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A Laboratory Investigation Regarding Storage Stability of the CRM-Modified Bitumen—CRM Processing Method (Untreated vs. Treated)

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Abstract: The aim of this study is to analyze the phase separation that occurs between treated and untreated rubber crumb particles produced by wet processes in the laboratory. The percentage of replacement used for both the treated and untreated crumb rubber-modified asphalt (CRMA) was 5%, 10%, 15%, and 20%. Tests to evaluate binder properties were performed using a rotational viscometer and a DSR, and the following properties were determined—viscosity, G*/sinδ, % recovery, and J_{nr}. The phase separation study was analyzed using the viscosity and G*/sinδ results. In general, the results of the study show the following. (1) The treated CRMA binders had higher viscosity values than untreated CRMA binders, although some values could not be measured due to the high viscosity values. (2) The G*/sinδ, % recovery and J_{nr} results also had a similar trend with viscosity results. (3) The viscosity and G*/sinδ phase separation values demonstrate that treated CRMA binders perform better than untreated CRMA binders. (4) Different experimental methods have shown variations in the calculated SI value; hence, a more improved approach should be explored to accurately assess the storage stability of asphalt binders containing various additives.

Keywords: CRMA; treated; untreated; phase separation

Citation: Yun, J.; Vigneswaran, S.; Lee, M.-S.; Lee, S.-J. A Laboratory Investigation Regarding Storage Stability of the CRM-Modified Bitumen—CRM Processing Method (Untreated vs. Treated). *Sustainability* **2023**, *15*, 10825. https://doi.org/10.3390/su151410825

Academic Editors: Antonio D'Andrea, Marinella Giunta, Ahmad Safuan A Rashid, Nor Zurairahetty Mohd Yunus and Norhidayah Abdul Hassan

Received: 31 March 2023
Revised: 9 June 2023
Accepted: 3 July 2023
Published: 10 July 2023

1. Introduction

Nowadays, road transport is one of the main modes of transport in the world, and as a result, there is an increasing reliance on automobiles for transporting goods and services, which obviously increases the demand for tires and the necessity to recycle end-of-life tires (ELTs) [1–3]. ELTs are always an environmental concern as they are non-biodegradable and pose a fire hazard. One of the techniques to recycle end-of-life tires is in the form of crumb rubber mixed with pure asphalt binders, and more than 30 countries around the world have combined the application of these crumb rubber-modified asphalt (CRMA) binders to prepare for both the airport and highway pavement [4–11]. In general, asphalt consists of the correlation of three-dimensional polar molecules [12]. Binding of the polar molecules of the shredded rubber after mixing with the basic asphalt binder leads to the crumb rubber modifier (CRM) in the asphalt binder absorbing the lighter fractions of the binders, causing the CRM to swell. This swelling leads to a decrease in the distance between rubber particles, resulting in the hardening of the residual binders after the interaction with the enhancement of properties such as adhesion, elasticity, resistance to heat, cold, and aging [13–19]. In general, these CRMA binders are produced by a wet process [20]. There is also unambiguous evidence from previous studies that the inclusion of crumb rubber to base binders through a wet process leads to a refinement of pavement life, decreased propensity to rutting and cracking, decrease in noise during traffic mobility, and reduced costs against periodic maintenance [21–33]. Despite all these merits, CRMA binders are more amenable to high phase separation, i.e., low storage stability [34–38], and past studies demonstrate that multiple parameters have an influence on the phase separation including the size of the crumb rubber particles, the type of base binder used for modification, and interaction conditions [39–43]. Furthermore, a prior project has revealed that increasing the proportion of substitution of crumb rubber to the weight of the original binder facilitates, to some extent, reducing the phase separation [15]. In addition, the inclusion of nanoclay in CRM asphalt binders was found to positively affect storage stability. Nanoclay improves the dispersion and interfacial bonding of crumb rubber particles within the binder matrix, reducing the potential for phase separation and sedimentation [44]. However, the impact of bio-oil on the storage stability of CRMA was positive [45].

Taking into consideration all of the above determinants that may contribute to reducing phase separation, experiments were conducted using both treated and untreated crumb rubber with examining their interplay, since there is a paucity of research discussing all these factors.

In addition, the determination of the separation index (SI) for assessing storage stability has been carried out following the ASTM D7173 standard [46]. This measurement is employed to evaluate modified binders for storage stability based on a softening point test [47], rheological properties analysis [48], and multiple stress creep tests [49]. However, in a previous study, it was found that the SI value was different between the test methods presented in the ASTM standards. Therefore, it is necessary to reconfirm that the evaluation of storage stability varies depending on the experimental method [50].

The following tests are performed to determine the phase separation of the modified CRMA binders generated with 5%, 10%, 15%, and 20% of treated and untreated crumb rubber to the original binder's weight: rotational viscometer (RV) to find out the effect of viscosity and phase separation when exposed to two different temperatures, DSR (Dynamic Shear Rheometer) tests, where the modified binder is tested for $G^*/\sin\delta$, % Recovery, and Creep Recovery when exposed to three different temperatures to find out its susceptibility to plastic deformation and the percentage of separation. The ideal test method chosen for this study based on previous research was the cigar tube test method [51]. Figure 1 illustrates a flow chart that emphasizes the empirical design adopted for this study.

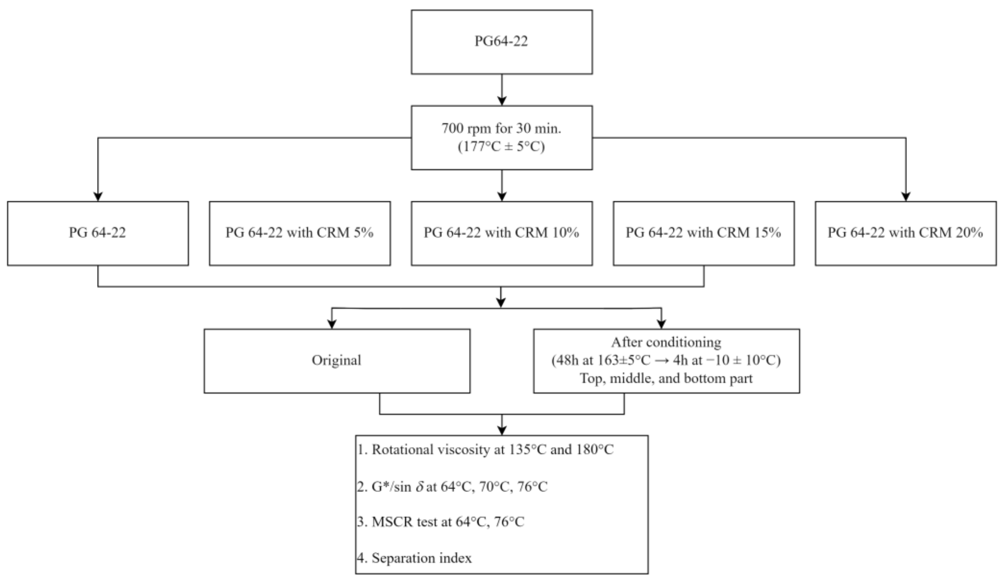

Figure 1. Flow chart of experimental design procedures.

2. Experimental Design

2.1. Materials

PG64-22 was the base asphalt binder used to formulate both treated and untreated CRMA binders. Table 1 shows the characteristics of the virgin asphalt binder, followed by Table 2, which shows the grading outcomes of both ground crumb rubber used for this study. Untreated crumb rubber is typically produced through mechanical shredding at ambient temperature. In this study, surface-treated crumb rubber was utilized to produce treated CRMA. The treated crumb rubber exhibited a gradation that closely resembled a single particle size in comparison to untreated crumb rubber. It is anticipated that the smaller amount of rectangular-shaped particles in the treated rubber sample will influence the performance of the microstructural properties of the crumb rubber [52]. Both untreated and treated crumb rubbers were provided by a local producer. An evaluation of storage stability was carried out using an aluminum tube according to ASTM D7173. The images of the cigar tube are listed in Figure 2.

Table 1. Properties of base asphalt binder (PG 64-22).

Aging States	Test Properties	Test Result	Standards
Unaged binder	Viscosity @ 135 °C G*/sinδ @ 64 °C	538 cP 1.38 kPa	<3000 1.1<
RTFO aged residual	G*/sinδ @ 64 °C	3.82 kPa	2.2<
RTFO + PAV aged residual	G*sinδ @ 25 °C Stiffness @ −12 °C m-value @ −12 °C	4402 kPa 205 MPa 0.323	<5000 <300 0.3<

Table 2. Gradation of CRM used in this study.

Sieve Number (µm) %	% Cumulative Passed of Untreated CRM	% Cumulative Passed of Treated CRM
30 (600)	100	100
40 (425)	91.0	96.4
50 (300)	59.1	27.9
80 (180)	26.2	16.8
100 (150)	18.3	6.8
200 (75)	0.0	0.7

(**a**) (**b**)

Figure 2. Aluminum tube (**a**) and vertically held tube (**b**).

2.2. Production of Treated and Untreated CRMA Binders

Both CRMA binders were prepared by a wet process in the laboratory, with modification temperatures maintained near 177 °C, and blended with a terminal velocity of 700 rpm utilizing a low-shear mixer for 30 min. The percentage of rubber crumb used to make both treated and untreated CRMA binders was 5%, 10%, 15%, and 20%, respectively, based on the weight of the virgin binder. To ensure consistency criteria for the blend produced, PG64-22 and crumb rubber (both treated and untreated) from the same batch were used for this study.

2.3. Preparation of Test Specimens

The aluminum tubes (Figure 2) utilized for conditioning purposes are cylindrical in shape, measuring 25 mm in diameter and 125 mm in length. These tubes are specifically designed to securely hold the test samples during the conditioning process. First, 50 ± 0.5 g of the modified binder was poured into each tube, and the tops were sealed to prevent any oxidation reaction and kept in the oven at a temperature of 163 ± 5 °C for a period of 48 ± 1 h. The sample should be kept undisturbed during this conditioning period. After the 48 ± 1 h conditioning period in the oven, the sample is transferred to a freezer maintained at -10 ± 10 °C and left undisturbed for a minimum period of 4 h to allow the sample to fully solidify. After the sample has attained its full conditioning, the sample cigar tube is divided into three equal halves. For this study, the top, middle, and bottom of the cigar tube are considered. The sample is then transferred to an oven maintained at 163 ± 5 °C, and the sample is held in the oven until it begins to be stable. In addition, we ensure that the sample should not be left in the oven for more than 30 min, as prolonged exposure will affect the result. Once the sample begins to liquify after 25 min, the sample is transferred to a hot plate where it is manually stirred and mixed with a spatula free of foreign materials until a cohesive mixture is observed, and test samples are used for both RV and DSR.

2.4. Binder Properties

2.4.1. Rotational Viscosity

To determine which CRMA binders had higher base property values among the treated and untreated, a Brookfield rotational viscometer was employed to evaluate their viscosity at two different temperatures, 135 °C and 180 °C, according to ASTM D4402. The spindle number used to compute the viscosity values is SPN-27 with a rotation speed of 20 rpm and an interval between each reading of 1 min.

2.4.2. Viscoelasticity by DSR Method

A Dynamic Shear Rheometer (DSR) was used to analyze the viscoelastic properties of the modified binders. Two types of tests were performed using this equipment: one was the original binder test, in which only the permanent set, i.e., $G^*/\sin\delta$ at 1.00 kPa (10 cycles at a frequency of 10 rad/s per Test Method D 7175) at three different temperatures of 64 °C, 70 °C, 76 °C, and the other was an MSCR test performed per Test Method D 7405 using only % recovery and unrecoverable creep J_{nr} per AASHTO TP70. The load cycle and measured test temperature were limited to 3.2 kPa and temperatures of 64 °C and 76 °C. This experiment was evaluated as an average of three samples tested.

2.4.3. Separation Index (SI)/Phase Separation

According to ASTM D7103, as an alternative method to softening point testing to analyze the rheological properties of the binder, a DSR test was conducted. In this, Superpave test specifications were used to find the percentage of separation through Equations (1) and (2) according to previous research studies [52–62]. This experiment was evaluated as an average of three samples tested.

$$\text{Separation index } = \frac{(\text{Viscosity})max - (\text{Viscosity})avg}{(\text{Viscosity})avg} \tag{1}$$

$$\text{Separation index } = \frac{(G*/sin\delta)max - (G*/sin\delta)avg}{((G*/sin\delta)avg)} \tag{2}$$

where

($G^*/\sin\delta$)max—Highest value between the upper and lower part of the cigar tube.
($G^*/\sin\delta$)avg—Mean value between the upper and lower part of the cigar tube.

2.5. Statistical Analysis Method

A statistical analysis of the results of the experimental samples was performed using SPSS (Statistical Package for the Social Sciences). To determine the variance, a one-way ANOVA (analysis of variance) test was performed, and post hoc data analysis was performed using the LSD (Least Significant Difference) method with 95% confidence intervals, i.e., say $\alpha = 0.05$. Key variables included the two types of rubber discs (treated and untreated) and their replacement rates (5%, 10%, 15%, and 20%), and a one-way ANOVA was performed on the data to find any significant differences in their means. In the analysis of this study, the level of significance was set at $\alpha = 0.05$, which specifies that each result has a 95% probability of being true. After determining that there is a difference between the sample means using one-way ANOVA, the LSD is calculated. LSD is defined as the observed difference between two sample means that should explain the difference in the respective population means. When calculating LSD, all pairs of sample means are compared. The population means are reported as statistically different if the difference between the two-sample means is greater than or equal to LSD [63].

3. Results and Discussion

3.1. Rotational Viscosity

The temperature necessitated to generate the asphalt at the mixing plant and the workability of the asphalt mixture during field compaction are all determined by the viscosity of the mixture. A high viscosity affects the optimal field density, which indirectly affects the service life of the pavement. Figure 3 shows the values found by RV at 135 °C and 180 °C for the original condition of both treated and untreated CRMA asphalt, with the percentage of crumb rubber by weight of virgin asphalt binder replaced immediately after 30 min of modification. At 135 °C, both the modified binder had higher viscosity values compared to the controlled state modified binder as the percentage of crumb rubber replacement increased. Compared to 0% controlled binder, the 20% untreated CRMA binder showed about a 6-fold increment in viscosity values. Treated CRMA binder values became so high that it was evidently indicating that the mix was unworkable, showing that the data were not measured due to its high viscosity. In particular, the treated rubber had higher viscosity values compared to untreated rubber, which means that the temperature of the asphalt production temperature has to be increased in order to achieve the optimal field compaction, but the viscosity decreased significantly, showing the influence of temperature; i.e., as the temperature increases, the viscosity decreases. Figure 4 shows the results for the top, middle, and bottom parts at 135 °C viscosity of CRMA binders for both treated and untreated crumb rubber CRMA after conditioning. It can be seen from the graph that as the substitute content for crumb rubber increased, the lower parts had higher viscosity values compared to the upper and middle parts. The treated rubber CRMA becomes almost unusable when the replacement percentage in the lower part of the sample exceeds 15% because of its high viscosity not being measured. This demonstrates the effect of sediment velocity on CRMA binders in terms of the increased replacement of crumb rubber content. The study shows that the treated rubber settles more at the bottom of the tube at 135 °C compared to untreated CRMA.

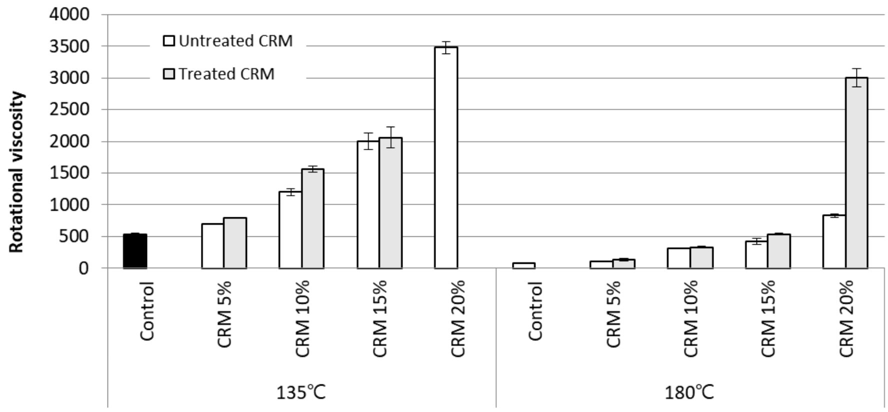

Figure 3. Viscosity of the CRM asphalt binders at 135 °C and 180 °C.

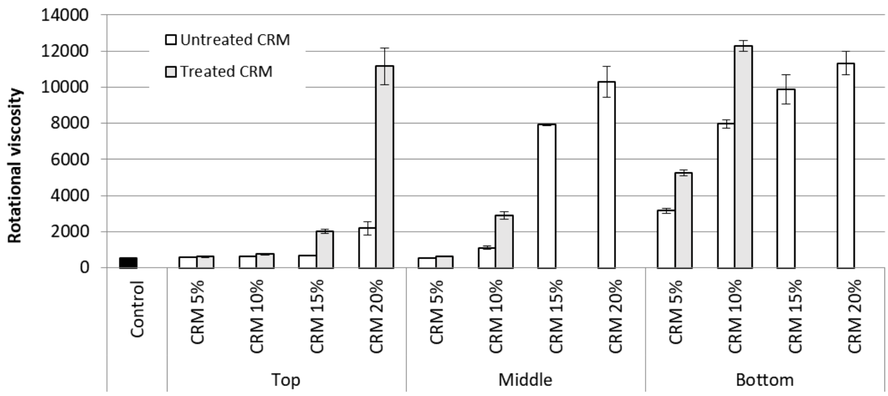

Figure 4. Viscosity at 135 °C of the CRM binders of top, middle and bottom parts after conditioning.

As in the case of increasing the temperature to 180 °C, the viscosity starts to decrease, and untreated rubber granules showed lower viscosity values at higher temperatures compared to treated rubber-modified asphalt. The treated CRMA exhibited an approximately 4-fold increase in viscosity values compared to untreated rubber. This clearly shows that the treated rubber has higher polar bonding with aromatic virgin binder compared to untreated ambient ground crumb rubber. Figure 5 shows the viscosity test values at 180 °C.

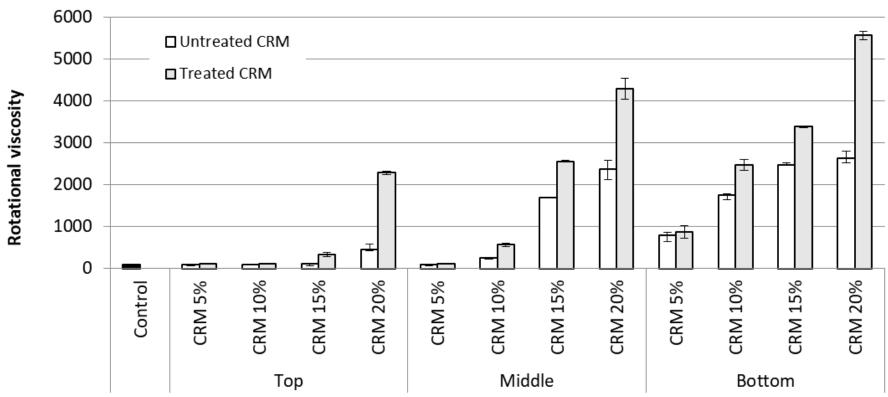

Figure 5. Viscosity at 180 °C of the CRM binders of top, middle and bottom parts after conditioning.

When a one-way ANOVA test was performed with a 95% confidence interval between the means of the same population of both the untreated and treated CRMA binder, considering only the upper and lower parts after the conditioning period, it was found that there was no significant difference between the statistics average to 15% or more in untreated binder and 20% in the treated binder in the top and bottom. Viscosity values were found to be significant between the untreated and treated binder. This means that the statistical analysis follows the above bar chart result. A statistical analysis of the results of viscosity of both untreated and treated CRMA binders as a function of tops and bottoms with a 95% confidence interval is shown in Table 3.

Table 3. Statistical analysis results of the viscosity at 135 °C of CRM binders as a function of top and bottom parts (α = 0.05).

Viscosity (135 °C)			**Untreated CRM**												**Treated CRM**											
			Original (%)				**Top (%)**				**Bottom (%)**				**Original (%)**				**Top (%)**				**Bottom (%)**			
			5	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**
Untreated ´CRM	Original (%)	5	-	N	S	S	N	N	N	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		10		-	S	S	N	N	N	S	S	S	S	S	N	N	S	S	N	N	S	S	S	S	S	S
		15			-	S	S	S	S	N	S	S	S	S	S	N	N	S	S	S	N	S	S	S	S	S
		20				-	S	S	S	S	N	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
	Top (%)	5					-	N	N	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		10						-	N	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		15							-	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		20								-	S	S	S	S	S	N	N	S	S	S	N	S	S	S	S	S
	Bottom (%)	5									-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		10										-	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		15											-	S	S	S	S	S	S	S	S	S	S	S	S	S
		20												-	S	S	S	S	S	S	S	N	S	S	S	S
Treated CRM	Original (%)	5													-	S	S	S	N	N	S	S	S	S	S	S
		10														-	N	S	S	S	N	S	S	S	S	S
		15															-	S	S	S	N	S	S	S	S	S
		20																-	S	S	S	S	S	S	S	S
	Top (%)	5																	-	N	S	S	S	S	S	S
		10																		-	S	S	S	S	S	S
		15																			-	S	S	S	S	S
		20																				-	S	S	S	S
	Bottom (%)	5																					-	S	S	S
		10																						-	S	S
		15																							-	N
		20																								-

S—Significant; N—Non-significant.

3.2. *Viscoelasticity by DSR Method*

The G*/sinδ property values from this test are generally used to analyze the permanent deformation or plastic deformation or rutting property or viscoelastic property when it is susceptible to the load of 1.00 kPa for a cycle of 10 rad/s at different temperatures. Figure 6 illustrates the experimental G*/sinδ values of both untreated and treated CRMA at various temperatures (64 °C, 70 °C, and 76 °C) after modification, using different proportions of replacement CRMA binders. It was noticed that when both treated and untreated CRMA binders were subjected to a higher replacement content of crumb rubber, its resistance to permanent deformation increased; especially, treated crumb rubber showed superior resistance to permanent deformation. In addition, a rise in temperature had a direct impact on resistance to permanent deformation. There is evidence that a high PG-76 can be obtained with an even much lower percentage of treated rubber compared to a higher percentage of untreated rubber.

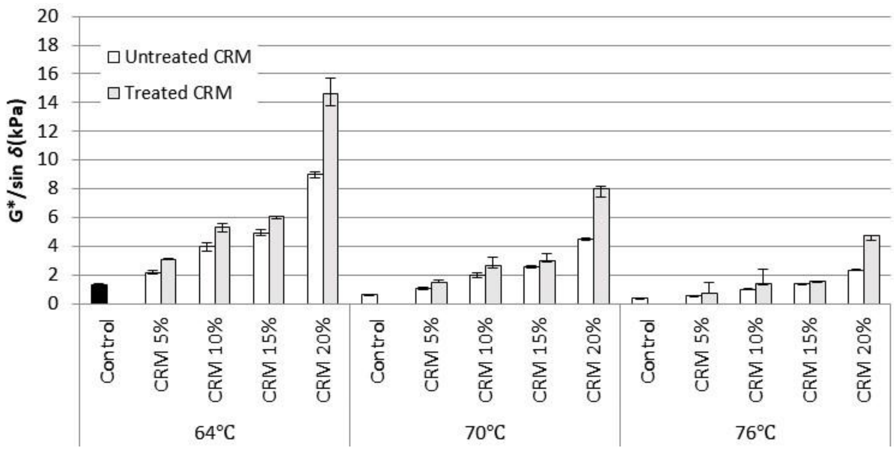

Figure 6. G*/sinδ values of the CRM asphalt binders at 64 °C, 70 °C and 76 °C.

The permanent deformation values of the top, middle, and bottom portion of CRMA binder with both untreated and treated rubber after the conditioning period measured at different temperatures ranging from 64 °C, 70 °C, and 76 °C is shown in the figure (Figures 7–9). As expected, the measured value decreased independently of the CRMA content with the increasing test temperature in both treated and untreated rubber. In addition, G*/sinδ increased at all test temperatures as the rubber particle content increased. However, the data for the top, middle, and bottom showed a different trend compared to the original CRM binders before conditioning. First, the upper G*/sinδ value maintained a similar level up to the 15% crumb rubber content in untreated rubber and then increased more than twice at 20%, and in the case of treated rubber, the values were found almost similar until 5% and increased almost four times even at high temperature of 76 °C. In the middle part, the value was found both for treated and untreated crumb rubber up to 5%, similar to the top part, but the value increased from 10% CRMA, and the value was about three times as high as the top part at 15% or more, and treated rubber witnessed superior values compared with untreated rubber. The lower part showed a higher performance rating (PG) of 76 or higher in both untreated and treated CRMA, which could be obtained when the rubber content was added even at percentages less than 5%, and thereafter, the value showed a tendency to increase gradually. The treated rubber witnessed almost more than a 100% increase up to 15% CRMA and almost more than 400% at 20% content at the higher temperature of 76 °C.

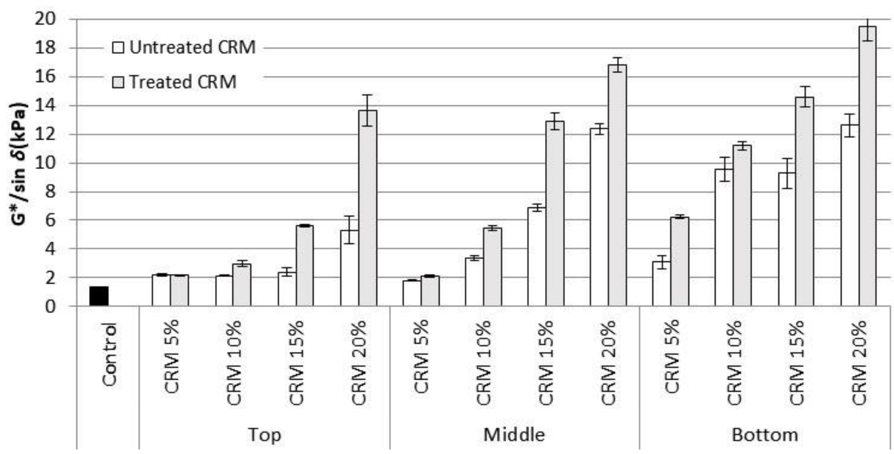

Figure 7. G*/sinδ at 64 °C of the CRM asphalt binders of top, middle and bottom parts after conditioning.

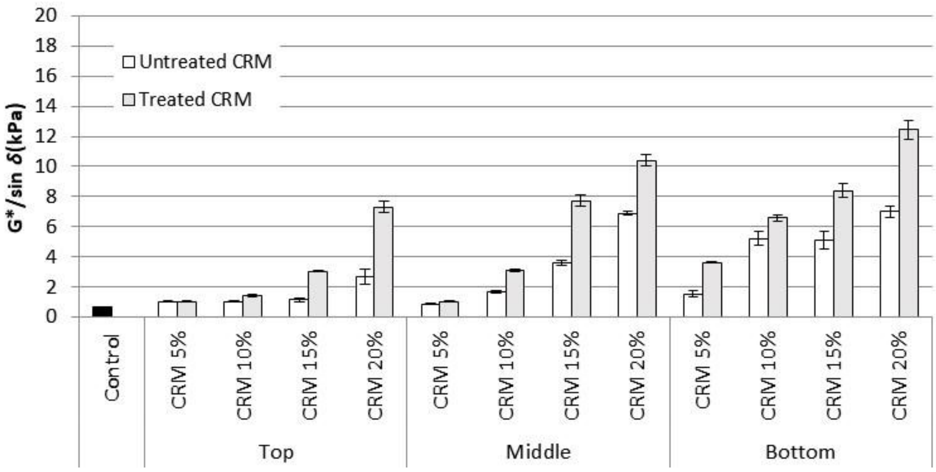

Figure 8. G*/sinδ at 70 °C of the CRM asphalt binders of top, middle and bottom parts after conditioning.

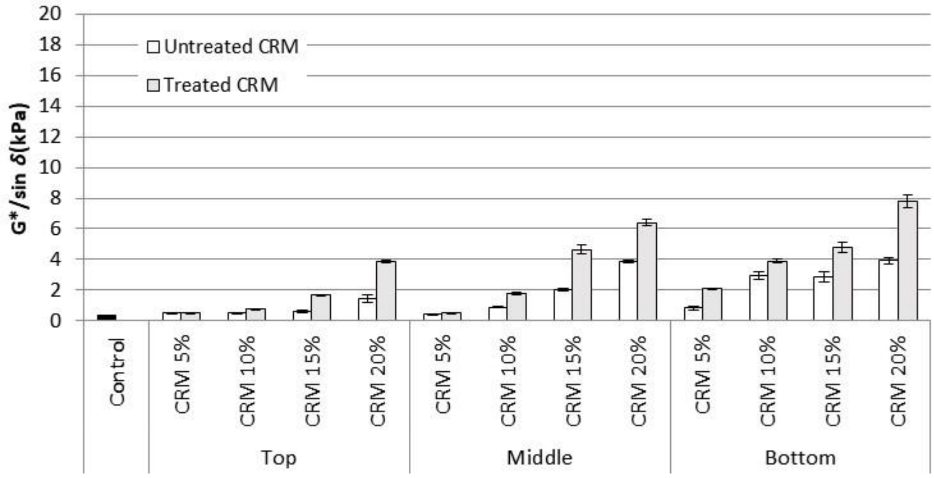

Figure 9. G*/sinδ at 76 °C of the CRM asphalt binders of top, middle and bottom parts after conditioning.

In general, the top, middle, and bottom readings at 70 °C and 76 °C showed the same trend as G*/sinδ at 64 °C.

The statistical importance of the variation in both untreated and treated CRMA content was studied by comparing the baseline caveat to the top, middle, and bottom, utilizing a one-way assessment of variance (Table 4). In general, the important difference within each pure grade was seen similarly at all temperatures, and for this study, only a statistical analysis for 64 °C based on this investigation is shown. The upper part verified that the quantified values were not statistically significant up to a CRM content of 10% compared to the result of 5% CRMA for the original condition. In the lower part, the values of 10%, 15%, and 20% CRMA binders were mostly significant among binders in each part at the 95% confidence level, meaning that the results of the bar chart are proven.

Table 4. Statistical analysis results of the G*/sinδ at 64 °C of CRM binders as a function of top and bottom parts (α = 0.05).

G*/sinδ			**Untreated CRM**												**Treated CRM**											
			Original (%)				**Top (%)**				**Bottom (%)**				**Original (%)**				**Top (%)**				**Bottom (%)**			
			5	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**
Untreated ´CRM	Original (%)	5	-	S	S	S	N	N	N	S	N	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		10		-	S	S	S	S	S	S	N	S	S	S	N	S	S	S	S	S	S	S	S	S	S	S
		15			-	S	S	S	S	N	S	S	S	S	S	N	S	S	S	S	N	S	S	S	S	S
		20				-	S	S	S	S	S	N	N	S	S	S	S	S	S	S	S	S	S	S	S	S
	Top (%)	5					-	N	N	S	N	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		10						-	N	S	S	S	S	S	S	S	S	S	N	N	S	S	S	S	S	S
		15							-	S	N	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		20								-	S	S	S	S	S	N	N	S	S	S	N	S	N	S	S	S
	Bottom (%)	5									-	S	S	S	N	S	S	S	S	N	S	S	S	S	S	S
		10										-	N	S	S	S	S	S	S	S	S	S	S	S	S	S
		15											-	S	S	S	S	S	S	S	S	S	S	S	S	S
		20												-	S	S	S	S	S	S	S	S	S	S	S	S
Treated CRM	Original (%)	5													-	S	S	S	N	N	S	S	S	S	S	S
		10														-	N	S	S	S	N	S	N	S	S	S
		15															-	S	S	S	N	S	N	S	S	S
		20																-	S	S	S	S	S	S	N	S
	Top (%)	5																	-	N	S	S	S	S	S	S
		10																		-	S	S	S	S	S	S
		15																			-	S	N	S	S	S
		20																				-	S	S	S	S
	Bottom (%)	5																					-	S	S	S
		10																						-	S	S
		15																							-	S
		20																								-

S—Significant; N—Non-significant.

3.3. Multi-Stress Creep and Recovery Test

MSCR (Test Method D7405) is an alternative test to the DSR test method used for storage stability. The MSCR test was performed at 64 °C and 76 °C according to AASHTOTP 70 by loading at 3.2 kPa to evaluate the viscoelastic properties of both untreated and treated CRMA binders under more extreme conditions than the DSR test. Figure 10 shows the results of J_{nr} and percent recovery of control CRMA binders. In general, increasing the crumb rubber content allowed a decrease in the J_{nr} and an increase in the % rec, meaning that the higher the CRM content, the higher the viscoelasticity of the binder. More specifically in the Figure 11, the data from 0% and 5% with untreated CRMA levels were not measured at 64 °C due to the low viscoelasticity of the samples, but the 5% treated CRMA binder showed higher J_{nr} and % recovery values. The J_{nr} steadily decreased after the 5% level for treated CRMA binders and up to the 20% level. It was also noted that the untreated CRMA binders also followed a similar trend but showed higher values at similar levels, except that at 5% and at 20%, the values for both treated and untreated CRMA were found to be similar. In addition, when the test temperature was increased to 76 °C, the data were only measured from the 15% CRM binder since the binders softened at higher temperatures as shown in the Figure 12. The value of 20% CRMA binder was only measured, and untreated CRMA binders exceeded the values of treated CRMA binders by a factor of two. In the % recovery study, both treated and untreated CRMA binders showed a similar trend at 64 °C, with the exception of crumb rubber at 20%, where the % recovery values of treated CRMA were comparatively better than untreated CRMA binders, but cut at a higher temperature, both treated and untreated CRMA is similar.

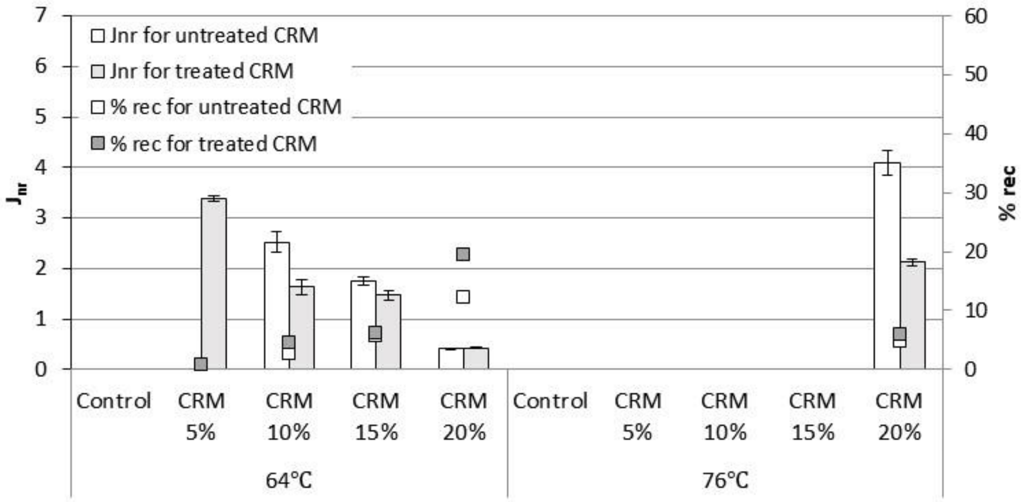

Figure 10. J_{nr} and % rec of the CRM asphalt binders at 64 °C and 76 °C.

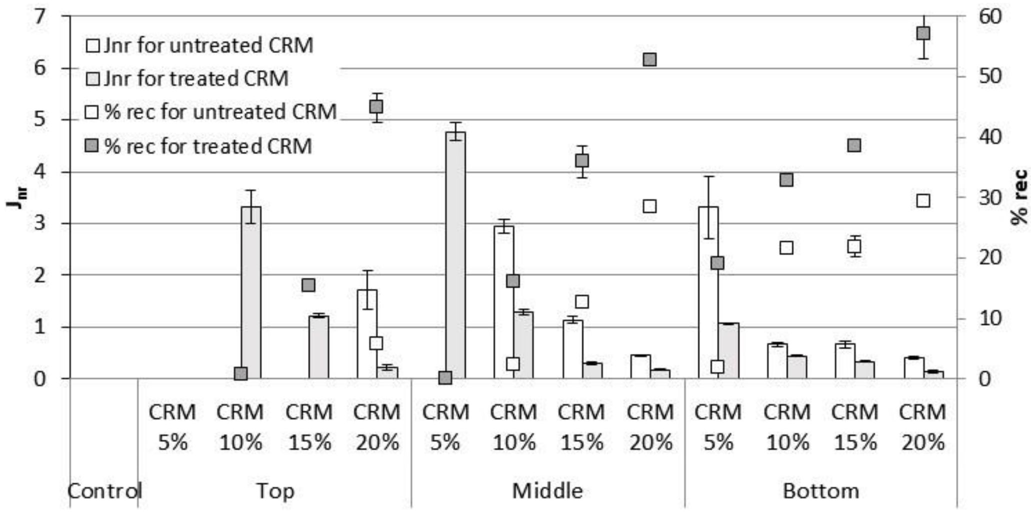

Figure 11. J_{nr} and % rec at 64 °C of the CRM asphalt binders of top, middle and bottom parts after conditioning.

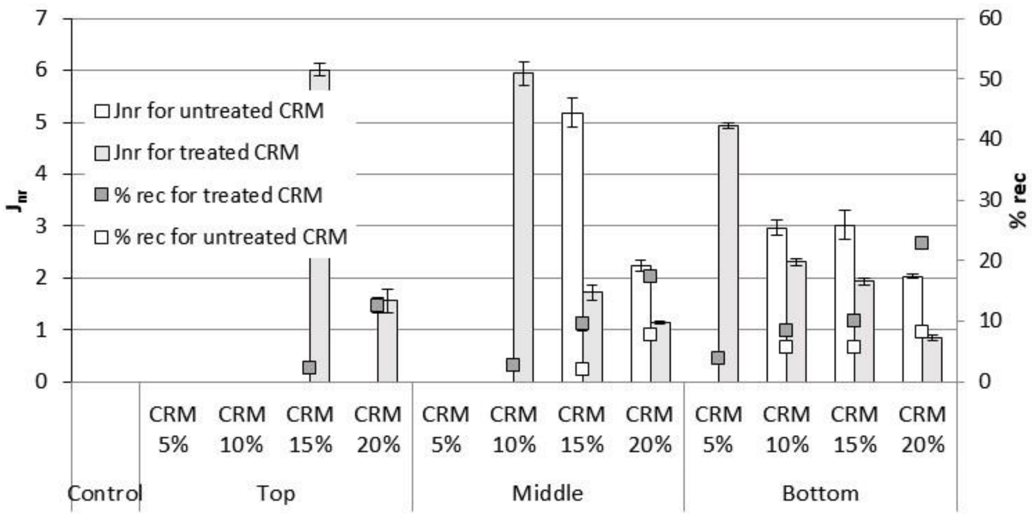

Figure 12. J_{nr} and % rec at 76 °C of the CRM asphalt binders of top, middle and bottom parts after conditioning.

Using a one-way analysis of variance, the statistical significance of the change in J_{nr} and % rec was examined for both treated and untreated CRMA binders, comparing the original state to the top and bottom portions (Tables 5 and 6). In general, the original condition J_{nr} values from the MSCR test were found to be 64 °C for untreated rubber, which were most significantly different compared to treated rubber depending on the crumb rubber levels. For the conditioned CRMA binders of the top and bottom pieces, a significant difference was observed within each piece compared to the original condition from top to bottom. In the case of statistical analysis for J_{nr} at 76 °C, due to the unmeasured results, there was an insignificant difference within each part of the original, top, and bottom. The % rec case showed a similar trend as the J_{nr} analysis.

Table 5. Statistical analysis results of the Jnr of CRM binders as a function of top, middle, and bottom parts (α = 0.05).

J_{nr}			**Untreated CRM**												**Treated CRM**											
			Original (%)				**Top (%)**				**Bottom (%)**				**Original (%)**				**Top (%)**				**Bottom (%)**			
			5	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**
Untreated ′CRM	Original (%)	5	-	S	S	S	N	N	N	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		10		-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		15			-	S	S	S	S	N	S	S	S	S	S	N	S	S	S	S	S	S	S	S	S	S
		20				-	S	S	S	S	S	N	N	N	S	S	S	N	S	S	S	N	S	N	N	N
	Top (%)	5					-	N	N	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		10						-	N	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		15							-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
		20								-	S	S	S	S	S	N	N	S	S	S	S	S	S	S	S	S
	Bottom (%)	5									-	S	S	S	N	S	S	S	S	N	S	S	S	S	S	S
		10										-	N	N	S	S	S	N	S	S	S	S	S	N	S	S
		15											-	N	S	S	S	N	S	S	S	S	S	N	S	S
		20												-	S	S	S	N	S	S	S	N	S	N	N	N
Treated CRM	Original (%)	5													-	S	S	S	S	N	S	S	S	S	S	S
		10														-	N	S	S	S	S	S	S	S	S	S
		15															-	S	S	S	N	S	S	S	S	S
		20																-	S	S	S	N	S	N	N	N
	Top (%)	5																	-	S	S	N	S	S	S	N
		10																		-	S	S	S	S	S	S
		15																			-	S	N	S	S	S
		20																				-	S	N	N	N
	Bottom (%)	5																					-	S	S	S
		10																						-	N	S
		15																							-	N
		20																								-

S—Significant; N—Non-significant.

Table 6. Statistical analysis results of the % rec of CRM binders as a function of top, middle, and bottom parts (α = 0.05).

% rec			Untreated CRM												Treated CRM											
			Original (%)				**Top (%)**				**Bottom (%)**				**Original (%)**				**Top (%)**				**Bottom (%)**			
			5	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**	**5**	**10**	**15**	**20**
Untreated 'CRM	Original (%)	5	-	S	S	S	N	N	N	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		10		-	S	S	S	S	S	S	N	S	S	S	S	S	S	S	S	N	S	S	S	S	S	S
		15			-	S	S	S	S	N	S	S	S	S	S	S	N	S	S	S	S	S	S	S	S	S
		20				-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
	Top (%)	5					-	N	N	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		10						-	N	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		15							-	S	S	S	S	S	N	S	S	S	N	N	S	S	S	S	S	S
		20								-	S	S	S	S	S	N	N	S	S	S	S	S	S	S	S	S
	Bottom (%)	5									-	S	S	S	N	S	S	S	S	N	S	S	S	S	S	S
		10										-	N	S	S	S	S	S	S	S	S	S	S	S	S	S
		15											-	S	S	S	S	S	S	S	S	S	S	S	S	S
		20												-	S	S	S	S	S	S	S	S	S	S	S	S
Treated CRM	Original (%)	5													-	S	S	S	N	N	S	S	S	S	S	S
		10														-	N	S	S	S	S	S	S	S	S	S
		15															-	S	S	S	S	S	S	S	S	S
		20																-	S	S	S	S	N	S	S	S
	Top (%)	5																	-	N	S	S	S	S	S	S
		10																		-	S	S	S	S	S	S
		15																			-	S	S	S	S	S
		20																				-	S	S	S	S
	Bottom (%)	5																					-	S	S	S
		10																						-	S	S
		15																							-	S
		20																								-

S—Significant; N—Non-significant.

3.4. Separation Index/Phase Separation Results

To study the storage phase separation of untreated and treated CRMA binders, viscosity values from the rotational viscometer test and $G^*/\sin\delta$ of the top and bottom parts after conditioning are considered for this separation index study. The percent separation index of both untreated and treated CRMA at temperatures of 135 °C and 180 °C is shown in Table 7. At 135 °C, the rejection percentage of the 5% and 20% crumb rubber content is lower compared to the 10% and 15% crumb rubber substitute. In untreated rubber and on the other hand, the content in treated rubber with 15% and 20% was not measurable, since their viscosity values were found to be high. At an elevated temperature of 180 °C, the treated modified CRMA binders had less phase separation compared to untreated rubber, and the 20% replacement rubber crumb content in both untreated and treated CRMA performed better in this study compared to others.

Table 7. Separation index from viscosity of CRM binders.

Binder			Viscosity		
			Top	**Bottom**	**% Separation**
135 °C	Untreated	CRM 5%	574.8	3162	69%
		CRM 10%	637.5	7968.5	85%
		CRM 15%	693.5	9879.5	87%
		CRM 20%	2193.5	11,323.5	68%
	Treated	CRM 5%	624.8	5259	79%
		CRM 10%	756.0	12287.5	88%
		CRM 15%	2031.0	None.	None.
		CRM 20%	11,149.5	None.	None.
180 °C	Untreated	CRM 5%	86.25	793.5	80%
		CRM 10%	93.75	1756	90%
		CRM 15%	112	2478.5	91%
		CRM 20%	449.5	2628.5	71%
	Treated	CRM 5%	112.5	871.5	77%
		CRM 10%	106	2474.5	92%
		CRM 15%	324.75	3381	82%
		CRM 20%	2282.5	5562	42%

Table 8 presents the G*/sinδ values of phase separation observed in CRMA binders at 64 °C. It was attested by this research that the CRMA binders at 5% and 20% grade had less phase separation compared to those at 15% and 20% grade in untreated rubber, which is similar to the viscosity study at 135. The treated rubber-modified asphalt, on the other hand, had less phase separation at 20% grade compared to the other substitute content of crumb rubber.

Table 8. Separation index from G*/sinδ of CRM binders.

Binder		G*/sinδ at 64 °C		
		Top	**Bottom**	**% Separation**
Untreated	CRM 5%	2.17	3.08	17%
	CRM 10%	2.13	9.53	63%
	CRM 15%	2.38	9.29	59%
	CRM 20%	5.31	12.6	41%
Treated	CRM 5%	2.16	6.23	48%
	CRM 10%	2.97	11.2	58%
	CRM 15%	5.62	14.6	44%
	CRM 20%	13.63	19.47	18%

4. Conclusions

To investigate the phase separation problems found during storage in regard to the stability of treated and untreated 5%, 10%, 15%, and 20% CRMA binders, the binders were oven conditioned for 48 h at 163 °C. The tests were undertaken using the rotational viscometer and the Dynamic Shear Rheometer to ascertain the properties and separation index (SI) of CRMA binders. From these results, the following outcomes were drawn for storage consistency in this study.

1. The addition of CRM increased viscosity at 135 °C and 180 °C for both treated and untreated CRMA. The conditioned CRMA binders showed higher viscosity in the bottom portion compared to the middle and top portions, which is due to the movement of rubber particles during conditioning. Treated rubber exhibited higher viscosity values than untreated CRMA, indicating the importance of high temperature in maintaining the performance of treated CRMA.

2. Increasing crumb rubber content led to higher $G^*/\sin\delta$ values, but with increasing temperature, $G^*/\sin\delta$ values decreased similar to the viscosity study. Treated rubber-modified binder had higher values compared to untreated CRMA, indicating better resistance to permanent deformation.

3. From the MSCR test, it is observed that the J_{nr} and % rec values show a similar trend with the $G^*/\sin\delta$ results. However, some data were not quantified due to the higher load than the DSR test.

4. Phase separation was significantly affected by temperature, with increasing temperature resulting in more severe phase separation. Treated rubber mixed with fresh asphalt binder had lower phase separation values compared to untreated CRMA at a replacement extent of 20%.

5. The statistical data obtained aligns with the findings shown in the bar chart, indicating an increasing separation index of the binder as the data illustrate the difference between each sample.

6. The study found a discrepancy in the SI value between the test method proposed in ASTM D7173 and the viscosity test method. As a result, it is recommended to reevaluate the SI value using various experimental methods for asphalt binders that incorporate different modifiers. This suggests the need for a comprehensive assessment of the SI value in conjunction with other relevant testing techniques to accurately evaluate the SI value of asphalt binders with different modifiers.

7. In future research, it is advisable to explore the use of various additives aimed at enhancing the storage stability of asphalt binders. Additionally, a comprehensive evaluation of asphalt mixtures based on the storage stability should be conducted, taking into account their rutting and cracking performance, to ensure practical application.

Author Contributions: Formal analysis, S.V.; Investigation, J.Y.; Supervision, S.-J.L.; Project administration, M.-S.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by a grant from a government funding project (2023 National Highway Pavement Management System).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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