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# Low Temperature Performance of Bio-Derived/ Chemical Additives in Warm Mix Asphalt

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# Low Temperature Performance of Bio-Derived/Chemical Additives in Warm Mix Asphalt

## Abstract

Corn and soy based bio-derived warm mix asphalt (WMA) additives are currently being developed. In the past, additives with similar properties have been shown to successfully reduce the mixing and compaction temperatures of asphalt by as much as 30°C. Isosorbide distillation bottoms (IDB), a WMA additive, is a co-product from the conversion of sorbitol to isosorbide, where sorbitol is derived by hydrogenating glucose from corn biomass. Past research utilizing IDB at several dosage rates showed there was improvement in low temperature binder performance using the bending beam rheometer (BBR) between dosage rates of 0.5% and 1.0% by weight of the binder. This research investigates whether low temperature improvement occurs with several new bio-derived material additives that have similar properties to materials used in past research, as well as compares their performance to two commercially available/bio-derived WMA additives from the forest products industry. In cold regions of the United States, the main observed distress in asphalt pavements is low temperature cracking. Characterization of binder performance at low temperature is possible with the use of the BBR. For asphalt mixtures, characterization is more challenging at low temperatures due to the response from the aggregate phase of a mixture. To examine low temperature performance of hot mix asphalt (HMA) and WMA, the semi-circular bend (SCB) test was used to characterize the fracture properties. SCB tests showed that additive choice was a statistically significant factor in fracture energy properties but not for stiffness and fracture toughness. All of the new additives were successfully used at reduced mixing and compaction temperatures and did not adversely impact low temperature mix fracture properties of WMA when compared against the control HMA. However, improvement of fracture energy was observed when comparing the epoxidized esterified fatty acid additive to the other five additives used in this work.

## Keywords

warm mix asphalt, bio-derived material additives, low temperature performance, semi-circular bend (SCB) text

## Disciplines

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## Comments

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# Low Temperature Performance of Bio-Derived/Chemical Additives in Warm Mix Asphalt

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**ABSTRACT:** Corn and soy based bio-derived warm mix asphalt (WMA) additives are currently being developed. In the past these additives with similar properties have been shown to successfully reduce the mixing and compaction temperatures by as much as 30°C. Isosorbide distillation bottoms (IDB) – a warm mix asphalt additive, is a co-product from the conversion of sorbitol to isosorbide where sorbitol is derived by hydrogenating glucose from corn biomass. Past research utilizing IDB at several dosage rates showed there was improvement in low temperature binder performance using the bending beam rheometer (BBR) between dosage rates of 0.5% and 1.0% by weight of the binder. This research investigates whether low temperature improvement occurs with several new bio-derived material additives which have similar properties to materials used in past research as well as compares their performance to two commercially available/bio-derived WMA additives from the forest products industry. In cold regions of the United States the main observed distress in asphalt pavements is low temperature cracking. Characterization of binder performance at low temperature is possible with use of the BBR. For asphalt mix, characterization is more challenging at low temperatures due to the response from the aggregate phase of a mixture. To examine low temperature performance of hot mix asphalt (HMA) and WMA, the semi-circular bend (SCB) test was used to characterize the fracture properties. SCB tests showed that additive choice was a statistically significant factor in the fracture energy properties but not for stiffness and fracture toughness. All the new additives were successfully used at reduced mixing and compaction temperatures and did not adversely impact low temperature mix fracture properties of warm mix asphalt when compared against the control HMA. However, improvement of fracture energy was observed when comparing the additive FA to the other five additives used in this work.

**KEY WORDS:** warm mix asphalt; bio-derived material additives; low temperature performance; semi-circular bend (SCB) test.

## 1 INTRODUCTION

Historically, growth of bio-based chemical products in the world market has typically been limited due to their higher production costs as compared to crude petroleum derived products. However due to the variability of crude petroleum pricing, increasing demand for environmentally friendly products from a growing population and limited amount of nonrenewable resources, growth for bio-based chemical products has increased. This market growth has propelled the number and size of bio-refineries to increase in the past 10 years. Depending on the production process, bio-refineries can produce a sizable amount of material with surfactant characteristics and thus these materials are candidates for use as bio-based warm mix asphalt (WMA) additive technologies.

WMA technologies are known to reduce binder viscosity, mixing and compaction temperatures. Reductions of 20°C-55°C during the production and laydown of asphalt mixtures can be realized. Temperature reductions lead to production cost savings due to reduced fuel use and lower emissions of greenhouse gases. Lower compaction temperatures due to reduced binder viscosity enables improved mix compactibility. This also enables contractors an extension in the paving season for colder climates, because longer haul distances are possible. An additional benefit of WMA is the ability to increase use of reclaimed asphalt pavement (RAP) in a mix. In concern to worker health in both the field and plant, temperature reductions exposes workers to less fumes (Button et al. 2007, D'Angelo et al. 2008, Gandhi 2008, Hassan 2009, Hurley and Prowell 2006, Jenkins et al. 1999, Kim et al. 2012, Kristjánsdóttir 2006, Kristjánsdóttir et al. 2007, Larsen et al. 2004, Perkins 2009, Prowell et al. 2007).

Isosorbide Distillation Bottoms (IDB) is a recently bio-derived co-product that has surfactant properties. IDB is produced from the conversion of sorbitol to isosorbide by using sorbitan to perform a dehydration reaction. Sorbitol is produced by hydrogenating glucose from corn biomass (Werpy et al. 2004). In previous studies IDB has shown great potential in improving the low temperature performance grade (PG) benefits (Podolsky et al. 2014, Podolsky 2014). At low temperatures the critical temperature has been decreased 2.5°C to 4.6°C through the addition of IDB to various binders from different source locations. However at high temperature, no effect was seen on high temperature grade in both original and short term aged binder, and viscosity remained unchanged when compared to the control (base) HMA binder (Podolsky 2014). Within this research work six bio-derived materials including IDB (at a dosage rate of 0.75% by weight of the binder) with similar properties at low temperatures are examined as warm mix asphalt additives against a control group using a PG 58-28 binder. Due to the past observation that low temperature binder properties are improved with the addition of IDB, it is proposed that there will be improvement in low temperature performance of WMA when modified with each of the six bio-derived WMA additive materials as compared to a control group. The semi-circular bend (SCB) test was used with long-term oven aged material to evaluate the low temperature performance.

## 2 OBJECTIVES

The main objective for this research work is to examine whether improvement in low temperature WMA performance is possible through the addition of the six bio-derived material additives when compared against the performance of a hot mix control. The secondary objective is to evaluate the differences between performances of bio-derived material additives at low temperature.

## 3 EXPERIMENTAL MATERIALS AND METHODS

### 3.1 Material Description

In this research work a crude source of binder from Montana, which is similar to a Canadian crude source was used as the control and the base asphalt for the six WMA additives. The Montana crude used was a PG 58-28 binder for the control HMA. The six WMA additives used in this research work were IDB, a reactor product (RP), crude isosorbide (CI), epoxidized esterified fatty acid derived from soy beans (FA), FP 1, and FP 2 – all at addition rates of 0.75% by weight of the binder to create six modified WMA mixtures. For blending the PG 58-28 binder with the six WMA additives, a Silverson shear mill was used with a blending speed of 3000 rpm at  $140^{\circ}\text{C}\pm 10^{\circ}\text{C}$  for one hour.

RP and CI are materials taken at various stages in the processing of isosorbide, while FA is derived from epoxidized soy bean oil. FP 1 and 2 are commercially available water-free chemical/bio-derived additives derived from pine tall oil that display surfactant properties. Properties of FP 1 and FP 2 are shown in Table 1. Addition of modified binder with FP 1 or FP 2 to aggregates causes a reduction in the friction between the aggregate-binder interface. The reduction of friction within aggregate interfaces allows use of lower mixing and compaction temperatures (Buss et al. 2014, Leng et al. 2013). Within this research work FP 1 and FP 2 are used as benchmarks to compare the performance of the new bio-derived WMA additives at low temperature.

Table 1. Properties of WMA additives FP 1 and FP 2 (Buss et al. 2014)

	FP 1	FP 2
Physical form	Dark amber liquid	Dark liquid
Specific Gravity at 25°C (77°F)	0.97	0.999
Conductivity at 25°C (77°F) ( $\mu\text{S}/\text{cm}$ )	2.2	4.3
Dielectric Constant at 25°C (77°F)	2 - 10	2 - 10
Viscosity ( $\text{Pa} \cdot \text{S}$ )		
at 27°C (80°F)	0.28 - 0.56	1.05 - 1.90
at 38°C (100°F)	0.15 - 0.30	0.47 - 0.85
at 49°C (120°F)	0.08 - 0.16	0.20 - 0.40

A surface mix with a 10 million ESAL design level approved for use from the Iowa Department of Transportation (DOT) was used to construct bulk specific gravity samples with air voids at

7% ± 0.5% and set heights at 115 mm. The blended aggregate gradation and source information used for this mix design is shown in Table 2. The job mix formula from the Iowa DOT was verified for each source aggregate's gradation.

Table 2. Mix Design Gradation and Supplier Information

Source		Martin Marietta (Ames)	Martin Marietta (Ames)	Manatts (Ames)	Hallet (Ames)	Martin Marietta (Ames)	Martin Marietta (Ames)	Blend
Aggregate		12.5 mm Limestone	9.5 mm Limestone	Quartzite	Natural Sand	Manuf. Sand	Agg Lime	
U.S. Sieve	Sieve, mm	29% % Passing	16% % Passing	15% % Passing	13% % Passing	15% % Passing	12% % Passing	100% % Passing
3/4"	19.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1/2"	12.5	79.7	100.0	100.0	100.0	100.0	100.0	94.1
3/8"	9.5	65.8	90.1	71.5	100.0	100.0	100.0	84.2
#4	4.75	37.2	20.5	5.1	96.8	95.2	99	53.6
#8	2.36	18.1	2.1	2.2	64.2	65.5	97	35.7
#16	1.18	12.5	0.7	2.0	33.7	36.3	75	22.9
#30	0.60	9.5	0.4	1.9	11.4	17.4	53	13.6
#50	0.30	7.5	0.3	1.9	0.9	6.5	38	8.2
#100	0.15	6.2	0.3	1.5	0.1	1.9	29	5.8
#200	0.075	5.2	0.3	1.2	0.0	0.8	22.3	4.5

### 3.2 Mixture Testing Methods

The SCB test has received a lot of attention among test procedures used for providing mix fracture characteristics at low temperature due to the simplicity and repeatability of specimen fabrication from standard laboratory compacted or field cored asphalt concrete samples (Chong and Kuruppu 1984, Krans et al. 1996, Marasteanu et al. 2004). Two modes of fracture can be studied using this testing method – mode I or mode II. The mode of fracture used in testing depends on the initial notch orientation. Within this research work, fracture from mode I will be examined and analyzed. Parameters found using this test are fracture energy ( $G_f$ ), fracture toughness ( $K_{IC}$ ), and stiffness ( $S$ ) (Li et al. 2008, Li and Marasteanu 2004, Li and Marasteanu 2010, Lim et al. 1993, Marasteanu 2012, Teshale 2012)

In 1984, the SCB test was first used to measure rock material fracture properties by Chong and Kuruppu. For this test a single edge notched semi-circular specimen is subjected to three-point loading as shown in Figure 1 (c). A constant crack mouth opening displacement (CMOD) of 0.0005 mm/s is achieved by applying a vertical compressive load at the top of each specimen. The CMOD is measured at the bottom of each specimen using an Epsilon clip gauge located between two buttons. More details on specific test conditions for the SCB are provided in AASHTO TP 105 - 13. The parameters fracture energy, toughness and stiffness are determined using load and load line displacement (LLD) results recorded for each tested specimen (AASHTO 2013). Within this research work the load line displacement was recorded through the displacement from a linear variable differential transformer (LVDT) built into the actuator (Marasteanu et al. 2012).

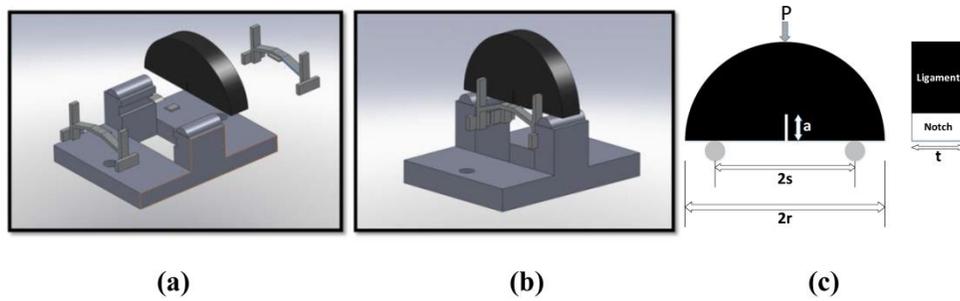


Figure 1. The SCB experiment setup (a) and (b) with one asphalt specimen (c) (Tang 2014).

The fracture energy, fracture toughness, and stiffness were calculated at  $-18^{\circ}\text{C}$ ;  $10^{\circ}\text{C}$  above the low temperature grade of the binder per AASHTO TP 105-13. Two bulk specific gravity ( $G_{mb}$ ) specimens for each WMA group were mixed and compacted at  $120^{\circ}\text{C}$  after two hours of curing to a height of 115 mm for a set mass to achieve  $7\% \pm 0.5\%$  air voids using a Superpave gyratory compactor (SGC) according to AASHTO T 312 and air voids were measured according to AASHTO T 166 (AASHTO 2011, AASHTO 2012). For the hot mix asphalt (HMA) control group two bulk specific gravity ( $G_{mb}$ ) specimens were mixed and compacted at  $140^{\circ}\text{C}$  after two hours of curing to a height of 115 mm for a set mass to achieve  $7\% \pm 0.5\%$  air voids using a SGC. Six SCB specimens were produced from each  $G_{mb}$  specimen with approximate dimensions of  $25 \pm 2\text{mm}$  in thickness, and  $150 \pm 9\text{mm}$  in diameter and notch length of  $15 \pm 0.5\text{mm}$  with the notch width being no wider than 1.5 mm. If the dimension limits were not met for a specimen then that specimen was discarded. If the air voids were not achieved to be  $7\% \pm 0.5\%$  then that specimen was also discarded. At least three specimens were randomly selected for testing from the twelve SCB specimens produced from each group's two  $G_{mb}$  specimens. Each specimen underwent preconditioning for two hours at  $-18^{\circ}\text{C}$  in the environmental chamber. At least three specimens were tested at  $-18^{\circ}\text{C}$  for each of the seven groups to take into account testing variability. For statistical analysis, an analysis of variance (ANOVA) was conducted to examine whether additives are significantly different from one another in terms of the parameters fracture energy, fracture toughness, and stiffness. A randomized complete block design was chosen to conduct the ANOVA with the block factor being Additive.

#### 4 DISCUSSION OF RESULTS AND ANALYSIS

The average fracture parameters (computed using at least three specimens) at the  $-18^{\circ}\text{C}$  test were computed for each additive group and are reported in Table 3. Table 3 reports the standard deviation and coefficient of variation for each additive group's average fracture parameters. The results are grouped from largest to smallest with respect to fracture energy (FA, None, IDB, FP 1, CI, FP 2, and RP).

The average fracture energy, fracture toughness, and stiffness values with their corresponding error bars (one standard deviation about the mean) for the test temperature  $-18^{\circ}\text{C}$  are shown in Figure 2. There appears to be differences between the additive groups. In order to analyze this,

statistical analysis was done according to a 95% confidence level to examine if there were statistically significant differences between the seven additive groups at -18°C.

Table 3. SCB Test Results

<b>Additive</b>	<b>Average <math>G_f</math> (<math>J/m^2 \times 10^{-3}</math>)</b>	<b>Stdev <math>G_f</math> (<math>J/m^2</math>)</b>	<b>COV <math>G_f</math> (%)</b>
FA	2.54	0.41	16.0
None	1.93	0.27	14.1
IDB	1.79	0.34	19.1
FP 1	1.56	0.40	25.4
CI	1.52	0.24	15.9
FP 2	1.36	0.07	5.1
RP	1.34	0.17	12.5
<b>Additive</b>	<b>Average <math>K_{IC}</math> (<math>Mpa \cdot m^{0.5}</math>)</b>	<b>Stdev <math>K_{IC}</math> (<math>Mpa \cdot m^{0.5}</math>)</b>	<b>COV <math>K_{IC}</math> (%)</b>
FA	1.00	0.04	3.8
None	1.04	0.11	11.0
IDB	0.93	0.22	23.8
FP 1	0.88	0.06	6.4
CI	0.91	0.06	6.7
FP 2	0.92	0.07	7.8
RP	0.81	0.17	20.8
<b>Additive</b>	<b>Average Stiffness (kN/mm)</b>	<b>Stdev Stiffness (kN/mm)</b>	<b>Stiffness COV (%)</b>
FA	3.90	0.03	0.9
None	4.57	0.92	20.0
IDB	4.57	0.61	13.4
FP 1	5.39	0.78	14.4
CI	5.07	0.54	10.6
FP 2	4.12	0.20	4.8
RP	4.84	1.18	24.4

As stated earlier a randomized complete block design was used to conduct the ANOVA, where the block factor examined is additive. The results of the ANOVA are shown in Table 4. Within the ANOVA air voids was not used as a factor because the air voids of the SCB specimens used in testing were  $7\% \pm 0.5\%$ . In Table 4, it is evident that Additive for the parameter fracture energy is a statistically significant source of variability. For Additive to be a statistical significant source of variability, the p-value must be less than or equal to 0.05. This means that the additives were not found to be statistically significantly different from one another overall according to a 95% confidence level for the parameters fracture toughness and stiffness.

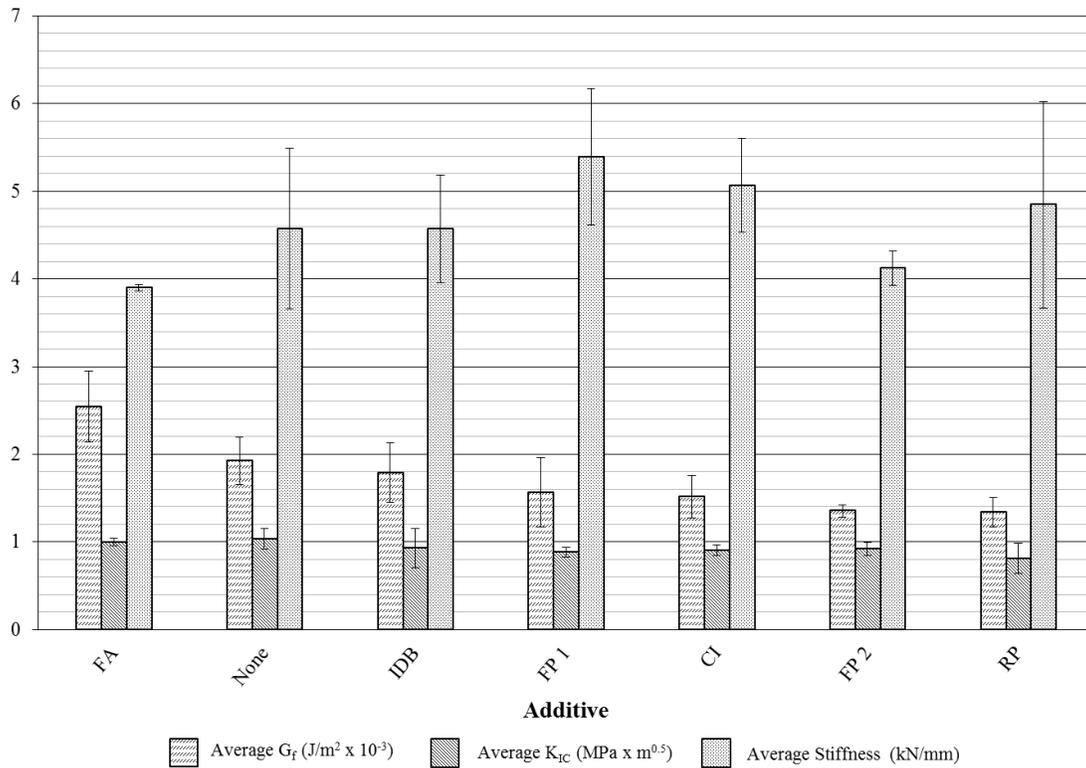


Figure 3. Average Fracture Energy ( $G_f$ ), Fracture Toughness ( $K_{IC}$ ), and Stiffness of Bio-derived/Chemical Additives.

Table 4. ANOVA results for  $G_f$ ,  $K_{IC}$ , and Stiffness

$G_f$ ( $J/m^2 \times 10^{-3}$ )	Source	DF	SS	MS	F Ratio	Prob > F
	Additive	6	3.3497325	0.558289	6.6141	0.0012*
	Error	16	1.3505505	0.084409		
	Total	22	4.7002831			
$K_{IC}$ ( $MPa \times m^{0.5}$ )	Source	DF	SS	MS	F Ratio	Prob > F
	Additive	6	0.11274573	0.018791	1.053	0.4291
	Error	16	0.28552362	0.017845		
	Total	22	0.39826935			
Stiffness (kN/mm)	Source	DF	SS	MS	F Ratio	Prob > F
	Additive	6	4.9012815	0.8168803	1.4766	0.2480
	Error	16	8.851678	0.55323		
	Total	22	13.752959			

Note: DF – degrees of freedom, SS – sum of squares, MS – mean square, F Ratio –  $MS_{additive}/MS_{error}$ .

From Table 4 it can be discerned that the best parameter for statistical analysis is fracture energy. The fracture energy is determined by dividing the work of fracture (area under the load vs. the load line displacement curve) by the ligament area (area of specimen prior to testing at which fracture will occur). Since fracture energy was found to be a statistically significant factor according to additive choice, closer examination is needed for the differences between the additives. To do this, a least square means plot was done using the Tukey Honestly Significantly Different (HSD) t-test for additive choice under the parameter fracture energy. This plot is shown in Figure 4. Additives that are not connected by the same capitalized letter are statistically significantly different from one another.

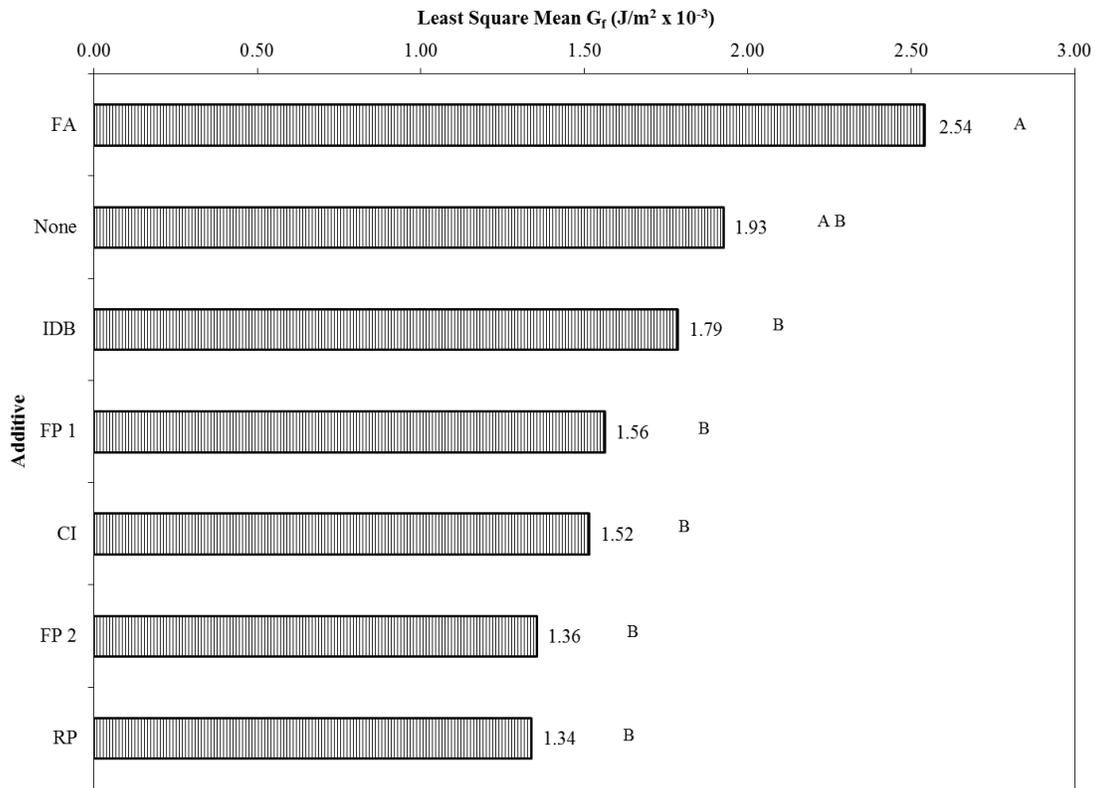


Figure 4. Tukey Honestly Significant Differences (HSD) between Additives for  $G_f$ .

For fracture energy it is shown that some of the additives are statistically significantly different from one another according to a 95% confidence interval. FA is shown to be statistically different from all the additives except the control group (None). However, the control group (None) is not statistically different from any of the six additives. From the statistical analysis the bio-derived additive FA performed equal to the control group (None) and better than the other five additives used in this research.

From this analysis it was determined that all the additives were not found to be different from the control HMA (none) for fracture energy. However, upon closer examination of the results FA appears to be different and performs better at low temperature than the other five additives in terms of fracture energy. It is hard to discern from these results if a chemical or physical

interaction or both instances is occurring between the additives, binder and aggregates at low temperature. Subsequent research work including analytical chemistry needs to be done to understand the mechanism for performance improvement and to evaluate all the additives at intermediate and high temperatures as well. For this to be done thorough chemical testing and analysis needs to be done.

## 5 CONCLUSIONS AND RECOMMENDATIONS

Warm mix asphalt performance during the semi-circular bend (SCB) test has shown that bio-derived materials can be used as WMA additives and are comparable to control hot mix asphalt (HMA). From an overall statistical analysis the additives are shown to be statistically significantly different from one another for fracture energy, but not for fracture toughness and stiffness results. Through closer examination it was found that all the additives are not found to be different from the control group (None) in terms of fracture energy. The additive FA is shown to be different from the other five additives for fracture energy and shows improved performance at low temperature. From the analysis of fracture energy for several additives with similar properties it was shown that there are no adverse impacts to low temperature properties of warm mix asphalt when it comes to fracture performance.

Limitations of this paper were that testing only took place at  $-18^{\circ}\text{C}$ , a polymer modified form of PG 58-28 binder was not used, and that other testing methods were not used to evaluate low temperature performance of the additives. In the future it would be beneficial to have testing take place at multiple temperatures to better understand how the additives impact mix performance, and to examine if relationships exist. Use of another binder in this study, e.g. a polymer modified form of the PG 58-28 binder would be useful as it would allow a look at whether the additives make an impact to mix performance through statistical analysis of interactions. Additional testing with other methods of measuring low temperature fracture would also be beneficial as results could be verified more thoroughly. These three factors would provide more emphasis to a statistical analysis of this research work. In the future, research will be done with the currently used bio-derived/chemical additives with a polymer modified form of the PG 58-28 binder (a PG 64-28 binder) at  $-18^{\circ}\text{C}$  with additional testing taking place at temperatures of  $-6^{\circ}\text{C}$  and  $-12^{\circ}\text{C}$  for both the PG 58-28 and the PG 64-28 binders, as well as testing all aforementioned groups with the disk compact tension (DCT) test. Subsequently, analytical chemical testing will be done on the additives by themselves, as well as the control binders and modified binders with WMA additives.

## 6 ACKNOWLEDGEMENT

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