Evaluation of the Effect of Crumb Rubber Properties on the Performance of Asphalt Binder

by

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Abstract

The purpose of the research was to determine the most influential factors of crumb rubber that affect the performance characteristics of crumb rubber modified binder. The first phase of the study required the characterization of crumb rubber products used for asphalt binder modification. The second phase of the study consisted of asphalt binder modification and quantification of the influence of crumb rubber properties through performance testing of the binder. The third phase of the study investigated strategies to reduce the separation tendency of crumb rubber modified binder. The study shows that asphalt binder is influenced by rubber content and the mean particle size of crumb rubber modifier. The study concluded that mean particle size is the most influential crumb rubber property characterizing modified asphalt binder separation tendency.

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Table of Contents

Abstract	i
Acknowledgments	iii
List of Tables	v
List of Figures	vi
Chapter 1: Introduction	1
Chapter 2: Literature Review	5
Chapter 3: Rubber Characterization	26
Chapter 4: Characterization of Crumb Rubber Modified Binders	40
Chapter 5: Statistical Analysis of Crumb Rubber Properties on Modified Binders	59
Chapter 6: Effect of Additive	67
Chapter 7: Conclusions and Recommendations	74
References	78
Appendix A	A-1
Appendix B	B-1

List of Tables

Table 1: RO-TAP Results for Crumb Rubber Particle Analysis	31
Table 2: Additional Crumb Rubber Properties	32
Table 3: Crumb Rubber Designated Mesh Sizes and Material Source	33
Table 4: Crumb Rubber Particle Size	34
Table 5: Thermogravimetric Results	36
Table 6: Surface Area Results	39
Table 7: Viscosity of Rubber Modified Blends	46
Table 8: Crumb Rubber Modified Binder Performance Grade	47
Table 9: AASHTO MP19-10 MSCR Performance Criteria	49
Table 10: MSCR Results	51
Table 11: Separation DSR Results	54
Table 12: Softening Point Results	59
Table 13: Pearson Correlation Table for Binder Performance	61
Table 14: True High Performance Grade ANOVA	63
Table 15: True Low Performance Grade ANOVA	64
Table 16: Separation Performance Grade Percent Difference ANOVA	65
Table 17: Separation Softening Point Percent Difference ANOVA	66

Table 18: Summary of Statistical Analyses	. 67
Table 19: Performance Grade of Additive Modified Materials	. 70
Table 20: Softening Point for Additive Modified Materials	. 72
Table 21: MSCR Results for Additive Modified Materials	. 73

List of Figures

Figure 1: Example Crumb Rubber Sample Preparation	41
Figure 2: Example Vortex Resulting in Properly Mixed Crumb Rubber Binder	43
Figure 3: 25mm Disc of Binder and DSR Testing Plates	44
Figure 4: Example 1mm Testing Gap	45
Figure 5: Example Set of Separated Materials	53
Figure 6: Ring and Ball Softening Point Apparatus	57

Chapter 1: Introduction

Background

Approximately 270 million scrap tires are produced annually in the United States. In addition to this annual waste, there are an estimated 800 million scrap tires stockpiled. Stockpiled tires are an environmental danger by providing fuel sources for unplanned fires as well as creating breeding habitats for vermin and mosquitoes (Rafat Siddique, 2004). Recycling or repurposing scrap tires alleviates many of the environmental issues associated with stockpiling and growing waste of the materials. An estimated 80 percent of the scrap tires produced annually since 2003 have been recycled or repurposed (Amirkhanian, 2006). The majority of the scrap tires have been utilized in tire derived fuel applications where tires are used as energy for heating. Civil engineering applications are the second largest sector utilizing scrap tires. These applications range from embankment fill to modifiers for asphalt pavements. Use of recycled rubber as an asphalt pavement modifier has been common for decades in several states. Field performance of thousands of projects has shown when used properly, scrap rubber can be successfully integrated and recycled in asphaltic mixtures.

While experience has shown using recycled rubber in asphalt mixtures is feasible, there are varying methods for incorporating the rubber into pavement which affect the overall performance of the mixture. The two main methods of rubber modification are the dry method and the wet method. The dry method was developed in 1986 by Takallou and utilizes coarse rubber particles as a portion of the aggregate gradation. Dry mixes can incorporate a relatively high amount of rubber with typical mixes consisting of approximately 3 percent rubber. The wet method stems from a binder modification process developed by Charles McDonald in 1964. This method utilizes finely ground rubber particles to modify asphalt binder used in asphalt paving mixtures. The wet method uses rubber contents up to 20 percent by the weight of the asphalt binder (Brown, 1993). While the dry method can incorporate more recycled rubber, inconsistent dispersion of the rubber particles throughout the mixture have led to poor performance on numerous projects. Conversely, the particle size of the rubber used in the wet processes and the addition of the rubber directly to the binder better disperses the rubber material allowing modified mixtures to have more effective rubber modification. This process has led to pavements that exhibit better performances compared to dry method modified pavements (Marvin Myhre, 2002).

Different grinding temperatures exist for producing crumb rubber products which ultimately can affect the texture of the rubber itself. The two main categories of crumb rubber production temperatures are ambient grinding and cryogenic grinding.

Ambient grinding occurs at normal or slightly elevated temperatures and yields materials with high surface areas and irregular shapes and textures. Cryogenic grinding

utilizes liquid nitrogen to make the rubber brittle for grinding. Cryogenic grinding results in materials with relatively low surface areas and regular blocky shapes (West, Randy, 1998). Common processes for grinding the materials are Crackermilling, Granulating, Micromilling, and Cryogenic. Past binder testing has shown that grinding method has a measurable influence on the binder properties and storage characteristics (West, Randy, 1998).

As crumb rubber can alleviate waste, reduce environmental hazards, and enhance the performance characteristics of asphalt pavements, it is important to characterize how crumb rubber properties influence the engineering properties of rubber modified asphalt binders. Research of the effect of crumb rubber properties on the pavement performance could lead to determining desirable grinding methods, particle sizes, and modification strategies. This could lead to a reduced dependence on synthetic polymers currently used for modifying binders to reduce the temperature susceptibility of asphalts. Assessing how the properties of the crumb rubber materials influence the performance of asphalt binder and asphaltic mixtures can assist states in ensuring high performing rubber pavements while continuing the use of recycled rubber as an asphalt modifier.

Objectives

The objective of this study was to evaluate how rubber properties affect the properties of an asphalt binder. The study utilized unique crumb rubber products to

modify a single asphalt binder. The modified binders were then characterized with performance grading (PG), multiple stress creep recovery testing (MSCR), separation testing (Cigar Test), and softening point testing (Ring-and-Ball). The objective was met by analyzing the performance characteristics of the modified binders to determine the effect of the rubber properties on the binder performance.

Scope

The study characterized twelve unique crumb rubber products and explored how those characteristics affect blends of the products with asphalt binder. The twelve unique crumb rubber products consisted of six ambient ground and six cryogenically ground materials. A single binder was used with each crumb rubber product to create modified asphalt binders. Each crumb rubber product was introduced at a rate of ten percent by weight of binder. In addition to the twelve modified blends at ten percent rubber content, two additional modified binders were created with a rubber content of fifteen percent. The two additional materials were modified with an ambient ground material and cryogenic ground material respectively. The fourteen modified materials were then tested to characterize the performance characteristics with performance grading (PG) and elastic response (MSCR). The stabilities or uniformities of the stored materials were also evaluated through the use of separation testing (Cigar Tube Testing) and softening point testing (Ring-and-Ball Testing). The results from the modified performance testing were used to determine the extent of the rubber property

influence on the binder properties. Statistical analyses were used to determine how the variability in binder performance can be described by measureable crumb rubber properties. In addition to the fourteen crumb rubber modified binders analyzed, a hybrid binder and Vestenamer modified binder were analyzed to determine the effect of stabilizers on crumb rubber modified materials.

Organization

The thesis consists of seven chapters. The second chapter contains the literature review and critical findings from previous research utilizing scrap tire rubber in asphalt mixtures. The third chapter of this study discusses the methodology and findings from the characterization of the crumb rubber products analyzed in this study. The fourth chapter discusses the methodologies used to create the modified binders. This chapter also includes the methodologies for conducting the binder performance testing as well as the results from the binder testing. The fifth chapter focuses on the statistical analyses used to determine the effect of the crumb rubber properties on the properties of the crumb rubber modified binders. The addition of stabilizers and the influence it has on the performance of crumb rubber modified binders is discussed in chapter six. Chapter seven focuses on the conclusions of the research as well as the recommendations that can be made from the study.

Chapter 2: Literature Review

Background

Approximately 270 million waste tires are generated annually in the United States. In addition to the continued waste, the United States has an estimated 800 million stockpiled waste tires (Amirkhanian, 2001). In the year 2000, 45 percent of the annual 270 million waste tires were found to be disposed in landfills or illegally stockpiled (Rafat Siddique, 2004). This number of stockpiled tires creates danger to the environment by providing a breeding ground for vermin and mosquitos while also creating a fuel source for unexpected fires (Rafat Siddique, 2004). To magnify the problem of the stockpiled tires, limited tire disposal options are available.

Due to these concerns, landfilling waste tires is not the practical long term solution for the disposing scrap tires. These issues have led many researchers and industries to evaluate the use of the waste tires as recyclable material and even fuel sources. As of 2001, the largest market for scrap tires was the cement industry that used the tires as fuel to heat kilns (Nongnard Sunthonpafasit, April 2003). Approximately 125 million tires

are used annually for tire derived fuel (Rafat Siddique, 2004). Other practical solutions for repurposing the waste material include civil engineering applications, producing carbon black, creating recycled rubber products and as modifiers for plastic goods.

A 1989 Transportation Research Board synthesis on potential civil engineering applications for scrap tires showed modifiers for asphalt binder, light weight embankments, retaining walls, safety hardware, and pavement subbase potential recycling uses (FHWA, 1992). Using scrap tires as a modifier for asphalt materials was found to be the most practical solution for repurposing the waste material which could successfully incorporate two to six tires per metric ton of modified asphalt binder. (FHWA, 1992). The use of scrap tires as a modifier for asphalt binder requires that the waste material be ground to a fine crumb-like size which is commonly referred to as crumb rubber modifier (CRM).

The process of modifying asphalt binder with crumb rubber is called the wet method. The name reflects that the additive or tire modifier is being introduced directly to the liquid or wet portion of the hot asphalt mixture, the asphalt binder. A second method for introducing rubber into asphalt is referred to as the dry method where rubber is directly added to the aggregate during the production process. The dry process uses aggregate-sized rubber particles. Additionally, the process will dictate the amount of rubber which can be used in the mix. The wet method typically uses a rubber content of 10 to 25 percent of crumb rubber modifier by weight of the binder. This results in the rubber comprising 0.5 to 1.3 percent of the final modified mixture. Conversely, the dry method integrates aggregate sized rubber at a rate of 3 percent by the weight of the

aggregate (Jay L.McQuillen, 1987). The dry method can only be applied to asphalt mixture applications whereas the wet method can be utilized in crack sealing, surface treatments, and other hot mix applications (FHWA, 1992).

One disadvantage of the dry method is mixture uniformity. Differences in rubber and aggregate densities cause the rubber and aggregate to separate during the mixing and hauling processes (Marvin Myhre, 2002). The separation can cause large pockets of rubber to exist in the pavement which lead to construction and long-term performance problems. Additionally, when rubber is exposed to the asphalt binder, the rubber absorbs some the light ends and oils of the binder. When this occurs, the rubber modified mixture is weakened (Marvin Myhre, 2002). Dry mix methodology does not provide any significant structural benefit when compared to traditional hot mixes and the size of the rubber limits the effectiveness of the rubber as a modifier in the mix.

While the quantity of rubber used in the wet process is not as great as that in the dry process, the wet method of directly modifying the asphalt binder has numerous benefits. By directly modifying the binder with crumb rubber, there is less rubber separation in the final mixture generating a more homogenous material when compared to the dry method mixtures. The smaller particles allow for better dispersion of the rubber and increase the influence of the rubber modification on the total mix lending itself to be highly durable, quieter and smoother (Marvin Myhre, 2002).

Despite the benefits of using CRM, obstacles related to the practicality and functionality of the product need to be overcome for widespread implementation.

Practical obstacles include the potential for higher costs, lack of specification, rubber-

modified mixture recyclability, and other environmental concerns (Amirkhanian, 2001). Functional obstacles include, but are not limited to product variability. Rubber properties vary with source and age. When integrated into asphalt, inconsistency in rubber source properties could create undesirable paving materials.

While many of the practical issues with the use of crumb rubber can be overcome through experience, the functionality of the material need continual evaluation. Sunthonpagasit (2003) suggests that particle size and quality should be evaluated to assess performance. In addition to particle size and quality, characteristics such as surface area might affect overall performance. Studies distinguishing critical crumb rubber factors could eliminate many functional obstacles. The results would also assist in a market impact studies as suggested by Sunthonpagasist.

History

Crumb rubber was first used as a binder modifier in the United States in the 1950's; however, the first application of crumb rubber as a modifier for asphalt pavements construction occurred in 1964. This material was created by Charles McDonald who applied CRM asphalt as a test patch at the Sky Harbor Airport of Phoenix, Arizona, to seal existing surface cracks from water exposure and to prevent reflective cracking. Upon evaluation, the experimental crumb rubber patch was deemed successful and the product was called McDonald Technology (Brown, 1993). This innovative technology led to the development of other liquid asphalt rubber

applications. Stress absorbing membranes (SAM) and stress absorbing membrane interlayers (SAMI) were developed in the early 1970's. Challenges with SAM and SAMI layers were the amount of loose aggregate on the roadway when used as a chip seal and high quality construction on rough existing pavements. The high viscosity of the CRM binder inhibited good construction practices. To reduce the viscosity, kerosene was used to cut the CRM binder. This led to the development of customized extender oils which modify the workability. Despite early success, the lack of understanding and equipment for producing the technology was a concern (Brown, 1993).

Despite concerns, the United States Congress passed legislation requiring state agencies to utilize and study the use of crumb rubber as a recycled paving material to counter the surplus of stockpiled waste tires. The Intermodal Surface Transportation Efficiency Act (ISTEA) of 1991 and the Resource Conservation Recovery Act were used to limit funding for state agencies that did not comply with the recycling requirements of the federal government. The requirement forced many states to be proactive in the evaluation of rubber asphalt. Many states reacted by passing legislation to ensure that the use of scrap tires was evaluated and implemented. By the time the federal law was passed, 44 states had already addressed the approaching legislation by formulating state specific laws and strategies for implementing crumb rubber (FHWA, 1992). The original federal mandate required that each state have a 5 percent minimum percentage of rubber modified asphalt of the total tonnage of asphalt produced in 1994. By 1996 the requirement CRM asphalt mixtures was eliminated from the 1991 ISTEA mandate. This withdrawal from the original mandate and removal of penalties for

compliance failure were integrated through the approval of the 1995 National Highway System Designation Act, Section 205b. While these items were retracted, the requirement of studying and developing technologies associated with researching the rubber material remained. This was reinforced by section 327 of the National Highway Designation Act by requiring continued research of crumb rubber modified material in the Strategic Highway Research Program (SHRP) specifications. This amendment also required that future procedures developed for the use of the crumb rubber materials include consultation of industry and interest parties associated with the process of recycling of the crumb rubber materials.

In 1988, Florida became one of the first states to pass legislation to evaluate the use of CRM in paving mixtures. FDOT partnered with The National Center for Asphalt Technology (NCAT) and conducted a CRM feasibility study using a continuous mixing process. The study titled *Investigation and Evaluation of Ground Tire Rubber in Hot Mix Asphalt* was focused on using the most technologically advanced practices in implementing the use of ground rubber tire into asphalt mixes to determine if the resulting material could conform to the necessary FDOT standard for performance (NCAT, 1989). The study addressed five specific areas. Two of the tasks of the study addressed the functional issues with the use of scrap tires such as Specification and Design Factors and Pavement Performance. The Specification and Design Factors section investigated the necessary changes needed in the current practices and designs that would be required to implement scrap tires into future FDOT projects. The Pavement Performance Issues section investigated the effect of rubber on the pavement

performance and longevity. The research conducted by NCAT found that rubber pavements could exhibit acceptable performance in the field. The study also found that modifications in the current practices and specifications would make the integration of rubber into designs feasible. The recommendations from NCAT proposed acceptable material types, material sizes, rubber concentrations, methods for handling rubber RAP, future research projects, and data collection strategies. The report led to FDOT constructing sections of rubberized pavement for data collection and observation (NCAT, 1989).

The Federal Highway Administration report titled *State of the Practice-Design* and *Construction of Asphalt Paving Materials with Crumb Rubber Modifier* reinforced the need for continued research by stating that two principal issues exist for the use of crumb rubber modifier, recycling asphalt pavement and continual CRM asphalt research. It was requested that performance evaluations be conducted on state-by-state basis due to differences in design and construction practices between states.

The recommendations made by this report led many states such as Arizona,

Kansas and Texas to evaluate their respective practices including pavement durability

and pavement-tire noise. A recent study titled the Quiet Pavement Pilot Program is an

example of a research program designed to address the performance issues of rubber

asphalt critical to the state by partnering the Arizona Department of Transportation with

the Federal Highway Administration to evaluate the effectiveness of rubber pavements

in reducing pavement noise. The study assessed if an asphalt rubber friction course

(ARFC) could reduce noise at the tire-pavement interface.

Another study titled *Utilization of Crumb Rubber in Asphaltic Concrete Mixtures-South Carolina's Experience* was developed to assess and monitor the performance of CRM test sections using three methods of CRM. These methods consisted of the wet method, the dry method, and a method described as trickle method where rubber was added manually to a pugmill. The research distinguished durability issues between the test sections. Ultimately, the results from the collaboration between the South Carolina Department of Transportation and the FHWA were used to dictate the future research and integration of crumb rubber for the state of South Carolina (Amirkhanian S. N., 2001).

Grinding Method

The method used to grind scrap tire into a usable crumb size has been suggested to be a factor in dictating the performance of rubber modified products. The primary rubber property controlled by grinding method is particle size. Grinding method has also been found to affect shape, texture, and surface area of the rubber. These rubber properties can influence the rubber binder interaction and can affect the viscosity and storage stability of rubber modified materials (West, Randy, 1998). Two basic strategies exist for producing crumb rubber. The first strategy utilizes grinding tools like steel drums, granulators or steel plates to grind the scrap tire into a usable size. These grinding tools typically operate at ambient temperatures and the material produced from ambient grinding is categorized as ambient ground rubber. The second strategy for

exposed to liquid nitrogen making the material brittle. Impact loading is then used to fracture the material into usable sizes. Crumb rubber produced through the use of liquid nitrogen and impact crushing is categorized as cryogenic ground rubber. Materials produced with the two methods have been found to have noticeable differences in shape and texture (FHWA, 1992). Because of the impact of the rubber particle characteristics on the modified binders, many studies have been conducted to quantify the influence of grinding method on the performance of rubber modified materials.

The 1989 report titled *Investigation and Evaluation of Ground Tire Rubber in Hot Mix Asphalt* conducted literature reviews on the methodologies used to create ground tire rubber. The investigation found that ambient ground materials are shredded and ground at temperatures near or above ordinary room temperatures. The resulting material was found to have a sponge-like surface that increased surface areas for any particular particle sizes. The study also determined that cryogenic ground materials are produced at temperatures below the embrittlement temperature of the rubber. The resulting materials were found to have flat surfaces that reduced the surface area of the materials when compared to similar particle sizes of ambient ground materials. The study reviewed research conducted by the Australian Road Research Board and found that increased surface areas increased the reaction rate of the crumb rubber with binder. The review also found that low surface area crumb rubber material was undesirable due to the morphology of the material which limited the rubber reaction in the binder. The findings from the research conducted by the Australian Road Research

Board were one reason ambient ground material was recommended by NCAT for use in future FDOT experimental sections. NCAT concluded that cryogenically ground materials were not satisfactory and should be prohibited from use in the proposed experimental designs.

A 1998 study titled Effect of Tire Rubber Grinding Method on Asphalt-Rubber Binder Characteristics investigated the influence of grinding method on asphalt rubber properties. This study was conducted for the Florida State Department of Transportation and the purpose of the report was to determine if any measurable differences could be identified between the materials produced with ambient and cryogenic grinding. The study listed four known methods that produced crumb rubber and identified how each method created uniquely different particles. These methods were listed as a Crackermill Process, Granular Process, Micromill Process, and Cryogenic Process. The report identified the Crackermill Process as the most common method for producing crumb rubber. Crackermilling was conducted by using rotating steel drums that were designed to tear scrap rubber into usable sizes. This method used ambient temperatures to produce irregularly shaped materials with high surface areas. The second method described in this report was the granulator process which used ambient temperatures along with revolving steel plates to cut the scrap tire into a useable size. The material from this process was described to have uniform shapes of a cubic nature. The Granulating process was found to produce materials with low surface areas. Micromilling was described as a finishing process that could be used to shred the material to a very fine size. This process requires adding water to the rubber to create

slurries that can be passed through an abrasive disc for grinding. The cryogenic process was identified as the method that produced materials with glassy smooth textures. The materials produced with this method were described as angular with uniform shape.

The description of the general material properties caused by each grinding method was an indicator of the variability caused by the grinding methods. The discrepancies in the material characteristics validated the need for a study to determine how each material type affects the binder performances.

The study investigated the effect of grinding methods on the binder characteristics of modified materials. To conduct the analysis, four crumb rubber manufacturing facilities were contacted to obtain unique specimens. Two ambient ground materials were obtained in addition to one cryogenic and one hybrid cryogenicambient material. The four specimens were subjected to rubber particle characterizations. The rubber materials were characterized with sieve analyses, surface area measurements and bulk density determinations. The properties of the binder modified with the four products were characterized through viscosity measurements, rubber particle settlement testing, and drain down testing. The study found that the differences in shape, texture and physical properties caused by the two grinding strategies were significant. Binder testing results showed that grinding processes caused measurable differences in the performance of the modified binder while highmagnification images of the four rubber products indicated visible differences in the texture and surface area. Imaging showed that ambient ground materials have the potential for high surface areas when compared to cryogenically ground materials. The

research also found that the wet grinding methods result in materials with higher surface areas when compared to the dry ambient ground and the cryogenic ground materials. Binder testing showed materials with the highest surface areas were found to create modified materials with higher viscosities. The high surface area materials were found to exhibit the least amount of rubber particle settlement during the separation testing. The investigation showed specific surface area to be the most significant factor for the rubber material. Because ambient materials were found to have the most texture and surface area of this study, modified binders produced from these methods were found to be more desirable.

A Clemson University study that was published in 2007 titled *The Effect of Crumb Rubber Modifier (CRM)* on the Performance Properties of Rubberized Binders in HMA Pavements investigated the effect of both grinding methods and rubber content of rubber on Superpave binder tests. The investigation utilized ambient and cryogenic ground rubber materials with similar particle sizes. Both materials were designated as a size 40 mesh material and used at rubber contents of 5, 10, 15, and 20 percent by weight of binder. The study also used three different sources of 64-22 performance grade binders. For each source binder and rubber content, the viscosities of the modified binders in the analysis indicated that the tire grinding method caused statistical differences. The results from the elastic response of the material showed that the grinding method affected the majority of the testing scenarios. The study corroborated the findings from previous research by showing that ambient ground rubber produced modified binders with higher viscosities and less temperature

susceptibility when compared to materials modified with cryogenically ground rubber. While this analysis showed that the high performance grade properties could be influenced by the grinding method used to generate the crumb rubber material, the study showed that the low temperature performance grade may not be affected showing statistically similar m-values.

Effect on Binder

The main purpose of binder modification is to limit the temperature sensitivity of the material. Many different methods of grinding or shearing rubber have been developed to create rubber modifier which decrease the temperature sensitivity of the asphalt binders. The primary crumb rubber properties that are influenced by production methods are particle size and surface area which can be critical for rubber-binder interaction. The addition of rubber has been shown to modify the viscosity, phase angle, complex shear modulus, and elastic response of crumb rubber modified binders which are indications of changes to the material's temperature susceptibility and loading (Marvin Myhre, 2002).

Due to numerous rubber sources and varying methods used to create crumb rubber, studies characterizing the impact of crumb rubber properties in modified binders are important to classify the performance of the modified materials as well as provide guidance for future uses of the materials. In addition to studying the effects of

rubber properties on the modified binder performance, the stability of stored modified binders warrants investigation.

A study titled Crumb Rubber Modification of Binders: Interaction and Particle Effects describes the fundamental interaction between crumb rubber particles and asphalt binder which occurs in two steps. First, the rubber particles are added to the binder and the particles begin to swell from absorbing the lighter oily fractions within the binder. The second step is an increase in the viscosity of the binder due to the absorption of the oily light ends. In addition to studying how crumb rubber and its measurable properties affects the binder, the report states that other non-rubber factors can attribute to the interaction between the rubber and the binder. The amount of light ends or oil fraction can limit the effect of the rubber. This indicates that the binder source is important and could limit the effectiveness of the rubber modifier. The temperature at which the two materials are blended can also influence the interaction. Heitzman (FHWA, 1992) reinforces the importance of reaction temperature by stating that temperature, length of exposure, and blending energy can significantly affect the reaction of any crumb rubber in a binder. The viscosity issues discussed by Putman and Amirkhanian indicate that compatibility between the rubber and the binder could be an issue in allowing the rubber to be properly activated during blending (Amirkhanian B. J., 2006).

The interaction and particle effects study utilized a single ambient ground crumb rubber source to identify the influence of the rubber on the binder. To create different particle sizes, one rubber source was sieved into multiple mesh sizes to represent the

range of crumb rubber sized materials available in the United States. After executing a modification strategy utilizing three binder sources, two rubber contents and three rubber mesh sizes the modified binders were subjected to viscosity testing and DSR testing. The results from the investigation were able to identify that rubber content was a critical component influencing the viscosity and complex shear modulus. The viscosities of the materials were found to increase with decreasing particle size while the complex shear modulus increased with increasing particle size. The use of the three binder sources showed that binder source can influence the particle effect of the crumb rubber on the binder. While the complex shear modulus was found to decrease with decreased particle size, this was only a general trend. The statistical analysis from this study showed that there was little to no change in complex shear modulus values between specimens from the same binder type modified with different mesh sizes.

Rubber content and particle size had the greatest statistical influence on the binder performance.

Sohee Kim, Ssu-Wei Loh, Huachun Zhai, and Hussaim U. Bahia investigated the effect of crumb rubber on modified binder viscosity. The study utilized three gradations of crumb rubber, two rubber contents and two grades of binder to determine the effect of particle size and rubber content on binder performance using viscosity testing, frequency sweeps, bending beam rheometer testing, and binder separation testing. The study found that both rubber content and particle size have a statistically significant effect on the viscosity. The critical findings from the viscosity analysis found that higher rubber contents and smaller particles resulted in higher viscosities. Rubber content was

testing illustrated that the rubber content had a statistically high impact on the effect of crumb rubber on the binder. Sensitivity analyses found that particle size had little to no effect on the results from the frequency sweeps. The study showed that base binder was the highest contributor to binder performance for all rubber contents and particle sizes. Particle size had the greatest effect on the BBR performance. Stability of CRM asphalt is a critical parameter for assessment. The laboratory separation testing found that all crumb rubber modified materials were susceptible to separation. While this study confirmed that crumb rubber modified materials were susceptible to separation, the results did not indicate which crumb rubber properties were critical in describing the behavior.

One study that determined the crumb rubber properties that affected the storage stability of modified binders utilized one binder source and one ground rubber tire source (F.J. Navarro, 2004). Fractions of the original rubber gradation were created by screening the material. This resulted in five different mean particle size blends. The modified materials then underwent rheological testing and storage stability testing. The viscosity of the modified materials in this study was shown to increase with increased rubber particle sizes. The study addressed the difference by stating that particle aspect ratio and morphology of any particle can lead to higher viscosities. After identifying this irregularity, the study evaluated the storage stability of the blends. The study illustrated that the particle size had an effect on separation showing that particle sizes 0.29mm or

smaller were more stable; however, this may not be true for all rubber types or rubber contents.

Effect on Mixture

The most important aspect of using crumb rubber is the performance evaluation of rubber modified asphalt pavements. The findings from the modified binder testing show that crumb rubber changes the rheological properties of the binder. This would suggest that the performance properties of the mixture could be enhanced by the rubber modification of the binder. Studies have shown rubber modification of asphalt pavement could enhance the performance of low temperature flexibility, higher strength when wet and more resistance to oxidative hardening (Myhre 2002). Other reports show addition of rubber can modify thermal cracking, rutting, reflective cracking, and aging (Amirkhanian 2006). While the logic between increased binder performance leads to increased pavement performance seems reasonable, many institutions like NCAT and FHWA indicate that laboratory material performance has not always correlated well with measured field performances. Because of the differences with laboratory and field performances, the need for field experiments is paramount.

It is well understood that CRM mixtures are different than conventional asphalt mixtures. CRM mixtures require more binder to be added to mixtures. The increase in binder along with the increase in viscosity of the binder due to the addition of rubber causes a thicker film of binder to be placed on the aggregate (FHWA, 1992). Laboratory

testing has shown the additional binder due to rubber content can cause the modified materials to absorb elastic stress better than conventional mixtures. While increased elastic properties are desirable, a common problem with the increased binder content is flushing and reduced air voids. With the increase of binder, existing mix designs may not provide enough void space to account for an increased volume of rubber modified binder. The necessary modifications to the aggregate structure could allow for the proper voids while limiting flushing. Slight modifications in mix designs can allow for rubber to be used in dense, gap-graded, and open-graded mixes. For open-graded mixtures, increased mixing temperature would be necessary due to increased binder viscosity.

The 1989 NCAT study that evaluated ground rubber tire in hot mix asphalt for FDOT found that three performance parameters were significantly affected by the addition of rubber to the mixture. The study utilized literature review, laboratory data and field experiments to determine that fatigue life, permanent deformation, and resilient moduli from asphalt rubber mixes behave differently than conventional mixes. The fatigue portion of the study found that dense graded mixtures modified with rubber decrease strain levels after repeated loading when compared to conventional mixtures. The permanent deformation portion of the study demonstrated that rubber modified mixtures exhibited higher moduli values than conventional mixtures at elevated temperatures. The study was also able to identify that the resilient moduli of mixtures can be increased through the addition of rubber modifier. While these conclusions show

promise in the use of crumb rubber materials, the findings were very climate specific and were determined specifically for FDOT to guide future research experiments.

Similar conclusions on field fatigue behavior of asphalt rubber pavements to the 1989 NCAT investigation on ground rubber tire in hot mix were found by Roberts, Kandhal, Brown and Dunning. A field aging study compared the fatigue life of a dense graded conventional mix to the fatigue life of a gap graded asphalt rubber mix. The two mixes were obtained from a 10 year old field section of a parking lot in California. The study determined the rubber asphalt section possessed lower stiffness for both aged and unaged specimens. This indicated that the rubber influenced the rate of aging; however, the effect of fatigue performance was negligible.

Raghu Ram Madapati evaluated the permanent deformation of materials with and without crumb rubber. The materials analyzed for this study were a control mix, two wet crumb rubber mixes, and one dry process crumb rubber mix. The evaluations were conducted through the use of VESYS 3A-M software that utilized elasto-plastic theory and seasonal inputs to predict pavement performances over 20 years. The analysis found the dry method of rubber modification to have the worst deformation followed by the control mixture. The wet method process for creating asphalt rubber was found to be the best performing or materials with the least amount of rutting in the prediction analyses. The study concluded that experimental crumb rubber sections should be constructed and evaluated.

Laboratory findings have also concluded that the addition of crumb rubber has significantly reduced the permanent deformation of asphaltic materials when compared

to conventional mixes. A study titled Rutting Resistance of Rubberized Asphalt Concrete Pavements Containing Reclaimed Asphalt Pavement Mixtures evaluated two methods of rutting, viscous flow and plastic deformation using indirect tensile strength (ITS) and the Asphalt Pavement Analyzer (APA). Both performance testing strategies were applied to a wide range of materials that varied in RAP content, percentage of rubber, rubber type, and rubber size. The indirect tension test was previously shown to have good correlation to rutting resistance by a joint study with The Pennsylvania Transportation Institute, The Pennsylvania State University, and Advanced Asphalt Technologies. The results from the indirect tension testing illustrated that increasing rubber contents would decrease the ITS values. A critical finding of the ITS analysis was the effect of the rubber content on the ITS values. The study showed that acceptable ITS values could be achieved up to 15 percent rubber by weight of the binder. While the ITS value trend was shown, the investigation showed that through correlations rubber modification could increase the resistance to rutting. This finding was reinforced by the Asphalt Pavement Analyzer testing. The APA results showed that increasing rates of rubber modification increased the ability of the material to resist deformation.

Chapter 3: Rubber Characterization

Introduction

Studies have shown that rubber properties such as particle size, grinding method, and surface area can influence the final characteristics of rubber modified binders (West, Randy, 1998). However, due to differing rubber sources and production methods it is important to characterize the properties of ground rubber. This study evaluated twelve unique crumb rubber products to characterize the gradation, chemical composition, and surface area of rubber materials. The results from the characterization assisted in the evaluation of the influence of a respective rubber material on a modified binder.

RO-TAP Gradation Analysis Methodology

To identify the particle size of the twelve crumb rubbers used in this study, a gradation analysis was conducted through the use of a RO-TAP apparatus by Lehigh Technologies Incorporated using methodology LTDCN-QC160, ASTM 5644-01, ASTM

26

E11-04, and ASTM D5603-01 to conduct the gradation analyses. From this gradation analysis, Lehigh Technologies reported the particle size distribution of the individual crumb rubber materials and the individual fiber contents found during the gradation process. The results from this analysis were then used to determine the mesh designation and mean particle size and particle size distribution.

The twelve individual particle analyses required that a zero screen and designation screen be selected. The zero screen was designated the smallest screen that allows for 99.9% of the sample to pass. The designation screen (mesh size) was identified as the smallest screen that allowed a minimum for 90% of the material to pass. The RO-TAP procedure indicated that the estimated particle size prior to the gradation analysis would provide guidance for selecting sieve sizes to be used for this analysis.

One hundred grams of a respective rubber material was weighed and prepared for gradation. Flow-aid, a P200 material used to ease the flow of the test material through the sieves was then added to the specimen. For rubber specimens coarser than 50 mesh, 5 grams of Flow-aid was added. For materials 50 mesh or finer, 15 grams of the Flow-aid was added to the test specimen due to the increase in surface area of the rubber particles. The combined material was then placed in a 500 milliliter covered jar and manually agitated for a minimum of one minute to distribute the Flow-aid.

Sieves were stacked in increasing mesh number with the last component of the sieve stack being a pan. The purpose of the pan was to catch all the fine material so that the total amount of fines (P200) could be determined. Each sieve contained two rubber

balls to help facilitate the flow of the material during the sieving process. The preagitated rubber specimen with prescribed Flow-aid was then placed in the top sieve along with the lid from the agitation jar. Lehigh Technologies had two prescribed agitation times for properly sieving the material. Rubber coarser than 50 mesh was agitated for 10 minutes while finer material was agitated for 20 minutes.

Upon completion of the agitation, each sieve was checked for fiber content by recording the weight of the fiber retained on each sieve. If no fiber was found in the top portion of the sieve, the sieve was prepared for rubber weight determination. The bottom or underneath portion of the sieve was brushed to release any rubber particles into the next sieve. This material clinging to the bottom of the sieve represented a portion of the rubber that had passed the respective sieve. The material retained in the top of the sieve and on the rubber balls were then brushed into a pan holder. The weight collected into the pan holder was recorded and the weight measurements were documented to the nearest one-hundredth of a gram for each sieve. Lastly, the sum of the weights on each sieve that was collected into the pan holder was tabulated.

An equation (Equation 1) was then used to determine the amount P200 rubber that was in the pan excluding the Flow-aid that was added to the rubber specimen prior the testing.

$$x = y - (z - 100)$$
 Equation 1

Where: x= the weight of rubber in the pan

y= weight in the bottom of the pan including Flow-aid

z= combined weight of rubber and Flow-aid prior to testing

The validity of the RO-TAP analysis required that the (z) weight deviate no more than 2 grams from the original weight of rubber and Flow-aid that was tested. This analysis provided an indication of the amount of fine rubber material in the tested specimens.

The last portion of the RO-TAP analysis was to determine the designated mesh size of the tested material. This process required that the first sieve retaining no more than 1% of the tested specimen be determined. The next screen was then analyzed to ensure that less than 10% of the specimen was cumulatively retained. If 90% or more of the material was found to pass this sieve, the entire gradation was determined to be designated as this sieve size. This was done in accordance with ASTM Method D5603-1. The classification of the material according to the ASTM Method indicates that 90% of the material tested is either the designated mesh size or smaller.

RO-TAP Gradation Analysis Results

The results from the RO-TAP analysis were tabulated and the percent retained values were determined for the sieves used for each respective material (Table 1). For

this analysis, the percent retained shown is a function of the entire specimen analyzed including the rubber and the Flow-aid. The table indicates that the majority of the materials analyzed were a size 30 mesh or smaller. Two materials analyzed were found to have a coarser designation mesh number than a 30 mesh. These two materials are Liberty Powderizers -16 (16 mesh) and Liberty -20 (20 mesh). The materials were provided by Liberty Tire Recycling and were produced at ambient temperatures. The entire weight of the sample including the weight of Flow-aid used was tabulated and is located in Table 2 which also contains the fiber content of the material, the P200 rubber weight, and the temperature process for crumb rubber manufacturing. The RO-TAP results from Lehigh Technologies can be found in Appendix A.

Table 1. RO-TAP Results for Crumb Rubber Particle Analysis

	Percent Retained														
Material	Pan	#200	#170	#140	#120	#100	#80	#60	#50	#40	#30	#20	#16	#14	#10
MD-402-TR	31.13	-	-	-	-	-	0.00	53.03	15.78	0.06	0.00	-	-	-	-
MD-180-TR	43.79	-	-	15.96	29.00	10.17	1.07	0.02	0.00	-	-	-	-	-	-
MD-105-TR	77.27	17.27	5.04	0.36	0.07	-	-	-	-	-	-	-	-	-	-
(-80/+140)	11.62	-	-	11.60	57.80	16.52	2.46	0.00	-	-	-	-	-	-	-
MD-400-AM	0	0	0	0	0	0	5.0	33.0	56.0	6.0	0.00	0.00	-	-	-
Liberty Powderizers - 16	43.14	-	-	-	-	-	-	0.00	0.00	6.61	21.17	29.03	0.04	0.02	0.00
Liberty -20	11.23	-	-	-	-	-	10.76	32.63	-	37.51	7.82	0.05	0.00	0.00	0.00
MD-400-TR	16.07	-	-	-	-	-	49.66	37.72	-	1.54	0.01	-	-	-	-
Cryohammer	15.61	-	-	-	-	-	16.84	50.48	-	16.78	0.27	0.01	-	-	-
Liberty -30	9.95	-	-	ı	ı	ı	14.98	49.58	-	25.45	0.05	0.00	ı	-	-
Liberty -30 Fine	24.98	-	-	-	-	-	22.17	40.53	-	12.28	0.04	0.00	-	-	-
Crackermill	23.07	-	-	-	-	-	18.88	45.42	-	12.64	0.00	0.00	-	-	-

Three materials were found to have measurable fiber contents. These materials are the Lehigh MD-402-TR, Liberty Powderizers -16, and the Liberty -20 materials which correspond to the coarsest gradations. Table 2 shows how the entire sample weight varied due to the weight of the Flow-aid added as a function of the anticipated particle size. The table also shows the true amount of rubber fines that were determined from each analysis. The Lehigh (-80/+140) was found to have no fine material since it was designed as a "cut" of rubber ranging between the #80 and #140 screens. After using equation 1, this material reported a negative value for rubber in the pan. The negative value indicated that the Flow-aid material was distributed on the surface of the materials retained above the 200 mesh sieve with little material passing to the pan.

Table 3 shows the designated material size as well as the manufacturer of the material and material sources.

Table 2. Additional Crumb Rubber Properties

Material	Entire Sample Weight	Fiber Content	Rubber in Pan	Material Type
MD-402-TR	104.42	0.025	28.09	Cryogenic
MD-180-TR	114.43	0	35.68	Cryogenic
MD-105-TR	114.2	0	74.04	Cryogenic
(-80/+140)	114.45	0	0	Cryogenic
Liberty Powderizers -16	104.41	0.01	40.63	Ambient
Liberty -20	104.05	0.034	7.63	Ambient
MD-400-TR	103.91	0	12.79	Cryogenic
Cryohammer	104.33	0	11.96	Cryogenic
Liberty -30	104.16	0	6.2	Ambient
Liberty -30 Fine	104.59	0	21.54	Ambient
Crackermill	104.14	0	19.88	Ambient

The finest rubber designation was placed on MD-105-TR which used a cryogenic grinding process.

Table 3. Crumb Rubber Designated Size and Material Source

Material	Manufacture	Size	Source
Iviaterial	ivialiulacture	SIZE	Source
MD-402-TR	Lehigh	-40	Truck Tire (TT)
MD-180-TR	Lehigh	-80	TT
MD-105-TR	Lehigh	-140	TT
(-80/+140)	Lehigh	-80	TT
MD-400-AM	Lehigh	-40	TT
MD-400-TR	Lehigh	-40	TT
Liberty -20	Liberty	-20	Passenger Car (PC)
Cryohammer	Liberty	-30	PC + TT
Liberty -30	Liberty	-30	PC + TT
Liberty -30 Fine	Liberty	-30	PC + TT
Crackermill	Liberty	-30	PC
Liberty Powderizers -16	Liberty	-16	PC + TT

In addition to describing the crumb rubber materials with a designated mesh size, the materials can be quantified using the mean particle size. Mean particle size can be a better representation of the particle sizes within a gradation due to mesh designation characterizing the coarsest 10% of the material. Table 4 shows how two different mesh designations can have the same or very similar mean particle sizes. This ultimately can be used in conjunction with the mesh designation to assess the particle size distribution of the rubber.

Table 4. Crumb Rubber Particle Size

Material	Mean Particle Size (microns)	Mesh Size
MD-402-TR	180	-40
MD-180-TR	105	-80
MD-105-TR	50	-140
(-80/+140)	125	-80
MD-400-AM	180	-40
MD-400-TR	180	-40
Liberty -20	250	-20
Cryohammer	250	-30
Liberty -30	250	-30
Liberty -30 Fine	250	-30
Crackermill	250	-30
Liberty Powderizers -16	600	-16

Thermogravimetric Analysis Methodology

The twelve crumb rubber products used in this study were also analyzed for their individual chemical compositions (extractables, polymer, carbon black, and ash) using Thermogravimetric Analysis (TGA). The TGAs were performed by Lehigh Technologies in accordance to ASTM E1131-03. To conduct the analyses, a 10 to 25 gram sample of a crumb rubber was placed in the controlled atmosphere of a thermal gravimetric analyzer. The specified pressure for this analysis was 25 psi. The initial testing temperature of the device was 30°C. The material was then heated to 530°C at a constant rate of 10°C per minute. The weight of material lost by 325°C was determined to be the percentage of extractable material from the sample. After reaching the desired temperature of 530°C the temperature was held constant for 10 minutes. The amount of material lost between the 325°C calculation and the 10 minutes of 530°C was

determined to be the percentage of polymer in the specimen. The sample was then heated from 530°C to 850°C at a constant rate of 10°C per minute. The material was then held constant at this desired temperature for 10 minutes. The material lost during the range of 530°C to 850°C was determined to be carbon black. The remaining weight of material from this analysis was classified as ash and unburned fibers. The results from this study were tabulated and the percentages of each of the four chemical components were calculated.

Thermogravimetric Analysis Results

The percentages of the extractable material, polymer, carbon black, and ash from the TGAs are presented in Table 5. The results show that polymer comprises more than half of the chemical composition of all the crumb rubber products analyzed. The data from the TGAs also shows little variability in the chemical components of the different rubber products. The coefficient of variation for the polymer and carbon black are both relatively low, less than 5 percent. The highest degree of variability from this study can be found in the ash property. Ash had a coefficient of variation of 21.05 percent. The shaded portion of Table 5 shows the highest values determined for each measurable TGA component from the twelve crumb rubbers evaluated. While the coefficient of variation is relatively low for the polymer content for the twelve materials, a slight change in polymer content could have a significant effect on the overall performance rubber when combined with asphalt. Comparing the highest polymer

content to the lowest polymer content of the materials in this study shows that a 6.2% difference in polymer modification can be achieved at 10 percent rubber content. The laboratory results from the chemical composition analyses can be found in Appendix A.

Table 5. Thermogravimetric Results

	Percentage					
Material	Extractables	Polymer	Carbon Black	Ash		
MD-400-AM	10.17	56.10	28.36	5.18		
MD-402-TR	6.78	58.11	30.13	4.80		
MD-180-TR	8.79	54.48	30.88	5.67		
MD-105-TR	10.97	52.43	30.17	6.26		
(-80/+140)	8.67	53.73	32.31	5.31		
MD-400-TR	7.35	57.95	29.86	4.70		
Liberty -20	9.86	54.36	30.65	5.03		
Cryohammer	10.37	51.89	31.40	6.20		
Liberty -30	9.69	52.29	31.54	6.36		
Liberty -30 Fine	7.73	58.46	27.56	5.96		
Crackermill	8.11	52.78	29.35	9.63		
Liberty Powderizers -16	9.49	55.53	28.43	6.44		
Average	9.00	54.84	30.05	5.96		
Standard Deviation	1.25	2.28	1.37	1.25		
Coefficient of Variation	13.92	4.16	4.55	21.05		

Surface Area Methodology

The surface area of each crumb rubber blend was also investigated to further characterize the rubber materials. Quantachrome, a company that specializes in characterizing materials and powders was contracted to conduct the surface area analysis of the twelve materials. Quantachrome utilized a proprietary surface area

analyzer and methodologies to conduct multipoint Brunauer-Emmett-Teller (BET) testing on each specimen. Samples from each material ranged in size between 1 and 3 grams.

The rubber sample was placed into the testing unit where all gas was removed from the testing chamber. The process prepared the material for testing by limiting the amount of impurities in the sample through the flow of the air during the degasification process. The degasification was conducted at ambient temperatures over a 16 hour test period. Once completed, the sample was weighed. The specimen then underwent BET testing. This testing involved the exposure of the rubber sample to ultra-high purity Krypton gas and variable relative pressures at a constant temperature of 77.35K. These relative pressures were 0.05, 0.075, 0.10, 0.15, 0.20, 0.25, and 0.30 of the atmospheric pressure. The exposure of the Krypton at the pressures allowed for the surface area of the rubber to be calculated by finding the difference in the absorbed and desorbed gas around the sample in the testing chamber (Equation 2).

$$S = W_m NA/M$$
 Equation 2

Where: $S = Surface area of the rubber (m^2/gram)$

W_m= Monolayer capacity of the Krypton

N= Avagadro's Number

A= Cross Sectional Area of the Krypton

M= Molecular Weight of the Krypton

Surface Area Results

The results from Quantachrome testing are given in Table 6. The material with the lowest surface area was found to be the Cryohammer Liberty crumb rubber. With the general trend of increases in surface area with decreased particle sizes, the surface area data agrees with the mean particle size findings in Table 4. The material with the highest surface area per gram of sample was found to be the Lehigh MD-105-TR material. This cryogenic temperature produced material was found have the lowest mean particle size of the rubbers in the study. The coefficient of variability for the surface area of the twelve materials was 98. This value indicates that a great deal of variability exists for the surface area of the materials. While many of the gradations appear to be similar as shown in Table 1, the degree of variability in the surface area is very high illustrating that grinding or fabrication method of the crumb rubber greatly influences the surface area properties of the materials. The laboratory data from the Quantachrome testing can be found in Appendix A.

Table 6. Surface Area Results

Surface Area (m^2/g)
0.104
0.275
0.079
0.407
0.751
0.056
0.092
0.079
0.104
0.044
0.114
0.4
0.209

Chapter 4: Characterizing Crumb Rubber Modified Binder

Introduction

The first step of the study required that the twelve crumb rubbers be analyzed for particle size, surface area and chemical composition. Once this phase was completed, the influence of the individual crumb rubber materials on the rheological properties of binder when used as a modifier was investigated. To conduct the second phase of the study a single asphalt binder (67-22) was modified by the twelve crumb rubbers. Performance testing consisting of performance grading, multiple stress creep-recovery testing, separation testing, and softening point testing was then used to assess how each rubber modified the binder.

Methodology for Blending

Blending was conducted by dividing five gallon buckets in gallon size batches. To prepare the smaller batch size quantities of binder, five gallon buckets were heated to 275°F or until the material was fluid and pourable. Once the material was homogeneous

and pourable the material was distributed into pre-weighed cans. Each five gallon bucket was distributed over six to seven gallon size cans to ensure enough space remained in the cans for the crumb rubber. The individual cans of binder were then weighed to determine the weight of binder in each respective can.

Once the binder was distributed appropriately, the crumb rubber material was prepared for blending. A representative sample was created by sampling a large portion of material from the stockpile provided. The material was then placed onto a sheet and manually blended to ensure the material was visually homogeneous (Figure 1). The weight needed for each individual blend was then a function of the weight of the binder in the can and the rubber content desired. For this study each rubber material was loaded at 10 percent of the weight of the binder. Two of the rubber products in this study were then loaded at 15 percent of the binder weight to create additional comparison for modified binder properties. This resulted in fourteen crumb rubber modified binder samples.



Figure 1. Example Crumb Rubber Sample Preparation

Prior to blending, each gallon was heated in a 300°F oven. Once homogeneous, the material was placed in a heating mantle. The heating mantle was set at 280 to 320°F for blending purposes. The temperature was monitored through the use of a temperature gauge. The corresponding dial setting of the heating mantle was used for the duration of the blending. The binder temperature was also measured using a thermometer. Care was taken to ensure that the binder did not receive excessive aging while in the oven and that the heating mantle did not reach undesirable temperatures.

enough torque to ensure 1000 rpms. To initiate blending, the stirring rod was placed in the binder. The depth of the paddle was set so that a 1 inch vortex could be achieved in the binder during blending to ensure proper stirring. The speed of the paddle was set to 700 rpms. The predetermined weight of rubber was then added at a constant rate over 2 minutes of blending. After the rubber was added to the binder, the rpms of the paddle was increased to 1000 rpms. The crumb rubber modified binder was then blended at this constant rate for an additional 30 minutes. The blending strategy of incorporating the rubber material over 2 minutes with additional blending for 30 minutes was modeled after the methodology for blending documented by a study titled *Crumb Rubber Modification of Binders: Interaction and Particle* Effects (Amirkhanian B. J., 2006). Separation tests were then conducted with the freshly blended material. The remaining material was covered and allowed to rest at room temperature.

To prepare pre-blended room temperature material for additional testing the specimens were gently reheated ensuring the binder temperature did not exceed 300°F.

The specimen was placed in the preheated heating mantle. The crumb rubber modified binder was then reblended for 10 minutes at 1000 rpms. Figure 2 shows the desired vortex needed for blending which corresponded to an appropriate height of the blending paddle.

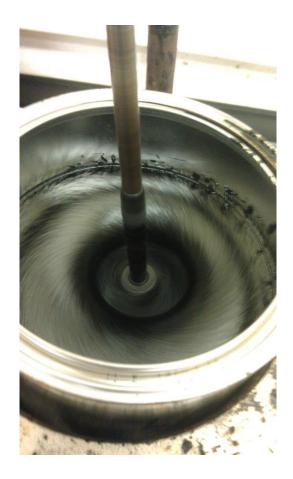


Figure 2. Example Vortex Resulting in Properly Mixed Crumb Rubber Binder

Method for Performance Grade

Performance grading of the fourteen crumb rubber modified binders was conducted with pre-blended material. The material was prepared for performance

grading by following the previously discussed reblending methodology. Modified binder specimens were poured into a 25mm diameter mold for unaged binder testing. The specimens were then removed from the mold and were placed on the dynamic shear rheometer (DSR) testing plate (Figure 3).

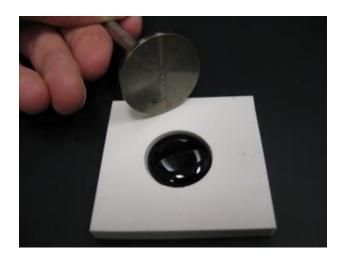


Figure 3. 25mm Disc of Binder and DSR Testing Plate (Dabbs, 2012)

The performance grading of the materials were performed according to AASHTO M320-10 using a 1mm gap between the testing plates when appropriate (Figure 4). The method requires testing of both unaged and aged binders to assess binder properties at different stages of aging. Aging of the binder was performed according to the Standard Test Method for Effect of Heat and Air on a Moving Film of Asphalt designated (ASTM D2872-04) and the Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel designated (ASTM D6521-08). The particle size of the Liberty Powderizers material caused a high degree of variability during the DSR performance

grading, so a 2mm gap was used with the 25mm specimens to generate more reliable high temperature grade results. The use of a 2mm testing gap strategy was previously evaluated and proposed from research conducted by H.U. Bahia and R. Davies (Amirkhanian, 2006). The particle size of this material did not appear to interfere with the viscosity determination of the binder nor the flexural creep stiffness results of the material.

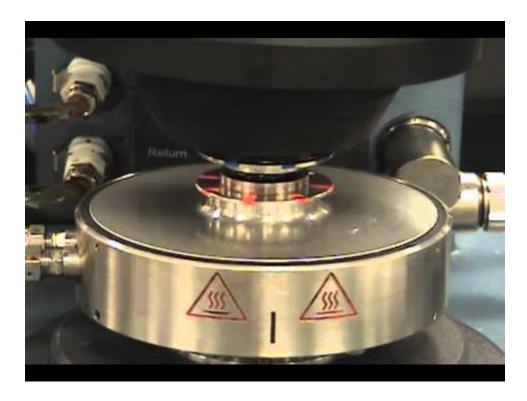


Figure 4. Example 1mm Testing Gap (Digparty, 2013)

Performance grade testing also requires viscosity testing, mass change analysis, and low temperature creep stiffness analysis. Viscosity testing was performed with freshly blended binder and was performed according to AASHTO T315-09. The flexural

creep stiffness of the binder was analyzed according to the AASHTO T313-10 specification.

Results from Performance Grading

Results from rotational viscometer analysis, DSR analysis, and bending beam rheometer analysis were used to grade each modified binder. The viscosity values were determined according to AASHTO 316 (Table 7). Most materials were found to be pumpable or workable as indicated by the viscosity determinations at 135°C. The -20 Liberty material loaded at 15 percent exceeded the maximum allowable viscosity of 3.00 PaS at 135°C. The most viscous material that did not exceed the maximum viscosity at this temperature was the 15 percent rubber Liberty Cryohammer material (2.912 PaS).

Table 7. Viscosity of Rubber Modified Blends Passing AASHTO 316 Criteria

Rubber Product	Rubber Content %	Viscosity, PaS at 135°C
80/140	10	1.425
MD-180-TR	10	0.825
MD-400-TR	10	1.425
MD-402-TR	10	1.300
MD-105-TR	10	1.425
-30 Liberty	10	1.400
-20 Liberty	10	1.887
Crackermill	10	1.990
Cryo-Hammer	10	1.675
-30 Liberty Fines	10	1.725
-16 Powderizers	10	1.600
MD-400-AM	10	1.887
Cryo-Hammer	15	2.912

Each freshly blended binder was analyzed with the DSR to determine the high temperature at which the binder had a minimum G*/sin δ (Table 8). Every rubber increased the high temperature grade of the base binder.

Table 8. Crumb Rubber Modified Binder Performance Grade

Tested Material	Grinding Method	Mean Particle Size**	Rubber Content	True Grade	Performance Grade	G* @82°C kPa	δ @82°C
Base Binder	NA	NA	0%	70.0 – 25.4	70 – 22	NA	NA
-80/140	Cryo	125	10%	83.6 – 24.9	82 – 22	1.14	81.8
MD-180-TR	Cryo	105	10%	81.2 – 25.4	76 – 22	0.92	85.3
MD-400-TR	Cryo	180	10%	80.4 – 24.2	76 – 22	0.85	85.5
MD-400-AM	Amb	180	10%	82.1-16.3	82-16	1.00	84.1
MD-402-TR	Cryo	180	10%	79.0 – 23.0	76 – 22	0.73	85.8
MD-105-TR	Cryo	50	10%	77.9 – 25.6	76 – 22	0.66	83.7
-30 Liberty	Amb	250	10%	80.7 – 23.6	76 – 22	0.87	84.8
-20 Liberty	Amb	250	10%	83.1 – 24.6	82 – 22	1.09	81.5
Crackermill	Amb	250	10%	82.8 – 23.1	82 – 22	1.07	82.1
Cryo-Hammer	Cryo	250	10%	82.2 – 23.2	82 – 22	1.01	82.8
-30 Liberty Fines	Amb	250	10%	79.8 – 20.4	76 – 16	0.80	84.4
-16 Powderizers (1mm gap)	Amb	600	10%	76.3 – 21.8	76 – 16	NA	NA
-16 Powderizers (2 mm gap)	Amb	600	10%	84.7 – 21.8	82 – 16	1.28	82.1
-20 Liberty	Amb	250	15%	87.9 – 21.3	82 – 16	1.63	79.5
Cryo-Hammer	Cryo	250	15%	86.7 – 19.3	82 – 16	1.52	81.2

^{**(}microns)

The results suggest that using 15 percent rubber had the greatest impact in increasing the high temperatures grade. This additional 5 percent rubber increased the critical high temperature by approximately 5°C when compared to the 10 percent rubber binders. The 10 percent crumb rubber modified materials indicate that the true high temperature grade of the base binder could be increased by 10°C.

The higher rubber dosage also appeared to affect the lower temperature grade. The bending beam rheometer results show that the low temperature grade of -22°C from the original binder was increased causing the modified material to be more susceptible to cracking at low temperatures. While the 15 percent loaded materials appeared negatively influenced by the low temperature grade, some of the 10 percent rubber modified binders like the MD-180-TR and MD-105-TR modified materials were found to have little or no effect on the low temperature grade when loaded at 10 percent.

Method for Multiple Stress Creep Recovery Analysis

The fourteen modified binders also underwent advanced performance testing according to AASHTO TP70-09 for multiple stress creep recovery (MSCR). The MSCR results were used to grade the modified binders according to AASHTO MP19-10. The MSCR test was used to determine the amount of elastic response of rolling thin film aged (RTFO) modified material. MSCR testing was typically run after conducting a performance grading of the oven aged materials. The material was tested at 64°C, the average 7-day high pavement design temperature for Auburn, Alabama.

For this analysis, each 25mm RTFO specimen was tested with the DSR to assess the permanent deformation characteristics of each material. The sample was subjected to two standardized procedures of stress that included cycles of constant loading with periods of zero loading allowing strain recovery. The two stress levels were 0.100kPa

and 3.200kPa. The testing of the RTFO material included ten cycles of constant stress applied to the binder for 1 second with 9 seconds of stress free conditions which allowed for partial recovery of the induced strain. The amount of recovery occurring during the rest cycle indicated the amount of elastic nature of the binder. The amount of strain remaining in the specimen was used along with recovery criteria to evaluate the material performance (J_{nr}). The acceptable J_{nr} at the 3.200kPa and the percent difference with J_{nr} at 0.100kPa was used to characterize the expected traffic level that could be used for each respective material. Table 9 shows the values from AASHTO MP19-10 that were used to classify the materials.

Table 9. AASHTO MP19-10 MSCR Performance Criteria

Traffic Level	Max J _{nr3.2} (kPa ⁻¹)	Max J _{nrdiff} (%)
Standard Traffic "S" Grade	4.0	75
Heavy Traffic "H" Grade	2.0	75
Very Heavy Traffic "V" Grade	1.0	75
Extremely Heavy Traffic "E" Grade	0.5	75

Results from MSCR Testing

As stated earlier, MSCR testing was conducted on RTFO binders using a 1mm testing gap with the exception of the -16 Liberty Powderizers. Two materials (-20 Liberty and 15% rubber Cryohammer) were found to not meet the Max J_{nrdiff} criteria when tested with a 1mm gap. The unacceptable Max J_{nrdiff} values were a function of the non-recoverable creep compliance values at the lower stress level. The calculation comparing the two materials indicates unrealistically large percent difference values for

these materials. The percent recoveries for these materials at both strain levels indicate that the materials have desirable permanent deformation characteristics. Even with the known issues in the calculation, the prescribed traffic level indicated by the MSCR results were determined and are shown in Table 10. The findings from the MSCR analysis indicated that every material was graded for "extremely heavy" traffic.

Table 10. MSCR Results

	Cuin din a	Mean	Rubber		J _{nr}		% Red	covery	
Rubber Product	Grinding Method	Particle Size***	Content,	0.1 kPa ⁻¹	3.2 kPa ⁻¹	% Diff	0.1 kPa ⁻¹	3.2 kPa ⁻¹	Traffic Level
Base Binder	NA	NA	0%	1.150	1.353	17.68	12.99	5.616	"H"
-80/140	Cryo	105	10%	0.123	0.190	46.39	58.44	41.87	"E"
MD-180-TR	Cryo	105	10%	0.175	0.201	14.90	44.66	38.02	"E"
MD-400-TR	Cryo	180	10%	0.139	0.166	19.19	51.23	43.55	"E"
MD-402-TR	Cryo	180	10%	0.178	0.202	13.52	42.90	36.69	"E"
MD-400-AM	Amb	180	10%	0.123	0.160	57.18	46.88	30.53	"E"
MD-105-TR	Cryo	50	10%	0.273	0.344	26.08	36.86	24.50	"E"
-30 Liberty	Amb	250	10%	0.201	0.233	15.95	43.56	36.42	"E"
-20 Liberty	Amb	250	10%	0.086	0.159	85.81	69.16	46.45	"E"*
-20 Liberty	Amb	250	15%	0.030	0.193	554.0	85.94	50.43	"E"*
Crackermill	Amb	250	10%	0.122	0.183	50.88	59.46	42.73	"E"
Cryo- Hammer	Cryo	250	10%	0.150	0.201	34.21	50.59	36.68	"E"
Cryo- Hammer	Cryo	250	15%	0.062	0.1949	216.2	68.87	44.72	"E"*
-30 Liberty Fines	Amb	250	10%	0.092	0.127	37.68	61.62	49.58	"E"
-16 Powderizers (1mm gap)	Amb	600	10%	0.096	0.720	652.5	95.20	66.45	"E"*
-16 Powderizers (2 mm gap)	Amb	600	10%	0.088	0.122	39.41	63.03	50.44	"E"

^{*} Did not meet Jnr % difference requirement (≤75%)

^{***}microns

Freshly blended crumb rubber modified materials were subjected to separation testing. Immediately after blending, material was sampled and tested according ASTM D7173-11, *The Standard Practice for Determining the Separation Tendency of Polymer from Polymer Modified Asphalt* to quantify the amount of separation or non-uniformity of the modified material after heated storage.

At the time of crumb rubber binder blending, 1 inch diameter conditioning tubes were not available for purchase so a $\frac{1}{2}$ inch aluminum tube was used as an alternative. The tubing had one closed end and an opposing open end that allowed freshly blended binder to be added to the tubing. The mass of binder added to each tube was 50 ± 0.5 grams. Once the material was poured into the aluminum container, the tubing was crimped and sealed. The tube was then placed vertically in a metal storage rack in an oven at 163 ± 5 °C oven for 48 ± 1 hour. Care was taken to ensure that the tubing remained plumb during the conditioning phase. After conditioning, the rack was removed from the oven and placed in a freezer for a minimum of 4 hours to solidify the binder. The frozen material was then cut into three equal portions. The middle portion was discarded while the top and bottom thirds were used for comparison.

To prepare the top and bottom portions of the binder, the tubing and frozen material was placed in individually labeled 3 ounce cans and heated until the binder could flow from the tubes. Pliers were used to help crimp and tubing and force any residual material that was attached to the inner wall from the tubing before stirring the

binder to ensure homogeneity. The top and bottom portions of the conditioned material were then tested in with the DSR to determine the critical high temperature grades of the top and bottom halves of the tubing. Figure 5 shows an example of the top and bottom portions of a separation tested material.



Figure 5. Example Set of Separated Materials

Results from Separation Test

The true high temperature grade of the conditioned sections was determined through the use of DSR testing (Table 11). The results of the separation test indicated varying degrees of separation occurred between the rubber products. The two materials with the highest uniformity were determined to be MD-180-TR and the -30 Liberty Fines. These materials had less than 12 percent difference of the high temperature

grade between the top and bottom sections. The material that was found to have the highest difference between the top and bottom portions for high temperature grade was determined to be the -16 Liberty Powderizers. The results from the entire analysis are shown in Table 11. The table documents the true high temperature grade from each test specimen as well as the percent difference providing a relative indicator of the true difference in the top and bottom portions of the conditioned material.

Table 11. Separation DSR Results

Dubbor	Crindina	Mean	Rubber		Critical Hi	gh Temperatui	re, °C
Rubber Product	Grinding Method	Particle	Content,	Ton	Pottom	Absolute	%
Product	Method	Size**	%	Тор	Bottom	Difference	Difference
-80/140	Cryo	125	10%	78.87	97.52	18.65	23.65
MD-180-TR	Cryo	105	10%	74.98	81.56	6.58	8.78
MD-400-TR	Cryo	180	10%	81.79	102.6	20.81	25.44
MD-402-TR	Cryo	180	10%	82.75	102.7	19.95	24.11
MD-400-AM	Amb	180	10%	86.10	99.90	13.8	13.81
MD-105-TR	Cryo	50	10%	79.93	92.53	12.6	16.61
-30 Liberty	Amb	250	10%	76.67	103.6	26.93	35.12
-20 Liberty	Amb	250	10%	75.84	101.7	25.86	34.10
-20 Liberty	Amb	250	15%	94.3	107.4	13.1	12.20
Crackermill	Amb	250	10%	77.04	102.4	25.36	32.90
Cryo-Hammer	Cryo	250	10%	77.71	102.1	24.39	23.89
Cryo-Hammer	Cryo	250	15%	79.85	110.5	30.65	38.40
-30 Liberty	Amb	250	10%	85.63	97.07	11.44	11.79
Fines	AIIID	230	1070	85.05	37.07	11.44	11.75
-16							
Powderizers	Amb	600	10%	79.78	197.5	117.72	147.56
(1mm)							

^{**(}microns)

Softening point analysis (ASTM D36-09) is another test commonly used to characterize the uniformity of the material using a Ring-and-Ball Apparatus. This test was utilized to experimentally determine the temperature at which modified binder specimens were able to soften or flow. The softening point test was particularly useful for doing a side-by-side analysis of the separation test conditioned samples to document the uniformity of the material after heated storage.

To conduct the tests, the preconditioned materials stored in the 3 ounce cans were heated until fluid. This required a short exposure to a 150°C oven. While the material was being heated, the pouring plate was prepared by applying a thin coat of release agent to assist the removal of the binder from the plate after pouring specimens into the rings. The ring molds for the binder were also heated during this time period. The binder was then removed from the oven and stirred to ensure homogeneity. The homogeneous material was then poured into the heated rings taking care to slightly overfill the brass molds. This pouring process occurred with the rings resting on the prepared pouring plate. The material was allowed to rest at room temperature for 30 minutes prior to trimming the top of the specimens flush with the square edge of the molds. The test specimens were prepared no earlier than 4 hours prior to testing.

Freshly boiled distilled water was used as the test bath medium. Water was chosen because of the acceptable range of testing temperatures available near the estimated softening point temperatures for the modified binders. After the water was

boiled, it was immediately cooled to ensure a test temperature of $5\pm1^{\circ}$ C. A freezer was used to assist in cooling the water from boiling temperatures to testing temperatures. The testing range for determining softening point with water is 30 to 80°C. Prior to testing, the entire Ring-and-Ball apparatus with testing specimens was assembled with the steel ball resting in the bottom of the container. These assemblies were allowed to rest at the test temperature for 15 minutes. After 15 minutes of stabilized test conditions, the balls were placed on the center of specimens and the entire assembly was ready for testing. Figure 6 shows an example of the Ring-and-Ball Apparatus setup that was used to determine the softening point of the separated materials.



Figure 6. Ring-and-Ball Softening Point Apparatus

To begin testing, the bath was slowly warmed at a rate of 5°C per minute with minimal variation from this rate of heating. The bath was heated through the use of an open flame and the rate of heating was controlled by the operator. A stop watch was used to help ensure a uniform rate of heat was applied to the testing vessel. Once the material was soft enough to allow the weight of the ball to sink and touch the bottom of the testing cylinder, the temperature of the "softening point" for the respective sample was recorded to the nearest tenth of a degree.

All materials were found to have a softening point within the 30 to 80°C range which justified the use of water as a testing bath medium. On average, this analysis provided a smaller value for percent difference between the top and bottom portions analyzed when compared to the DSR testing of the conditioned specimens. The materials with the lowest percent difference for softening point from this analysis were found to be MD-105-TR and MD-180-TR. The material that possessed the highest percent difference for the softening point was determined to be the MD-402-TR material. The increase in rubber content did not appear to have measureable effects on the softening point of the material. The entire set of results from the softening point analysis is available in Table 12.

Table 12. Softening Point Results

Dubbor	Crinding	Mean	Dubbor		Softening Point, °C					
Rubber Product	Grinding Method	Particle Size**	Rubber Content, %	Тор	Bottom	Absolute Difference	% Difference			
-80/140	Cryo	125	10%	61.4	78.0	16.6	23.8			
MD-180-TR	Cryo	105	10%	58.6	63.0	4.4	7.2			
MD-400-TR	Cryo	180	10%	61.7	73.3	11.6	17.2			
MD-402-TR	Cryo	180	10%	59.7	77.2	17.5	25.6			
MD-400-AM	Amb	180	10%	62.2	71.4	9.2	13.8			
MD-105-TR	Cryo	50	10%	60.6	61.1	0.5	0.8			
-30 Liberty	Amb	250	10%	58.9	72.5	13.6	20.7			
-20 Liberty	Amb	250	10%	58.9	69.4	10.5	16.4			
-20 Liberty	Amb	250	15%	66.4	73.3	6.9	9.9			
Crackermill	Amb	250	10%	58.9	66.7	7.8	12.4			
Cryo-Hammer	Cryo	250	10%	59.7	70.7	10.3	15.9			
Cryo-Hammer	Cryo	250	15%	61.9	75.0	13.1	19.1			
-30 Liberty Fines	Amb	250	10%	62.8	72.8	10.0	14.7			
-16 Powderizers	Amb	600	10%	59.2	69.7	10.5	16.3			

^{**(}microns)

Chapter 5: Statistical Analysis of Crumb Rubber Properties on Modified Binders

Introduction

Statistical analyses were conducted to determine the degree of influence of each crumb rubber property on the modified binder performance. The six crumb rubber properties characterized in Chapter 3 were used as parameters to assess the explainable variability of the performance data of the fourteen modified binders (Chapter 4).

Correlation Analysis of the Binder Performances

Before assessing the effect of rubber properties of binder performance, a correlation analysis was conducted to determine if any two binder performances (Chapter 4) were correlated to each other. Pearson product moment correlation coefficients were developed for the binder performance test results using Minitab 16. These coefficients represent linear trends between any two sets of data. Pearson correlation coefficients can range between values of -1 to 1. A correlation of 1 represented that the data has a linear trend or positive slope with both variables

analyzed trending similarly. A value of -1 indicated that the two sets of data are linear with a negative slope and opposing trends. A Pearson correlation of 0 indicated that no true linear relationship existed for the two variables. This could indicate scattered data or non-linear relationships.

To quantify the statistical significance of the results, p-values were also determined for each paired correlation coefficient. The null hypothesis for this analysis was that the correlation value was 0. The alternative hypothesis was that the correlation value was not equal to 0. P-values less than 0.05 indicated that the null hypothesis should be rejected while p-values greater than 0.05 indicated that the alternative hypothesis should be rejected. Table 13 shows the results from the correlation analysis for the 4 binder performance responses. The coefficients for each analysis are represented by the variable (r) while the p-values are represented by the variable (p).

As seen in the table, the statistics failed to reject all the null hypotheses suggesting that the correlation between the any two parameters of the analysis are statistically insignificant.

Table 13. Pearson Correlation Table for Binder Performance

	Softening Point %	% Difference in True High	True Low
	Difference	Grade After Conditioning	Grade
% Difference in True High	r=0.187		
Grade After Conditioning	p=0.522		
True Low Grade	r=0.213	r=0.172	
	p=0.465	p=0.557	
True High Grade	r=0.116	r=0.290	r=0.491
	p=0.692	p=0.314	p=0.075

The general regression analysis tool within Minitab 16 was used to conduct a general linear regression analysis with the crumb rubber factors influencing the true high performance grade. A linear model was formed through the general regression analysis which utilized the crumb rubber properties for the true high performance grade of the rubber modified blends. An analysis of the variance (ANOVA) was included with the regression and the results are presented in Table 14. The linear model described 81.65 percent of the variability in the true high performance grade. The analysis found that the two sources describing the majority of the variability of the true high performance grade were rubber content and mean particle size. Rubber content was found to describe 33.84 percent of the variability while mean particle size was found to account for 20.63 percent of the true high performance grade variability.

In addition to assessing sources of the variability, p-values were calculated to determine the statistical significance of each rubber characteristic. The null hypothesis for this analysis was that the linear coefficient for any source material from the regression was 0. The alternative hypothesis was that the coefficient was not equal to 0.

Rubber content was the most statistically significant property affecting the true high performance grade of the rubber modified binders. Surface area was found to be the second most influential property in determining the true high temperature grade of the binder; however, this variable was not statistically significant.

Table 14. True High Performance Grade ANOVA

Property	DF	Seq SS	Adj SS	Adj MS	F	Р
Mean Particle Size	1	21.25	3.14	3.14	1.16	0.317
Surface Area	1	17.40	5.80	5.80	2.15	0.186
Percent Polymer	1	9.67	3.06	3.06	1.13	0.322
Temperature	1	0.32	1.10	1.10	0.41	0.544
Rubber Content	1	34.85	35.38	35.38	13.10	0.009
Tire Source	1	0.60	0.60	0.60	0.22	0.651
Error	7	18.90	18.90	2.70		
Total	13	102.99				

Analysis of Crumb Rubber Properties on True Low Performance Grade

A similar analysis methodology was conducted to determine what rubber properties had the greatest influence on the true low performance grade of the rubber modified binders. The general linear model (GLM) found to the six variables accounted for 61.62 percent of the variability in the true low performance grade (Table 15). Similar to the true high temperature performance grade, rubber content and mean particle size were found to contribute the greatest amount of variability in the analysis. The mean particle size was determined to account for 21.05 percent of the variability in the model while rubber content accounted for 34.84 percent of the variability. No other crumb rubber source influenced the test results. The p-values from this analysis indicated that only rubber content had a statistically significant coefficient (p=0.039) in the linear regression.

Table 15. True Low Performance Grade ANOVA

Property	DF	Seq SS	Adj SS	Adj MS	F	Р
Mean Particle Size	1	10.55	2.57	2.57	0.94	0.366
Surface Area	1	0.44	0.01	0.01	0.00	0.967
Percent Polymer	1	1.10	2.07	2.07	0.75	0.414
Temperature	1	1.11	1.60	1.60	0.58	0.470
Rubber Content	1	17.46	17.62	17.62	6.41	0.039
Tire Source	1	0.23	0.23	0.23	0.08	0.782
Error	7	19.23	19.23	2.75		
Total	13	50.11				

Analysis of Crumb Rubber Properties on Performance Grade of Conditioned Materials

The DSR separation tube test results were analyzed to determine if any of the properties describing the crumb rubber materials were influential using a linear regression model. The six crumb rubber parameters were used to model the percent difference in the true high performance grade between the top third and bottom third of the conditioned materials (Table 16). The percent difference of the performance grades within the sample was used to place a relative value on the settlement occurring within the material.

The regression analysis conducted through the use of the Minitab 16 software found that the six properties were able to describe 95.32 percent of the variability in the in the model. The mean particle size was found to explain 78.69 percent of the variability in the analysis. No other parameter described a practical amount of variability compared to this result.

Both mean particle size and percent polymer were statistically significant parameters of the model. The third lowest p-value was calculated for the tire rubber

source (0.059). This suggests that the tire source could have some effect on the test results; however, it may have been overshadowed by the mean particle size.

Table 16. Separation Performance Grade Percent Difference ANOVA

Property	DF	Seq SS	Adj SS	Adj MS	F	Р
Mean Particle Size	1	12242.6	12473.7	12473.7	119.80	0.000
Surface Area	1	851.7	162.4	162.4	1.56	0.252
Percent Polymer	1	177.4	610.1	610.1	5.86	0.046
Temperature	1	740.3	143.2	143.2	1.38	0.279
Rubber Content	1	291.0	161.5	161.5	1.55	0.253
Tire Source	1	526.3	526.3	526.3	5.06	0.059
Error	6	728.8	728.8	104.1		
Total	13	15558.2				

Analysis of Crumb Rubber Properties on Softening Point of Conditioned Materials

Softening point test results were also statistically analyzed for the influence of rubber characteristics using a general linear regression. The analysis utilized the percent difference in softening point temperatures between the results from the top third and bottom third of every specimen.

The general linear regression analysis (Table 17) explained only 44.92 percent of the test result variability. The analysis showed that the source describing the majority of the variability in the model was surface area; however, it only accounted for 19.30 percent of the variability in the model. The p-values generated for the statistical significance of each regression coefficient showed that the six properties had no influence on the softening point. The lowest p-value was determined for surface area. The value of 0.100 indicated that the surface area of the crumb rubber material had the

highest likelihood of any parameter in affecting the temperature difference between the softening points.

Table 17. Separation Softening Point Percent Difference ANOVA

Property	DF	Seq SS	Adj SS	Adj MS	F	Р
Mean Particle Size	1	32.89	2.66	2.66	0.06	0.810
Surface Area	1	105.00	153.37	153.37	3.58	0.100
Percent Polymer	1	42.09	7.25	7.25	0.17	0.693
Temperature	1	33.84	7.04	7.04	0.16	0.697
Rubber Content	1	8.70	4.29	4.29	0.10	0.760
Tire Source	1	21.91	21.91	21.91	0.51	0.498
Error	6	299.72	299.72	42.82		
Total	13	544.15				

Summary

The findings from the correlation analysis indicated that no two sets of binder performance had statistically similar trends. The highest correlation was determined to be the relationship between true high performance grade and the true low performance grade. While the correlation was the highest, the analysis showed that the two parameters do not share statistically similar trends. This indicates that the high and low temperature binder performances are influenced independently. When the six crumb rubber properties were used to model the true high and low performance grades, the analyses indicated that rubber content contributed to the majority variability described in both analyses. The coefficients for rubber content indicated that performance grade increased with rubber content.

The regression analysis yielding the most statistically significant influence of a crumb rubber parameter through linear modeling was the true grade separation analysis. The regression indicated that mean particle size was statistically significant. The coefficient for mean particle size from this regression showed that separation increased with increased mean particle size. The results from all four linear regression analyses indicated that the mean particle size, rubber content, and polymer content affect the binder performance and binder stability. Table 18 shows the statistically significant parameters found from the four analyses.

Table 18. Summary of Statistical Analyses

Crumb Rubber Parameter	Analysis	Statistical Significance (p-value)
Rubber Content	High Temperature PG	0.009
Rubber Content	Low Temperature PG	0.039
Mean Particle Size	Separation PG	0.000
Percent Polymer	Separation PG	0.046

Chapter 6: Effect of Additives

Introduction

Additives can be used to assist in the stabilization of blended crumb rubber in the asphalt binder. Two current methods for achieving this stabilization are adding styrene-butadiene-styrene (SBS) polymer or Vestenamer, a trans-polyoctenamer rubber, directly to the crumb rubber modified binder. SBS is a common polymer used for binder modification to assist in elastic response of the material and increase the performance grade while Vestenamer is a proprietary performance modifier developed by the Degussa Corporation. Vestenamer is a double bond chemical intended to create a more uniform rubber-composite material. It was designed to link the sulfur components of the asphalt binder to the sulfur components of the rubber particles. The use of the SBS polymer with the crumb rubber binder yields a product termed hybrid binder. Because both SBS-GTR hybrid binder and Vestenamer modified material are considered solutions to creating stabilized rubber modified binders, the two additives were analyzed to determine the effect of the additives on the performance grade, softening point, and MSCR performance.

Vestenamer was added in a similar method to the crumb rubber after the entirety of the rubber modifier was blended into the binder. For this study, the Vestenamer was loaded at a rate of 0.5 percent of the weight of the binder and blended for 30 minutes using the previously described methods. This portion of the study only evaluated two rubber products with the Vestenamer modifier. The Vestenamer was blended with the Liberty 30 Mesh material and the Lehigh MD-400-TR material. Both of the materials were loaded at a rate of 10 percent of the weight of binder as conducted in prior analyses. Once blended, the modified binders were then subjected to separation testing, performance grading, and MSCR testing.

The hybrid binder product analyzed for this study was developed by Blacklidge Emulsions Inc. The hybrid binder was created through the use of a proprietary combination of 40 mesh ambient ground rubber and SBS polymer. The material was prepared and provided by Blacklidge Emulsions Inc. and the exact proportions of modifier or blending instructions were not provided. To prepare the material for performance testing, the re-blending procedure previously described was used.

Results from Additive Modified Binder Performance Grading

The Vestenomer and hybrid modified crumb rubber binders were subjected to performance grading, softening point testing, and MSCR testing. Performance grading

was conducted using the methodology outlined in Chapter 4 and the results of the performance grade testing with and without the additives are given in Table 19.

Vestenamer increased the true high temperature grade of the binders. At times, this stiffening would increase the actual performance grade of the binder. The true grading of the material modified with Vestenamer also indicates that the additive may increase the true low grade of the binder and potentially affect the performance grade of the material as demonstrated by the MD-400-TR material.

Table 19 also includes performance grading results for the Blacklidge hybrid product. The analysis shows that the Blacklidge product containing SBS polymer and GTR possessed a performance grade of 82-22. This product illustrates relatively high performance grade range when compared to performance grade ranges determined from the materials without additives.

Table 19. Performance Grade of Additive Modified Materials

Rubber Product	Rubber Content. %	Additive	True Grade	Performance Grade
#30 Liberty	10%	None	80.7 – 23.6	76 – 22
	10%	Vestenamer	82.3 – 22.4	82 – 22
MD 400 TD	100/	None	80.4 – 24.2	76 – 22
MD 400 TR	10%	Vestenamer	81.8 – 19.5	76 – 16
Blacklidge Hybrid	NA	Polymer	82.8 – 23.5	82 – 22

Results from Additive Modified Binder Softening Point Test

Softening point testing was conducted according to the methodology presented in Chapter 4 and the results are presented in Table 20. The results represent the

temperature at which the separated materials were softened to the point of flow within the test bath. The values in this table allow for comparison between the materials with and without additives. The Vestenamer comparisons provided mixed results. The MD-400-TR comparison indicated that the Vestenamer increased the amount of separation that occurred in the material while the Liberty 30 Mesh comparisons demonstrated that Vestenamer assisted in reducing the amount of separation of the material. In both cases, the softening point temperature for the top portion of the test specimen was increased with the Vestenamer added when compared to the other materials. This is consistent with the performance grade testing results.

The separation results of the Blacklidge material is also shown in Table 20. The material was found to have a relatively low percent difference of 13.8 percent between the top and bottom portions of the conditioned material. With respect to the other material and analyses presented in Chapter 4, the 13.8 percent difference for this material was found to be on the lower end (23rd percentile) for the percent difference between the softening points of the conditioned materials. When compared to the Vestenamer modified materials, the Blacklidge binder was found to provide similar softening point differences between the top and bottom conditioned specimens.

Table 20. Softening Point for Additive Modified Materials

Rubber	Rubber		Softening Point, °C			
Product	Content, %	Additive	Тор	Bottom	Absolute	%
Product	Content, 70		τορ	БОШОП	Difference	Difference
NAD 400 TD	100/	None	61.7	73.3	11.6	17.2
MD-400-TR	10%	Vestenamer	77.6	114.3	36.7	38.2
-30 Liberty	100/	None	58.9	72.5	13.6	20.7
-50 Liberty	10%	Vestenamer	92.0	104.5	12.5	12.7
Blacklidge Hybrid	NA	Polymer	80.4	94.2	13.8	15.8

Results from Additive Modified Binder MSCR Testing

The three materials containing modifier underwent MSCR testing after performance grading as explained in the methodologies in Chapter 4. Again, the additive material was reported against its respective additive free crumb rubber binder. The results from this analysis are shown in Table 21. The analysis of the materials found that the designated traffic level was not altered due to the additive when compared to the materials containing no additive. The analysis showed that the J_{nr} value was reduced for the material with additive at both stress conditions tested. This material response illustrated that the materials with Vestenamer possessed slightly higher elastic response when compared to the same materials without Vestenamer. The Blacklidge material performed similarly to the other materials of this study and was found to have a traffic level grading for "Extremely Heavy" traffic as suggested by AASHTO MP19-10.

Table 21. MSCR Results for Additive Modified Materials

				J_{nr}		% Rec	overy	
Rubber Product	Rubber Content, %	Additive	0.1 kPa ⁻¹	3.2 kPa ⁻¹	% Diff	0.1 kPa ⁻¹	3.2 kPa ⁻¹	Traffic Level
MD-400-TR	10%	None	0.139	0.166	19.19	51.23	43.55	"E"
1VID-400-1K	10%	Vestenamer	0.091	0.157	71.65	62.44	41.37	"E"
20 Liborty	100/	None	0.201	0.233	15.95	43.56	36.42	"E"
-30 Liberty	10%	Vestenamer	0.092	0.118	28.84	57.66	48.44	"E"
Blacklidge Hybrid	NA	Polymer	0.196	0.240	22.31	57.91	50.03	"E"

Summary

Comparison of the crumb rubber modified binder with additive versus crumb rubber modified binder without additives illustrated that an additive had a measurable effect on performance characteristics of the materials. The performance grading of the additive modified material showed that Vestenamer increased the true high temperature grade of the modified binder. Softening point testing of the Vestenamer modified materials showed mixed results with all materials having varying degrees of separation. The results from the softening point test could be related to other variables other than the additive such as particles size or particle alignment. MSCR testing of the materials containing Vestenamer showed that the material had slightly higher elastic properties when compared to materials without additives. The Blacklidge hybrid material was found to possess similar performance grade, uniformity, and elastic recovery when compared to the Vestenamer modified materials. Ultimately, analysis of the materials with additive showed that a higher uniformity of stored material could be achieved along with potentially higher performance grades when compared to materials

without additives. The individual complete binder gradings for the hybrid and Vestenamer modified materials are located in Appendix B.

Chapter 7: Conclusions and Recommendations

Conclusions

Results from the crumb rubber modified binder testing indicate that all fourteen modified binders have an increased high temperature performance grade due the rubber modification. Each of the twelve rubber products loaded at 10 percent increased the high temperature performance grade of the binder from PG70 to PG76. In addition to every rubber product being able to increase the grade to PG76, six of the twelve products loaded at 10 percent were able to increase the base binder grade by two grades from PG70 to PG82. The materials loaded to 15 percent also resulted in high temperature performance grades of PG82, two grades above the base binder grade of PG70. The rubber modification was also able to show that crumb rubber influences the low temperature grade of the binder. The low temperature grade was increased from -22°C to -16°C for 3 of the twelve materials loaded with 10 percent rubber and for both modified binders loaded at 15 percent rubber.

The MSCR testing of the fourteen modified materials indicated that each material could be classified for "extremely heavy" trafficking for the regional climate as

indicated by the AASHTO MP19-10 specification. The testing also indicated that the four modified binders that did not pass the percent difference in non-recoverable creep compliance between the two stress levels still exhibited high resistance to permanent deformation through the percent of recovered strain. The percent difference calculation failures were caused by the low stress level non-recoverable creep compliance values which skewed the percent difference calculations.

Separation testing and softening point testing indicated that the materials separate during heated storage regardless of particle size or rubber content. Only one material of the fourteen blends passed the softening point criteria. The use of an additive like Vestenamer with crumb rubber indicated that additives were not effective in preventing settlement or the separation of the rubber during heated storage.

Performance testing of the hybrid materials illustrated potential for hybrid binders through the stiffening of the crumb rubber modified binders and reduction in the high temperature susceptibility of the mixtures.

Statistical analyses of the modified binder performance testing illustrated that rubber content was a statistically significant factor in describing the variability in the performance grade of the materials. Mean particle size of the crumb rubber materials was also found to be significant in describing the variability in the performance grades of the materials. Statistical analysis of the high performance grades of the separated materials indicated that mean particle size was the critical factor in describing the separation of the materials. Mean particle size was found to account for 78.69 percent of the variability in the DSR graded data. Larger particle sizes indicated larger differences

in the high performance grade between the top and bottom portion of the specimens. The percentage of polymer in the crumb rubber was also found to be a statistically significant contributor in describing the separation tendencies of the modified binders. As the amount of rubber polymer increased, the differences in the high grade between the top and bottom portions of the specimen reduced. Grinding method, tire source, and surface area were not found to have any statistical significance describing the variability in the performance characteristics of the modified materials.

Recommendations

The results from the binder testing and laboratory performance testing allow for the following recommendations to be made:

- Ground tire rubber should be considered as an asphalt binder modifier for applications requiring increases in high temperature performance grades.
- State specifications restricting the use of crumb rubber modification of binder to ambient ground materials should be opened to allow the use properly processed cryogenic ground rubber.
- 3. Crumb rubber content should be restricted to 10 percent in PG67-22 binders to limit the effect of the crumb rubber on low temperature performance grade.
- Continuous blending should be used when blending materials mesh size #100 or larger.

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Appendix A: Crumb Rubber Data

RO-TAP Gradations

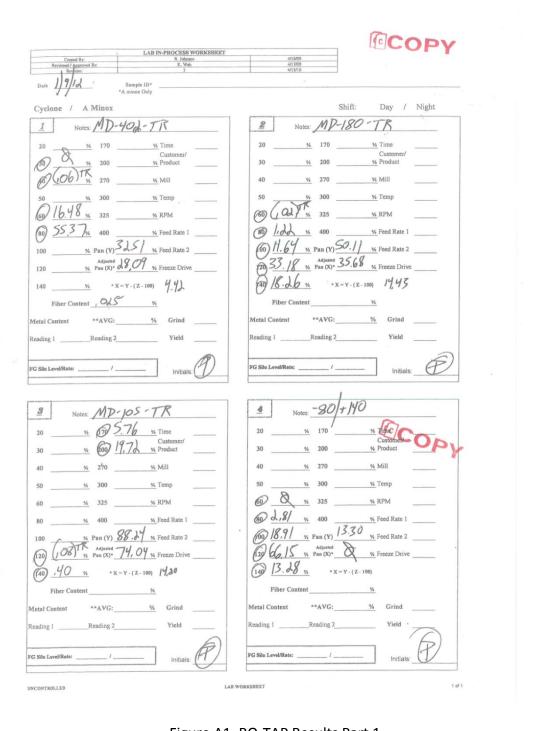


Figure A1. RO-TAP Results Part 1

Created By:	N-PROCESS WORKSHEET R. Johnson	4/16/09	(T) = -
Reviewed / Approved By: Revision:	K. Wah	4/17/09 4/13/10	©COPY
Date //9// Sample ID* *A minox Only			
Cyclone / A Minox		Shi	ft: Day / Night
1 Notes: Cryohammer		2 Notes: Liberty	30 -
NOITE "	Time	20 & % 170	% Time
0.28	Customer/	COM	Customer/ % Product
(30) 100 % 200 %	Product	30 (05) % 200	% Floduct
	Mill	(40) <u>Ab. > 1 %</u> 270	% Mill
50 % 300 %		50 % 300	% Temp
60 52.6 / % 325%	RPM	60 5/.67 % 325	% RPM
(80) 17.57% 400 %	Feed Rate 1	(80) 15,60 % 400 _	% Feed Rate 1
100% Pan (Y) 16.29 %	Feed Rate 2	100% Pan (Y) /O	36 % Feed Rate 2
120 % Pan (X)* 11.96 %	Freeze Drive	120 % Pan (X)* 6,0	60 % Freeze Drive
140 <u>%</u> * X = Y - (Z - 100)	-4,33	140% * X = Y	-(Z-100) · 4,16
Fiber Content %		Fiber Content	%
Metal Content **AVG:%	Grind	Metal Content **AVG:	% Grind
Reading 1Reading 2	Yield	Reading 1Reading 2	Yield
FG Silo Level/Rate:/	Initials:	FG Silo Level/Rate: //	Initials:
Notes: Liberty -30	Fine	4 Notes: Crack	ermil/
28	Time	(20 & % 170 <u></u>	% Time
O Cay TR	Customer/	(30) & % 200	Customer/ % Product
0/104	Product	(40) 13.16 % 270	% Mill
	Гетр	50 % 300	% Temp
24129	RPM	60 47,30% 325	% RPM
D12 19	Feed Rate 1	80) 19.66 % 400	% Feed Rate 1
16.13	Feed Rate 2	100 % Pan (Y) 4,	% Feed Rate 2
Adjusted \1/1/		Adjusted 190	50
	Freeze Drive	120% Pan (X)*] /, Z	% Freeze Drive
140 <u>%</u> * X = Y - (Z - 100)	-4,59	140 <u>%</u> * X = Y -	1,1]
Fiber Content		Fiber Content	
Metal Content **AVG:%	Grind	Metal Content **AVG:	
Reading 1Reading 2	Yield	Reading 1Reading 2	Yield
FG Silo Level/Rate: /	Initials:	FG Silo Level/Rate: /	Initials:
UNCONTROLLED	LAB WOR	KSHEET	1 of 1

Figure A2. RO-TAP Results Part 2

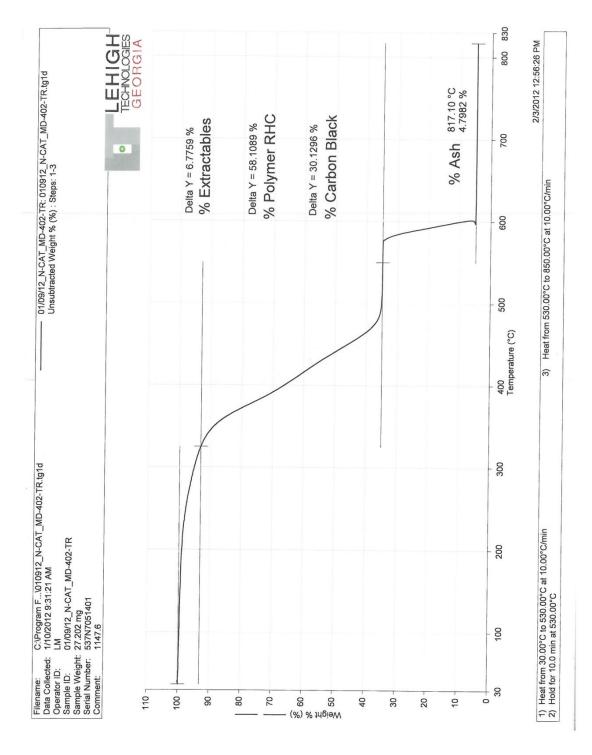


Figure A3. TGA Results MD-402-TR

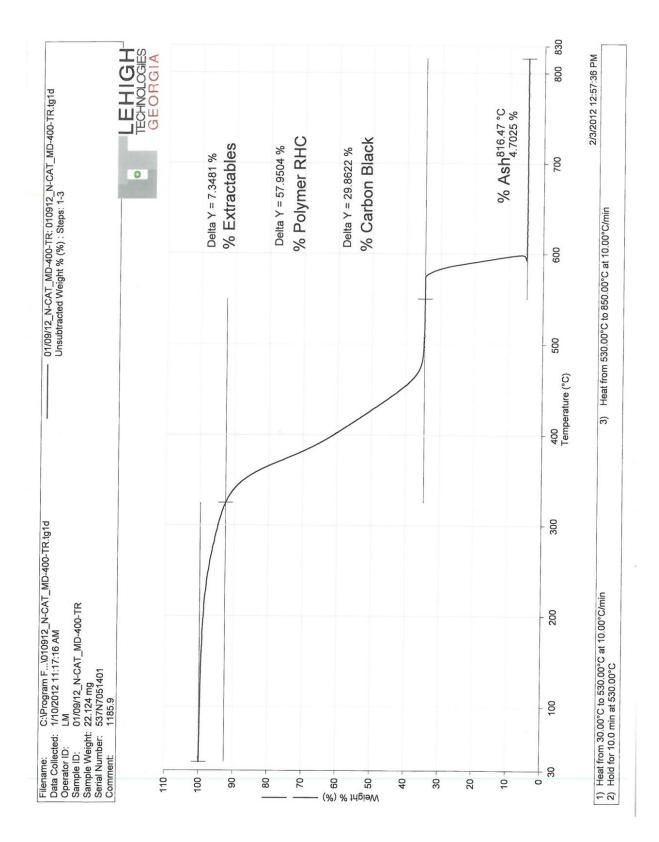


Figure A4. TGA Results MD-400-TR

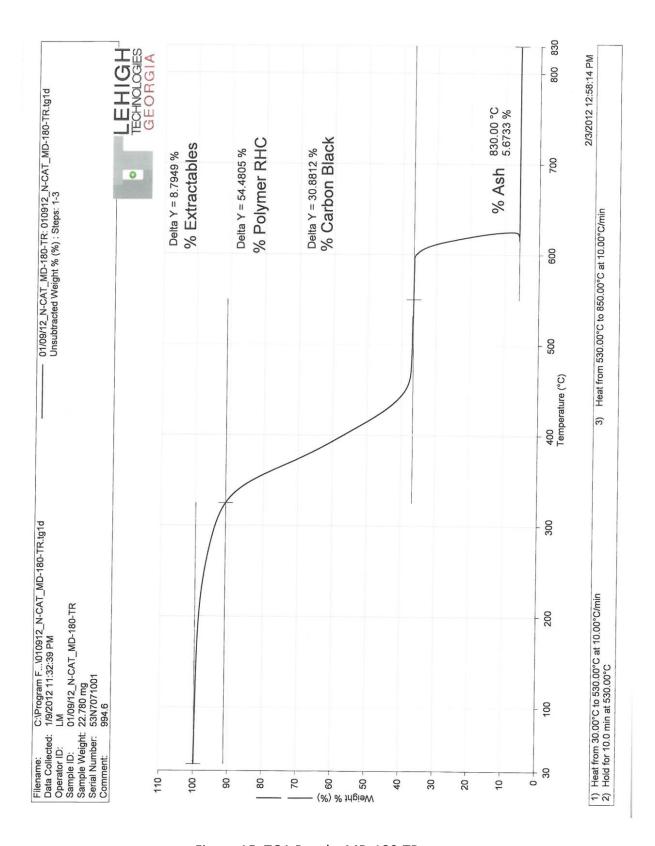


Figure A5. TGA Results MD-180-TR

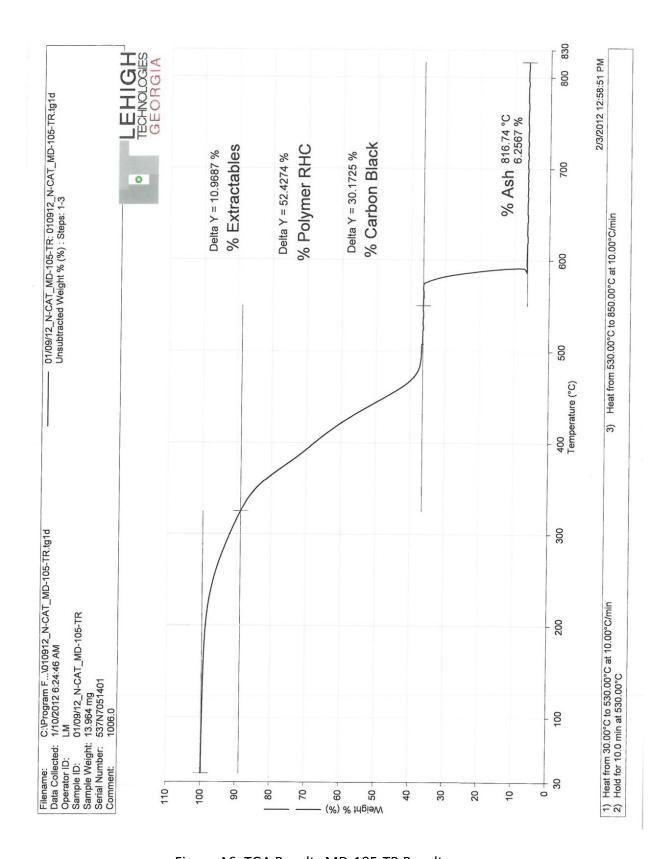


Figure A6. TGA Results MD-105-TR Results

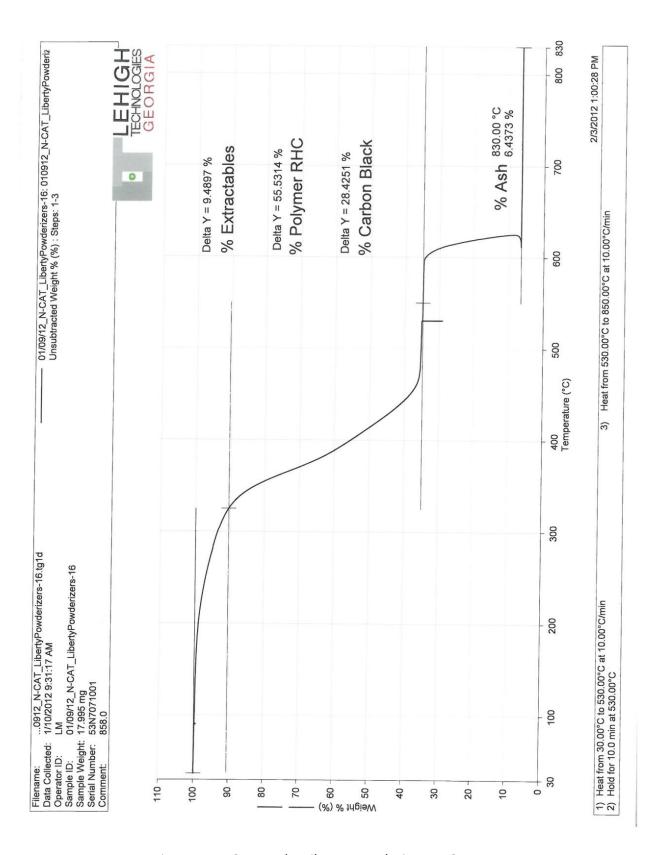


Figure A7. TGA Results Liberty Powderizers -16

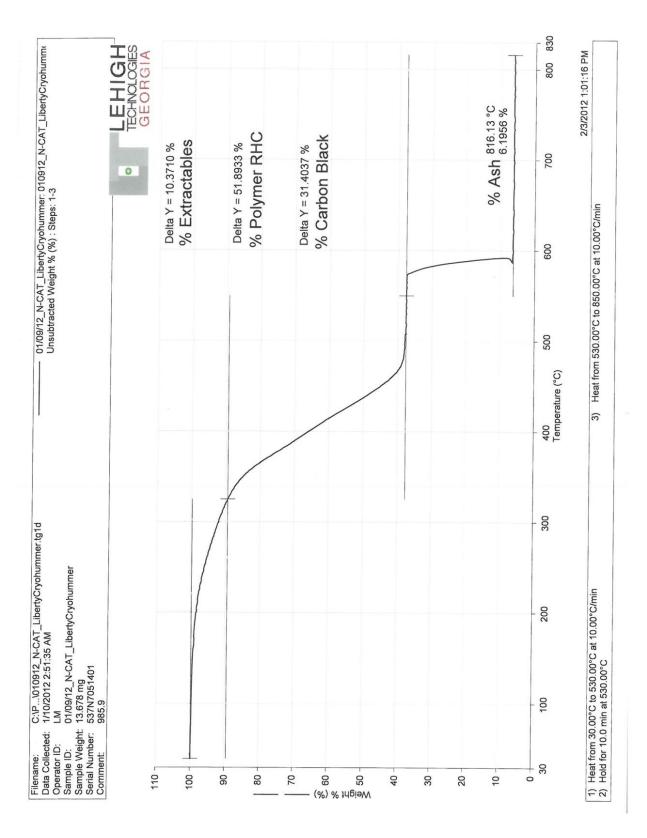


Figure A8. TGA Results Liberty Cryohammer

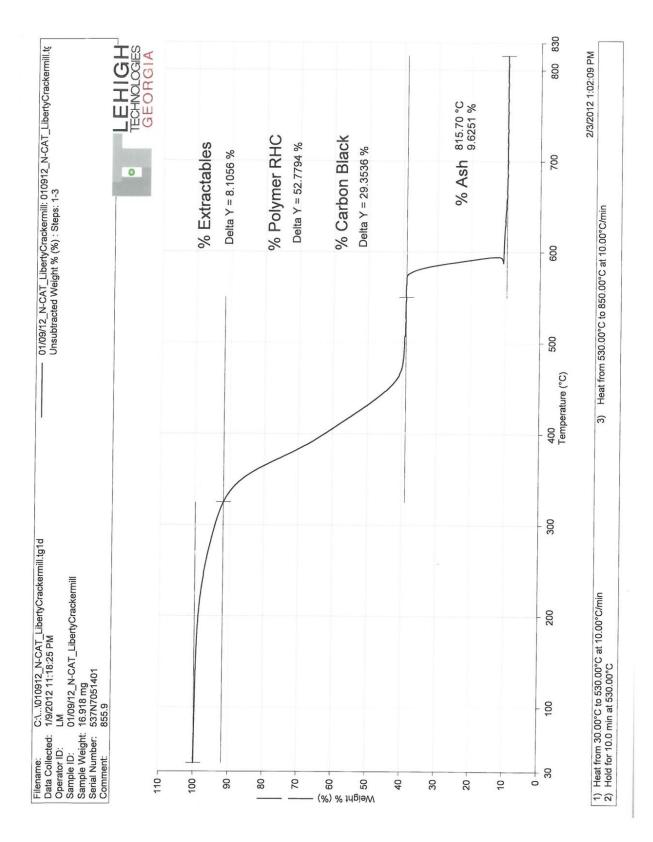


Figure A9. TGA Results Liberty Crackermill

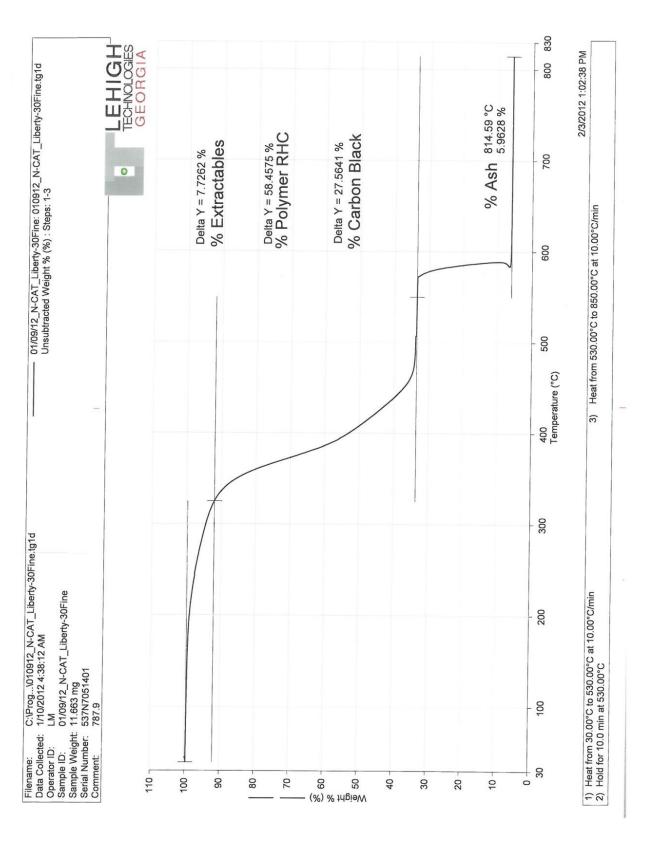


Figure A10. TGA Results Liberty -30 fines

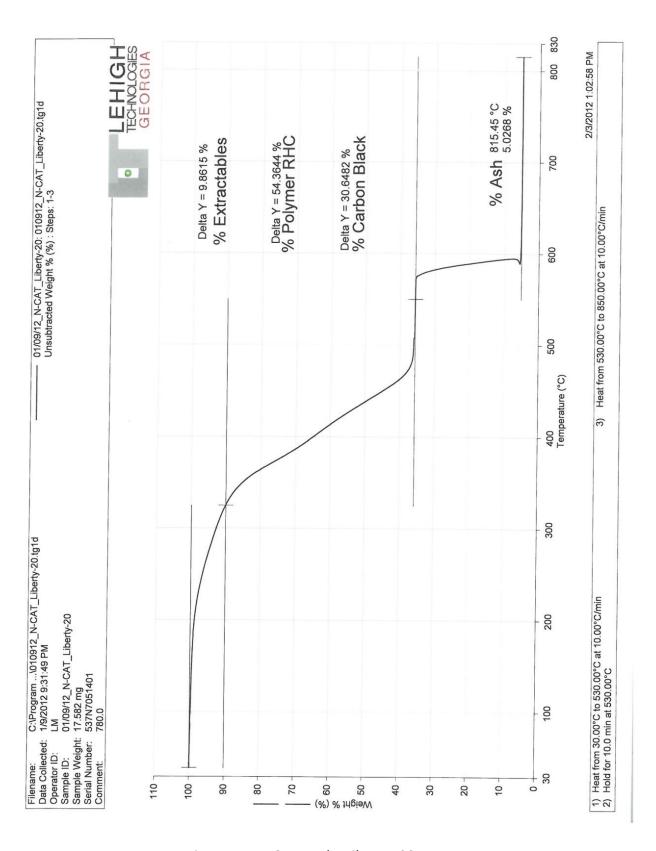


Figure A11. TGA Results Liberty -20

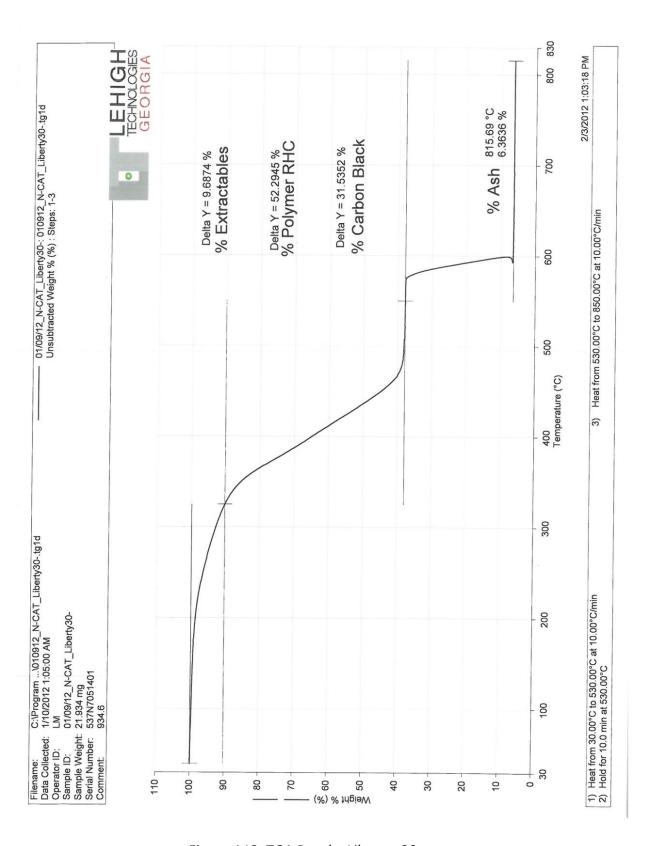


Figure A12. TGA Results Liberty -30

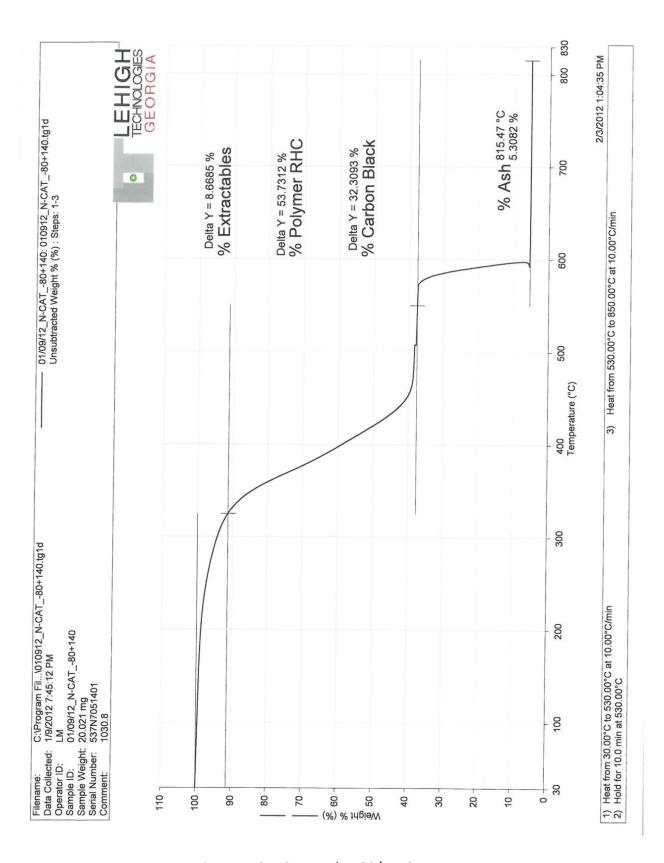


Figure A13. TGA Results -80/+140

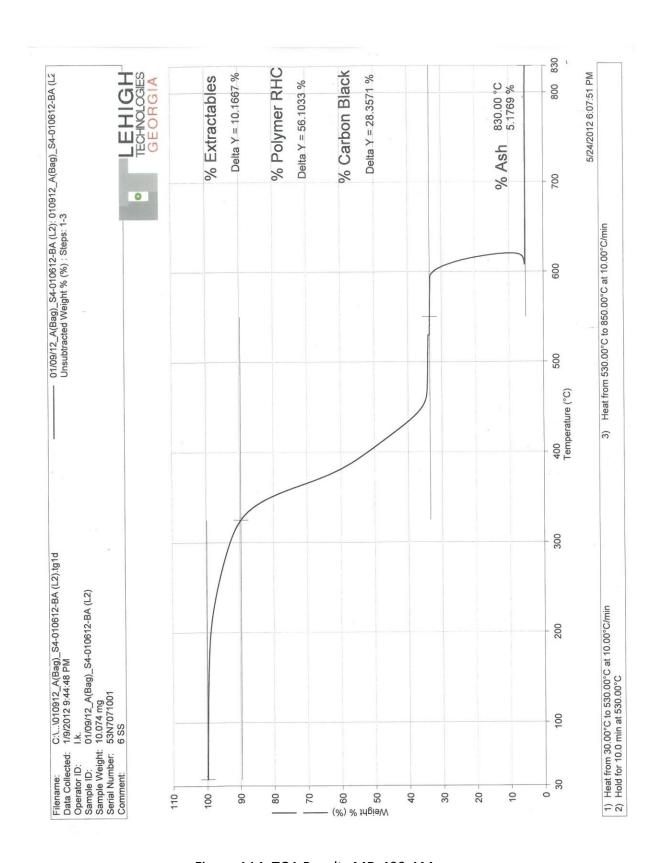


Figure A14. TGA Results MD-400-AM



Analysis			Report		
Operator:	operator Date:5	/16/2012	Operator: ra	Date:2	012/05/22
Sample ID:	-20	Filename:	Liberty Tire Recycling20_Lab	3646_051612.qps	
Sample Desc:	Ground rubber	Comment:	Liberty Tire Recycling, Lab #36	46	
Sample weight:	2.3800 g				
Analysis Time:	131.5 min	End of run:	5/16/2012 19:21:00	Instrument:	QuadraSorb Station 1
Void Vol.:	He Mode.Cell: 9mm large bulb	Run mode	Standard	Instrument version:	5.06
Outgas Time:	16.0 hrs	OutgasTemp:	25.0 C		
Analysis gas:	Krypton	Bath Temp:	77.3K		
Press. Tolerance:	0.050/0.000 (ads/des)	Equil time:	200/0 sec (ads/des)	Equil timeout:	400/0 sec (ads/des)
	•	Multi-P	oint BET	-	

			—Data Reduction Parameter	ters Data		
Adsorbate	Krypton Molec. Wt.:	83.800		350K 500 A=	Liquid Density:	2.413 g/cc
	molec. W	03.000	Closs Section. 20.0	00 A-	Elquiu Delisity.	2.413 g/dc

	Multi-Point BET Data —						
Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]		
6.39756e-02 9.14304e-02 1.19875e-01 1.48883e-01 1.77412e-01	0.0046 0.0064 0.0079 0.0094 0.0107	3.9399e+03 4.2328e+03 4.6016e+03 5.0011e+03 5.3775e+03	2.05317e-01 2.29452e-01 2.58084e-01 2.86017e-01	0.0121 0.0132 0.0145 0.0158	5.7263e+03 6.0198e+03 6.4135e+03 6.7824e+03		

BET su	mmary
Slope =	12907.941
Intercept =	3.077e+03
Correlation coefficient, r =	0.999794
C constant=	5.195
Surface Area =	0.092 m²/g

Figure A15. Surface Area Analysis Summary Liberty -20



Report
Operator: ra
Liberty Tire Recycling_30_Lab 3646_051612.qps
Liberty Tire Recycling, Lab #3646 Date:5/16/2012 Date:2012/05/22 operator 30-

Sample ID: Filename:

sample ID: Sample Desc: Sample weight: Ground rubber Comment 2.7228 g

126.4 min He Mode.Cell: 9mm large bulb 16.0 hrs 5/16/2012 11:39:11 Standard 25.0 C End of run: Run mode Analysis Time: Instrument: Instrument version: QuadraSorb Station 1

OutgasTemp: Bath Temp: Outgas Time: 77.3 K

Analysis gas: Press. Tolerance: Krypton 0.050/0.000 (ads/des) Equil timeout: Equil time: 200/0 sec (ads/des) 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

Adsorbate Krypton Temperature Molec. Wt.: 83.800 20.500 Å: Liquid Density: Cross Section: 2.413 g/cc

Multi-Point BET Data -Relative Volume @ STP 1 / [W((Po/P) - 1)] Relative Volume @ STP 1 / [W((Po/P) - 1)] Pressure Pressure [P/Po] [P/Po] [cc/g] [cc/g] 5.14314e-02 9.00871e-02 1.23499e-01 1.57646e-01 0.0019 0.0031 0.0040 0.0050 7.7972e+03 8.6424e+03 9.3359e+03 9.9939e+03 1.88518e-01 2.17805e-01 2.41751e-01 2.69852e-01 0.0059 0.0067 0.0074 0.0082 1.0543e+04 1.1127e+04 1.1588e+04 1.2121e+04

BET summary ne = 19802.332 Slope = Intercept = 6.859e+03 Correlation coefficient, r = C constant= 0.999636 3.858 0.056 m²/g Surface Area =

Figure A16. Surface Area Analysis Summary Liberty -30



Analysis Operator: Sample ID: Report
Operator: ra Date:2012/05/22
Liberty Tire Recycling _-30Fine _Lab 3646_051612.qps operator -30 Fines Date:5/15/2012

Filename:

Sample Desc: Sample weight: Ground rubber 1.8816 g Liberty Tire Recycling, Lab #3646

Analysis Time: Void Vol.: 125.8 min End of run: 5/15/2012 18:54:07 Instrument: Instrument version: QuadraSorb Station 1 He Mode.Cell: 9mm large bulb 16.0 hrs Run mode OutgasTemp: Standard 25.0 C 5.08

Outgas Time:

Krypton 0.050/0.000 (ads/des) 77.3 K 200/0 sec (ads/des) Equil timeout: 400/0 sec (ads/des) Equil time:

Multi-Point BET

Data Reduction Parameters Data

<u>Adsorbate</u> Krypton Molec. Wt.: Temperature Cross Section: 77.350K 2.413 g/cc 83.800 20.500 Å= Liquid Density:

Multi-Point BET Data -1 / [W((Po/P) - 1)] Volume @ STP Volume @ STP 1 / [W((Po/P) - 1)] Relative Relative Pressure Pressure [P/Po] [cc/g] [P/Po] [cc/g] 9.34647e-02 1.23314e-01 1.52054e-01 1.79345e-01 0.0080 0.0099 0.0116 0.0133 2.06852e-01 2.28826e-01 2.57412e-01 2.86567e-01 0.0150 0.0163 0.0179 0.0195 3.4588e+03 3.7947e+03 4.1205e+03 4.3867e+03 4.6487e+03 4.8619e+03 5.1680e+03 5.5159e+03

BET summary = 10425.072 Slope = 2.503e+03 0.999490 Intercept = Correlation coefficient, r = C constant= 5.164 Surface Area = 0.114 m²/g

Figure A17. Surface Area Analysis Summary Liberty -30 Fines



Report
Operator: ra
Lehigh Tech_80+140_050812.qps
Lehigh Technologies, Lab #3847 Analysis Operator: Sample ID: Date:5/8/2012 Date:2012/05/16 operator -80+140 Filename:

Ground Rubber 2.3997 g Sample Desc: Sample weight:

127.7 min He Mode.Cell: 9mm large bulb 16.0 hrs 5/8/2012 18:06:59 Instrument: Instrument version: QuadraSorb Station 1 End of run: Analysis Time: 5.06

Run mode OutgasTemp: Standard 25.0 C Outgas Time:

Bath Temp: Equil time: Analysis gas: Press. Tolerance: Krypton 0.050/0.000 (ads/des) 77.3 K 200/0 sec (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

Krypton Molec. Wt.: Temperature Cross Section: 77.350K 20.500 Å <u>Adsorbate</u> 83.800 Liquid Density: 2.413 g/cc

Multi-Point BET Data -Relative Pressure Volume @ STP 1 / [W((Po/P) - 1)] Relative Pressure Volume @ STP 1 / [W((Po/P) - 1)] [P/Po] [cc/g] [P/Po] [cc/g] 8.03850e-02 1.09878e-01 1.38447e-01 1.66670e-01 1.94625e-01 0.0074 0.0092 0.0110 0.0125 3.1722e+03 3.5783e+03 3.9249e+03 4.2660e+03 4.5764e+03 2.17459e-01 2.46664e-01 2.76043e-01 3.03861e-01 0.0154 0.0170 0.0184 0.0198 4.8297e+03 5.1652e+03 5.5396e+03 5.8876e+03

BET summary 11928.567 2.251e+03 Slope = Intercept = Correlation coefficient, r = 0.999686 C constant= 6.300 0.104 m²/g Surface Area =

Figure A18. Surface Area Analysis Summary -80/+140



Report
Operator: ra Date:2012/05/22
Liberty Tire Recycling_Asphalt Blend_Lab 3646_051512.qps
Liberty Tire Recycling, Lab #3646 operator Asphalt Blend Ground rubber 2.3424 g Date:5/15/2012 mple ID: Filename:

Sample Desc:

2.3424 g 123.7 min He Mode.Cell: 9mm large bulb 16.0 hrs 5/15/2012 15:38:37 End of run: Instrument: Instrument version: QuadraSorb Station 1 Analysis Time: Run mode OutgasTemp: Standard 25.0 C 5.06

Outgas Time: Analysis gas: Press. Tolerance: Bath Temp: 77.3 K

Krypton 0.050/0.000 (ads/des) Equil timeout: 200/0 sec (ads/des) 400/0 sec (ads/des) Equil time:

Multi-Point BET

Data Reduction Parameters Data

Krypton Molec. Wt.: Temperature Cross Section: 77.350K 20.500 Å: <u>Adsorbate</u> 2.413 g/cc 83.800 Liquid Density:

Multi-Point BET Data -1 / [W((Po/P) - 1)] 1 / [W((Po/P) - 1)] Relative Pressure Volume @ STP Relative Volume @ STP Pressure [P/Po] [cc/g] [P/Po] [cc/g] 5.71688e-02 9.03911e-02 1.19233e-01 1.51277e-01 0.0034 0.0050 0.0064 0.0077 4.7371e+03 5.2717e+03 5.7007e+03 6.1975e+03 1.81936e-01 2.11265e-01 2.34041e-01 2.63235e-01 6.6551e+03 7.0576e+03 7.3835e+03 7.8054e+03 0.0089 0.0102 0.0111 0.0122

BET summary Slope = 14830.880 Intercept = Correlation coefficient, r = 3.925e+03 0.999754 C constant= 0.079 m²/g Surface Area =

Figure A19. Surface Area Analysis Summary Liberty Powderizers -16



Report
Operator: ra Date:2012/05/22
Liberty Tire Recycling_Crackermill_Lab 3646_051612.qps
Liberty Tire Recycling, Lab #3646 operator Crackermill Date:5/16/2012 nple ID:

Filename: Comment: Ground rubber nple Desc:

2.4212 g 5/16/2012 16:01:05 126.3 min End of run: Instrument: Instrument version: QuadraSorb Station 1 He Mode.Cell: 9mm large bulb 16.0 hrs Standard 25.0 C Run mode 5.06

OutgasTemp: Bath Temp: Equil time: Outgas Time: 77.3 K

Krypton 0.050/0.000 (ads/des) Analysis gas: Press. Tolerance: 200/0 sec (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

Krypton Molec. Wt.: 77.350K 20.500 Å <u>Adsorbate</u> Temperature 83.800 Cross Section: Liquid Density: 2.413 g/cc

Multi-Point BET Data -Relative Pressure 1 / [W((Po/P) - 1)] Relative Pressure Volume @ STP 1 / [W((Po/P) - 1)] Volume @ STP [P/Po] [cc/g] [P/Po] [cc/g] 5.84235e-02 8.56368e-02 1.10115e-01 1.39301e-01 0.0051 0.0069 0.0085 0.0102 3.2729e+03 3.6060e+03 3.9125e+03 4.2641e+03 4.6193e+03 1.96243e-01 2.18910e-01 2.47943e-01 2.75679e-01 0.0133 0.0146 0.0161 0.0175 4.9054e+03 5.1399e+03 5.4748e+03 5.8112e+03

BET summary 11566.585 2.628e+03 Slope = Intercept = Correlation coefficient, r = C constant= 5.402 0.104 m²/g Surface Area =

Figure A20. Surface Area Analysis Summary Crackermill



Report
Operator: ra D
Lehigh Tech_Cry-30-GTR_Lab 3647_051412.qps
Lehigh Technologies, Lab #3647 Analysis Operator: Date:5/14/2012 Date:2012/05/16

operator Cry-30 GTR Filename:

Sample ID: Sample Desc: Sample weight: Ground Rubber 2.7235 g Comment

5/14/2012 17:42:20 Standard 25.0 C 121.9 min He Mode.Cell: 9mm large bulb 16.0 hrs Analysis Time: End of run: Run mode Instrument: Instrument version: QuadraSorb Station 1

OutgasTemp: Bath Temp: Outgas Time: 77.3 K

Krypton 0.050/0.000 (ads/des) Analysis gas: Press. Tolerance: Equil time: 200/0 sec (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

Krypton Molec. Wt.: <u>Adsorbate</u> Temperature 20.500 Å: 83.800 Liquid Density: Cross Section: 2.413 g/cc

Multi-Point BET Data -Relative Volume @ STP 1 / [W((Po/P) - 1)] Relative Volume @ STP 1 / [W((Po/P) - 1)] Pressure Pressure [P/Po] [P/Po] [cc/g] [cc/g] 5.59507e-02 9.57755e-02 1.27140e-01 1.62761e-01 1.95174e-01 0.0015 0.0025 0.0032 0.0040 0.0047 1.0289e+04 1.1275e+04 1.2093e+04 1.3054e+04 1.3820e+04 2.25649e-01 2.48072e-01 2.78101e-01 3.06622e-01 0.0054 0.0059 0.0065 0.0071 1.4494e+04 1.5058e+04 1.5746e+04 1.6562e+04

BET summary = 24754.870 8.936e+03 Slope = Intercept = 0.999706 3.770 Correlation coefficient, r = C constant= Surface Area = 0.044 m²/g

Figure A21. Surface Area Analysis Summary Cryohammer



Report
Operator: ra
Lehigh Tech_MD-105-TR_Lab 3647_051012.qps
Lehigh Technologies, Lab #3647 operator MD-105-TR Date:5/10/2012 Date:2012/05/16

Sample ID: Sample Desc: Sample weight: Filename:

Ground Rubber 2.7419 g Comment

End of run: Run mode OutgasTemp: Bath Temp: Equil time: 5/10/2012 20:21:52 Standard 25.0 C 201.3 min He Mode.Cell: 9mm large bulb 16.0 hrs QuadraSorb Station 1 5.06 Analysis Time: Instrument: Instrument version:

Outgas Time:

77.3 K 200/0 sec (ads/des) Analysis gas: Press. Tolerance: Krypton 0.050/0.000 (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

Krypton Molec. Wt.: Temperature Cross Section: 77.350K 20.500 Å <u>Adsorbate</u> 83.800 Liquid Density: 2.413 g/cc

Multi-Point BET Data					
Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]
4.86630e-02 7.44080e-02 1.02627e-01 1.20568e-01 1.44748e-01 1.68548e-01	0.0413 0.0562 0.0710 0.0803 0.0910 0.1012	3.3161e+02 3.8239e+02 4.3062e+02 4.5666e+02 4.9754e+02 5.3584e+02	1.94844e-01 2.23567e-01 2.40732e-01 2.71950e-01 2.90007e-01	0.1114 0.1222 0.1286 0.1391 0.1454	5.8088e+02 6.3025e+02 6.5968e+02 7.1818e+02 7.5150e+02

BET summary - 1710.176 Slope = 2.508e+02 0.999715 Intercept = Correlation coefficient, r = C constant= 7.819 Surface Area = 0.751 m²/g

Figure A22. Surface Area Analysis Summary MD-105-TR



Report
Operator: ra
Lehigh Tech_MD-180-TR_Lab 3847_051012.qps
Lehigh Technologies, Lab #3847 Analysis Operator: Date:5/10/2012 Date:2012/05/16 operator MD-180-TR

mple ID:

Ground Rubber 3.2087 g Sample Desc Comment

End of run: Run mode OutgasTemp: Bath Temp: Equil time: 158.7 min He Mode.Cell: 9mm large bulb 16.0 hrs Analysis Time: Void Vol.: 5/10/2012 16:22:02 Instrument: Instrument version: QuadraSorb Station 1 5.08

Standard 25.0 C

77.3 K 200/0 sec (ads/des) Analysis gas: Press. Tolerance: Krypton 0.050/0.000 (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

77.350K 20.500 Å Krypton Molec. Wt.: Temperature Cross Section: Adsorbate Liquid Density: 83.800 2.413 g/cc

Multi-Point BET Data —					
Relative Pressure [P/Po]	Volume @ STP	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP	1 / [W((Po/P) - 1)]
6.24007e-02	0.0189	9.4091e+02	2.02446e-01	0.0424	1.6027e+03
7.31546e-02 9.60094e-02	0.0211 0.0255	1.0010e+03 1.1128e+03	2.17884e-01 2.52479e-01	0.0445 0.0490	1.6734e+03 1.8420e+03
1.22970e-01 1.51280e-01	0.0302	1.2427e+03 1.3717e+03	2.69048e-01 2.86074e-01	0.0512	1.9234e+03
1.73381e-01	0.0348 0.0382	1.3/1/e+03 1.4678e+03	2.80U/4e-U1	0.0534	2.0078e+03

<u>BET su</u>	
Slope =	4706.949
Intercept =	6.554e+02
Correlation coefficient, r =	0.999880
C constant=	8.182
Surface Area =	0.275 m³/g

Figure A23. Surface Area Analysis Summary MD-180-TR



Analysis Operator: Sample ID: Report
Operator: ra D
Lehigh Tech_MD-400-AM_Lab 3647_050812.qps
Lehigh Technologies, Lab #3647 operator MD-400-AM Date:5/9/2012 Date:2012/05/23

Filename:

Sample Desc: Sample weight: Ground Rubber 2.3062 g Comment

156.6 min 5/9/2012 16:51:56 QuadraSorb Station 1 End of run: Instrument: Instrument version: Analysis Time: 5.06

Void Vol.: Outgas Time: Analysis gas: Press. Tolerance: Run mode OutgasTemp: Bath Temp: Equil time: He Mode.Cell: 9mm large bulb 24.0 hrs Standard 200.0 C 77.3 K

Krypton 0.050/0.000 (ads/des) Equil timeout: 200/0 sec (ads/des) 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

<u>Adsorbate</u> Krypton Molec. Wt.: Temperature Cross Section: 77.350K 20.500 Å 83.800 Liquid Density: 2.413 g/cc

	Multi-Point BET Data					
Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	
6.32462e-02 7.33299e-02 9.51805e-02 1.20562e-01 1.48546e-01	0.0264 0.0296 0.0360 0.0425 0.0491	6.8322e+02 7.1561e+02 7.8223e+02 8.6188e+02 9.5036e+02	1.74812e-01 2.03480e-01 2.19377e-01 2.52978e-01 2.69462e-01	0.0549 0.0608 0.0640 0.0701 0.0732	1.0316e+03 1.1234e+03 1.1752e+03 1.2920e+03 1.3485e+03	

BET summary = 3205.994 4.767e+02 Slope = Intercept = Correlation coefficient, r = C constant= 0.999798 7.725 0.400 m²/g Surface Area =

Figure A24. Surface Area Analysis Summary MD-400-AM



Analysis
Operator:
Sample ID:
Sample Desc:
Sample weight: Report
Operator: ra
Lehigh Tech_MD-400-TR_Lab 3847_050812.qps
Lehigh Technologies, Lab #3847 Date:5/8/2012 Date:2012/05/16 operator MD-400-TR

Filename:

Ground Rubber Comment

2.1951 g Analysis Time: Void Vol.: 131.2 min He Mode.Cell: 9mm large bulb 16.0 hrs 5/8/2012 11:00:40 End of run: QuadraSorb Station 1 Instrument:

Standard 25.0 C Run mode Instrument version: 5.06 Outgas Time: OutgasTemp:

Krypton 0.050/0.000 (ads/des) Analysis gas: Press. Tolerance: Bath Temp: Equil time: 200/0 sec (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

Data Reduction Parameters Data

Temperature Cross Section: 77.350K 20.500 Å: <u>Adsorbate</u> Krypton 83.800 Liquid Density: Molec. Wt.: 2.413 g/cc

Multi-Point BET Data Relative Pressure Volume @ STP 1 / [W((Po/P) - 1)] Relative Pressure Volume @ STP 1 / [W((Po/P) - 1)] [P/Po] [cc/g] [P/Po] [cc/g] 0.0055 0.0068 0.0082 0.0095 8.99974e-02 1.18573e-01 1.49350e-01 1.79402e-01 4.8129e+03 5.2831e+03 5.7282e+03 6.1646e+03 2.08516e-01 2.33145e-01 2.63815e-01 2.92393e-01 0.0107 0.0117 0.0128 0.0139 6.5979e+03 6.9703e+03 7.4612e+03 7.9366e+03

BET summary 15230.611 Slope = 3.447e+03 0.999759 Intercept = Correlation coefficient, r = C constant= 5.419 Surface Area = 0.079 m²/g

Figure A25. Surface Area Analysis Summary MD-400-TR



Analysis Operator: Sample ID: <u>Report</u> Operator: ra Lehigh Tech_MD-402-TR_Lab 3647_051012.qps Date:5/10/2012 Date:2012/05/16 operator MD-402-TR Sample Desc Ground Rubber Comment Lehigh Technologies, Lab #3647 Ground Rubber 3.9141 g 195.8 min He Mode.Cell: 9mm large bulb 16.0 hrs 5/10/2012 12:59:15 Standard End of run: Run mode QuadraSorb Station 1 5.06 Instrument version: OutgasTemp: Bath Temp: Equil time: Outgas Time: 25.0 C Analysis gas: Press. Tolerance: Krypton 0.050/0.000 (ads/des) 77.3 K 200/0 sec (ads/des) Equil timeout: 400/0 sec (ads/des)

Multi-Point BET

—Data Reduction Parameters Data

 Adsorbate
 Krypton
 Temperature
 77.350K

 Molec. Wt.:
 83.800
 Cross Section:
 20.500 Å*
 Liquid Density:
 2.413 g/cc

Multi-Point BET Data					
Relative Pressure	Volume @ STP	1 / [W((Po/P) - 1)]	Relative Pressure	Volume @ STP	1 / [W((Po/P) - 1)]
[P/Po]	[cc/g]		[P/Po]	[cc/g]	
5.91930e-02 7.31343e-02 9.65647e-02 1.25492e-01 1.50624e-01 1.67274e-01	0.0258 0.0310 0.0374 0.0446 0.0509 0.0547	6.5299e+02 6.8106e+02 7.6522e+02 8.6045e+02 9.3232e+02 9.8193e+02	2.00840e-01 2.21326e-01 2.42199e-01 2.64666e-01 2.88369e-01	0.0617 0.0659 0.0701 0.0743 0.0786	1.0891e+03 1.1534e+03 1.2198e+03 1.2954e+03 1.3785e+03

Figure A26. Surface Area Analysis Summary MD-402-TR

Appendix B: Binder Data

AASHTO M320 Binder Grading Results

Table B1. Base Binder

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	316, PaS	0.6	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
70	1.00	85.0	1.00	≥ 1.00 kPa
76	0.51	85.9	0.51]
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		•
Mass Change, %			-0.195	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
70	2.98	79.5	3.03	≥ 2.20 kPa
76	1.472	81.98	1.49	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	-
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC 22	G*, kPa 6000	Phase Angle δ, ° 39.8	G* sinδ, kPa 3842	≤ 5,000 kPa
19	8749	37.6	5336	_ = 5,000 Ki a
. •	ometer (BBR) AASHTO	I .	0000	1
Test Temperature, °C				
-12	Stiffness, Mpa		146	≤ 300 Mpa
	m-value		0.337	≥ 0.300
-18	Stiffness, Mpa		282	
	m-value		0.272	
True Grade	70.0	-25.4		
PG Grade	70 -	22		

Table B2. -80/+140

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	316, PaS	1.425	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.14	81.8	1.16	≥ 1.00 kPa
88	0.66	82.9	0.67	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		•
Mass Change, %			-0.164	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		•
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.34	70.5	3.549	≥ 2.20 kPa
88	1.915	74.35	1.988	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2		•
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 19	G*, kPa 7091	Phase Angle δ, ° 36	G* sinδ, kPa 4168	≤ 5,000 kPa
16	9864	34.2	5543	
Bending Beam Rhe	ometer (BBR) AASHTO	D T313	T	•
Test Temperature, ºC				
-12	Stiffness, Mpa		89	≤ 300 Mpa
	m-value		0.322	≥ 0.300
-18	Stiffness, Mpa		188	_
	m-value		0.276	
True Grade	83.6			
PG Grade	82 -	22		

Table B3. MD-180-TR

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	[,] @ 135°C, AASHTO T 3	316, PaS	0.825	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
76	1.71	83.2	1.73	≥ 1.00 kPa
82	0.92	85.3	0.92	
Rolling Thin Film ((RTFO) Aged Binder, A	ASHTO T 240	•	
Mass Change, %			-0.183	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.33	72.5	3.50	≥ 2.20 kPa
88	1.86	76.8	1.91	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C	G*, kPa 6161	Phase Angle δ, ° 38.9	G* sinδ, kPa 3965	_ ≤ 5,000 kPa
19	8604	37	5177	_ = 0,000 Ki u
	ometer (BBR) AASHT(_		
Test Temperature, °C				
-12	Stiffness, Mpa		112	≤ 300 Mpa
	m-value		0.325	≥ 0.300
-18	Stiffness, Mpa		234	
	m-value		0.276	
True Grade	81.2			
PG Grade	76 -	22		

Table B4. MD-400-TR

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	316, PaS	1.425	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
76	1.55	83.9	1.55	≥ 1.00 kPa
82	0.85	85.5	0.85	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.215	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.71	70.6	3.938	≥ 2.20 kPa
88	2.131	74.8	2.209	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 16	G*, kPa 8715	Phase Angle δ, ° 33.7	G* sinδ, kPa 4829	≤ 5,000 kPa
13	12050	32	6383	
Bending Beam Rhe	ometer (BBR) AASHTO	J 1313	<u> </u>	
Test Temperature, °C				
-12	Stiffness, Mpa		89	≤ 300 Mpa
	m-value		0.316	≥ 0.300
-18	Stiffness, Mpa		179	
	m-value		0.273	
True Grade	80.4			
PG Grade	76 -	22		

Table B5. MD-402-TR

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	316, PaS	1.3	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
76	1.36	84.6	1.37	≥ 1.00 kPa
82	0.73	85.8	0.73	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.236	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.35	73.3	3.501	≥ 2.20 kPa
88	1.88	77.2	1.928	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 22	G*, kPa 5192	Phase Angle δ, ° 36.9	G* sinδ, kPa 3118	≤ 5,000 kPa
19	7233	35.2	4165	
Bending Beam Rhe	ometer (BBR) AASHT(D T313	Т	
Test Temperature, ºC				
-12	Stiffness, Mpa		96	≤ 300 Mpa
	m-value		0.306	≥ 0.300
-18	Stiffness, Mpa		186	
	m-value		0.27	
True Grade	79.0	-23.0		
PG Grade	76 -	22		

Table B6. MD-105-TR

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	316, PaS	1.425	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
76	1.20	82.9	1.21	≥ 1.00 kPa
82	0.66	83.7	0.66	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		•
Mass Change, %			-0.174	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	2.14	76.92	2.192	≥ 2.20 kPa
88	1.198	79.9	1.217	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 19	G*, kPa 7319	Phase Angle δ, ° 36.4	G* sinδ, kPa 4339	≤ 5,000 kPa
16	10390	34.4	5864	
Bending Beam Rhe	ometer (BBR) AASHTO	O T313	1	
Test Temperature, °C				
-12	Stiffness, Mpa		93	≤ 300 Mpa
	m-value		0.317	≥ 0.300
-18	Stiffness, Mpa		183	
	m-value		0.289	
True Grade	77.9	-25.6		
PG Grade	76 -	22		

Table B7. -30 Liberty

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	[,] @ 135°C, AASHTO T 3	316, PaS	1.4	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
76	1.61	82.9	1.62	≥ 1.00 kPa
82	0.87	84.8	0.87	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.23	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.00	73.2	3.128	≥ 2.20 kPa
88	1.689	77.1	1.733	1
Pressure Aging Ve	essel (PAV) Aged Bind	er, AASHTO R2	8	•
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, °C 19	G*, kPa 7989	Phase Angle δ, ° 34	G* sinδ, kPa 4471	≤ 5,000 kPa
16	10810	32.5	5805	
Bending Beam Rhe	ometer (BBR) AASHT	O T313	Ι	1
Test Temperature, °C				
-12	Stiffness, Mpa		94	≤ 300 Mpa
	m-value		0.309	≥ 0.300
-18	Stiffness, Mpa		193	4
	m-value		0.276	
True Grade	80.7	-23.6		
PG Grade	76 -	22		

Table B8. -20 Liberty

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T	316, PaS	1.887	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.09	81.5	1.10	≥ 1.00 kPa
88	0.63	83.1	0.64	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.237	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ∘C	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.23	69.2	3.46	≥ 2.20 kPa
88	1.929	72.6	2.022	1
Pressure Aging Ve	essel (PAV) Aged Bind	er, AASHTO R2	8	•
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, °C 16	G*, kPa 9057	Phase Angle δ, ° 33.1	G* sinδ, kPa 4942	≤ 5,000 kPa
13	12410	31.5	6476	
Bending Beam Rhe	ometer (BBR) AASHT	O T313	I	1
Test Temperature, °C				_
-12	Stiffness, Mpa		92	≤ 300 Mpa
	m-value		0.315	≥ 0.300
-18	Stiffness, Mpa		176	1
	m-value		0.28	
True Grade	83.1	-24.6		
PG Grade	82 -	22		

Table B9. Liberty Powderizers -16 (1mm gap)

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	16, PaS	1.6	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
94	0.94	36.2	1.60	≥ 1.00 kPa
100	0.89	28.34	1.87	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.227	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
94	2.65	45.72	3.696	≥ 2.20 kPa
100	2.131	39	3.384	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	=4
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 19	G*, kPa 6852	Phase Angle δ, ° 35.1	G* sinδ, kPa 3935	≤ 5,000 kPa
16	9509	33.3	5226	
Bending Beam Rhe	ometer (BBR) AASHTO	T313		
Test Temperature, ºC				
-6	Stiffness, Mpa		55	≤ 300 Mpa
	m-value		0.33	≥ 0.300
-12	Stiffness, Mpa		102	
	m-value		0.299	
True Grade	76.3			
PG Grade	76 -	16		

Table B10. Liberty Powderizers -16 (2mm gap)

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	16, PaS	1.6	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.28	82.1	1.29	≥ 1.00 kPa
88	0.73	83.6	0.74	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.227	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
88	2.18	72.8	2.282	≥ 2.20 kPa
94	1.288	76.1	1.327	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315			
Test Temperature, °C 19	G*, kPa 6852	Phase Angle δ, ° 35.1	G* sinδ, kPa 3935	≤ 5,000 kPa
16	9509	33.3	5226	
Bending Beam Rhe	ometer (BBR) AASHTO	T313		
Test Temperature, °C				
-6	Stiffness, Mpa		55	≤ 300 Mpa
	m-value		0.33	≥ 0.300
-12	Stiffness, Mpa		102	
	m-value		0.299	
True Grade	84.7			
PG Grade	82 -	16		

Table B11. Liberty Crackermill

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity @ 135°C, AASHTO T 316, PaS			1.99	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.07	82.1	1.08	≥ 1.00 kPa
88	0.61	83.9	0.61	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.193	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.21	69.7	3.42	≥ 2.20 kPa
88	1.899	73.4	1.98	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 16	G*, kPa 10020	Phase Angle δ, ° 33.9	G* sinδ, kPa 5594	≤ 5,000 kPa
19	7186	35.6	4186	_ 3,000 KF a
_	ometer (BBR) AASHT(4100	
Test Temperature, °C	(==-)			
-12	Stiffness, Mpa		99	≤ 300 Mpa
	m-value		0.306	≥ 0.300
-18	Stiffness, Mpa		199	_
	m-value		0.273	
True Grade	82.8	-23.1		
PG Grade	82 -	22		

Table B12. Liberty Cryohammer

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	[,] @ 135°C, AASHTO T 3	316, PaS	1.675	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.01	82.8	1.02	≥ 1.00 kPa
88	0.58	84.2	0.58	
Rolling Thin Film ((RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.193	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.14	72.5	3.294	≥ 2.20 kPa
88	1.793	76.4	1.84	
Pressure Aging Ve	essel (PAV) Aged Bind	er, AASHTO R2	8	•
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, °C 16	G*, kPa 9482	Phase Angle δ, ° 33.2	G* sinδ, kPa 5186	_ ≤ 5,000 kPa
19	6777	34.95	3883	
Bending Beam Rhe	ometer (BBR) AASHTO	O T313		
Test Temperature, ºC				
-12	Stiffness, Mpa		100	≤ 300 Mpa
	m-value		0.307	≥ 0.300
-18	Stiffness, Mpa		184	
	m-value		0.273	
True Grade	82.2			
PG Grade	82 -	22		

Table B13. Liberty Cryohammer at 15% Rubber Content

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	@ 135°C, AASHTO T 3	316, PaS	2.912	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.52	81.2	1.54	≥ 1.00 kPa
88	0.88	82.8	0.89	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		•
Mass Change, %			-0.259	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
88	2.76	73.6	2.88	≥ 2.20 kPa
94	1.641	77.4	1.682	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, °C 16	G*, kPa 10310	Phase Angle δ, ° 32.1	G* sinδ, kPa 5471	≤ 5,000 kPa
19	7744	33.4	4262	
Bending Beam Rhe	ometer (BBR) AASHTO) T313	Ī	
Test Temperature, °C				
-6	Stiffness, Mpa		46	≤ 300 Mpa
	m-value		0.338	≥ 0.300
-12	Stiffness, Mpa		302	
	m-value		0.269	
True Grade	86.7			
PG Grade	82 -	16		

Table B14. Liberty 20 Mesh at 15% Rubber Content

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity	[,] @ 135°C, AASHTO T 3	316, PaS	4.050	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	1.63	79.5	1.66	≥ 1.00 kPa
88	0.98	80.4	1.00	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		
Mass Change, %			-0.259	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
88	3.20	68.8	3.43	≥ 2.20 kPa
94	1.955	72	2.06	
Pressure Aging Ve	essel (PAV) Aged Bind	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5	<u>r</u>	
Test Temperature, °C	G*, kPa	Phase Angle δ, °	G* sinδ, kPa	< 5 000 l-D-
16 19	10500 7645	31.4 33.1	5471 4172	_ ≤ 5,000 kPa
	ometer (BBR) AASHT(4172	
	ometer (bbit) AASITI	J 1313		I
Test Temperature, °C				
-6	Stiffness, Mpa		43	≤ 300 Mpa
	m-value		0.324	≥ 0.300
-12	Stiffness, Mpa		85	_
	m-value		0.297	
True Grade	87.9	-21.3		
PG Grade	82 -	16		

Table B15. -30 Liberty Fines

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity @ 135°C, AASHTO T 316, PaS			1.725	≤ 3 PaS
Dynamic Shear Rhe	eometer AASHTO T 31	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
76	1.44	82.8	1.45	≥ 1.00 kPa
82	0.80	84.4	0.80	
Rolling Thin Film (RTFO) Aged Binder, A	ASHTO T 240		•
Mass Change, %			-0.256	≤ 1.00%
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC	G*, kPa	Phase Angle δ, °	G* / sinδ, kPa	
82	3.65	69.2	3.90	≥ 2.20 kPa
88	2.13	73.2	2.23	
Pressure Aging Ve	essel (PAV) Aged Binde	er, AASHTO R2	8	
Dynamic Shear Rhe	eometer AASHTO T 315	5		
Test Temperature, ºC 13	G*, kPa 10900	Phase Angle δ, ° 32.4	G* sinδ, kPa 5835	≤ 5,000 kPa
16	7959	33.9	4434	3 5,000 KFa
_	ometer (BBR) AASHT(1 1101	1
Test Temperature, °C				
-6	Stiffness, Mpa		54	≤ 300 Mpa
	m-value	-	0.324	≥ 0.300
-12	Stiffness, Mpa		109	_
	m-value		0.291	
True Grade	79.8	-20.4		
PG Grade	76 -	16		

Table B16. MD-400-AM

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity @	135°C, AASHTO T 316,	PaS	1.887	≤ 3 PaS
Dynamic Shear Rheom	neter AASHTO T 315			
		Phase Angle		
Test Temperature, °C	G*, kPa	δ, °	G* / sinδ, kPa]
82	1.00	84.1	1.01	≥ 1.00 kPa
88	0.58	85.3	0.58	
Rolling Thin Film (RT	FO) Aged Binder, AAS	SHTO T 240		
Mass Change, %			-0.281	≤ 1.00%
Dynamic Shear Rheom	eter AASHTO T 315			
		Phase Angle		
Test Temperature, °C	G*, kPa	δ, °	G* / sinδ, kPa	
88	2.15	73.5	2.24	≥ 2.20 kPa
94	1.281	76.7	1.32	
Pressure Aging Vess	el (PAV) Aged Binder,	AASHTO R28		
Dynamic Shear Rheom	eter AASHTO T 315			
Test Temperature, °C				
		Phase Angle		
Test Temperature, °C	G*, kPa	δ, °	G* sinδ, kPa	
19	7520	32.2	4008	≤ 5,000 kPa
16	9903	31.3	5144	
Bending Beam Rheom	eter (BBR) AASHTO T	313		
Test Temperature, °C				
-6	Stiffness, Mpa		53	≤ 300 Mpa
	m-value		0.323	≥ 0.300
-12	Stiffness, Mpa		102	
	m-value		0.294	
True Grade	82.1	-20.8		
PG Grade	82 -	16		

Table B17. MD-400-TR with Vestenamer

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity @ 135°C, AASHTO T 316, PaS			1.712	≤ 3 PaS
Dynamic Shear Rheom	eter AASHTO T 315			
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* / sinδ, kPa	
76	1.79	83.4	1.80	≥ 1.00 kPa
82	0.98	85.1	0.98	
Rolling Thin Film (RT	FO) Aged Binder, AAS	HTO T 240		
Mass Change, %			-0.268	≤ 1.00%
Dynamic Shear Rheom	eter AASHTO T 315			
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* / sinδ, kPa	
88	2.70	73.8	2.807	≥ 2.20 kPa
94	1.55	77.5	1.585	
	el (PAV) Aged Binder,	AASHTO R28		
Dynamic Shear Rheom	eter AASHTO T 315			1
Test Temperature, °C		1		
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* sinδ, kPa	
16	9715	34.1	5067	≤ 5,000 kPa
19	7248	32.9	3938	
Bending Beam Rheom	eter (BBR) AASHTO T	313		
Test Temperature, °C				
-6	Stiffness, Mpa		51	≤ 300 Mpa
	m-value		0.322	≥ 0.300
-12	Stiffness, Mpa		105	
	m-value		0.284	
True Grade	81.8	-19.5		
PG Grade	76 -	16		

Table B18. Liberty -30 with Vestenamer

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity @	135°C, AASHTO T 316	, PaS	1.650	≤ 3 PaS
Dynamic Shear Rheom	eter AASHTO T 315			
	0* 1.5	Phase Angle δ,	0# / : 5 D	
Test Temperature, °C	G*, kPa	-	G* / sinδ, kPa	
82	1.02	84.1	1.03	≥ 1.00 kPa
88	0.58	85.5	0.59	
<u> </u>	FO) Aged Binder, AAS	НТО Т 240		1
Mass Change, %			-0.281	≤ 1.00%
Dynamic Shear Rheom	eter AASHTO T 315			1
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* / sinδ, kPa	
88	2.33	69.9	2.48	≥ 2.20 kPa
94	1.40	69.1	1.50	
Pressure Aging Vess	el (PAV) Aged Binder,	AASHTO R28		
Dynamic Shear Rheom	<u> </u>			
Test Temperature, °C				
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* sinδ, kPa	
16	9964	32.3	5319	≤ 5,000 kPa
19	7254	33.8	4037	
Bending Beam Rheom	eter (BBR) AASHTO T	313		
Test Temperature, °C				
-12	Stiffness, Mpa		98	≤ 300 Mpa
	m-value		0.303	≥ 0.300
-18	Stiffness, Mpa		201	
	m-value		0.256	
True Grade	82.3	-22.4		
PG Grade	82 -	22		

Table B19. Blacklidge Hybrid 88-22

Original Binder				
Test, Method			Test Results	Specification
Rotational Viscosity @	135°C, AASHTO T 316	, PaS	1.937	≤ 3 PaS
Dynamic Shear Rheom	neter AASHTO T 315			
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* / sinδ, kPa	
82	1.04	75.1	1.08	≥ 1.00 kPa
88	0.62	77.5	0.64	1
Rolling Thin Film (RT	FO) Aged Binder, AAS	HTO T 240		
Mass Change, %			-0.209	≤ 1.00%
Dynamic Shear Rheom	neter AASHTO T 315			
Test Temperature, °C	G*, kPa	Phase Angle δ,	G* / sinδ, kPa	
82	2.23	69.3	2.39	≥ 2.20 kPa
88	1.34	71.8	1.41	
Pressure Aging Vesse	el (PAV) Aged Binder,	AASHTO R28		
Dynamic Shear Rheom	eter AASHTO T 315			
Test Temperature, °C				
Test Temperature, °C 19	G*, kPa 8459	Phase Angle δ, °	G* sinδ, kPa 5056	≤ 5,000 kPa
22	5974	38.6	3708	3,000 KF a
<u></u>	eter (BBR) AASHTO T	l.	0,00	1
Test Temperature, °C	oto. (BBIT) PROTITO I	0.10		
-12	Stiffness, Mpa		117	≤ 300 Mpa
	m-value		0.310	≥ 0.300
-18	Stiffness, Mpa		323	-
	m-value		0.269	
True Grade	82.8	-23.5		
PG Grade	82 -	22		