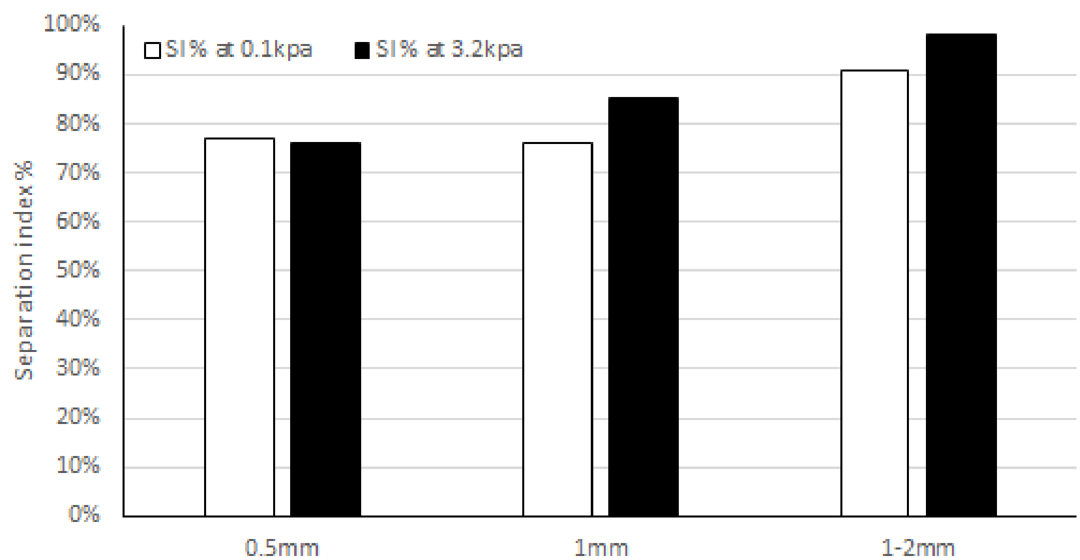

Figure.9 Separation Index % at 64°C of effective particle size of crumb rubber for 0.1 kPa and 3.2 kPa

Storage stability results

The evaluation of storage stability for the above modified crumb rubber asphalt with different particle size was conducted as per super pave method i.e., G*/sin δ and % recovery top and bottom parts of the conditioned samples were used to find out the separation index percentage. Tables 8 to 10 clearly proves the effect of particle size on the CRM binder properties i.e., SI % is directly proportional to the particle size of the crumb rubber.

Table.8 Separation index from $G^*/\sin\delta$ of CRM binders

CRMB size (mm)	G^*/ in δ (kPa)			
	Temperature	Top	Bottom	% Separation
≤0.5	64°C	4.25	9.39	38
≤1	64°C	3.58	11.63	53
1-2	64°C	2.59	17.16	74

Table.9 Separation index from % recovery for 0.1 kPa of CRM binders

CRMB size (mm)	% Recovery			
	Temperature	Top	Bottom	% Separation
≤0.5	64°C	10.46	79.11	77
≤1	64°C	12.10	89.70	76
1-2	64°C	4.71	97.93	91

Table.10 Separation index from % recovery for 3.2 kPa of CRM binders

CRMB size (mm)	% Recovery			
	Temperature	Top	Bottom	% Separation
≤0.5	64°C	3.74	27.73	76
≤1	64°C	2.76	34.74	85
1-2	64°C	0.60	53.50	98

5. SUMMARY AND CONCLUSION

To evaluate the effect of crumb rubber particles on viscosity and viscoelastic properties, a rubberized asphalt binder was prepared in the laboratory using 10% CRM by weight of fresh binder. The base binder in this study is PG64-22 mixed with rubber crumbs of various particle sizes ranging from ≤0.5mm, ≤1mm and 1-2mm. Samples were taken both from the modified original binder and after conditioning. Rotational viscosity tests were performed at temperatures of 135°C and 180°C, and viscoelastic properties were measured with DSR using two methods: original binder test at a temperature of 64°C and MSCR at 64°C with under different load cycles 0.1kPa and 3.2kPa. Based on the tests performed, the following conclusions were drawn:

1. The sample tested in original condition showed higher property values compared to the top portion but not greater than bottom portion of the sample. The original condition sample values were higher significantly than tested top portion sample because the mixing time was limited to 30 minutes. Also, the modified binder was cohesive as the modified binder sample was taken immediately and the particles did not have sufficient time to settle, resulting in lower values compared to bottom portion. But it was clearly observed the conditioned sample after 48 hours especially the bottom part of the sample had higher property value. This clearly shows the influence of time.

2. The $G^*/\sin\delta$ results tested at 64°C also showed a similar pattern of increasing characteristic values at the bottom compared to the top and middle of the sample.

3. The % recovery result from MSCR test on the sample tested at 64°C using DSR showed that 0.1 kPa has a higher value than the sample tested at 3.2 kPa, which clearly indicates the increasing load cycles increases binder deterioration, resulting in lower recovery percentages.

4. The SI% value, especially from the rotational viscosity test, was considered insignificant for the study because the top part had a higher SI% compared to the middle part and therefore the Super Pave method was used to analyze the SI%. G*/ Sin δ and % recovery was found ideal for the study of SI% and only top and bottom portion of the sample were finally considered for this analysis.

5. The SI% value clearly proves that as the particle size and temperature increase, the SI% is higher, and the decrease in the particle size of the crumb rubber leads to a decrease in the SI%, which determines the importance of this study. The higher particle size of the crumb rubber and the high proportion of the crumb rubber substitute in relation to the weight of the original binder can be ruled out for the present study, since the purpose of the study has already been determined.

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