

LABORATORY EVALUATION OF STORAGE STABILITY
FOR CRM ASPHALT BINDERS

by

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DEDICATION

I would like to dedicate this dissertation to God, my family.

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First of all, through the many events that occurred in the two or three years that God prepared, I knew that my life was moving in God's plan and that he wanted to bring me closer to you. Therefore, I want to give all thanks to God.

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ABSTRACT

This study was performed to quantify a storage stability depending on CRM binder content. The percentages of crumb rubber used for CRM binder were 0%, 5%, 10%, 15% and 20%. According to ASTM D7173, the CRM binder sample was prepared to pour into the vertically held aluminum tube. This tube was conditioned in a vertical position for 48 hours at a temperature of $163 \pm 5^\circ\text{C}$ and placed immediately in the freezer at $-10 \pm 10^\circ\text{C}$ for at least 4 hours to completely solidify the sample. Finally, test specimens were arranged into three parts of approximately equal length. In order to evaluate the properties of each part of binders, tests were carried out through the rotational viscosity and viscoelasticity, and the separation index (SI) was assessed with the $G^*/\sin \delta$ and % rec. In general, the results of this study revealed that (1) the conditioned CRM binders are found to have higher viscosity in the bottom part compared to the middle and top parts, as expected; (2) similar to the viscosity results, the CRM binders after conditioning showed the highest $G^*/\sin \delta$ value in the bottom part; (3) the SI from $G^*/\sin \delta$ generally increased as the test temperature increased; (4) the SI increased up to 10% CRM, and then decreased as the CRM content was further increased; (5) the SI from $G^*/\sin \delta$ was suitable for evaluating the storage stability of CRM asphalt binders, compared to the SI from % rec.

1. INTRODUCTION

Background

With the high demand for improvement in the pavement industry to resist aggravative axis loads and prevent damages like rutting and cracking (Wang et al. 2019), polymer modifiers application is growing. Asphalt binder modification via different polymers has been contemplated to enhance pavement durability (Ren et al. 2020). Common modifiers include crumb rubber modifier (CRM), styrene-butadiene-rubber (SBR), and styrene-butadiene-styrene (SBS), which are considered because of their efficient adhesive and cohesive performance (Yu et al. 2018) and can improve the resistance of the asphalt pavements against defects such as cracking and rutting under traffic loading. Since CRM has the reasonable performance to enhance the resistance of the asphalt binder while reducing the sound of the asphalt pavement, it is more and more popular to be applied as an asphalt modifier.

Workability and storage stability of modified asphalt binders is a concern during application caused by the interaction between polymers and bitumen liquid phase. (Akisetty et al. 2009; Yu et al. 2013). Asphalt is structured out of continuous three-dimensional polar molecules spread in a fluid of nonpolar or low polar molecules (Wekumbura et al. 2007). During the mixing process of asphalt modification, the absorption of the low molecular weight oil fraction of base asphalt by polymer strands occurs (Ragab and Abdelrahman 2018). Based on weak physical interactions and nonchemical interactions between polymer modifiers and asphalt binders, the modified asphalt binders perform poor storage stability at high temperatures (Perez et al. 2007). The separation concern tends to occur in elevated temperatures and causes varying

polymer concentrations in asphalt. Comparing the density of the modifiers and virgin asphalt, CRM is likely the most susceptible polymer, which tends to sink in the liquid phase of asphalt. In contrast, SBR and SBS tend to be floating according to their lower density (Ren et al. 2020). Considering the compaction and mixing temperature of the CRM asphalt binders compared to the natural asphalt binders, the chance of separation rises significantly (Wang et al. 2020). The separation between asphalt and modifier, which may occur after storage, affects the rheological and composition of asphalt binders' top and bottom portions. Consequently, transportation and pumping through to the pipelines will suffer difficulties, and the final product would last less than expected.

Many factors affect the storage stability of the CRM binders, such as density, concentration, additives, and bitumen characteristics (Sienkiewicz et al. 2017). Besides preparation condition controlling, there are generally two concepts to improve the storage stability of CRM asphalt. First method is adding various chemical compounds into CRM asphalt binder to enhance the bonding forces between the binder and polymer networks (Sienkiewicz et al. 2017). The second option is treating the CRM surface to achieve desired interaction between CRM and the asphalt binder network (Hosseinnezhad et al. 2019; Xiao et al. 2020). Thermomechanical, thermochemical, biological, plasma and microwave are ways to treat the CRM surface.

Scope of Study

The objective of this study is to quantify the storage stability properties of CRM asphalt binders. According to the rubber content used and the test temperature, in order to check how the storage stability changes, CRM binders were prepared using five different contents (0%, 5%, 10%, 15% and 20%), and the properties after treatment were evaluated based on the viscosity, $G^*/\sin \delta$, J_{nr} and % recovery.

2. LITERATURE REVIEW

Crumb Rubber

With the significant increase in the number of automobiles worldwide, the accumulation of huge amounts of scrap tires has become a problem in waste management. (Najim and Hall 2010). For example, in the United States, the scrap tires are about 300 million, and 66 million scrap tires were utilized as ground tires. Also, 11 million ground tires were consumed for the asphalt mixture (U.S. tire manufacturers association 2019). Many governments around the world are trying to reuse this industrial by-product as alternative energy. The general compositions for tires are natural and synthetic rubbers, carbon black, metal, textile, and additives. These potential elements can be reutilized to yield other materials by preventing negative impact on the environment caused by tire incineration (Nehdi and Khan 2001). The most beneficial one of the use of by-products from industry is its environmental value as an alternative to concrete by decreasing landfills. In case of negative impact for landfills, Landfills release harmful chemicals to the environment and emit methane gas, and even more, can kill beneficial bacteria for soil. These efforts to use scrap tires will not only profit governments in reducing landfills but will also increase economic growth in various sectors, particularly the construction industry (Noor 2015). Crumb rubber is manufactured by two main methods: room temperature grinding and cryogenic grinding. Each method can produce crumb rubber of similar particle size. However, the main difference between them is the particle surface texture. Crumb rubber particles from room temperature processing have irregular shapes with rough texture due to the tearing and shredding action of rubber particles in the cracker mills. On the other hand, the

crumb rubber particles produced by the cryogenic method have a smooth surface and resemble shattered glass. This difference in particle surface texture results in surrounding particles having a larger surface area than cryogenic crumb rubber (Putman 2005).

CRM Asphalt Binder

In the 1840s, the earliest experiments involved the incorporation of natural rubber into asphalt binders to improve the property of engineering performance. The asphalt modification with natural and synthetic rubbers was introduced as early as 1843 (Thompson and Hoiberg 1979). In 1923, the modification with natural and synthetic rubber in asphalt were more improved (Isacsson and Lu 1999; Yildirim 2007). The first attempt by Guedmberg to modify asphalt binders by adding rubber was made in 1898 at France (Mahrez 1999). Over time, the use of crumb rubber for mixing asphalt mixture is divided into two processes. The two processes used in designing and constructing an asphalt-rubber mixture are referred to as "wet" and "dry" processes. In the wet process, rubber is added to the asphalt binder. In the dry process, crumbed rubber is added to the asphalt mixture (Amirkhanian 1993; Kim et al. 2014). In the early 1960s, Charles Mc Donald completed mixing the crumb rubber and virgin asphalt binder as a wet process and allowed it to mix for 45-60 minutes. The rubber particles expanded in size at higher temperatures, resulting in a higher concentration of liquid asphalt in the asphalt mixture (Huffman 1980). Application of rubber modified asphalt mixture began in Alaska in 1979 with plus ride dry mix which is the trade name of the mix marketed under patent by the Swedish companies Skega AB. Between 1979 and 1981, seven rubber pavement layouts were reported for a total of 4 lane km. Moreover, asphalt rubber using the wet method was first applied in Alaska in 1988 (Raad and Saboundjian 1998; Esch 1982). Lundy et

al. (Lundy 1993) published three case studies using crumb rubber in both wet and dry processes. As a result, the material showed excellent resistance to thermal cracking even after 10 years. Moreover, another advantage of using asphalt rubber is that it can extend the service life. (Huang et al. 2007). The advantages of using crumb rubber modified asphalt are lower sensitivity to daily changing temperatures and are rutting and aging resistance, and bonding property between aggregate and binder. Since then, by reason of the various benefits of using crumb rubber into the asphalt binder, the use of crumb rubber has drawn interest in modifier for asphalt binder (Brown et al. 1997; Maupin 1996; Charania et al. 1991; Adhikari et al. 2000).

Storage Stability

The problem of phase separation goes back to the earliest descriptions of Polymer Modified Asphalt (PMA). In 1980, Kraus and Rollman (Kraus and Rollmann 1981) described the biphasic properties of asphalt/SBS mixtures and were probably the first to introduce the concepts of an Asphaltene-rich phase and a polymer-rich phase. The tendency of these two phases to become macroscopically separated during high-temperature storage has been known since the first studies of PMA. However, the interpretation of gravitational action is very recent. (Lu et al.1999). Storage stability can somehow be microscopically characterized for compatibility, which is certainly the most important requirement of PMA technology. The difference between Polymer-Rich Phase (PRP) and Asphaltene-Rich Phase (ARP) is oriented towards possible tests to confirm and quantify these phenomena. In order to confirm the segregation index, the first step is to simulate hot storage and a second step is to verify if and how much a separation took place (Polacco et al. 2015). In 1995, testing and evaluation of polymer modified bitumen

was reported and described three methods for testing compatibility and storage stability. However, none of them were standardized. The first is called the "tube test", which has survived to this day and is now included in the specifications in several countries (Isacsson and Lu 1995). The simple idea of a tube test is to place a sample of modified asphalt in a hot vertical container (tube) for a specific period of time. This simulates high temperature storage without agitation and shear where PRP and ARP can be separated. The lighter PRP stage moves to the upper part of the tube and the ARP settles to the lower part. The tube is then removed from the oven and cooled to room temperature, keeping the tube vertical. Finally, the sample is horizontally cut into three equal sections. The characteristics of the upper and lower parts are tested and compared, but the middle part is removed. In the last few years, various testing procedures were suggested with different storage periods and temperatures. In the first one to evaluate the asphalt binder, the testing method was visually conducted, determining the viscosity difference and flow of the materials. In the second procedure, the compatibility is then assessed by comparing the softening point and/or penetration of the top and bottom parts. It is increasingly common practice to evaluate both parts in terms of their rheological properties as an alternative to softening point and penetration. The Strategic Highway Research Program (SHRP) specification introduces the following "separation index":

$$Separation\ index = \log \frac{(|G|_b^*)}{(|G|_t^*)} \quad (1)$$

where $|G|_b^*$ and $|G|_t^*$ are the complex moduli at 25 °C and frequency of 10 rad/s of the bottom and top phases, respectively. Analogously, the viscosity or the ratio $|G^*|/\sin \delta$ (where $|G^*|$ is the complex modulus and δ is the phase angle), also taken from the SHRP specifications, can be used. For example, the latter is used (Kim and Lee 2013), which

defines the percentage of separation as the ratio where $(|G^*|/\sin \delta)_{\max}$ represents the higher value of either the top or bottom section of the tube and $(|G^*|/\sin \delta)_{\text{avg}}$ is the average value of both sections.

3. EXPERIMENTAL DESIGN

Methods

Chapter 3 provides the experimental procedures included in this study and the description of the materials used to achieve the research objectives. Figure 1 shows the flow chart of the experimental design applied in this study. This chart presents the overall research process to assess the storage stability of CRM asphalt binders. The summary below shows full names and the abbreviation used in this study.

PG: Performance Grade

64-22: The binder meets the high temperature properties up to 64°C and low temperature properties down to -22°C

CRM: Crumb Rubber Modified

MSCR: Multiple Stress Creep Recovery

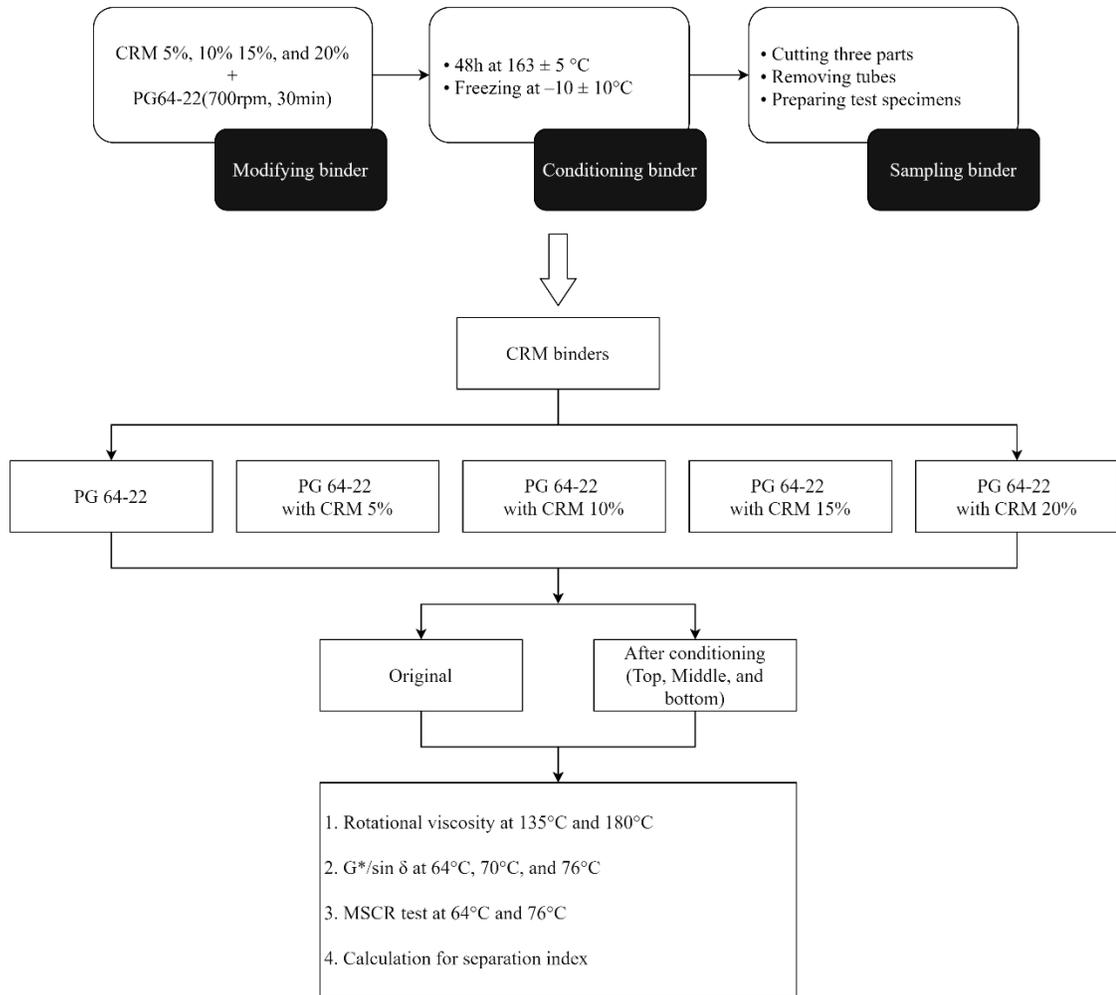


Figure 1. Experimental Design

Material

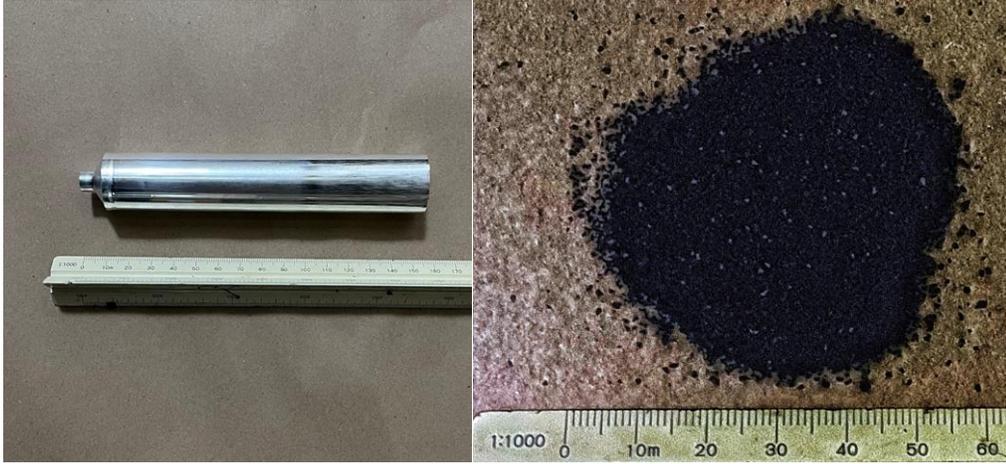
In this study, performance grade (PG) 64-22 asphalt binder was used to make crumb rubber modified (CRM) binders. Table 1 shows the properties of the asphalt binder. Crumb rubber is used in order to modify the binder. Table 2 shows the gradation of material. For the storage stability evaluation, the aluminum tube suggested in ASTM D7173 was used. The aluminum tube and crumb rubber were shown in Figure 2.

Table 1. Properties of Base Asphalt Binder (PG 64-22)

Aging states	Test properties	Test result
Unaged binder	Viscosity @ 135°C (cP)	538
	$G^*/\sin \delta$ @ 64°C (kPa)	1.38
RTFO aged residual	$G^*/\sin \delta$ @ 64°C (kPa)	3.82
RTFO+PAV aged residual	$G^* \sin \delta$ @ 25°C (kPa)	4402
	Stiffness @ -12°C (MPa)	205
	m-value @ -12°C	0.323

Table 2. Gradation of CRM

Sieve Number (μm)	% Cumulative Passed
30 (600)	100
40 (425)	91.0
50 (300)	59.1
80 (180)	26.2
100 (150)	18.3
200 (75)	0.0



(a) **(b)**
Figure 2. Aluminum Tube (a) and Crumb Rubber (b)

Production of CRM Asphalt Binders

CRM asphalt binder was produced through the wet process in the laboratory at 177°C for 30 minutes by an open blade mixer at a blending speed of 700 rpm (Lee et al. 2007; Lee et al. 2006; Shen et al. 2006). The percentages of crumb rubber used for rubberized binder were 5%, 10%, 15% and 20% by weight of the base binder. In order to ensure that the consistency of the CRM asphalt binder was maintained throughout the study, only one batch of crumb rubber was used in this study.

Preparation of Test Specimens

The CRM binder sample was thoroughly stirred and poured 50 ± 0.5 g into the vertically held tube according to ASTM D7173 (Figure 3). The modified binder in a sealed aluminum tube was treated in a vertical position for 48 hours at a temperature of 163 ± 5 °C. At the end of the treating period, the tube was removed from the oven and placed immediately in the freezer at -10 ± 10 °C, taking care to keep the tube in a vertical position at all times. This tube was placed in the freezer for at least 4 hours to completely solidify the sample. After hardening the tube, the tube was removed from the freezer and placed on a hard, flat surface, then cut into three parts of approximately equal length. Each asphalt binder was placed in the 163 ± 5 °C ovens until the asphalt is fluid enough to remove the pieces of aluminum tube, but no more than 30 minutes. Finally, test specimens were prepared for each test.



Figure 3. Tube Positioning after Conditioning

Rotational Viscosity

In order to evaluate the basic property of CRM binders, A Brookfield rotational viscometer was utilized to measure the viscosity at 135°C and 180°C by applying 27 cylindrical spindles and a constant speed of 20 rpm with a weight of 10.5 g of the binder sample. The time taken to acquire data was considered 20 minutes for each sample.

Figure 4 shows a set of rotational viscometer.

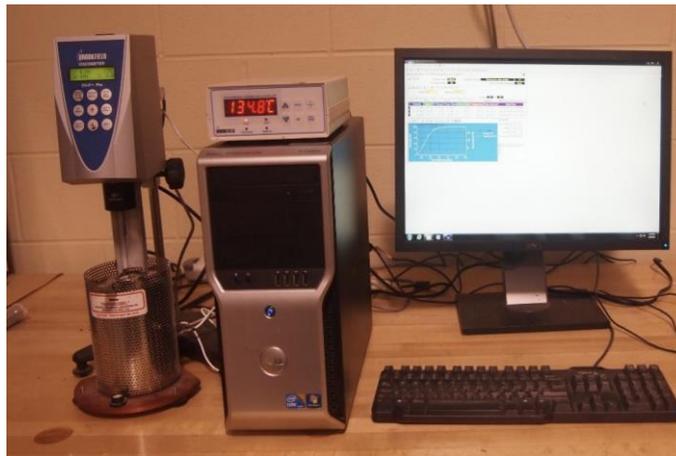


Figure 4. Rotational Viscometer

Viscoelasticity

To measure the viscoelasticity of the asphalt binder, dynamic shear rheometer (DSR) was used to result in $G^*/\sin \delta$, J_{nr} , and % rec. $G^*/\sin \delta$ was calculated from the complex shear modulus (G^*) and the sine (δ) of the phase angle at 64°C, 70°C and 76°C temperatures. The multi-stress creep and recovery (Test Method D7405) test was performed in order to draw J_{nr} and % rec according to AASHTO TP 70, loading 3.2 kPa to evaluate the viscoelasticity of the binder at 64°C and 76°C temperatures. Figure 5 shows a set of DSR.



Figure 5. Dynamic Shear Rheometer

Separation Index (SI)

It is increasingly common practice to evaluate both fractions in terms of their rheological properties from the DSR test as an alternative to softening point and penetration. In this study, $G^*/\sin \delta$ ratio, referencing the Superpave specifications, was used. For instance, the previous research (Kim and Lee 2013) mentioned the ratio, using the equation (2), where $(G^*/\sin \delta)_{max}$ presents the higher value between the top and bottom parts and $(G^*/\sin \delta)_{avg}$ is the average value for both parts. In addition, the SI was calculated and evaluated with the % rec, using the equation (3).

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

$$\textit{Separation index} = \frac{(\% \text{ rec})_{\max} - (\% \text{ rec})_{\text{avg}}}{(\% \text{ rec})_{\text{avg}}} \quad (3)$$

4. RESULTS

Rotational Viscosity

The viscosity of asphalt binder affects the workability to production, delivery and compaction of asphalt mixtures. If the viscosity is too high, it may be difficult to achieve the optimum in-field density, which is also related to pavement performance life. Figure 6 indicates the viscosity values at 135°C and 180°C for the original CRM asphalt binders immediately after blending. It is clear that the viscosity of CRM binders decreased as the testing temperature increased, and as the CRM content increased, the binder viscosity increased, as expected. At 135°C, compared to 0% CRM binder (control), 20% CRM binder showed an increase in viscosity of approximately 7 times. In particular, in the case of 20% CRM binder, it showed a viscosity value higher than 3,000 cP at 135°C, meaning that the production and construction temperatures for 20% CRM mixtures must be increased for proper workability and compaction.

Comparing the viscosity values at 180°C, compared with the control binder, 20% CRM asphalt resulted in a viscosity increase of about 10 times or more. It was found that the viscosity value at 180°C decreased, but the viscosity increase rate was higher compared to 135°C. This result is thought to be because the viscosity of rubber powder is less sensitive to temperature increase.

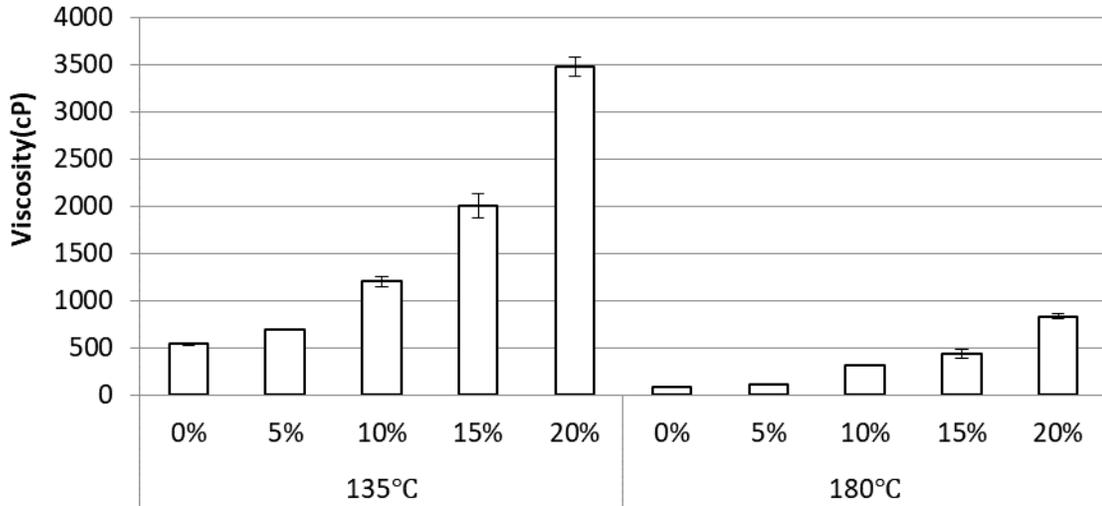


Figure 6. Viscosity of CRM Asphalt Binders at 135°C and 180°C

Figure 7 depicts the viscosity results at 135°C of CRM binders of top, middle and bottom parts after conditioning. The data of the CRM binders after conditioning for 48 hours in an oven at 163°C showed that the bottom part had a higher viscosity than the top and middle parts in all samples. As expected, rubber particles appear to have sunk to the bottom. In the top part, as the CRM content increased, the viscosity value gradually increased (0% CRM binder - 539, 5% CRM - 575, 10% CRM - 638, 15% CRM - 694 and 20% CRM - 2194 cP). Similar to the results in the top part, the middle part showed a tendency to increase in viscosity as the CRM contents increased. In particular, the 15% CRM binder resulted in 11 times higher viscosity, compared to the top part, and the 20% CRM binders showed a viscosity value of 10,000 cP or more.

As for the viscosity of the bottom part, it was confirmed that the viscosity of the 20% CRM binder increased by approximately 21 times compared to that of 0% CRM binder (the viscosity of the CRM binder increased by about 7 times at the same temperature before the conditioning process). This result is considered to have caused the

rubber particles to settle to the middle and bottom during the treatment process, resulting in an increase in viscosity.

Figure 8 shows the results of the viscosity test of the conditioned CRM binders at 180°C. It appeared the lowest value in the top part, and exhibited a tendency to increase in viscosity toward the middle part and the bottom part. In general, in the case of the wet process, the CRM binder is continuously rotated using a propeller until just before making the CRM asphalt mixture in order to prevent the settlement of rubber particles. For this reason, CRM binder should be made immediately prior to the production of CRM mixture in the asphalt plant.

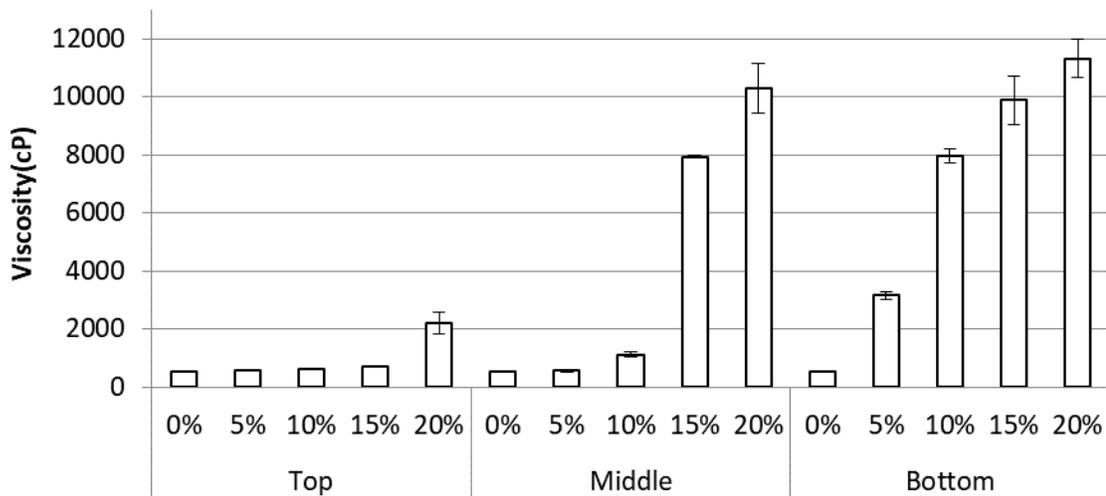


Figure 7. Viscosity at 135°C of CRM binders of Top, Middle and Bottom Parts after Conditioning

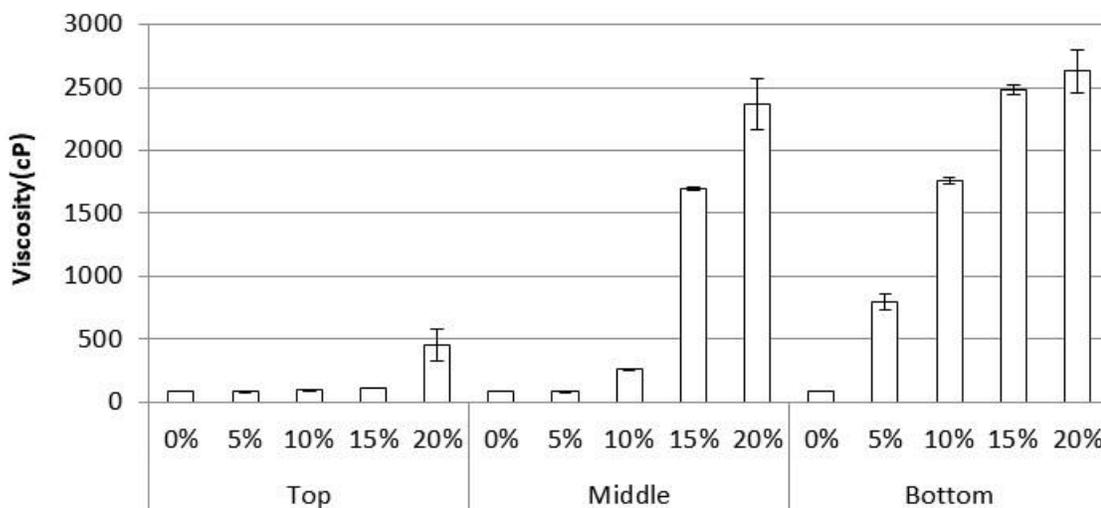


Figure 8. Viscosity at 180°C of CRM Binders of Top, Middle and Bottom Parts after Conditioning

Using one-way analysis of variance, the statistical significance of the CRM binder viscosity values was examined depending on the top, middle and bottom parts after the treatment (Table 3). In the top part, there was no statistically significant difference in viscosity values up to 15% CRM at both temperatures. In the middle part, it was evident that the viscosity values showed a significant difference at 15% or more CRM binders regardless of the measured temperatures. In the bottom part, the viscosity values with statistical significance were confirmed in all CRM contents.

Table 3. Statistical Analysis Results of Viscosity of CRM Binders as a Function of Top, Middle and Bottom Parts ($\alpha=0.05$).

		Viscosity at 135°C																			
		Original (%)					Top (%)					Middle (%)					Bottom (%)				
		0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20
Original (%)	0	-	N	N	S	S	N	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	5		-	N	S	S	N	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	10			-	N	S	N	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	15				-	S	S	S	S	S	N	S	S	N	S	S	S	S	S	S	S
	20					-	S	S	S	S	S	S	S	S	S	S	S	N	S	S	S
Top (%)	0						-	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	5							-	N	N	S	N	N	N	S	S	N	S	S	S	S
	10								-	N	S	N	N	N	S	S	N	S	S	S	S
	15									-	S	N	N	N	S	S	N	S	S	S	S
	20										-	S	S	N	S	S	S	N	S	S	S
Middle (%)	0											-	N	N	S	S	N	S	S	S	S
	5												-	N	S	S	N	S	S	S	S
	10													-	S	S	N	S	S	S	S
	15														-	S	S	S	N	S	S
	20															-	S	S	S	N	S
Bottom (%)	0																-	S	S	S	S
	5																	-	S	S	S
	10																		-	S	S
	15																			-	S
	20																				-
		Viscosity at 180°C																			
		Original (%)					Top (%)					Middle (%)					Bottom (%)				
		0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20
Original (%)	0	-	N	N	S	S	N	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	5		-	N	S	S	N	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	10			-	N	S	N	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	15				-	S	S	S	S	S	N	S	S	N	S	S	S	S	S	S	S
	20					-	S	S	S	S	S	S	S	S	S	S	S	N	S	S	S
Top (%)	0						-	N	N	N	S	N	N	N	S	S	N	S	S	S	S
	5							-	N	N	S	N	N	N	S	S	N	S	S	S	S
	10								-	N	S	N	N	N	S	S	N	S	S	S	S
	15									-	S	N	N	N	S	S	N	S	S	S	S
	20										-	S	S	N	S	S	S	N	S	S	S
Middle (%)	0											-	N	N	S	S	N	S	S	S	S
	5												-	N	S	S	N	S	S	S	S
	10													-	S	S	N	S	S	S	S
	15														-	S	S	S	N	S	S
	20															-	S	S	S	N	S
Bottom (%)	0																-	S	S	S	S
	5																	-	S	S	S
	10																		-	S	S
	15																			-	S
	20																				-

N: non-significant, S: significant

$G^*/\sin \delta$ Property

$G^*/\sin \delta$ values obtained from DSR equipment are most commonly used to evaluate the storage stability of polymer modified asphalt (PMA) binders. Figure 9 illustrates the $G^*/\sin \delta$ values measured immediately after making CRM binders. As expected, the values tended to increase as the CRM content increased. In addition, the value decreased as the testing temperature increased from 64°C to 70°C and to 76°C. It was expected that a high performance grade (PG) of 76 or higher could be obtained when the rubber content was added at more than 10%.

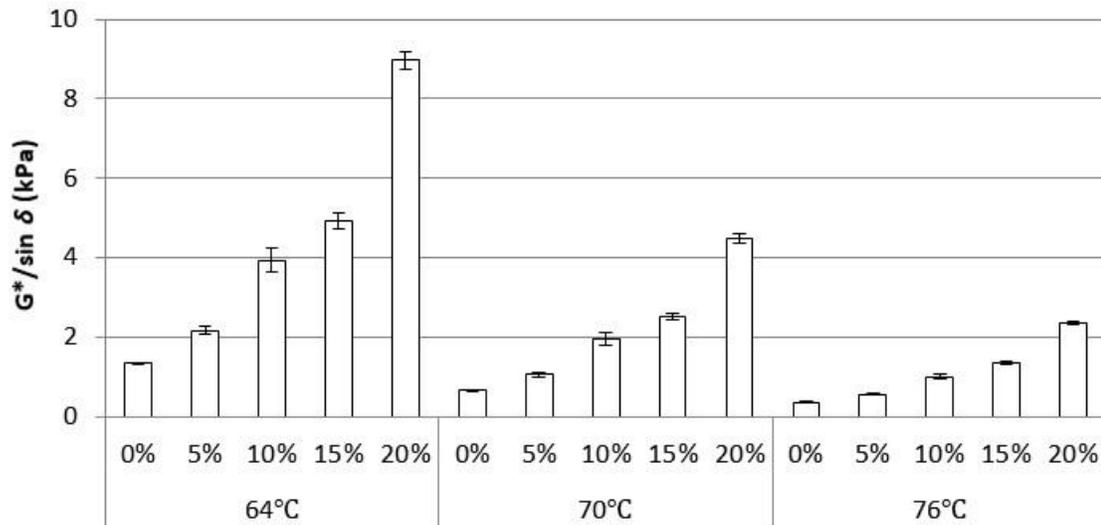


Figure 9. $G^*/\sin \delta$ Values of CRM Asphalt Binders at 64°C, 70°C and 76°C

The $G^*/\sin \delta$ values for the top, middle and bottom parts of CRM binders after conditioning at 163°C for 48 hours were measured at 64°C, 70°C and 76°C (Figures 10~12). As expected, as the testing temperature increased, the measured value decreased regardless of CRM contents. Also, as the rubber particle content increased, the $G^*/\sin \delta$ value increased at all testing temperatures. However, the data of top, middle and bottom parts showed a different trend, compared with the original CRM binders before conditioning. First, the top $G^*/\sin \delta$ value maintained a similar level up to the 15% CRM content, and then increased more than twice at 20% CRM. In the middle part, the value was similar to the top part up to 5%, but the value increased from 10% CRM, and the value was approximately three times higher than the top part in 15% or more. The bottom part showed a higher value compared to the top part and middle part from 5% CRM, and the value increased rapidly at 10% CRM, and thereafter, the value showed a tendency to gradually increase.

Comparing the middle and bottom parts, the bottom part resulted in a higher value up to 15% CRM, but revealed a similar value at 20%. This is thought to be because, at 20% CRM binder, the rubber particles sunk from the top part were distributed at a similar rate in the middle and bottom parts. In general, the measured values for the top, middle and bottom parts at 70°C and 76°C demonstrated the same trend as $G^*/\sin \delta$ at 64°C.

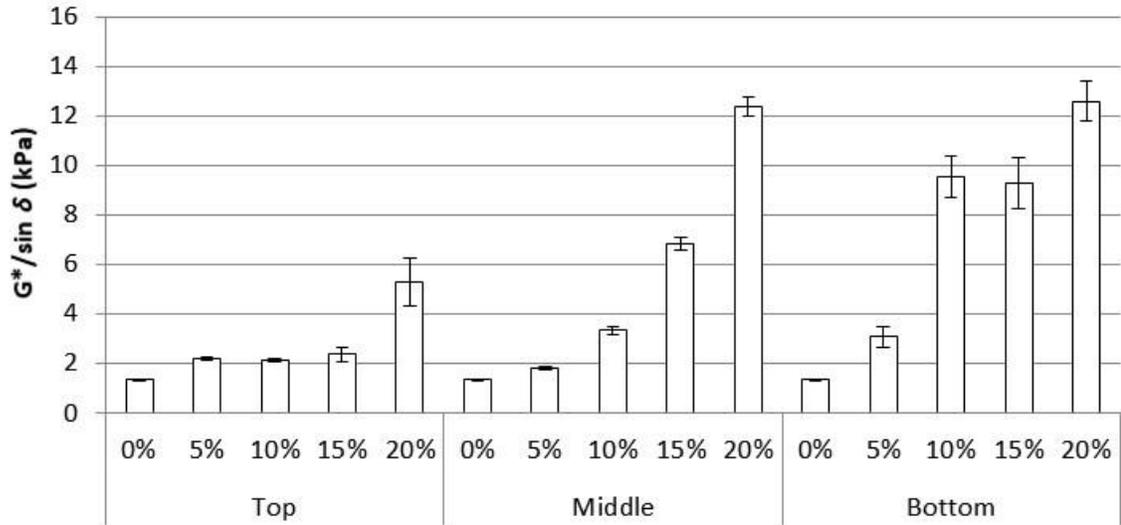


Figure 10. $G^*/\sin \delta$ at 64°C of CRM Asphalt Binders of Top, Middle and Bottom Parts after Conditioning

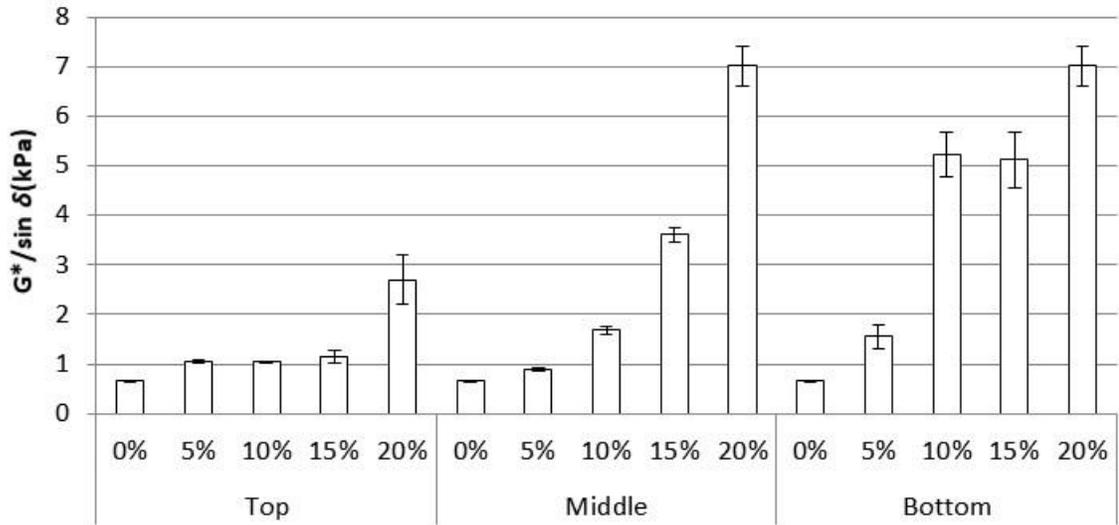


Figure 11. $G^*/\sin \delta$ at 70°C of CRM Asphalt Binders of Top, Middle and Bottom Parts after Conditioning

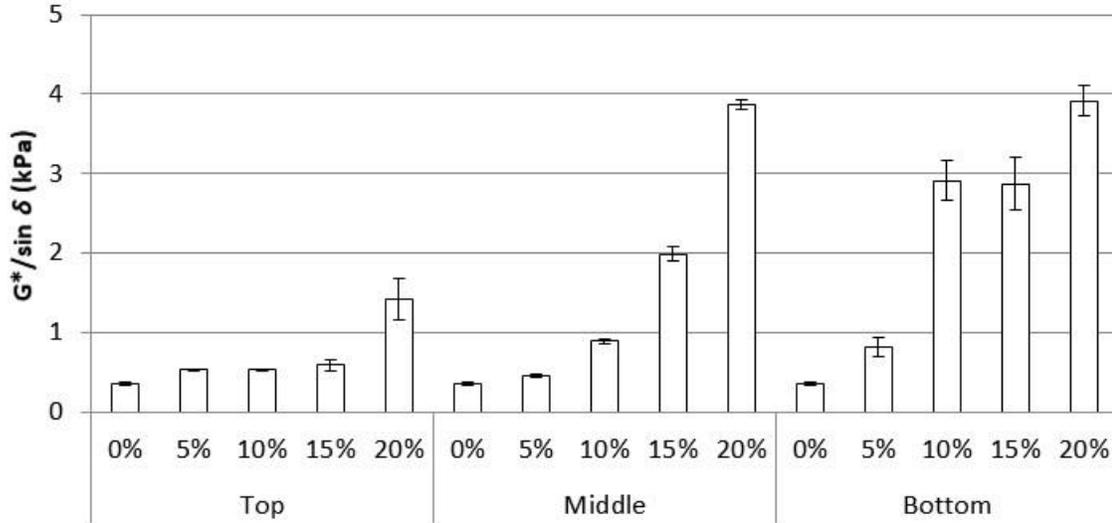


Figure 12. $G^*/\sin \delta$ at 76°C of CRM Asphalt Binders of Top, Middle and Bottom Parts after Conditioning

The statistical significance of the change in the CRM contents was examined, comparing the original condition to the top, middle, and bottom parts, using one-way analysis of variance (Tables 4~6). In general, the significant difference within each content of original condition at all temperatures was observed. In the top part, it was confirmed that the measured values were not statistically significant up to 10% CRM content within each testing temperature. In the bottom part, the values of 10% and 15% CRM binders were statistically similar at the 95% confidence level.

Table 4. Statistical Analysis Results of $G^*/\sin \delta$ at 64°C of CRM Binders as a Function of Top, Middle and Bottom Parts ($\alpha=0.05$).

	Original (%)					Top (%)					Middle (%)					Bottom (%)					
	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	
Original (%)	0	-	S	S	S	S	N	N	N	S	S	N	N	S	S	S	N	S	S	S	S
	5		-	S	S	S	S	N	N	N	S	S	N	S	S	S	S	S	S	S	S
	10			-	S	S	S	S	S	S	S	S	S	N	S	S	S	N	S	S	S
	15				-	S	S	S	S	N	S	S	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
Top (%)	0					-	N	N	S	S	N	N	S	S	S	N	S	S	S	S	
	5						-	N	N	S	N	N	S	S	S	N	S	S	S	S	
	10							-	N	S	N	N	S	S	S	N	S	S	S	S	
	15								-	S	S	N	S	S	S	S	S	S	S	S	
	20									-	S	S	S	S	S	S	S	S	S	S	
Middle (%)	0										-	N	S	S	S	N	S	S	S	S	
	5											-	S	S	S	N	S	S	S	S	
	10												-	S	S	S	N	S	S	S	
	15													-	S	S	S	S	S	S	
	20														-	S	S	S	S	S	
Bottom (%)	0															-	S	S	S	S	
	5																-	S	S	S	
	10																	-	N	S	
	15																		-	S	
	20																				-

N: non-significant, S: significant.

Table 5. Statistical Analysis Results of $G^*/\sin \delta$ at 70°C of CRM Binders as a Function of Top, Middle and Bottom Parts ($\alpha=0.05$).

	Original (%)					Top (%)					Middle (%)					Bottom (%)					
	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	
Original (%)	0	-	S	S	S	S	N	N	N	S	S	N	N	S	S	S	N	S	S	S	S
	5		-	S	S	S	S	N	N	N	S	S	N	S	S	S	S	S	S	S	S
	10			-	S	S	S	S	S	S	S	S	S	N	S	S	S	N	S	S	S
	15				-	S	S	S	S	N	S	S	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
Top (%)	0					-	N	N	S	S	N	N	S	S	S	N	S	S	S	S	
	5						-	N	N	S	N	N	S	S	S	N	S	S	S	S	
	10							-	N	S	N	N	S	S	S	N	S	S	S	S	
	15								-	S	S	N	S	S	S	S	S	S	S	S	
	20									-	S	S	S	S	S	S	S	S	S	S	
Middle (%)	0										-	N	S	S	S	N	S	S	S	S	
	5											-	S	S	S	N	S	S	S	S	
	10												-	S	S	S	N	S	S	S	
	15													-	S	S	S	S	S	S	
	20														-	S	S	S	S	S	
Bottom (%)	0															-	S	S	S	S	
	5																-	S	S	S	
	10																	-	N	S	
	15																			-	S
	20																				

N: non-significant, S: significant.

Table 6. Statistical Analysis Results of $G^*/\sin \delta$ at 76°C of CRM Binders as a Function of Top, Middle and Bottom Parts ($\alpha=0.05$).

	Original (%)					Top (%)					Middle (%)					Bottom (%)					
	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	
Original (%)	0	-	N	S	S	S	N	N	N	S	S	N	N	S	S	S	N	S	S	S	S
	5		-	S	S	S	N	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	10			-	S	S	S	S	S	S	S	S	S	N	S	S	S	N	S	S	S
	15				-	S	S	S	S	N	S	S	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
Top (%)	0					-	N	N	S	S	N	N	S	S	S	N	S	S	S	S	
	5						-	N	N	S	N	N	S	S	S	N	S	S	S	S	
	10							-	N	S	N	N	S	S	S	N	S	S	S	S	
	15								-	S	S	N	S	S	S	S	S	S	S	S	
	20									-	S	S	S	S	S	S	S	S	S	S	
Middle (%)	0										-	S	S	S	S	N	S	S	S	S	
	5											-	S	S	S	N	S	S	S	S	
	10												-	S	S	S	N	S	S	S	
	15													-	S	S	S	S	S	S	
	20														-	S	S	S	S	N	
Bottom (%)	0															-	S	S	S	S	
	5																-	S	S	S	
	10																	-	N	S	
	15																		-	S	
	20																			-	

N: non-significant, S: significant.

Multiple Stress Creep and Recovery Property

MSCR (Test Method D7405) is an alternate test to the DSR test method used for the storage stability. The MSCR test was performed at 64°C and 76°C according to AASHTO TP 70 by loading 3.2 kPa to evaluate the viscoelasticity of the CRM binders under more extreme conditions than DSR test.

Figure 13 presented the results of J_{nr} and % recovery of control CRM binders. In general, increasing CRM contents made it possible to decline J_{nr} value and enhance % rec, which means the higher CRM contents, the higher viscoelasticity of binder. In more

detail, the data from CRM contents of 0% and 5% were not measured due to the samples' low viscosity at 64°C. The J_{nr} value decreased steadily, with the data reaching 2.52 in 10% CRM, 1.75 in 15% CRM, and 0.41 in 20% CRM while the data for % recovery increased gradually by up to over 12.3% from 2.5%. In addition, by increasing the testing temperature to 76°C, the data were not measured until 15% CRM binders as the binders become softer at higher temperature. The value of 20% CRM binder was only measured.

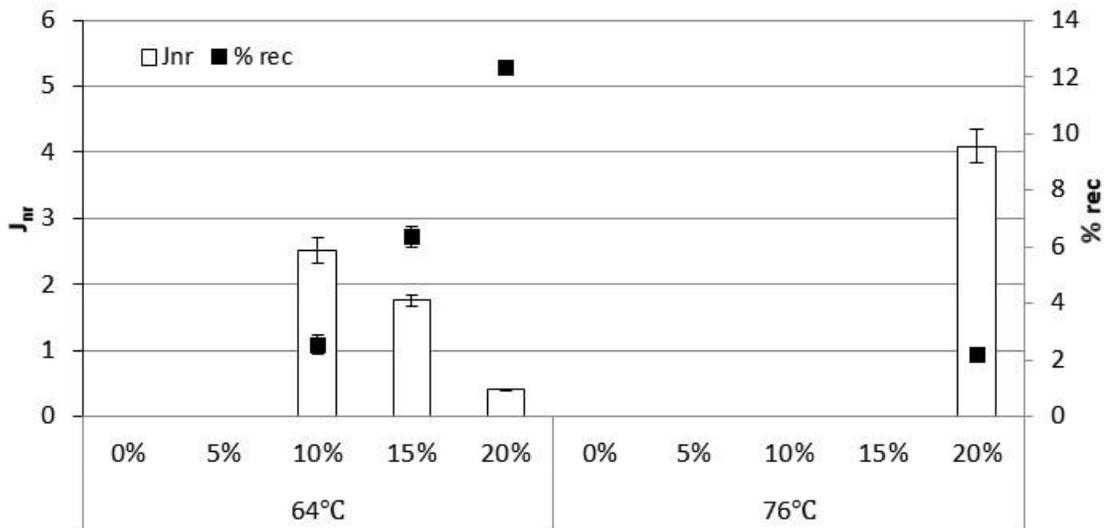


Figure 13. J_{nr} and % rec of CRM Asphalt Binders at 64°C and 76°C

Figures 14 and 15 show the J_{nr} and % rec of the CRM asphalt binders at 64°C and 76°C, respectively. At 64°C, no values were recorded in the binders except for 20% at the top part. The values were measured from 10% in the middle part and from 5% in the bottom part. It is considered that the viscosity at 64°C increased from the top to the middle to the bottom. As the CRM content increased, the J_{nr} value decreased and the % rec value increased. In the case of 20% CRM binder, the middle part and the bottom part resulted in similar values.

When 76°C was used as the testing temperature, it was not measured in the top part. The J_{nr} and % rec of the CRM asphalt binders were obtained from 15% in the

middle part and from 10% in the bottom part. It is thought that it is because the viscosity of CRM binders became lower compared with 64°C. The general trend was similar to that at 64°C.

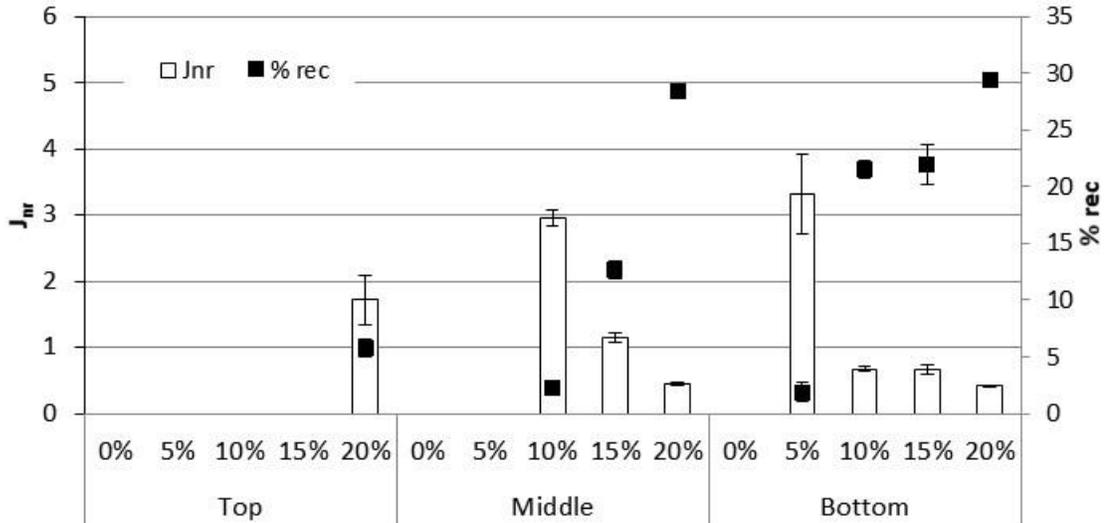


Figure 14. J_{nr} and % rec at 64°C of CRM Asphalt Binders of Top, Middle and Bottom Parts after Conditioning

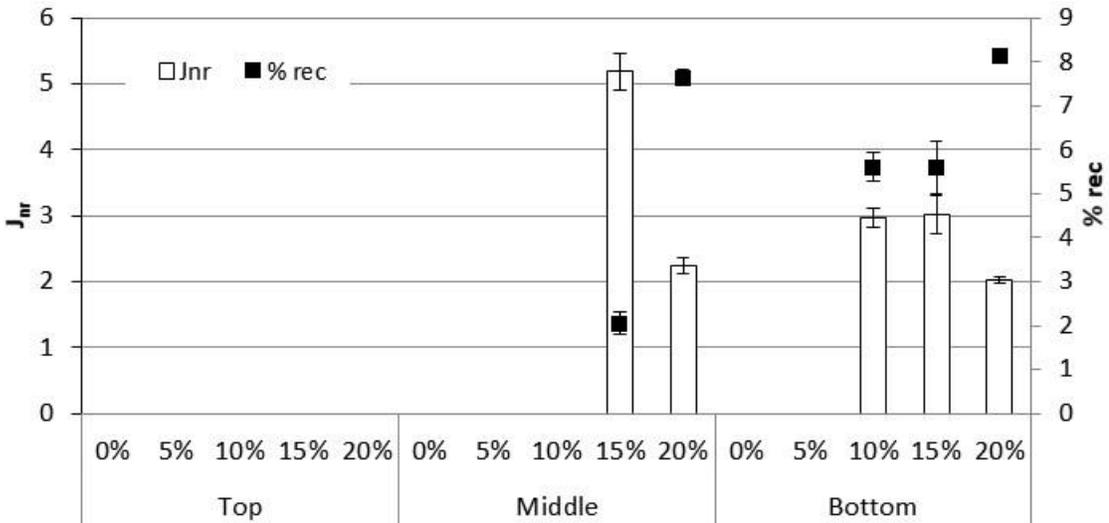


Figure 15. J_{nr} and % rec at 76°C of CRM Asphalt Binders of Top, Middle and Bottom Parts after Conditioning

Using one-way analysis of variance, the statistical significance of the change in the J_{nr} and % rec was examined, comparing the original condition to the top, middle, and bottom parts (Tables 7 and 8). In general, the J_{nr} values within original condition from the

MSCR test at 64°C were significantly different depending on CRM contents. For the conditioned CRM binders of the top, middle, and bottom parts, the significant difference was observed within each part compared to the original condition from the top to the bottom. In the case of statistical analysis for J_{nr} at 76°C, there was insignificant difference within each part of original, top, and bottom due to the non-measured results. The case of % rec showed a similar trend to the J_{nr} analysis.

Table 7. Statistical Analysis Results of J_{nr} of CRM binders as a Function of Top, Middle and Bottom Parts ($\alpha=0.05$).

		J_{nr} at 64°C																			
		Original (%)					Top (%)					Middle (%)					Bottom (%)				
		0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20
Original (%)	0	-	N	S	S	S	N	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	5		-	S	S	S	N	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	10			-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
	15				-	S	S	S	S	S	N	S	S	S	S	S	S	S	S	S	S
	20					-	S	S	S	S	S	S	S	S	S	N	S	S	N	N	N
Top (%)	0						-	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	5							-	N	N	S	N	N	S	S	S	N	S	S	S	S
	10								-	N	S	N	N	S	S	S	N	S	S	S	S
	15									-	S	N	N	S	S	S	N	S	S	S	S
	20										-	S	S	S	S	S	S	S	S	S	S
Middle (%)	0											-	N	S	S	S	N	S	S	S	S
	5												-	S	S	S	N	S	S	S	S
	10													-	S	S	S	N	S	S	S
	15														-	S	S	S	S	S	S
	20															-	S	S	N	N	N
Bottom (%)	0																-	S	S	S	S
	5																	-	S	S	S
	10																		-	N	N
	15																			-	N
	20																				-
		J_{nr} at 76°C																			
Original (%)	0	-	N	N	N	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	5		-	N	N	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	10			-	N	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	15				-	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	20					-	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Top (%)	0						-	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	5							-	N	N	N	N	N	N	S	S	N	N	S	S	S
	10								-	N	N	N	N	N	S	S	N	N	S	S	S
	15									-	N	N	N	N	S	S	N	N	S	S	S
	20										-	N	N	N	S	S	N	N	S	S	S
Middle (%)	0											-	N	N	S	S	N	N	S	S	S
	5												-	N	S	S	N	N	S	S	S
	10													-	S	S	N	N	S	S	S
	15														-	S	S	S	S	S	S
	20															-	S	S	S	S	S
Bottom (%)	0																-	N	S	S	S
	5																	-	S	S	S
	10																		-	N	S
	15																			-	S
	20																				-

N: non-significant, S: significant.

Table 8. Statistical Analysis Results of % rec of CRM Binders as a Function of Top, Middle and Bottom Parts ($\alpha=0.05$).

		% rec at 64°C																			
		Original (%)					Top (%)					Middle (%)					Bottom (%)				
		0	5	10	15	20	0	5	10	15	20	0	5	10	15	20	0	5	10	15	20
Original (%)	0	-	N	S	S	S	N	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	5		-	S	S	S	N	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	10			-	S	S	S	S	S	S	S	S	S	N	S	S	S	N	S	S	S
	15				-	S	S	S	S	S	N	S	S	S	S	S	S	S	S	S	S
	20					-	S	S	S	S	S	S	S	S	N	S	S	S	S	S	S
Top (%)	0						-	N	N	N	S	N	N	S	S	S	N	S	S	S	S
	5							-	N	N	S	N	N	S	S	S	N	S	S	S	S
	10								-	N	S	N	N	S	S	S	N	S	S	S	S
	15									-	S	N	N	S	S	S	N	S	S	S	S
	20										-	S	S	S	S	S	S	S	S	S	S
Middle (%)	0											-	N	S	S	S	N	S	S	S	S
	5												-	S	S	S	N	S	S	S	S
	10													-	S	S	S	N	S	S	S
	15														-	S	S	S	S	S	S
	20															-	S	S	S	S	N
Bottom (%)	0																-	S	S	S	S
	5																	-	S	S	S
	10																		-	N	S
	15																			-	S
	20																				-
		% rec at 76°C																			
Original (%)	0	-	N	N	N	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	5		-	N	N	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	10			-	N	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	15				-	S	N	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	20					-	S	S	S	S	S	S	S	S	N	S	S	S	S	S	S
Top (%)	0						-	N	N	N	N	N	N	N	S	S	N	N	S	S	S
	5							-	N	N	N	N	N	N	S	S	N	N	S	S	S
	10								-	N	N	N	N	N	S	S	N	N	S	S	S
	15									-	N	N	N	N	S	S	N	N	S	S	S
	20										-	N	N	N	S	S	N	N	S	S	S
Middle (%)	0											-	N	N	S	S	N	N	S	S	S
	5												-	N	S	S	N	N	S	S	S
	10													-	S	S	N	N	S	S	S
	15														-	S	S	S	S	S	S
	20															-	S	S	S	S	S
Bottom (%)	0																-	N	S	S	S
	5																	-	S	S	S
	10																		-	N	S
	15																			-	S
	20																				-

N: non-significant, S: significant.

Storage Stability Results

In order to evaluate the storage stability of CRM binders, $G^*/\sin \delta$ and % rec of top and bottom parts after conditioning, were used to calculate separation index (SI) suggested by Superpave.

As shown in Table 9, as the testing temperature increased from 64°C to 76°C, SI value generally increased in all contents of CRM binders. It showed the lowest value at 5% CRM, and the highest at 10%. The values were slightly lower at 15% and further decreased at 20%. The reason for the highest value at 10% is thought to be that the rubber particles that sunk from the top part were mainly at the bottom part. The relatively lower value at 20% is considered to be due to the high content of CRM used, and there was no more space to sink at the bottom part.

Table 10 shows the SI value for % rec after conditioning. It was observed that the SI for % rec is not suitable for evaluating the storage stability of CRM binders used in this study.

Table 9. Separation Index from $G^*/\sin \delta$ of CRM Binders

Binder	$G^*/\sin \delta$ (kPa)			% Separation
	Temperature	Top	Bottom	
CRM 5%	64°C	2.17	3.08	17
	70°C	1.04	1.55	20
	76°C	0.53	0.82	21
CRM 10%	64°C	2.13	9.53	63
	70°C	1.04	5.22	67
	76°C	0.53	2.91	69
CRM 15%	64°C	2.38	9.29	59
	70°C	1.15	5.12	63
	76°C	0.59	2.87	66
CRM 20%	64°C	5.31	12.60	41
	70°C	2.70	7.01	44
	76°C	1.42	3.92	47

Table 10. Separation Index from % rec of CRM Binders

Binder	% rec			
	Temperature	Top	Bottom	% Separation
CRM 5%	64°C	0.00	1.90	100
	76°C	0.00	0.00	None
CRM 10%	64°C	0.00	21.54	100
	76°C	0.00	5.61	100
CRM 15%	64°C	0.00	21.91	100
	76°C	0.00	5.60	100
CRM 20%	64°C	5.83	29.39	67
	76°C	0.00	8.15	100

5. SUMMARY AND CONCLUSION

In order to investigate the storage stability of CRM asphalt binders containing 5%, 10%, 15%, and 20%, the binders were conditioned for 48 hours in the oven at 163°C. The tests were carried out using the rotational viscosity and the dynamic shear rheometer to determine the properties and separation index (SI) of CRM binders. From these results, the following conclusions were drawn for the storage stability in this study.

- 1) The addition of CRM increased the viscosity at 135°C and 180°C, as expected. The conditioned CRM binders appeared to have higher viscosity in the bottom part compared to the middle and top parts. This is caused that rubber particles fill from the bottom part to the middle part while conditioning.
- 2) From the DSR test at high temperatures, it is found that increasing CRM content made it possible to increase $G^*/\sin \delta$ in original condition. Similar to the viscosity results, the CRM binders after conditioning showed the highest $G^*/\sin \delta$ value in the bottom part.
- 3) From the MSCR test, J_{nr} and % rec values are observed to have a similar trend with $G^*/\sin \delta$ results. However, some of the data were not measured due to the higher load than the DSR test.
- 4) The SI from $G^*/\sin \delta$ generally increased as the test temperature increased. The SI increased up to 10% CRM, and then decreased as the CRM content was further increased.
- 5) It was observed that the SI from $G^*/\sin \delta$ generally used was suitable for evaluating the storage stability of CRM asphalt binders, compared to the SI from % rec.

- 6) The results are limited to the CRM particles and asphalt binders used in this study, and are intended to show the change of storage stability according to CRM contents. To draw more general conclusion, it is recommended to use different types of rubber powder and asphalt binders.

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