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Evaluation of Carbon Char in Hot-Mix Asphalt Mixture

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16 Abstract

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The following conclusions were drawn based on the analysis of the test results. Modified Lottman test results illustrated that all mixtures achieved a higher tensile strength ratio (TSR) than the minimum 80% KDOT requirement. Moisture resistance slightly decreased with 5% carbon char, but TSR increased with 10% carbon char in the HMA. The Hamburg wheel tracking device (HWTD) test results showed that all mixtures failed at 20-mm rut depth. However, based on number of wheel passes to failure, we can also conclude that the mixture without carbon char has a higher rutting resistance than the mixtures with it. HWTD output parameters of creep slope, stripping slope, and stripping reflection point also indicate a low moisture resistance of HMA mixtures with 5% carbon char. The HWTD test results of 3% air voids mixtures showed that all mixtures failed at 20-mm rut depth. As carbon char content increased, rutting and moisture resistance slightly improved. However, the mixtures with 3% air voids are less rutting and moisture resistant than the mixtures with 4% air voids.

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Abstract

Carbon char is a by-product of the pyrolysis of waste tires. The use of carbon char would promote sustainability and economy. Carbon char is similar to carbon black, a potential additive for hot-mix asphalt (HMA). Previously carbon black in HMA showed the potential to reduce rutting potential, temperature susceptibility, and low-temperature cracking. The primary objective of this study was to evaluate carbon char, resulting from the pyrolysis of waste tires, in Superpave HMA. Carbon char was blended with the Superpave performance grading (PG) asphalt binder at various percentages (5%, 10%, 15%, and 20%). A rotational viscosity test was performed on the blend to see what percentages of carbon char by mass of asphalt binder would be workable. Five percent and 10 percent were found to be viable. Those carbon char percentages were used with a PG 58-28 binder in a Superpave HMA mix design. Modified Lottman and Hamburg wheel tracking tests were then conducted on this Superpave mixture.

The following conclusions were drawn based on the analysis of the test results. Modified Lottman test results illustrated that all mixtures achieved a higher tensile strength ratio (TSR) than the minimum 80% KDOT requirement. Moisture resistance slightly decreased with 5% carbon char, but TSR increased with 10% carbon char in the HMA. The Hamburg wheel tracking device (HWTD) test results showed that all mixtures failed at 20-mm rut depth. However, based on number of wheel passes to failure, we can also conclude that the mixture without carbon char has a higher rutting resistance than the mixtures with it. HWTD output parameters of creep slope, stripping slope, and stripping reflection point also indicate a low moisture resistance of HMA mixtures with 5% carbon char. The HWTD test results of 3% air voids mixtures showed that all mixtures failed at 20-mm rut depth. As carbon char content increased, rutting and moisture resistance slightly improved. However, the mixtures with 3% air voids are less rutting and moisture resistant than the mixtures with 4% air voids.

Acknowledgements

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Chapter 1: Introduction

1.1 Background

Carbon char, also known as pyrolyzed carbon black, is a by-product from the process of combusting waste tires to recover crude oil. Usually, the pyrolysis process of tires produces 55% oil, 25% pyrolyzed carbon black, 9% steel, 5% fiber, and 6% gas (Park, Coree, & Lovell, 1996). Carbon black has previously shown benefits in asphalt mixtures. Since the 1960s many researchers investigated the use of carbon black in HMA (Rostler, White, & Dannenberg, 1977; Vallerga & Gridley, 1980). It was proven to improve rutting resistance, reduce temperature susceptibility, and decrease low-temperature cracking potential (Park et al., 1996). Vallerga and Gridley (1980) and Yao and Monismith (1986) found that the use of carbon black increased the rutting resistance at high temperature and decreased the temperature susceptibility and low temperature cracking of HMA. However, the use of carbon black was somewhat limited due to the high cost (Park et al., 1996). Because of the relatively high content of carbon black in pyrolyzed carbon char, it has the potential to become an additive in asphalt mixture. The use of carbon char would also promote the use of a waste product for sustainability and economy.

1.2 Objective

This study focused on investigating performance of HMA mixtures with various carbon char contents and then comparing with the performance of a control mixture (with no carbon char). Modified Lottman and Hamburg wheel tracking device (HWTD) tests were conducted on Superpave mixtures for evaluation.

1.3 Report Outline

This report is divided into four chapters. Chapter 1 states the background, problem statement, and objective of the research. Chapter 2 describes the materials and experimental work performed. Chapter 3 presents the results and analysis. Finally, Chapter 4 summarizes conclusions based on this study.

Chapter 2: Materials and Laboratory Experiments

2.1 Materials

In this study, carbon char powder, as shown in Figure 2.1 and produced from pyrolysis of waste tires, was provided by Blizzard Energy, Inc., in Kansas. It was blended with a PG 58-28 asphalt binder at various percentages (5%, 10%, 15%, and 20%) by mass of asphalt cement.

Figure 2.1: Carbon Char

The scanning electron microscopy (SEM) test was conducted to explore the microstructure characteristics. Blizzard Energy Inc. performed the tests and provided test results. Figure 2.2 shows the SEM image of carbon char with 1,000 magnification. The main elements and components detected by SEM are shown in Table 2.1. The main detected elements are carbon (C), oxygen (O), sulfur (S), and iron (Fe); and the components of carbon char are elemental carbon (C), elemental sulfur (S), iron carbonate ($FeCO_3$), and iron sulfur (FeS).

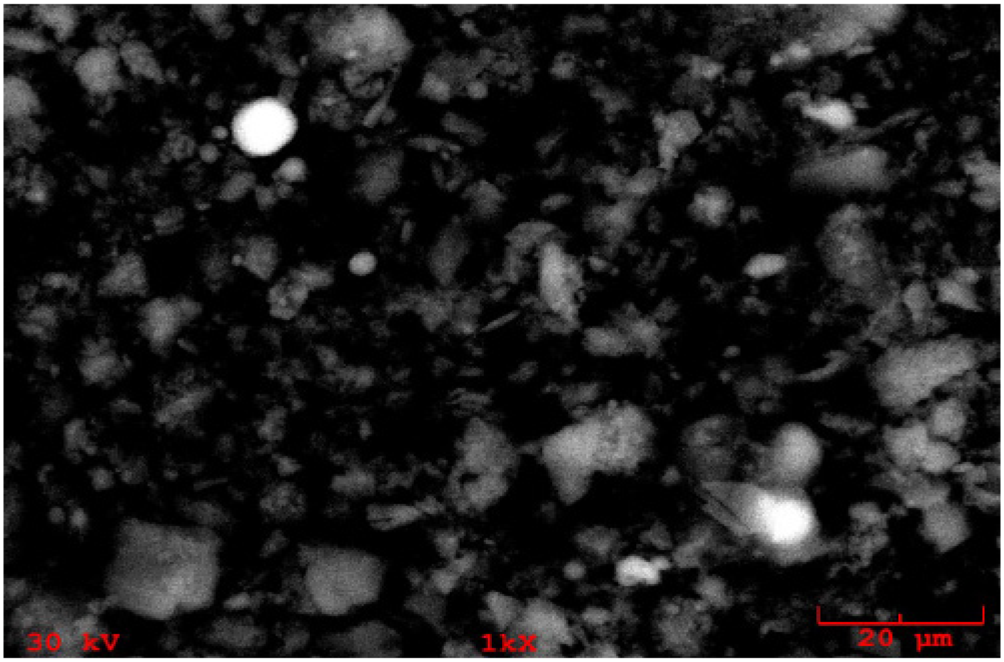

Figure 2.2: SEM Result of Carbon Char
Source: Blizzard Energy, Inc.

Table 2.1: Main Elements and Components of Carbon Char

Element	%	Component	%	Comment
C	58.50	C	55.14	Elemental Carbon
O	13.40	S	10.40	Elemental Sulfur
S	11.17	$FeCO_3$	32.35	Iron Carbonate
Fe	16.93	FeS	2.11	Iron Sulfide
Total	100	Total	100	

Source: Blizzard Energy, Inc.

In this study, HMA mixtures containing RAP also used five virgin aggregates (CS-1, CS-2A, CS-2, SSG, and SSG-1). Virgin aggregates and RAP were collected from Shilling Construction Co. Inc. in Riley County, Kansas. The gradation of each aggregate is shown in Table 2.2. A PG 58-28 asphalt binder was used in this research for the HMA mixtures. The viscosity of the asphalt binder was 310 centipoise (cP).

Table 2.2: Gradation of Each Aggregate

Aggregate Type	1/2	3/8	#4	#8	#16	#30	#50	#100	#200
CS-1	36	73	95	99	99	99	99	99	99.0
CS-2A			5	48	78	88	92	94	95.8
CS-2			24	50	62	69	75	78	80.2
SSG				25	60	80	91	98	99.0
SSG-1		7	73	99	99	99	99	99	99.0
RAP	4	10	26	44	60	71	83	88	90.6

2.2 Experimental Work

2.2.1 Binder Preparation

Carbon char at mass percentages of 5%, 10%, 15%, and 20% were blended with heated asphalt cement. First the asphalt binder was heated to the mixing temperature. Then carbon char was added to the binder. A spiral mixer installed on a drill was used to mix the carbon char and binder together. After mixing, a rotational viscometer (RV) test was performed on the blends to see what percentages of carbon char would be workable. The test for the Superpave PG asphalt binder was always conducted at 135 °C, and typical viscosity values for the asphalt binder at this temperature are 200 cP to 2,000 cP (Pavement Interactive, n.d.). Based on the test results, tabulated in Table 2.3, viscosity values of the blends with 5% and 10% carbon char fall in this viscosity range, indicating good workability. However, we could not get any reading on the viscometer for blends with 15% and 20% carbon char. As shown in Figure 2.3, some carbon char seemed to "bunch" up around the test spindle, resulting in viscosity readings of these two blends that were out of range of the RV used. Therefore, blends of 5% and 10% carbon char were adopted for this study. Furthermore, as time went by, carbon char settled down in the binder. Therefore, blending of carbon char with the binder was done just before putting in the asphalt mixtures.

Table 2.3: Viscosity Test Results

Carbon Char Content (%)	Viscosity (cP)
0	310
5	340
10	352

Figure 2.1: Bunch Up of Carbon Char around Test Spindle

2.2.2 Mix Design

HMA mixtures were developed in the laboratory following KDOT requirements. KDOT defines mixtures by their nominal maximum aggregate size (NMAS). In this study, 12.5-mm NMAS mix design was developed. The mixture gradation is shown in Figure 2.4. Table 2.4 shows the percentages of aggregates used, gradation of the combined blend, and KDOT requirements. In this study, PG 58-28 was needed in the mixture due to the use of 15% recycled asphalt pavement (RAP) materials. All aggregates were heated within the mixing temperature range as was the binder. RAP was heated at 60 °C. Aggregates were mixed with various binder percentages using a mechanical mixer. After mixing, the loose mixtures were aged for two hours at the compaction temperature. Then a Superpave gyratory compactor was used to compact the asphalt mixtures with the maximum number of gyrations selected based on the design equivalent single axel loads (ESALs). The compacted cylindrical samples with 150-mm diameter were expected to achieve the target of 4% air voids. Two compacted samples and 1,500 g of loose mixture for each asphalt mixture, respectively, were prepared for the bulk specific gravity (Gmb) and theoretical maximum specific gravity (Gmm) tests to compute air voids. Based on the required air voids, the control mix (0% carbon char) was found to have 6.1% asphalt binder. Asphalt content was 6.7% in mixtures with 5% and 10% carbon char.

Table 2.4: Percentages of Aggregates, Gradation of Blend, and KDOT Requirements

Aggregate Type	% in Mix	1/2	3/8	#4	#8	#16	#30	#50	#100	#200
CS-1	18	6	13	17	18	18	18	18	18	17.8
CS-2A	24			1	12	19	21	22	23	23.0
CS-2	8			2	4	5	6	6	6	6.4
SSG	27			0	7	16	22	25	26	26.7
SSG-1	8		1	6	8	8	8	8	8	7.9
RAP	15	1	2	4	7	9	11	12	13	13.6
Design Single Point		7	15	30	55	75	85	91	94	95.5
SR-12.5A Master Limits		0-10	10 Min.		42-61					90-98

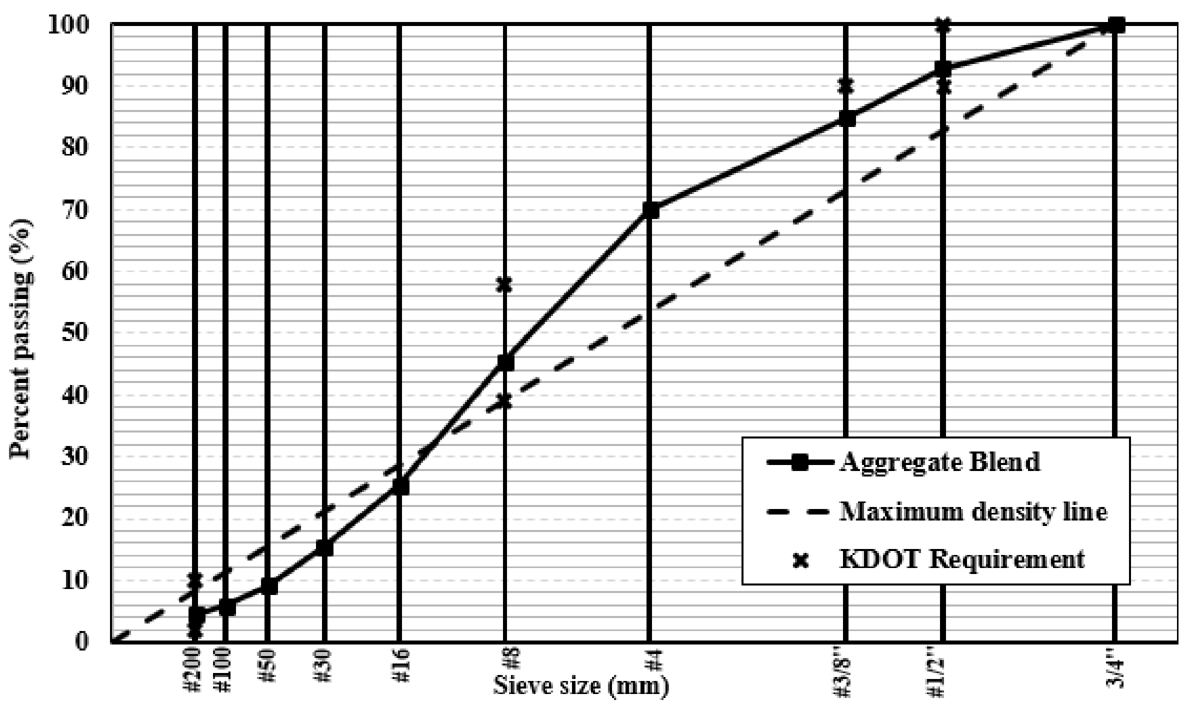

Figure 2.4: 0.45 Power Chart for Blended Aggregates

2.2.3 Modified Lottman Test

The modified Lottman test evaluates the moisture susceptibility or stripping potential of HMA mixtures. In this study, the test procedure followed was KDOT Test Method KT-56 (2014), "Resistance of Compacted Asphalt Mixture to Moisture-included Damage." Six samples with 150-mm diameter, 95±5-mm height, and 7±0.5% air voids were required in this moisture susceptibility test. The samples were sorted into two subsets of three specimens each by getting approximately equal average air voids. One of the subsets was taken as a control or unconditioned group which was tested when dry. The other subset was conditioned through a freeze-thaw cycle. The conditioned samples were first vacuum saturated until the volume of water was between 70% and 80% of the volume of air. After saturation, the specimens were frozen at -18±3 °C for a minimum of 16 hours. Then the specimens were placed in a hot water bath with a temperature of 60±1 °C for 24±1 hours, followed by placing the samples in a 25±0.5 °C water tank for 2 hours ± 10 minutes. Finally, the indirect tensile strength (ITS) test was conducted on conditioned and unconditioned samples with a loading speed of 50 mm per minutes until failure. The peak loads were recorded to calculate ITS by using Equation 2.1.

$$\mathrm{ITS} = \frac{2{,}000 \times \mathrm{P}}{\pi \times \mathrm{t} \times \mathrm{D}}$$ **Equation 2.1**

Where:

ITS = indirect tensile strength (kPa),

P = maximum load (N),

t = specimen thickness (mm), and

D = specimen diameter.

Then the tensile strength ratio (TSR) was calculated using Equation 2.2.

$$\mathrm{TSR} = \frac{\mathrm{ITS}_{C}}{\mathrm{ITS}_{UC}}$$ **Equation 2.2**

Where:

TSR = tensile strength ratio,

ITS_{c} = average indirect tensile strength of conditioned subset, and

ITS_{uc} = average indirect tensile strength of unconditioned subset.

2.2.4 Hamburg Wheel Tracking Device (HWTD) Test

The HWTD is one of the most common tests for evaluating the effects of rutting and moisture damage. The HWTD simulates the wheel load by rolling a pair of steel wheels back and forth over the surface of HMA specimens submerged in hot water with a temperature of 50 ℃. The test procedure followed is Tex-242-F (2014) test method of the Texas Department of Transportation (TxDOT). Test specimens were compacted to 7±1% air voids with 150-mm diameter and 62±2-mm height. A set of tests required four specimens with three replicates; therefore, a total of 12 specimens were prepared for each mixture. The edges of test specimens were trimmed according to the fabricated molds to form the test specimen configuration. Before starting the test, the number of maximum wheel passes and the maximum rut depth were required to input into the operation software. The test criteria of the Tex-242-F test method are summarized in Table 2.5.

Table 2.5: Hamburg Wheel Tracking Test Criteria

Binder Grade	Number of Wheel Passes	Maximum Rut Depth (mm)
PG 64-22	10,000	12.5
PG 70-22	15,000	12.5
PG 76-22	20,000	12.5

For this study, 40,000 wheel passes and a 20-mm maximum rut depth were set as the failure criteria. The test started once the water temperature reached 50 ℃. A linear variable differential transformer (LVDT) measured the rut depth every 100 wheel passes at 11 points along the wheel path. The test automatically stopped when either the desired number of wheel passes was reached or the maximum rut depth was reached, and the number of passes to failure and rut depth were recorded at the end of test.

Chapter 3: Results and Discussion

3.1 Modified Lottman (KT-56) Test Results

The KT-56 test results for all mixtures are listed in Table 3.1. As shown in Table 3.1, all mixtures met the KDOT criterion of a minimum 80% tensile strength ratio. Thus, all mixtures exhibited good moisture susceptibility. Mixtures containing 10% carbon char had the highest TSR, which indicated that adding 10% carbon char can improve moisture resistance to a limited extent. Based on the average tensile strength of conditioned and unconditioned samples as function of carbon char content, the highest tensile strength was observed for the control mix (0% carbon char). The average tensile strength slightly decreased when the carbon char content increased.

Table 3.1: Modified Lottman Test Results

Carbon Char Content		Air Voids (%)	Tensile Strength (kPa)	Avg. (kPa)	TSR (%)
0%	Conditioned	7.1	320	275	86.6
		7.0	243		
		6.8	262		
	Unconditioned	6.9	274	318	
		6.7	300		
		7.2	379		
5%	Conditioned	7.2	220	237	82.0
		7.0	287		
		7.0	205		
	Unconditioned	7.1	215	290	
		7.0	276		
		6.9	378		
10%	Conditioned	7.0	228	262	92.7
		7.0	193		
		7.0	364		
	Unconditioned	7.0	242	282	
		7.0	251		
		7.0	353		

3.2 HWTD Test Results

The HWTD test results are shown in Table 3.2. All the mixtures with carbon char failed to pass the HWTD test. The control mix (0% carbon char) failed with 20-mm rut depth at 9,849 passes. The mixtures with 5% and 10% carbon char failed at 6,717 and 6,979 passes, respectively. Therefore, adding carbon char in an HMA mixture would reduce the rutting resistance.

Table 3.2: HWTD Test Results

Carbon Char Content	Left Wheel		Right Wheel		Average		Average No. of Passes
	No. of Passes	Rut Depth (mm)	No. of Passes	Rut Depth (mm)	No. of Passes.	Rut Depth (mm)	
0%	12,147	20.02	10,163	20.00	11,155	20.01	9,849
	10,153	20.29	6,931	20.01	8,542	20.15	
	-*	-	-	-	-	-	
5%	6,820	20.07	5,794	20.00	6,307	20.04	6,717
	5,212	20.02	6,920	20.02	6,066	20.02	
	8,733	20.40	6,001	20.02	7,367	20.21	
10%	7,896	20.10	8,104	20.01	8,000	20.06	6,979
	5,886	20.08	6,030	20.01	5,958	20.05	
	-*	-	-	-	-	-	

* Data missing due to an unexpected machine problem during the test

To further evaluate mixture performance, the post-compaction consolidation, the creep slope, the stripping slope, and the stripping inflection point can be obtained by plotting a curve between rut depth and number of wheel passes, as shown in Figure 3.1. The deformation at 1,000 wheel passes is called post-compaction consolidation. It is assumed that the wheel is densifying the mixture within the first 1,000 wheel passes (Yildirim et al., 2007). The creep slope is the inverse of the rate of deformation with the linear region of the curve from post compaction to stripping (if it occurs). It is the number of wheel passes required to create 1-mm of rut depth. Creep slope is related to rutting susceptibility. The stripping slope is the inverse of the deformation rate with the linear region of the deformation curve after stripping starts, and it is the number of wheel passes creating 1-mm of rut depth after the stripping reflection point. Stripping slope is related to moisture damage. The stripping reflection point is the number of wheel passes at the interaction point of creep slope and stripping slope. In general, high creep slope, stripping reflection point, and stripping slope indicate less moisture susceptibility of a mixture (Yildirim et al., 2007).

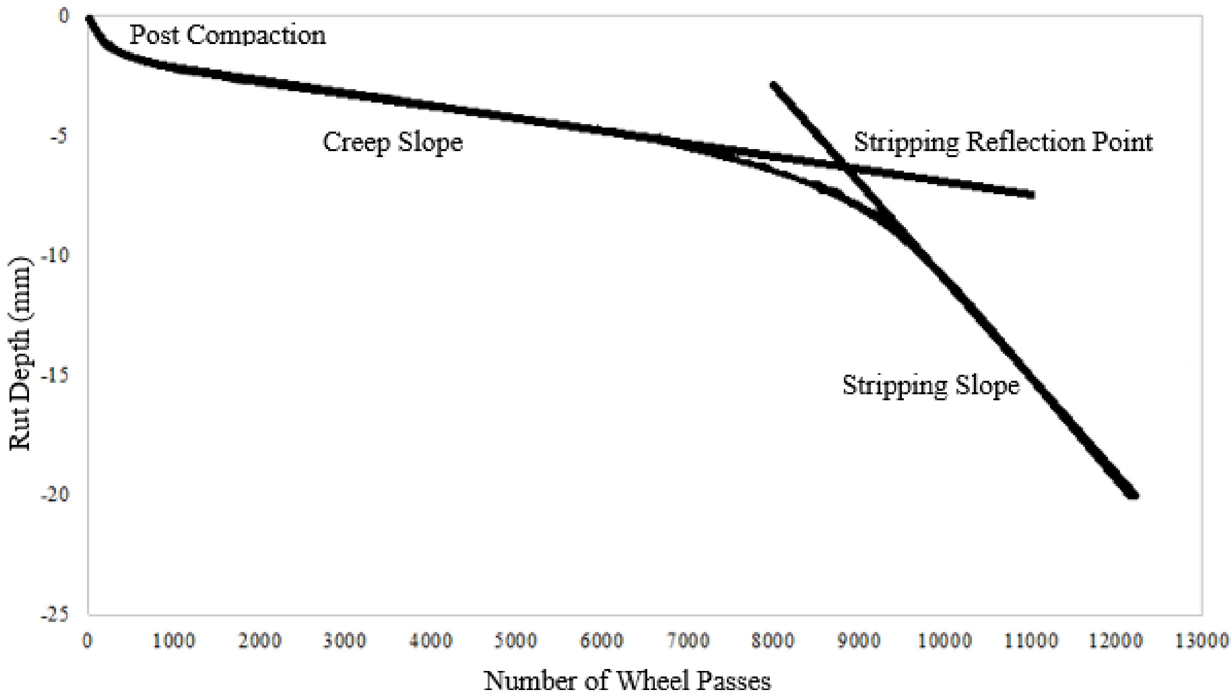

Figure 3.1: HWTD Outputs

Table 3.3 shows the test results of creep slope, stripping reflection point, and stripping slope of mixtures with various contents of carbon char. For the mixture without carbon char, the highest creep slope, stripping slope, and stripping inflection point were observed, as well as the wheel passing number at failure. Therefore, the mixture without carbon char is more resistant to rutting and moisture damage. However, the creep slope, stripping slope, and stripping inflection point of the mixture with 10% carbon char are higher than the mixture with 5% carbon char.

Table 3.3: HWTD Test Output Parameters

Carbon Char Content	Creep Slope	Stripping Reflection Point	Stripping Slope
0%	5,671	6,151	832
5%	1,867	2,948	301
10%	2,415	3,988	774

3.3 Statistical Analysis

This study utilized Statistical Analysis System (SAS)® software to perform statistical analysis of KT-56 and HWTD test results.

A generalized linear mixed model (GLMM) approach was used to analyze the data. This approach enables statisticians to incorporate both fixed and random effects in a model (Milliken & Johnson, 2009). In this test, there were two treatment factors: carbon char content at three levels (0%, 5%, and 10%), and condition state with two levels (conditioned and unconditioned.) The following model was used to investigate the strength differences among various carbon char contents in mixture:

$$y_{ijk} = \mu + \alpha_i + \beta(\alpha)_{ij} + \varepsilon_{ijk}$$ **Equation 3.1**

Where:

y is the response variable (ITS);

μ is the intercept;

α_i is the effect of i^{th} level of carbon char content, I = 1, 2, 3;

β is the effect of j^{th} level of condition state, j = 1, 2; and

ε_{ijk} is the response error for the k^{th} sample from the i^{th} carbon char content and j^{th} condition state.

Statistical Analysis System (SAS) software was used to perform the statistical analysis. The results are shown in Table 3.4 and Figure 3.2.

Table 3.4: Differences of Least Squares Means (of Carbon Char Content) for Multiple Comparisons

Carbon Char Content (%)	Carbon Char Content (%)	Estimate	Standard Error	DF	t value	Pr > |t|
0	5	33.12	37.43	12	0.88	0.39
0	10	24.73	37.43	12	0.66	0.52
5	10	-8.38	37.43	12	-0.22	0.82

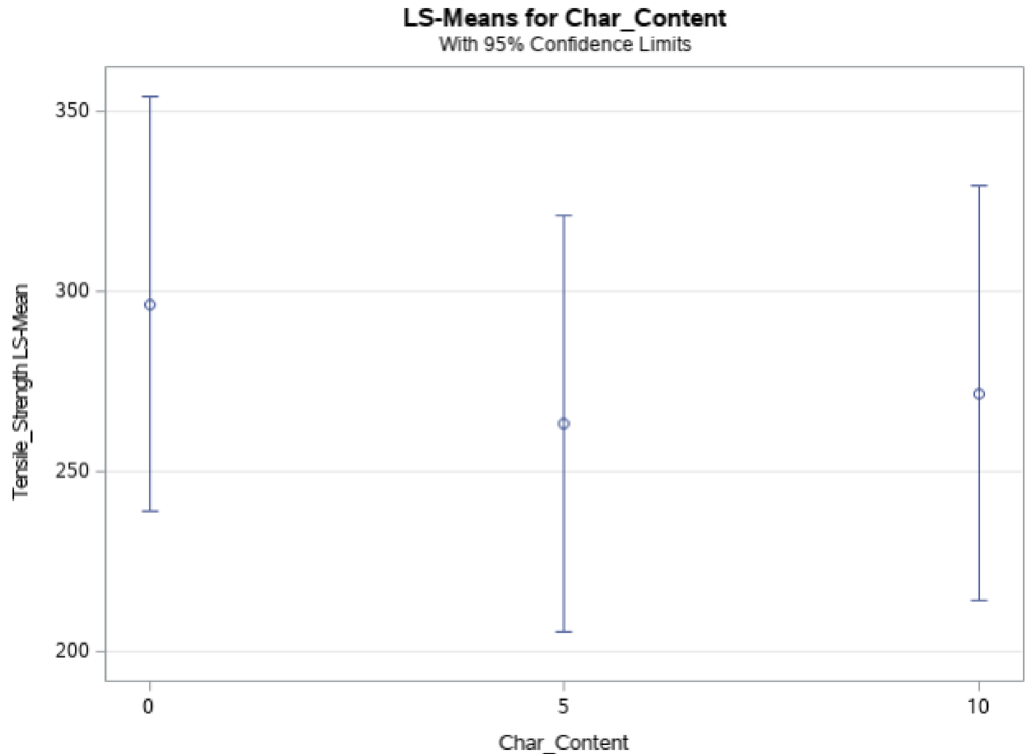

Figure 3.2: Difference in Tensile Strengths of Different Carbon Char Contents

Based on Figure 3.2, there is no significant difference between the least square means of tensile strengths derived from two different carbon char contents. The p-values showed in Table 3.4 support the same conclusion. The p-values are all larger than 0.05, which means there is no significant difference of tensile strength for two different carbon char contents. Adding carbon char will not significantly affect the mixture tensile strength.

A GLMM approach was also used to analyze the HWTD test data. In this test, there was only one treatment factor: carbon char content with three levels: 0%, 5%, and 10%. SAS software was used again to perform the statistical analysis. The results are shown in Table 3.5 and Figure 3.3.

Table 3.5: Differences in Least Squares Means for Multiple Comparisons

Carbon Char Content (%)	Carbon Char Content (%)	Estimate	Standard Error	DF	t value	Pr > |t|
0	5	3268.5	1159.82	4	2.82	0.0479
0	10	2869.5	1270.52	4	2.26	0.0868
5	10	-1159.82	1159.82	4	-0.34	0.7482

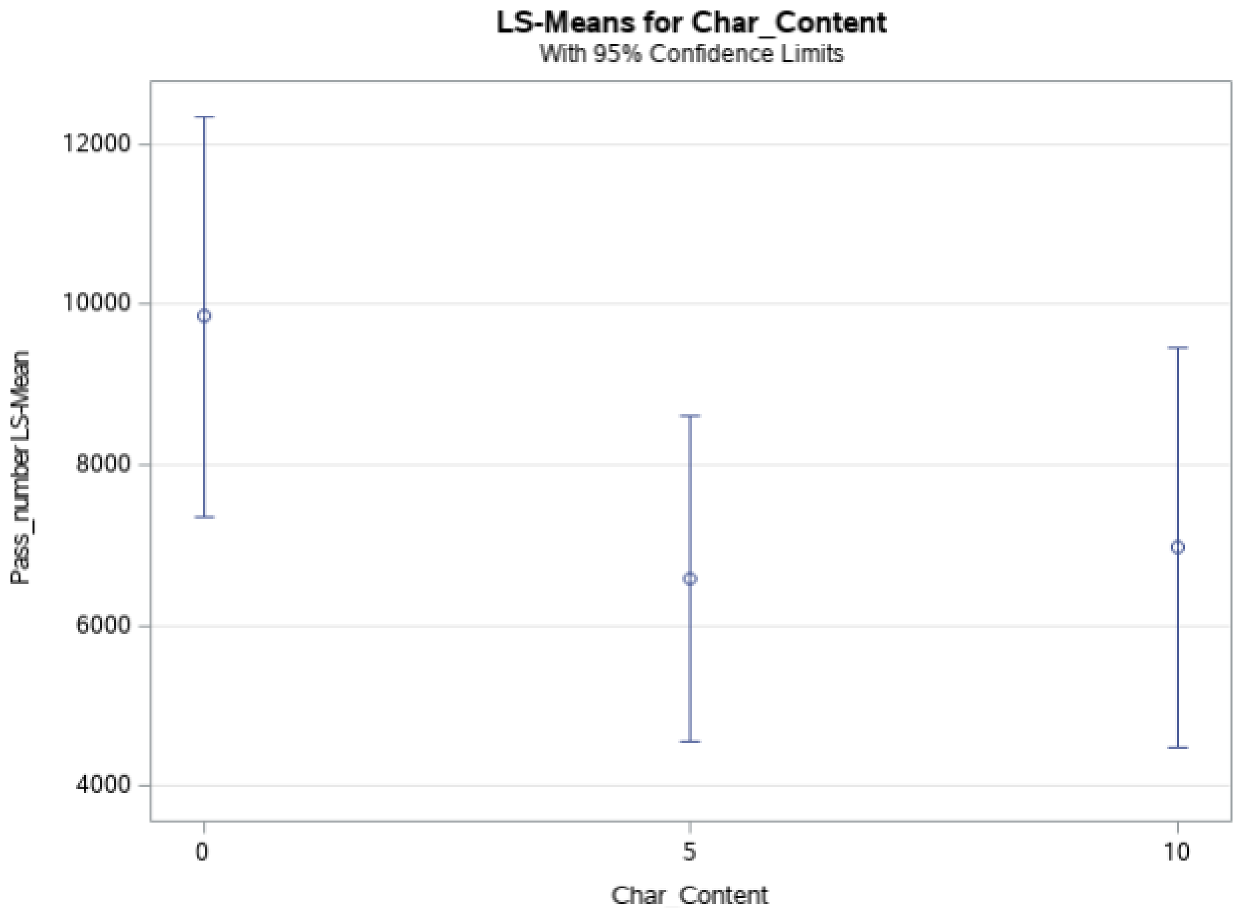

Figure 3.3: Differences in Number of Passes for Various Carbon Char Contents

Figure 3.3 clearly shows that there is a significant difference between the least square means of the pass number at failure of the mixture without carbon char and the mixtures with carbon char, regardless of carbon char content. The analysis results in Table 3.5 illustrate the same conclusion. The p-value of comparing 0% carbon char to 5% carbon char is smaller than 0.05, and the p-value of comparing 0% carbon char to 10% carbon char is slightly larger than 0.05, but much smaller than the p-value of comparing 5% carbon char to 10% carbon char. Therefore, adding carbon char significantly affects the rutting potential irrespective of the quantity of carbon char.

3.4 Design and Evaluation of HMA Mixtures with 3% Air Voids

To further evaluate the effects of carbon char on HMA mixtures, mixtures with 3% air voids were designed and tested. The same 12.5-mm NMAS mix design for mixtures with 4% air voids was used to achieve 3% air voids by adding more asphalt binder. The carbon char was mixed with a PG 58-28 binder at different percentages (0%, 5%, and 10%).

Hamburg wheel tracking device (HWTD) test was conducted on these new mixtures with 3% air voids. The test results are shown in Table 3.6. All the mixtures failed to pass the HWTD test criteria. The control mix (0% carbon char) failed with 20-mm rut depth at 3,584 passes. The mixtures with 5% and 10% carbon char failed at 3,733 and 4,263 passes, respectively. The average number of passes increased as the carbon char content increased. Adding carbon char in the HMA mixture would slightly increase the rutting resistance. However, when compared with the HWTD test results of 4% air voids mixtures, the overall number of passed dropped significantly. Thus, the mixtures with 3% air voids are less rutting resistant than the mixtures with 4% air voids.

Table 3.6: HWTD Test Results for 3% Air Voids Mixtures

Carbon Char Content (%)	Left Wheel		Right Wheel		Average		Average No. of Passes
	No. of Passes	Rut Depth (mm)	No. of Passes	Rut Depth (mm)	No. of Passes.	Rut Depth (mm)	
0	3,639	-20.05	3,519	-20.22	3,579	-20.14	3,584
	2,762	-20.01	4,256	-20.07	3,509	-20.04	
	3,350	-20.03	3,976	-20.04	3,663	-20.04	
5	3,261	-20.02	3,669	-20.02	3,465	-20.02	3,733
	3,222	-20.05	3,344	-20.05	3,283	-20.05	
	5,104	-20.04	3,800	-19.24	4,452	-19.64	
10	3,900	-20.05	4,406	-20.02	4,153	-20.04	4,263
	3,466	-20.05	3,600	-20.09	3,533	-20.07	
	6,134	-20.42	4,072	-20.02	5,103	-20.22	

Table 3.7 shows the creep slope, stripping reflection point, and stripping slope of mixtures with various carbon char contents. For the mixture without carbon char, the lowest creep slope, stripping slope, and stripping inflection point were observed. The creep slope, stripping slope, and stripping inflection point of the mixtures increased as the carbon char content increased. The mixture with 3% air voids became more moisture resistant when more carbon char was added. However, the values of creep slope, stripping slope, and stripping inflection point with 3% air voids were much lower than those for mixtures with 4% air voids. Therefore, mixtures with 3% air voids are more moisture susceptible than the mixtures with 4% air voids.

Table 3.7: HWTD Test Output Parameters

Carbon Char Content (%)	Creep Slope	Stripping Reflection Point	Stripping Slope
0	369	1,890	159
5	422	1,962	174
10	514	2,456	247

Conclusions

4.1 Conclusions

The objective of this research was to investigate asphalt mixture performance with various carbon char content at 5%, 10%, 15%, and 20%. Since no reading could be found on the viscometer for blends with 15% and 20% carbon char, they were dropped from this analysis. Mixtures with 0%, 5%, and 10% carbon char were tested in modified Lottman and HWTD tests. The following conclusions were drawn based on the analysis of the test results:

- Modified Lottman test results illustrated that all mixtures achieved a higher TSR than the minimum 80% KDOT requirement. Moisture resistance slightly decreased with 5% carbon char, but TSR increased with 10% carbon char in the HMA.
- The HWTD test results showed that all mixtures failed at 20-mm rut depth. However, based on number of wheel passes to failure, we can also conclude that the mixture without carbon char has a higher rutting resistance than the mixtures with it. HWTD output parameters of creep slope, stripping slope, and stripping reflection point also indicate a low moisture resistance of HMA mixtures with 5% carbon char.
- The HWTD test results of 3% air voids mixtures showed that all mixtures failed at 20-mm rut depth. As carbon char content increased, rutting and moisture resistance slightly improved. However, the mixtures with 3% air voids are less rutting and moisture resistant than the mixtures with 4% air voids.

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