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The Guayule Plant: A Renewable, Domestic Source of Binder Materials for Flexible Pavement Mixtures

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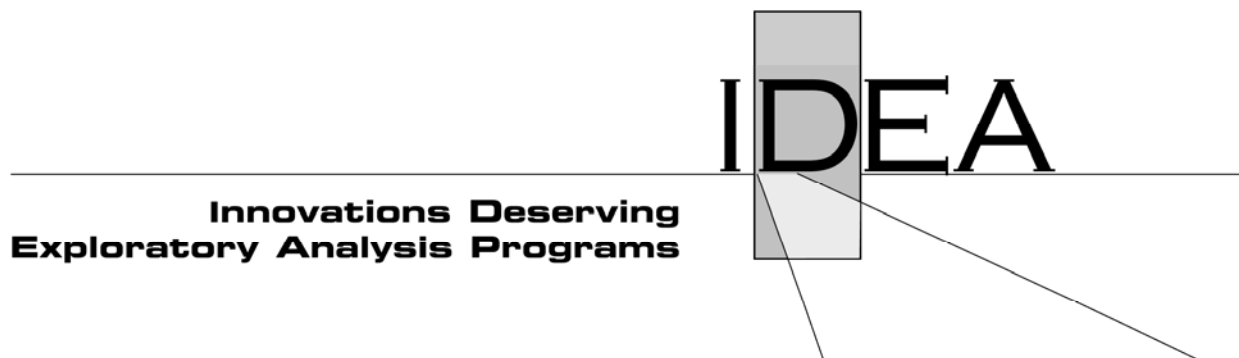
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The Guayule Plant a Renewable Domestic Source of Binder Materials for Flexible Pavement Mixtures

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THE GUAYULE PLANT: A RENEWABLE, DOMESTIC SOURCE OF BINDER MATERIALS FOR FLEXIBLE PAVEMENT MIXTURES

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January 2013

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EXECUTIVE SUMMARY

Due to the rising price of crude oil, flexible pavement costs have increased significantly. This price pressure has resulted in the increased use of reclaimed asphalt pavement (RAP) and/or reclaimed asphalt roofing shingles (RAS) because of the binder (asphalt cement) they contain. This has increased demand for recycling (rejuvenating) agents which return the RAP/RAS binders to their original state by 1) restoring maltenes (petroleum oils and resins) that have been depleted due to age-hardening/oxidation, and 2) reducing their viscosity.

The project concept was to design a flexible pavement mixture (FPM) produced with little-to-no virgin petroleum-based binder which implied the use of high percentages of RAP and/or RAS, and a bio-based virgin binder. The potential impacts of a renewable (bio-based), domestic source of FPM binder on highway construction could be lower costs and, perhaps more importantly, a decreased dependence on foreign oil.

The guayule (pronounced 'why-YOU-lee') plant is a perennial that grows in arid and semi-arid regions (e.g. the southwestern U.S.) and is a source of natural rubber. Three basic products can be obtained from the guayule plant: 1) rubber in latex or bulk (dried latex) form, 2) bagasse (post-rubber-extraction fibrous residue), and 3) resin. Guayule is currently being cultivated and processed primarily for the manufacture of hypo-allergenic latex used in medical products (e.g. gloves and catheters), but it has a long history including the use of the bulk rubber during World War II.

To validate the project concept, the use of guayule-based material in combination with high percentages of RAP and/or RAS was investigated. Two of the many guayule-based materials produced during this project became the focus of the work: 1) an acetone-extracted resin that can be an undesirable residuum in the bulk rubber but useful for FPM, and 2) a hexane-extract from the waste-stream leaves and attached stems. Comparisons were made of these two materials to two petroleum-based binders with similar viscosity-temperature relationships: 1) the acetone-extracted rubber resin (RR) was compared to a commercially-available recycling agent, Cyclogen® L (CycL), and 2) the hexane-extract from the leaf/stems (LF) was compared to a performance-graded (PG) 52-28 binder often utilized for high-RAP/RAS FPMs where the specified binder grade is stiffer than a PG52-28.

Binder-blending mixture experiments, useful for comparative analyses and necessary for FPM design, required creating many different blends of the RAP/RAS binders with the RR and LF, recreating those same blends but substituting the RR and LF with the appropriate petroleum-based binders, testing each blend, then generating response surface models (RSMs) using those test results. Binder-blending experiment conclusions are as follows:

- The RR-RAP/RAS blends did perform the same as proportionally-identical CycL-RAP/RAS blends in terms of high-temperature stiffness (as measured by the high critical temperature, T_cH), but did not perform exactly the same in terms of cold-temperature cracking resistance (as measured by the low critical temperature, T_cL). In terms more familiar to the FPM designer, the difference between the absolute temperature spread ($T_cH - T_cL$) for the RR-RAP/RAS blends relative to proportionally-identical CycL-RAP/RAS blends was small, but statistically different.

- The absolute temperature spreads ($T_cH - T_cL$) for the LF-RAP/RAS blends were, to a large degree, significantly different than the proportionally-identical PG52-28-RAP/RAS blends. This finding turned the remaining investigative effort solely onto the RR.

- The RR and LF suffer significant mass loss upon short-term aging at 163°C. However, when used as a recycling agent, the RR or LF would be considerably diluted by the RAP/RAS binder. Additionally, the RR can auto-oxidize at room

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temperature if exposed to the air for prolonged periods of time, and may dissolve if exposed to hot water for prolonged periods of time even if it is blended with petroleum-based binder.

FPM design began by using the RSMs to generate a RR-RAP/RAS blend that would meet specifications for a PG64-22 binder. Using an optimization procedure in the experimental mixture design software, a blend of 42.4% RAP1 binder, 17.8% RAP2 binder, 15.0% RAS binder, and 24.8% RR (note: no virgin petroleum-based binder) was produced that, through verification testing, met all specifications for a PG64-22 binder, except mass change. The maximum allowable mass change for a PG binder is ±1.0% but the blend experienced a mass loss of 3.1%.

Using this binder blend, a RR-based FPM was designed, produced, and tested that 1) met the gradation specification for a Missouri Department of Transportation (MoDOT) moderate-quality FPM, but 2) nearly met all of the volumetric requirements. This FPM utilized 53% reclaimed RAP/RAS aggregate and, therefore, 47% virgin aggregate. More importantly, and in relation to the project concept, only 0.23% of the total FPM mass (or 5.62% of total binder mass) was virgin PG64-22 binder. The rest of the binder was RAP/RAS binder and RR. A CycL-based FPM using the same design proportions was also produced and tested for comparative analyses. Conclusions from the FPM testing are as follows:

- Standard Hamburg Wheel-Track testing, which specifies full submersion of the specimens in 50°C water during the approximately 7 hours of rut testing, was performed. The results indicated that the RR-based FPM performed as well as the CycL-based FPM in regard to rutting and stripping (moisture damage) resistance.

- Standard Tensile Strength Ratio (TSR) testing, which specifies a 24 hour full submersion of the specimens in 60°C water, was performed. The results of this moisture-susceptibility test, however, indicated that the RR-based FPM may be more prone to stripping than the CycL-based FPM.

- A (currently) non-standard, but promising test protocol for determining low-temperature, FPM flexural creep stiffness was performed on the RR- and CycL-based FPMs. At -12°C, statistical analyses showed that the creep stiffness of the RR-based FPM was not significantly different than the CycL-based FPM. However, the m-value of the RR-based FPM was statistically different (lower) than the CycL-based FPM m-value which indicates a somewhat higher cold-temperature cracking potential for the RR-based FPM relative to the CycL-based FPM. The RR-based FPM met and nearly met recommended limits for creep stiffness and m-value, respectively.

Based on the test results, the RR produced in this study could, in a practical sense, be used as a recycling agent. FPM-production and paving industry representatives have shown interest in the results of this study and the future viability of guayule-based materials. The Yulex® Corporation, the only U.S. company currently cultivating and processing the guayule plant for commercial purposes, recently indicated that they are still in the economic modeling phase for resins and are planning to add more processing facilities in the near future. Bridgestone and Cooper Tire are also planning to begin research into and production of guayule-based products.

So, although the economic viability of guayule products for FPM applications is difficult to determine at this point, the potential availability of guayule in the U.S. as a feedstock is substantial; there are about 124 million acres of land in the U.S., mostly in the southwest, environmentally suited for guayule agricultural practices. This project was a first step and it verified that the guayule shrub, a perennial industrial plant, holds great potential as a renewable, domestic source of FPM binder additives such as resins, oils, and polymers.

IDEA PRODUCT

Due to the rising price of crude oil, flexible pavement costs have increased significantly. This price pressure has resulted in the increased use of reclaimed asphalt pavement (RAP) and/or reclaimed asphalt roofing shingles (RAS) because of the binder they contain. This has increased demand for recycling (rejuvenating) agents which return the RAP/RAS binders to their original state by 1) restoring maltenes (petroleum oils and resins) that have been depleted due to age-hardening/oxidation, and 2) reducing their viscosity. Note that the term "recycling agent" has been used interchangeably with terms such as rejuvenator, softening agent, aromatic oil, and others. However, Roberts et. al make the distinction that a softening agent lowers the viscosity of the aged binder while a rejuvenatorrestores the aged binder's viscous properties as well as the chemical constituency (*1*). Note that the term "recycling agent" will be used in this report to identify a material that "rejuvenates" the aged binder.

The project concept was to design a flexible pavement mixture (FPM) produced with little-to-no virgin petroleum-based binder (asphalt cement) which implied using high percentages of RAP and/or RAS, and a bio-based virgin binder. Note that the term "flexible pavement mixture" will be used throughout this report instead of "asphalt pavement mixture" in an effort to begin the terminology transition due to the inevitable depletion of crude oil.

The guayule (pronounced 'why-YOU-lee') plant is a perennial that grows in arid and semi-arid regions (e.g. the southwestern U.S.) and is a source of natural rubber. Three basic products can be obtained from the guayule plant: 1) rubber in latex or bulk (dried latex) form, 2) bagasse (post-rubber-extraction fibrous residue), and 3) resin. The guayule plant is currently being cultivated and processed primarily for the manufacture of hypo-allergenic latex used in medical products such as gloves and catheters, but it has a long history including the use of the guayule rubber during the World War II rubber shortage.

FIGURE 1: Arizona Guayule Fields (permission from Yulex® Corp.)

The potential impact of a renewable (bio-based), domestic source of FPM binder on highway construction could be lower costs. Perhaps more importantly, national security could be enhanced by decreasing the dependence on foreign oil.

In order to validate the project concept, the use of guayule-based material in combination with high percentages of RAP and/or RAS was investigated. Numerous extraction/recovery procedures were developed and performed using different guayule feedstocks and solvents. For each feedstock/solvent combination, the recovered product was evaluated through testing. Equipment was developed, and some additional equipment purchases were made that exceeded the expectations enumerated in the project proposal.

Two of the many guayule-based materials produced during this project became the focus of the work: 1) an acetone-extracted resin that can be an undesirable residuum in the bulk rubber but useful for FPM, and 2) a hexane-extract from the leaves and attached stems (currently removed before the commercial, water-based, rubber-extraction process, and considered a waste product). Comparisons were made of the two guayule-based materials to two petroleum-based binders (currently used in FPMs) with similar viscosity-temperature relationships: 1) the acetone-extracted rubber resin (RR) was compared to a commercially-available recycling agent, Cyclogen® L (CycL), and 2) the hexane-extract from the leaf/stems (LF) was compared to a performance-graded (PG) 52-28 binder often utilized for high-RAP/RAS FPMs where the specified binder grade is stiffer than a PG52-28.

CONCEPT AND INNOVATION

The project concept was to design a FPM produced with little-to-no virgin petroleum-based binder requiring the use of 1) high percentages of RAP and/or RAS, and 2) some type of non-petroleum based recycling agent. Both of the concept requirements have driven this research. Although RAP usage in paving applications became popular during the 1970s oil embargo, **high**-RAP FPM design has only recently become more common due to increasing crude oil and aggregate costs, and environmental concerns. However, the design procedure is still evolving (*3, 4, 5*). RAS usage also began in the 1970s and is on the rise with a large increase in the utilization of tear-off roofing shingles (*6*). The state of the art in FPM design using RAS, however, is in its relative infancy (*7*).

Therefore, FPM design using high percentages of RAP and/or RAS is a challenge unto itself and is leading to innovation on its own. For example, new designs and construction of asphalt mixing facilities/equipment are occurring regularly that are marketed for the purpose of utilizing RAP percentages as high as 100% (*8, 9, 10*). Currently, the upper limit for RAP usage at Missouri FPM mixing facilities is about 50% with a more practical upper limit of 40%.

Although the use of the guayule plant as a potential source of binder materials for FPM is a unique concept, there are other instances of the investigation/use of other renewable and/or waste bio-materials as FPM binder. For example, researchers at Iowa State University are investigating various biomass materials as sources of FPM binder (*11*), research engineers for a Leicester county, United Kingdom company (Aggregate Industries) are looking to use "chip" fat (i.e. waste vegetable oil) as a binder (*12*), and a French road construction company, Colas SA, has patented a binder made from sunflower oil called Végécol (*13*). Shell Bitumen has entered the fray with a product called Floraphalte (*14*). Earlier work on bio-based asphalt additives occurred in India where resin from the Anacardium Occidentale tree was processed to be used as a recycling agent (*15*). Bitu Tech RAP is a product recently available from Engineered Additives, LLC and is marketed as a recycling agent (*16*). EcoPave in Australia has also developed bio-based binders for paving purposes (*17*). Recently marketed products are based on crude tall oil, a paper manufacturing co-product (e.g. Hydrogreen®).

The primary innovation in this research is the development and use of guayule- or bio-based materials with the following attributes:

- They are obtained from a perennial plant specifically cultivated for industrial uses, not food production.
- Their source is renewable and of a USA domestic origin.

- One of the bio-based materials, a hexane-extract (LF), is derived from material that is currently waste (i.e. the leaves and attached stems are removed prior to commercial latex production from the remaining woody parts of the plant) and may serve to enhance other FPM performance parameters due to the rubber content of the LF.
- The second bio-based material, the rubber resin (RR), is a co-product removed by extraction from a secondary product (i.e. the bulk rubber) in order to improve said product. According to the Yulex Corporation, the residual, amber-colored resin in the bulk rubber is undesirable in certain applications (*18*).
- The solvent extraction methods used to produce this study's bio-based recycling agents are relatively simple in that only one hydrocarbon solvent per material is required which would minimize production costs. Alternatively, supercritical fluid extraction, although more complex, would make hydrocarbon solvents unnecessary and decrease the environmental impact of processing.
- They blend well with petroleum-based binders at typical (low to moderate) dosages.
- They perform as intended by 1) reducing the viscosity of the hardened RAP/RAS binder and 2) replenishing depleted polar and aromatic compounds.

What makes the guayule plant a unique source for bio-based FPM additives is the presence of high-quality natural rubber. Although the focus of this work has been on guayule-based materials as a recycling agent, it is important to stress the potential use of the natural rubber as a bio-polymer modifier to increase the high-temperature performance of FPMs.

INVESTIGATION

The project work was divided into three (3) tasks:

Task 1: Procurement of raw materials and equipment, and development of equipment, laboratory infrastructure, and test protocols.

Raw materials obtained for the project include:
- RAP1: reclaimed asphalt pavement obtained from Interstate 44 in Rolla, Missouri
- RAP2: reclaimed asphalt pavement obtained from a city street in Washington, Missouri
- RAS: processed tear-off roofing shingles obtained from local hot mix asphalt producer
- United States Department of Agriculture (USDA)-supplied, chipped, whole-shrub guayule plant
- Yulex Corporation-supplied pelletized whole-shrub guayule plant
- Yulex Corporation-supplied post-rubber-extraction (PRE) bagasse (and later in the project, the rubber itself)
- Yulex Corporaton-supplied waste-stream guayule leaves and attached stems
- Six different grades of polymer-modified and non-modified performance-graded (PG) binders
- Two different commercially-available, petroleum-based recycling agents

Significant equipment/supply items purchased are as follows:
- Bohlin Gemini 150 Dynamic Shear Rheometer (DSR) and Cannon Instruments temperature probe
- Applied Testing Systems Bending Beam Rheometer (BBR) and 10 BBR beam molds

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- Cox and Sons Rolling Thin Film Oven (RTFO), along with standard and large-lipped RTFO bottles
- Prentex Pressure Aging Vessel and bottled, compressed breathable air
- 20 liter Oregon Environmental Systems Solvent Recovery Device for large-scale primary distillations
- Clay-Gel Chromatography glassware, clay, and silica gel
- Large centrifuge for solvent extraction/recovery procedures
- Significant quantities of acetone, trichloroethylene, hexane, and lesser quantities of pentane, toluene, ethanol, and methanol
- Bottled, compressed carbon dioxide and nitrogen for gas purges during material extraction/recovery procedures

Equipment, laboratory infrastructure, and test protocol development included:

- Installation of an air compressor dedicated to the DSR, BBR, and RTFO
- Fabrication/installation of a fume hood in an isolated room with an independent air-exhaust system: the solvent recovery device was installed under the fume hood, and the room served as an area to work with and store the solvents
- Installation of compressed air lines and electrical power wiring/conduit for all new equipment
- Fabrication of a required clean and dry air filtering system for the RTFO
- Development of large-scale (20 liters of solvent) extraction and recovery procedures for RAP/RAS binders and guayule-based materials

Task 2: Individual test material characterization and binder-blending mixture experiments
Task 3: Flexible pavement mixture (FPM) design, production, and testing.

The impetus to this research began in 2004 with the viewing of a History Channel *Modern Marvels* television program about the history of rubber. The guayule plant was featured in this program and interest was piqued due to the increasing use of rubberized asphalt in the paving industry. After making several inquiries into the status of the guayule rubber industry, contact was established with Dr. Francis Nakayama, a United States Department of Agriculture (USDA) research chemist. Dr. Nakayama arranged to provide samples of guayule rubber that had been produced approximately 20 years earlier. The package arrived and contained a large piece of black bulk rubber and a one gallon can labeled, "derubberized guayule resin." The resin was a very dark green, almost black, fluid with the consistency of honey at room temperature, and it had a piney, pleasant odor. At that time it seemed obvious that the resin had promise as a softening agent in hardened petroleum-based binder. Preliminary testing using the resin in combination with petroleum-based binder led to the submittal of a proposal to the NCHRP-IDEA program which resulted in a research grant.

Originally, the research plan was to use the ~20 year old, USDA-supplied, de-rubberized guayule resin. However, the decision was made to extract and recover guayule-based material from freshly harvested, whole-shrub guayule plants because questions arose during attempts to perform a clay-gel chromatography procedure (*19*) on the resin as to whether it contained mineral oil and/or an antioxidant, Santoflex 134. Extensive communication with researchers familiar with the history of the USDA resin, and analysis of the pertinent literature (*20, 21, 22*) made it clear that further use of the USDA-supplied guayule resin was unwise. This turn of events proved to be a major unforeseen setback to the research and forced a significant increase in the scope of the project.

The decision to extract and recover guayule-based material in the laboratory was a major turning-point in the research; it drastically increased the proposed work plan. Instead of focusing on the materials characterization and binder-blending mixture experiments (to be discussed later), followed by FPM design, production, and testing, the majority of the research time was dedicated to obtaining a guayule-based material with the properties required for use as a recycling agent. Only then could the initially-planned Tasks 2 and 3 be started.

Another significant setback occurred because the first laboratory-obtained resins (using acetone-only extraction of various guayule plant feedstocks) were too stiff, making them ill-suited as a recycling agent. These resins, however, may hold promise as stiffening agents to improve the high-temperature performance of FPMs.

The next step was to investigate different solvents either alone or in combination with other solvents. Liquid-liquid extraction using pentane and aqueous methanol, and cold-filtered ethanol partitioning were also investigated. Although both of these procedures produced guayule-based materials with viscous properties sufficient for use as a recycling agent, neither yielded amounts considered sufficient to make it economical. In addition, these methods were much more complex to perform, relatively speaking, than single solvent extractions.

Despite the above setbacks, however, much was learned. It has been said that much of research is determining what does not work. This certainly was the case for this portion of the study.

Table 1 shows the results of the overall investigation into obtaining a guayule-based material with the properties required for use as a recycling agent, and reveals the reasons why two (2) were selected and nine (9) were rejected.

TABLE 1: Summary Table of Guayule-Based Material Generation Investigations

Plant or Plant Extract Precursor Materials	Solvent	Results
Whole-Shrub (WS)	Acetone	The WS resin is much stiffer and more temperature susceptible than a PG52-28 binder.
PRE Bagasse (PRE)	Acetone	The PRE resin is very stiff. Viscosity-temperature relationship similar to RAP binders.
Whole-Shrub (WS)	Pentane	Visual inspection only. The extract demonstrates moderate ductility, high elasticity. No follow-up work: pentane hazard-risk and high cost.
PRE Bagasse (PRE)	Pentane	Visual inspection only. The extract demonstrates moderate ductility and elasticity. No follow-up work: pentane hazard-risk and high cost.
PRE Bagasse (PRE)	Toluene	The resin is similar to the acetone-extracted PRE resin in terms of viscosity but is slightly less temperature susceptible.
Waste-Stream Leaf/Stem	***Hexane***	***The extract (LF) has viscosity similar to a PG52-28 but is significantly less temperature susceptible. Simple production method and moderate yield. Demonstrates high ductility, moderate elasticity upon visual inspection. Contains some rubber and is tacky.***
PRE Bagasse (PRE)	Hexane	The extract has a viscosity-temperature relationship similar to the LF material with higher viscosity; i.e. too stiff.
Waste-Stream Leaf/Stem	Acetone	Visual inspection only. Visual observation was similar to acetone-extracted WS resin. Too glassy (brittle) at room temperature.
Acetone-extracted PRE Bagasse Resin	Pentane-partition	The extract has good viscous properties comparable to Cyclogen L. No follow-up work: pentane hazard-risk and high cost, and a very complex production method. Very small yield.
Hexane-extracted LF	Ethanol-partition	The extract is less viscous than a PG46-28 binder but more viscous than Cyclogen L, and more temperature susceptible. Production less complex than pentane-partitioning. Small yield
Bulk Rubber (dried latex)	***Acetone***	***The rubber resin (RR) has viscosity similar to Cyclogen L but is slightly less temperature-susceptible. Relatively simple production method and high yield.***

The plant or plant-extract precursor materials investigated were three different plant feedstocks, two guayule-based materials resulting from the solvent extraction of one of the plant feedstocks, and the raw bulk rubber. The plant feedstocks were USDA-supplied chipped whole-shrub (WS), post-rubber-extraction (PRE) bagasse (fibrous residue leftover after 90-95% of the high molecular weight rubber was removed), and waste-stream leaf and attached stems, both of which were supplied by the Yulex Corporation. The whole-shrub and leaf/stem feedstocks were pulverized prior to solvent extraction using a horizontal shaft impactor. The PRE bagasse was already in a finely-macerated state.

Table 1 also refers to Cyclogen L® which is a petroleum-based aromatic oil used as a recycling agent. The PG52-28 and PG46-28 are softer performance graded (PG) petroleum-based binders.

The decision was made to focus on the LF material, the hexane-extract from the waste-stream leaves and attached stems (bolded and italicized in Table 1). The extraction procedure was simple, the yield averaged 4.4% by weight of the oven-dry (60°C) waste-stream leaf/stem, and the product satisfied the practical (but not all technical) requirements for a recycling agent. The LF is dark-green in color, semi-solid at room temperature, and has a piney, pleasant odor. However, the LF also contains some natural rubber which increases the tackiness of any material in which it is present.

Soon after beginning testing using the LF material, the Yulex Corporation suggested acetone-extraction on the natural bulk rubber they produce as a secondary product by drying the latex. The rubber has residual, amber-colored resin in it that 1) is undesirable for producing a near-white rubber that enables the creation of various grades of rubber, and 2) can be extracted using acetone. Yulex Corporation supplied about 50 pounds of the rubber and a method was devised to extract the resin by cutting the rubber into approximately 2 inch cubes, freezing the cubes in liquid nitrogen, pulverizing the frozen cubes, soaking the flaked rubber in acetone for an extended period of time, then de-solventizing the acetone-resin solution in a two-step distillation process. The acetone-extracted rubber resin (RR) is amber in color, flows at room temperature, and has a slightly pungent, but not unpleasant odor (bolded and italicized in Table 1). The acetone-extraction process was relatively simple and the yield averaged about 13% by weight of the rubber. The RR, too, satisfied the practical (but not all technical) requirements for a recycling agent.

Simultaneous to generating a desirable guayule-based material for use as a recycling agent, Task 2 was initiated: statistically-based, binder-blending mixture experiments were set up that 1) would allow for comparative analysis between a guayule-based material and a petroleum-based recycling agent, and 2) would be necessary for FPM design. Using the software program DesignExpert®, an optimal mixture experimental design was created that generated blend proportions for four components: two RAPs, one RAS, and one virgin binder, which is a generalized term here for any one of the guayule-based materials or petroleum-based binders to be utilized as recycling agents. The experiment required physically creating many different blends of the RAP/RAS binders with the guayule-based materials, recreating those blends by substituting the guayule-based materials with the appropriate petroleum-based binders, testing each blend, and then generating response surface models (RSMs) using those test results. The RSMs were then used to determine several alternative sets of proportions for optimizing a select binder property. These optimal blend proportions were then used for FPM design.

Comparisons of the two guayule-based materials to two petroleum-based binders of similar viscosity were made: 1) RR was compared to Cyclogen L (CycL), and 2) the LF was compared to a PG52-28 binder, a soft binder often used for high-RAP/RAS FPMs where the contract-specified, mixture binder grade is stiffer than a PG52-28.

LABORATORY INVESTIGATION

Only test methods and the associated final results that are relevant to the research objective are presented in this section. There was testing performed over the course of the investigation that was necessary in order to answer questions that arose but that do not require presentation. Most of the unreported results from this testing pertain to properties of the solvent-extracted-materials rejected as potential recycling agents. Those properties include viscosity, mass change during shear and/or at variable temperatures, and volatiles composition.

Material Characterization (Task 2)

Material characterization required testing that identified individual material properties required for classification purposes or that will be used for FPM design purposes. Those test methods and results are presented in this section.

Raw Material Descriptions

RAP1 came from a Missouri Department of Transportation (MoDOT) project on Interstate 44 in Phelps County, Missouri. RAP2 came from a city-street project in the city of Washington, Missouri.

The RAPs were further processed in the laboratory by first shaking them across a ¾ inch screen, running the +¾ inch material through a horizontal-shaft impactor, then running the pulverized RAP through the ¾ inch screen again such that the sample used for obtaining test specimens contained only RAP that had passed through the ¾ inch screen.

The RAS was supplied by a local asphalt contractor and had already been processed such that it would pass the ½ inch screen. However, MoDOT's specification for RAS is that it must pass the ⅜ inch screen. Therefore, the RAS was processed in the laboratory in the same manner as the RAP such that the sample used for obtaining test specimens contained only RAS that had passed through the ⅜ inch screen.

Virgin aggregate used in the FPMs was a dolomite from the Gasconade formation, obtained from a local asphalt producer. Three different size fractions were obtained: ½ inch clean, ⅜ inch clean, and manufactured sand.

The origins and basic methodology for processing of the precursor materials for the LF and RR materials were discussed in the previous section.

RAP/RAS Binder Contents and Guayule-Based Materials Yields (Extraction and Recovery)

Procedures ultimately used for solvent-based extraction and recovery procedures for the RAP/RAS binders, and the RR and LF materials were based on guidance given in standard test methods and in the literature (*23, 24, 25, 26, 27*). However, due to the amount of material needed to perform the required testing, certain aspects and/or portions of the standard test procedures were combined to develop the procedures utilized. The evolutionary development of the

procedures that were eventually used account for a very large portion of the time dedicated to this project. Detailed extraction/recovery procedures are beyond the scope of this report. Recovery (yield) Rresults are given in Table 2.

TABLE 2: Solvent-Based Extraction/Recovery Yield Results

Material	Batch ID	Amount Recovered (gm)	Content or Yield (%)	Mean (%)	St. Dev. (%)
Acetone-Extracted Guayule Rubber Resin (RR)	RR-1	226.6	11.5	11.9	0.30
	RR-2	247.0	12.2		
	RR-3	237.6	11.8		
	RR-4	248.2	12.0		
	RR-5	301.3	14.6	14.0	0.45
	RR-6	410.5	13.9		
	RR-7	312.5	14.0		
	RR-8	339.8	13.5		
Hexane-Extract from Waste Stream Guayule Leaves and Attached Stems (LF)	LF-1	215.9	4.5	4.5	0.09
	LF-2	210.4	4.4		
	LF-3	219.4	4.6		
	LF-4	211.2	4.4		
	LF-5	212.0	4.4		
RAP1 Binder (State Hwy Material: -3/4″)	RAP1-1	478.6	4.8	4.8	0.07
	RAP1-2	482.0	4.8		
	RAP1-3	492.3	4.9		
	RAP1-4	492.5	4.9		
	RAP1-5	512.1	4.8	4.7	0.14
	RAP1-6	488.6	4.7		
	RAP1-7	492.4	4.7		
	RAP1-8	462.1	4.5		
RAP2 Binder (Municipal Street Material: -3/4″)	RAP2-1	550.3	5.5	5.4	0.03
	RAP2-2	547.1	5.5		
	RAP2-3	543.3	5.4		
	RAP2-4	541.7	5.4		
	RAP2-5	558.8	5.5	5.5	0.01
	RAP2-6	569.1	5.5		
	RAP2-7	562.2	5.4		
	RAP2-8	559.1	5.5		
RAS Binder (-3/8″)	RAS-1	465.1	22.8	22.8	0.08
	RAS-2	468.7	22.9		
	RAS-3	459.8	22.7		
	RAS-4	459.8	22.8		

NOTE: The binder contents of the RAPs and RAS, and the yields of the LF material are based on the weight of the material after drying/warming overnight at 60°C. Yields of the RR material are based on the weight of the air-dry rubber subjected to acetone-extraction. Batches 1 – 4 (and LF batches 1 – 5) were extracted much earlier than batches 5 – 8.

Binder contents (or more correctly, percent losses) of the RAP/RAS were also determined using a standard solvent-extraction method, AASHTO T 164 (*23*), and the binder ignition oven based on AASHTO test method T 308 (*28*). Results are given in Table 3.

TABLE 3: T 164 and T 308 Binder Contents

Material	RAP1			RAP2			RAS		
Test/Property	T 164	T 308	Difference	T 164	T 308	Difference	T 164	T 308	Difference
Trial #1	4.92	7.19	2.27	5.24	6.14	0.90	23.20	26.45	3.25
Trial #2	5.06	6.99	1.93	5.08	6.33	1.25	22.01	26.53	4.52
Mean	4.99	7.09	2.10	5.16	6.24	1.08	22.61	26.49	3.89
Std. Dev.	0.099	0.141	0.240	0.113	0.134	0.247	0.841	0.057	0.898

The RAP1 and RAS binder content results in Table 2 relative to the T 164 results in Table 3 compare fairly well. However, the results for RAP2 in Table 2 (determined using the project-developed extraction/recovery methods) ran somewhat higher than those in Table 3.

The differences between T 308 and T 164 shown in Table 3 are large. The differences are primarily due to a combination of factors: aggregate mass change (a loss in most cases; probably the largest contributor to the differences), incomplete binder extraction in T 164 (aggregates absorb binder to differing degrees and the extent of removal of said binder using a solvent is variable), and in the case of the RAS, the burning of small amounts of paper, plastic, and wood included in the reclaimed shingles that show up as a mass loss in T 308. Note that the difference for RAP2 is considerably smaller than RAP1 and may be due to 1) the fact that RAP2 contains limestone and some trap rock (hard, low absorption aggregate used in many street resurfacing projects in Missouri), and/or 2) the RAP1 aggregate is dolomite, a common Missouri mineral that can cause excessive, and increasing mass loss with increasing temperature during ignition oven testing.

The temperature for binder ignition oven testing of the reclaimed materials should be the same as that used during binder ignition testing of the production FPM (*29*). However, because no FPM production had occurred prior to generating the T 308 data in Table 3, the binder ignition testing of the RAPs and RAS was performed at 538°C, the default temperature specified in T 308.

RAP/RAS Aggregates Gradations

Gradation analyses (*30, 31*) were performed on the recovered RAP and RAS aggregates from both the AASHTO T 308 and AASHTO T 164 procedures used to develop the results in Table 3. Gradation, or particle size distribution, is necessary for FPM design. The gradation results are given in Table 4.

TABLE 4: Gradation Results for Recovered RAP/RAS Aggregates

Material	RAP1		RAP2		RAS	
Aggregate Recovery Method	Solvent	Ignition	Solvent	Ignition	Solvent	Ignition
Sieve Size	% Pass	% Pass	% Pass	% Pass	% Pass	% Pass
¾ in. (19.0mm)	100	100	100	100	100	100
½ in. (12.5mm)	97	96	100	100	100	100
3/8 in. (9.5mm)	90	91	98	98	100	100
#4 (4.75mm)	69	71	75	74	99	99
#8 (2.36mm)	50	52	50	49	97	97
#16 (1.18mm)	40	42	36	36	81	81
#30 (600µm)	33	34	28	27	59	57
#50 (300µm)	25	27	19	18	51	50
#100 (150µm)	15	16	13	12	45	44
#200 (75µm)	9	10	11	9	37	35

The difference between the solvent- and ignition-recovered-aggregate gradations in Table 4 is small, but there is some evidence that the RAP1 dolomite breaks down more than the RAP2 and RAS aggregates during ignition testing. For FPM design, the gradations of the solvent-recovered RAP/RAS aggregates from the Table 2 extractions were assumed to be the same as the average of the solvent- and ignition-recovered-aggregate gradations in Table 4..Although not required for FPM design, the RAS fiber content was determined to be approximately 0.5% by weight of the RAS as a consequence of the washed sieve analysis of the recovered RAS aggregates.

Recycling Agent Classification

To properly perform FPM design that includes reclaimed binders, standard practice calls for classification of materials intended to be used as recycling agents. To this end, a standard material specification, AASHTO R 14 (*32*), is being referenced. The specification calls for several properties to be determined: viscosity, flash point, viscosity ratio (based on thin film oven (TFO) or rolling thin film oven (RTFO) residue viscosity relative to the original viscosity), weight (mass) change, specific gravity (relative density), and percent saturates. Table 5 summarizes all test results for recycling agent classification. Discussion of individual property testing follows Table 5.

TABLE 5: Recycling Agent (RA) Classification Results

Property or Parameter	LF	PG52-28	RR	CycL
RTFO viscosity @ 60°C (centipoise)	21450	160000	618	624
Original viscosity @ 60°C (centipoise)	36750	62125	218	438
Viscosity ratio (RTFO/original): maximum = 3	0.6	2.6	2.8	1.4
AASHTO R 14 RA Classification	RA 250↔500	NA (>RA 500)	RA 5	RA 5
Flash Point (°C): minimum = 218°C	~200	Not Determined*	213**	254***
Saturates (weight %): maximum = 25%	11.26***	Not Determined*	1.08***	16.38**
Weight (mass) change (%): maximum = ±4% for RA1 and RA5 RAs, ±3% for all others	-8.475	-0.066	-11.065	-1.817
Specific gravity (to be reported: no spec. limits)	1.003	1.012	1.015	1.004

NA = Not applicable; *Details in following discussions; **Average based on two replicate tests; ***Based on testing only one specimen

Percent Saturates (Clay-Gel Chromatography Testing)

According to AASHTO R 14, saturates are limited to a maximum of 25% (or 30% if all other specified criteria are met) and are to be determined using the clay-gel chromatography test method ASTM D 2007 (*19*). From this standardized test, one can determine the quantity of four different hydrocarbon types and structural groups in the recycling agent: saturates, aromatics, polar compounds (or polar aromatics), and asphaltenes (n-pentane insolubles). The relative proportions of these four groups affect the properties of the petroleum-based binder, and presumably, any blend of a guayule-based material and petroleum-based binder.

Clay-gel chromatography was performed on the RR, LF, and the Cyclogen L. Although the PG52-28 binder was used as a recycling agent in this study, clay-gel chromatography testing was not deemed necessary because the PG52-28 does not technically fall within the purview of R 14.

As shown in Table 5, all materials tested for percent saturates meet the criteria specified in AASHTO R 14 in that they are below 25%. Although not shown in Table 5, the LF and RR have a much higher polar compound content and considerably smaller aromatic compound content than the Cyclogen L. In a petroleum-based binder, age-hardening (simply stated) is a process by which the polar and aromatic compounds, over time, oxidize and become asphaltenes which are very large, complex molecules (i.e. microscopic solids) that serve as a bodying agent in the binder. Therefore, blends containing the LF or RR (both with higher polar compound contents than Cyclogen L) could possibly have an increased rate of age-hardening relative to blends with Cyclogen L. Saturates (also known as paraffins) serve as non-solvents or a gelling agent in a colloidal system in which the asphaltenes are dispersed or peptized by the polar compounds (also known as nitrogen bases), and the aromatics (also known as first and second acidaffins) serve as a solvent for the dispersed asphaltenes (*33, 34*).

Mass Change

Mass change testing of the LF, RR, Cyclogen L, and PG52-28 was conducted using the rolling thin film oven (RTFO) method AASHTO T 240 (*35*). The results are presented in Table 5.

The mass change (loss in this case) for the RR and LF materials exceeds the maximum allowable (4%) for any class of recycling agent (RA) based on AASHTO R 14 specifications. The PG52-28 and Cyclogen L met the specification. However, it should be noted that AASHTO R 14, as well as most every specification referenced for this research, pertains to petroleum-based binders, not necessarily a bio-based alternative. Mass change testing occurs at $163\pm1°C$. Many, if not most, of the chemical compounds in the guayule-based materials are terpenes (compounds with isoprene, C_5H_8, as the basic building block) and terpenoid alcohols. Gas chromatography – mass spectrometry test results (presented later) show some of the predominant compounds present in the RR and LF materials. Most of these compounds are volatile and, even though they may have high boiling points in their pure form (terpenes especially), they have low flash points.

Specific Gravity (Relative Density)

AASHTO T 228 (*36*) was followed in performing specific gravity tests on the PG52-28, Cyclogen L, RR, and LF materials. Specific gravity was determined at 25°C. The results are presented in Table 5.

Viscosity and Viscosity Ratio

A Brookfield rotational viscometer was used to determine viscosity-temperature relationships per AASHTO T 316 (*37*). Note that this is a deviation from specifications in AASHTO R 14 which calls for viscosity testing at 60°C using AASHTO T 201 or T 202 and then reporting the results in the "stoke" unit of viscosity. The stoke is equal to the poise (viscosity units determined using T 316) divided by the density (specific gravity) of the liquid. However, because the density or specific gravity of the petroleum-based binders and guayule-based materials are very nearly one (1), stoke and

poise values are very nearly the same. Therefore, because R 14 classifications are based on a range of viscosity, and because the calculated viscosity ratio is not dependent on the actual viscosity unit used, viscosity results shown in Table 5 are given in centipoise. The results in Table 5 show that all four materials meet the viscosity ratio specification in R 14.

The RA classifications in Table 5 line up fairly well between the materials that were compared. Both the RR and the Cyclogen L are classified as RA 5, but the LF and PG52-28 classifications are somewhat different. It is important to remember, however, that the classifications are based on viscosity at only one temperature. Viscosity measurements at more temperatures for each material will be presented later in the report, and the reason will become clearer as to why PG52-28 was chosen as the comparative petroleum-based binder to the LF material.

Flash Point

Flash point testing is required as a safety issue and was performed per AASHTO T 48 (*38*). As shown in Table 5, the LF and RR materials did not meet the flash point specification in AASHTO R 14, but they did not fail by a large margin. In fact, the margin of failure of the LF (~18°C) was essentially the same as the multi-laboratory precision d2s (i.e. reproducibility) value of 18°C, and the margin of failure of the RR (5°C) was less than the single operator precision 1s (i.e. repeatability) value of 8°C. As discussed in earlier reports in regard to the large mass loss (volatilization) experienced by the guayule-based materials during heating at typical FPM mixing temperatures, it may be necessary to pre-condition or modify the processing of the guayule-based materials to make them more thermally stable and reduce the mass loss. This action would also, presumably, increase the flash point. Testing of the PG52-28 binder was not performed because it was assumed that the flash point would be higher than that of the Cyclogen L which has a flash point of 254°C, well above the specified minimum of 218°C.

Gas Chromatography – Mass Spectrometry

Working with colleagues in the Environmental Engineering section of the Department of Civil, Architectural, and Environmental Engineering at the Missouri University of Science & Technology, testing was performed to evaluate the efficiency of the hexane and acetone de-solventization process, and to identify major volatiles in the RR and LF materials. The identification method used is called "headspace sampling using solid phase micro-extraction (SPME) with gas chromatography mass spectrometry (GC-MS)" (*39*). The media that was used to sample the guayule-based materials was a polydimethylsiloxane (PDMS) SPME fiber. The testing was done on an operator-availability basis and not all variables (e.g. specimen size) were carefully controlled. The results are shown in Figure 2.

Most of the compounds identified in Figure 2 are terpenes, terpenoid alcohols, or essential oils. The mass spectrometry software indicated uncertainty when identifying the peak labeled as unknown, and generated a "best guess" which is the compound in parenthesis. The acetone and hexane levels (labeled low and to the left in the appropriate chromatogram) indicate that the de-solvenitzation procedures were very good at removing the solvents.
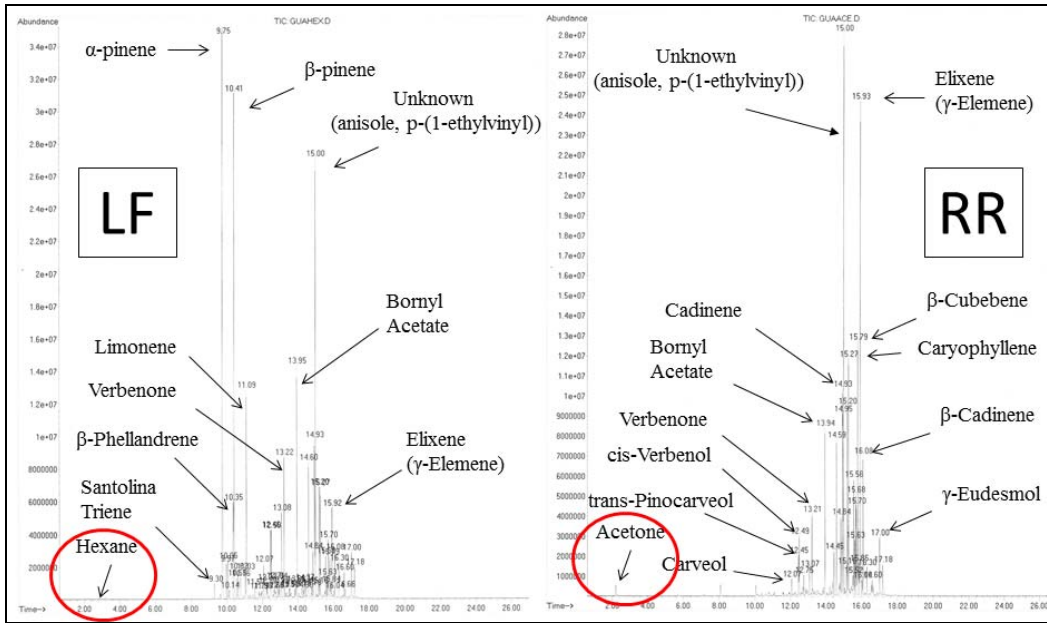
FIGURE 2: Chromatograms of LF (left) and RR (right) Materials

Viscosity Testing

AASHTO T 316 (*37*) was used for the viscosity testing presented in this section. Figure 3 shows the viscosity results for the original (not-RTFO-aged) RAP1, RAP2, RAS, PG52-28, and PG46-28 binders. The PG46-28 was included because at the time these tests were performed, it was not clear which petroleum-based binder(s) would be utilized in a comparative analyses.
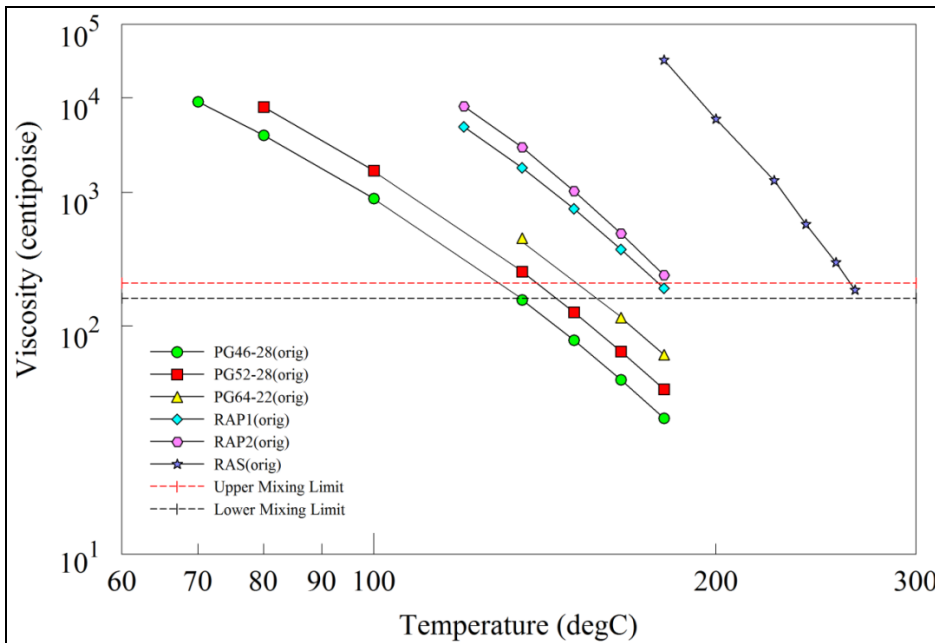
FIGURE 3: RAP, RAS, and PG Binder Viscosities

The choice of comparing the RR to the CycL and the LF to the PG52-28 was based on their relative viscosity-temperature relationships. It seemed to be the only parameter that could logically be used as a basis of comparison because the reduction of viscosity of the hardened binder in the RAP/RAS is a primary reason for utilizing a recycling agent. The viscosity results for these comparisons are given in Figure 4.
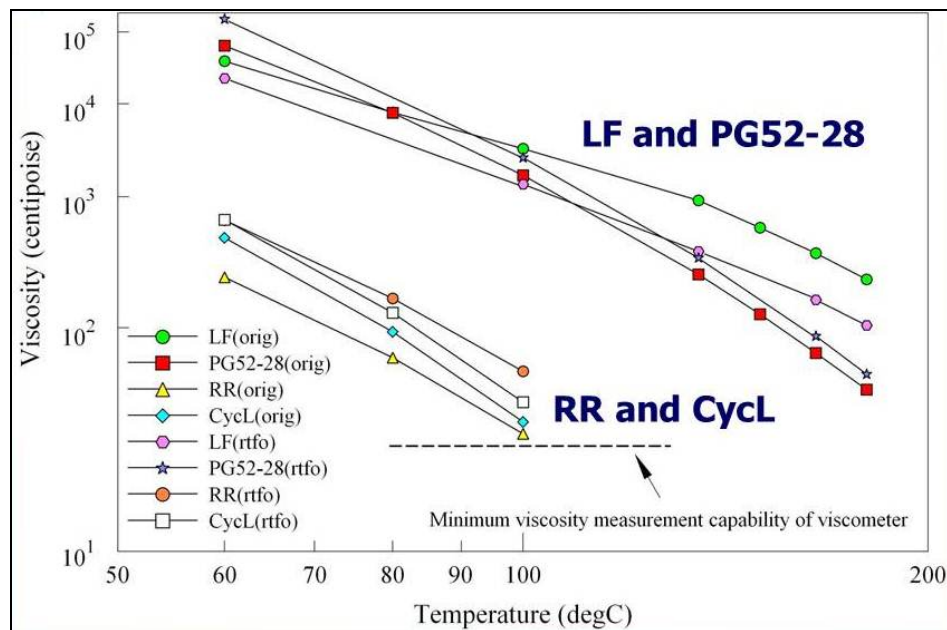
FIGURE 4: Comparison of Viscosity-Temperature Relationships

Figure 3 shows, as expected, the RAS(orig) binder is much more viscous than any of the other materials. As a pure blend (i.e. a non-blended material), the mixing temperature is ~265°C (~510°F) meaning its presence in a blend in any proportion will raise the mixing temperature of that blend, relative to what it would be without it. The Asphalt Institute has for a long time published literature in which they present recommended mixing and compaction viscosities for FPMs. The recommended binder viscosity for FPM mixing is 0.17 ± 0.02 Pa·sec or 170 ± 20 centipoise (*40*).

There are two significant features about the curves presented in Figure 4. One is the difference in the slopes for the LF and PG52-28 materials, both original and RTFO-aged. The slope of a viscosity-temperature curve shows a material's temperature susceptibility which is the change in viscosity for a given change in temperature. Low temperature susceptibility is desirable in a binder material and the LF material is superior to PG52-28 in this respect across the entire temperature range in which they were tested. Also, the relative location of the LF and PG52-28 curves shows why these two materials were chosen to be compared; they are fairly similar over a wide range of temperatures. The original and RTFO-aged RR and CycL are also very similar in terms of viscosity and the RR shows a slight advantage in temperature susceptibility over the CycL.

The second significant feature in Figure 4 is the viscosity reduction of the LF upon RTFO-aging. This behavior runs counter to that of petroleum-based binders. Discussion of possible reasons for this phenomenon is beyond the scope of this report. The RR, on the other hand, behaves typically in that the viscosity increases upon RTFO-aging. Also note the relative distance between the original and RTFO curves for all four materials. The guayule-based materials have a wider

separation than the petroleum-based binders which one would expect based upon the radical differences between the guayule-based materials and petroleum-based binders in terms of mass change upon RTFO-aging.

Binder-Blending Mixture Experiment

In order to determine the relative effects of the guayule-based materials vs. the petroleum-based binders when blended with RAP/RAS binders, and to develop response surface models (RSMs) required for FPM design, binder-blending mixture experiments were developed. The experimental design matrices for the comparison of the LF and PG52-28 binders are given in Tables 6 and 7, and the matrices for the comparison of the RR and Cyclogen L (CycL) binders are given in Tables 8 and 9. The "design" blend proportions were generated using the Design-Expert® software program based on constraints imposed on the proportion limits of each individual component, and were based on the literature and practical considerations. For the LF vs. PG52-28 and the RR vs. CycL binder-blending mixture experiments, the RAP1 and RAP2 design proportion limits were chosen to be 0 – 100%. For the LF vs. PG52-28 blends, the RAS design limits were chosen to be 0 – 50% and the VB design limits chosen to be 0 – 70%. For the RR vs. CycL blends, the RAS design limits were chosen to be 0 – 15% and the VB design limits chosen to be 0 – 30%. Each blend (row) was physically created using a modified Brookfield rotational viscometer as a mixing device. Each blend was then tested to produce the appropriate response data. All response data is the average of at least two repeated measurements; i.e. tests performed on specimens sampled from the same batch. Explanation of the column headings, etc. follows the tables.

TABLE 6: Binder-Blending Mixture Experiment Results: LF as Virgin Binder (VB)

| Data | Blended-Binder Components/Proportions | | | | Responses | |
Type	RAP1	RAP2	RAS	LF	TcH (°C)	TcL (°C)
No -VB (shared) Data	1.000	0.000	0.000	0.000	86.9	-8.3
	1.000	0.000	0.000	0.000	87.0	-8.1
	0.000	1.000	0.000	0.000	95.4	-0.9
	0.000	1.000	0.000	0.000	95.2	-1.0
	0.000	0.500	0.500	0.000	127.7	10.5
	0.000	0.500	0.500	0.000	127.3	10.6
	0.500	0.500	0.000	0.000	91.7	-4.4
	0.500	0.500	0.000	0.000	91.9	-4.2
	0.500	0.000	0.500	0.000	122.4	6.5
	0.750	0.000	0.250	0.000	102.1	-1.9
VB-Specific Data	0.000	0.300	0.000	0.700	55.5	-14.5
	0.150	0.000	0.150	0.700	54.1	-16.5
	0.650	0.000	0.000	0.350	71.5	-9.0
	0.000	0.000	0.500	0.500	90.3	-4.8
	0.000	0.360	0.260	0.380	93.7	-1.6
	0.325	0.325	0.000	0.350	74.2	-6.2
	0.167	0.167	0.500	0.167	121.4	4.8
	0.000	0.650	0.000	0.350	78.4	-6.3
	0.360	0.000	0.260	0.380	88.5	-1.8

Table column heading definitions are as follows:

<u>Mixture components</u>

RAP1 = RAP from I-44 project

RAP2 = RAP from a Washington, Missouri street project

RAS = Processed tear-off roofing shingles

LF, PG52-28, RR, and CycL = VB = Virgin binder; either a guayule-based material or petroleum-based binder.

TABLE 7: Binder-Blending Mixture Experiment Results: PG52-28 as Virgin Binder (VB)

Data	Blended-Binder Components/Proportions				Responses	
Type	RAP1	RAP2	RAS	PG52-28	TcH (°C)	TcL (°C)
No -VB (shared) Data	1.000	0.000	0.000	0.000	86.9	-8.3
	1.000	0.000	0.000	0.000	87.0	-8.1
	0.000	1.000	0.000	0.000	95.4	-0.9
	0.000	1.000	0.000	0.000	95.2	-1.0
	0.000	0.500	0.500	0.000	127.7	10.5
	0.000	0.500	0.500	0.000	127.3	10.6
	0.500	0.500	0.000	0.000	91.7	-4.4
	0.500	0.500	0.000	0.000	91.9	-4.2
	0.500	0.000	0.500	0.000	122.4	6.5
	0.750	0.000	0.250	0.000	102.1	-1.9
VB-Specific Data	0.000	0.300	0.000	0.700	69.1	-19.3
	0.150	0.000	0.150	0.700	73.9	-18.3
	0.650	0.000	0.000	0.350	76.5	-14.6
	0.000	0.000	0.500	0.500	103.3	-4.7
	0.000	0.360	0.260	0.380	94.6	-5.3
	0.325	0.325	0.000	0.350	79.8	-12.4
	0.167	0.167	0.500	0.167	117.3	4.3
	0.000	0.650	0.000	0.350	83.4	-9.6
	0.360	0.000	0.260	0.380	91.0	-9.2

Responses determined through Dynamic Shear Rheometer (DSR) testing (*41, 42, 43*)

T_cH = High critical temperature: An indicator of FPM rutting potential and the lowest (coolest) of the two temperatures determined under the following conditions:

1. The temperature at which the ratio of the complex modulus (G*) to the sine of the phase angle (sinδ) equals 1.00 kPa, as determined during DSR testing at 10 radians/second on original binder.

2. The temperature at which the ratio of the complex modulus (G*) to the sine of the phase angle (sinδ) equals 2.20 kPa, as determined during DSR testing at 10 radians/second on RTFO-aged binder.

Responses determined through Bending Beam Rheometer (BBR) testing (*44, 42, 43*)

T_cL = Low critical temperature: An indicator of FPM cold-temperature (thermal) cracking potential and the highest (warmest) of the two temperatures determined under the following conditions:

1. The temperature at which the creep stiffness (S) equals 300 MPa at 60 seconds of loading during BBR testing of pressure aging vessel(PAV)-aged binder.

2. The temperature at which the m-value (the absolute value of the slope of the logarithm of the stiffness curves versus the logarithm of the time) equals 0.300 at 60 seconds of loading during BBR testing of PAV-aged binder.

The mixture experiment is a special type of methodology for generating response surface models (RSMs) for design. What sets the mixture experiment apart from other RSM designs is the fact that the level of each component (factor) is dependent on the levels of the other components; i.e. the percentages of each component must add up to 100%. To generate the RSMs, ten (10) experimental runs (i.e. data points or blends) were required in order to fit the desired model to the data (in this case, a Scheffe quadratic model), at least three (3) additional unique blends were created to check for lack of fit, and at least four (4) replicates tests (duplicates of some of the other blends) were generated to calculate pure

error. By definition, a "replicate test" is one in which all process variability is included in the replicate blend to be tested. Listed below each component are the proportions (by weight) of each component per blend or run, and the colored blends indicate replicate tests where each pair of replicate tests is the same color.

TABLE 8: Binder-Blending Mixture Experiment Results: RR as Virgin Binder (VB)

Data	Blended-Binder Components/Proportions				Responses	
Type	RAP1	RAP2	RAS	RR	TcH (°C)	TcL (°C)
No -VB (shared) Data	1.000	0.000	0.000	0.000	87.7	-6.2
	1.000	0.000	0.000	0.000	87.5	-6.3
	0.000	1.000	0.000	0.000	93.9	-1.0
	0.000	1.000	0.000	0.000	94.5	-1.3
	0.210	0.718	0.072	0.000	97.1	-1.1
	0.210	0.718	0.072	0.000	97.0	-1.5
	0.648	0.202	0.150	0.000	97.8	-2.6
	0.832	0.162	0.006	0.000	90.1	-5.4
VB-Specific Data	0.000	0.768	0.150	0.082	91.0	-5.8
	0.425	0.454	0.003	0.119	77.7	-13.0
	0.425	0.454	0.003	0.119	77.5	-14.4
	0.215	0.513	0.150	0.122	83.7	-10.4
	0.740	0.000	0.092	0.168	72.4	-17.0
	0.740	0.000	0.092	0.168	72.6	-17.4
	0.000	0.685	0.043	0.272	62.4	-22.5
	0.631	0.069	0.000	0.300	56.3	-22.5
	0.401	0.149	0.150	0.300	62.0	-22.4
	0.204	0.346	0.150	0.300	62.2	-22.6

TABLE 9: Binder-Blending Mixture Experiment Results: CycL as Virgin Binder (VB)

Data	Blended-Binder Components/Proportions				Responses	
Type	RAP1	RAP2	RAS	CycL	TcH (°C)	TcL (°C)
No -VB (shared) Data	1.000	0.000	0.000	0.000	87.7	-6.2
	1.000	0.000	0.000	0.000	87.5	-6.3
	0.000	1.000	0.000	0.000	93.9	-1.0
	0.000	1.000	0.000	0.000	94.5	-1.3
	0.210	0.718	0.072	0.000	97.1	-1.1
	0.210	0.718	0.072	0.000	97.0	-1.5
	0.648	0.202	0.150	0.000	97.8	-2.6
	0.832	0.162	0.006	0.000	90.1	-5.4
VB-Specific Data	0.000	0.768	0.150	0.082	91.3	-6.4
	0.425	0.454	0.003	0.119	76.1	-14.4
	0.425	0.454	0.003	0.119	76.3	-15.2
	0.215	0.513	0.150	0.122	84.0	-11.7
	0.740	0.000	0.092	0.168	72.0	-19.1
	0.740	0.000	0.092	0.168	72.2	-19.6
	0.000	0.685	0.043	0.272	62.7	-23.5
	0.631	0.069	0.000	0.300	54.9	-23.7
	0.401	0.149	0.150	0.300	61.0	-23.8
	0.204	0.346	0.150	0.300	62.1	-23.9

For each of the four (4) tables above, two regression analyses were performed: one to generate a T_cH RSM and another to generate a T_cL RSM. The component (RAP1, etc.) percentages (in decimal form) were the independent variables, and the responses (T_cH and T_cL) were the dependent variables. Please note that for both comparisons (LF vs. PG52-28, and RR vs. CycL), the blends that do not include virgin binder (i.e. No-VB or reclaimed binders only) were only created and tested once; i.e. that data was "shared" between the guayule-based VB and petroleum-based VB analyses.

Once the RSMs are established, DesignExpert can be used to "optimize" the component proportions to meet a desired T_cH and/or T_cL value or range of values. If possible, many different optimization solutions can be generated by DesignExpert imparting more flexibility in the FPM design process.

The term "original binder," as it is used above, means that the reclaimed RAP/RAS binder or blend, or a RAP/RAS/VB blend has not been subjected to any aging (i.e. RTFO or PAV) in the laboratory. In reality, however, the reclaimed binders have already experienced significant aging as a result of their in-service history. With this in mind, it is important to point out that none of the blends shown in the above tables were PAV-aged, although this is required for standard BBR testing, as described above. The reason for this is because approximately half of the blends per table did not include a virgin binder, and the AASHTO 323 Appendix (*43*) specifies that when characterizing reclaimed binders, RTFO-aging is sufficient and is used in-lieu of PAV-aging. Thus to keep from injecting another variable into the mixture experimental designs, all blends, those that had a virgin binder and those that did not (i.e. No-VB), were RTFO-aged only when aging was required for testing. Therefore, the T_cL RSMs generated are based on RTFO-aged binder which means that estimated T_cL values using the RSMs would be lower (cooler) than actual T_cL values based on PAV-aged binder.

Finally, any blend ultimately chosen to be used in FPM design should first be verified as to whether or not it meets the required specifications using the appropriate test procedures, which would include testing of the PAV-aged blend. The following response descriptions discuss verification testing (in addition to BBR testing of PAV-aged binder) to be performed on an optimized blend that has been chosen for FPM design.

Response determined through Dynamic Shear Rheometer (DSR) testing (*41, 42, 43*)

T_cInt = Intermediate critical temperature: An indicator of FPM fatigue cracking potential and the temperature at which the product of G^* and $\sin\delta$ equals 5000 kPa as determined during DSR testing at 10 radians/second on PAV-aged binder.

Response determined through Brookfield Rotational Viscometer testing (*37, 45*)

Maximum viscosity at 135°C = 3 Pa·sec = 3000 centipoise

Response determined through Flash Point testing (*38, 45*)

Minimum flash point = 230°C

Response determined through Mass Change testing (*34, 45*)

Maximum mass change = ±1.00%

Responses determined on RTFO-aged binder using the Multiple Stress Creep Recovery (MSCR) test which utilizes the DSR: All averages based on 10 creep – recovery cycles (*46, 47*)

$J_{nr(3.2)}$ [$J_{nr(0.1)}$] = Average non-recoverable creep compliance at 3.2 kPa of creep stress [and 0.1 kPa, respectively]

$J_{nr(diff)}$ = Percent difference between $J_{nr(0.1)}$ and $J_{nr(3.2)}$

$R_{(3.2)}$ [$R_{(0.1)}$] = Average percent recovery at 3.2 kPa of creep stress [and 0.1 kPa, respectively]

$R_{(diff)}$ = Percent difference between $R_{(0.1)}$ and $R_{(3.2)}$

The high, intermediate, and low critical temperatures (T_c) are the primary bases for the grading of a PG binder under specifications set forth in AASHTO M 320 (*45*), which is still the predominant petroleum-based binder specification utilized by most state DOTs.

The MSCR test responses, $J_{nr(3.2)}$, $J_{nr(diff)}$, $R_{(3.2)}$, and $R_{(diff)}$ are determined through DSR testing as described in the AASHTO provisional test method TP 70 (*46*). These responses are indicators of rutting potential, stress sensitivity, the presence of a polymer and, if present, the quality of blending. The MSCR test is specified to be performed on RTFO-aged

binder and the test temperature depends on the environmental high pavement temperature and is usually selected by the specifying agency. For example, MoDOT has implemented the AASHTO specification, MP 19 (*47*), and specifies that the MSCR test be performed at 64°C on FPM binders used on Missouri state highways. In MP 19, traffic loading is the only other criteria for grading a binder when using the MSCR test. Traffic loading designations are "S" for standard, "H" for heavy, "V" for very heavy, and "E" for extremely heavy traffic. This traffic loading designation system replaces the "grade bumping" practice traditionally performed when following the PG binder specification, AASHTO M 320 (*46*). Under MP 19, $J_{nr(3.2)}$ and $J_{nr(diff)}$ are the only criteria required for making pass/fail determinations. For all temperatures and traffic loading designations, $J_{nr(diff)}$ cannot exceed 75%. The maximum $J_{nr(3.2)}$ value, on the other hand, is a function of traffic but not temperature. For example, $J_{nr(3.2)}$ cannot exceed 4.0 kPa^{-1} for the standard traffic "S" grade but the maximum $J_{nr(3.2)}$ is lowered to 1.0 kPa^{-1} for the very heavy traffic "V" grade. $R_{(3.2)}$ and $R_{(diff)}$ are obtained through MSCR testing and are used to determine the elastic response and stress dependence of polymer modified and neat binders, but they are not required as part of MP 19.

Results of the LF vs. PG52-28 and RR vs. Cyclogen L Comparisons

The VB-specific data in Tables 6 – 9 (i.e. those blends that include a virgin binder) was used to develop Figures 5 – 10. These plots show the correlations between the T_cH and T_cL, and the absolute temperature spread ($T_cH - T_cL$) of the materials being compared. Figures 5 – 7 shows the LF vs. PG52-28 direct comparisons.
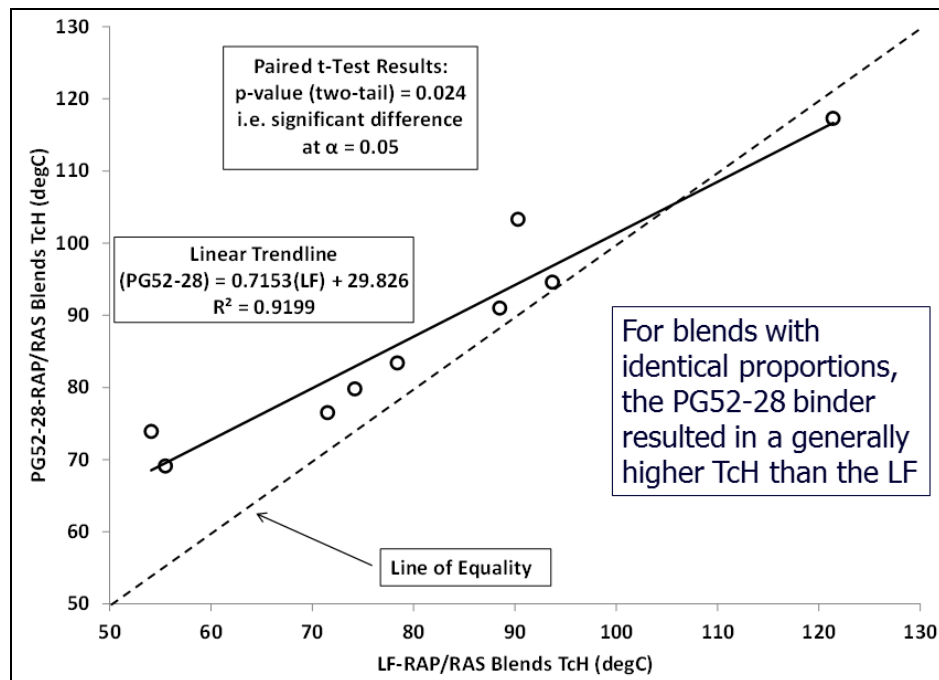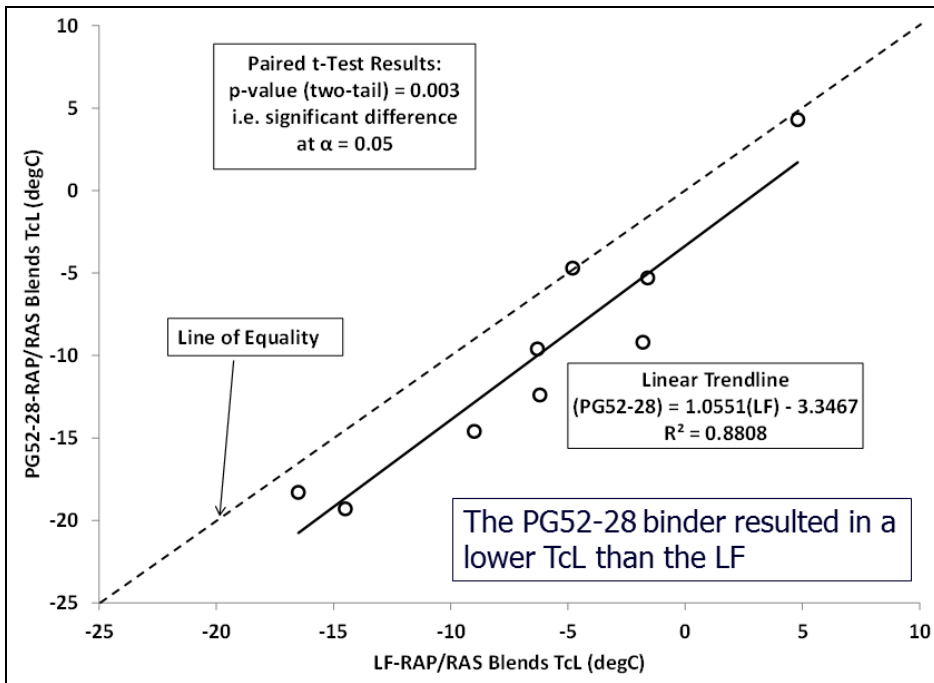

FIGURE 5: T_cH Correlation (LF vs. PG52-28)

FIGURE 6: T_cL Correlation (LF vs. PG52-28)
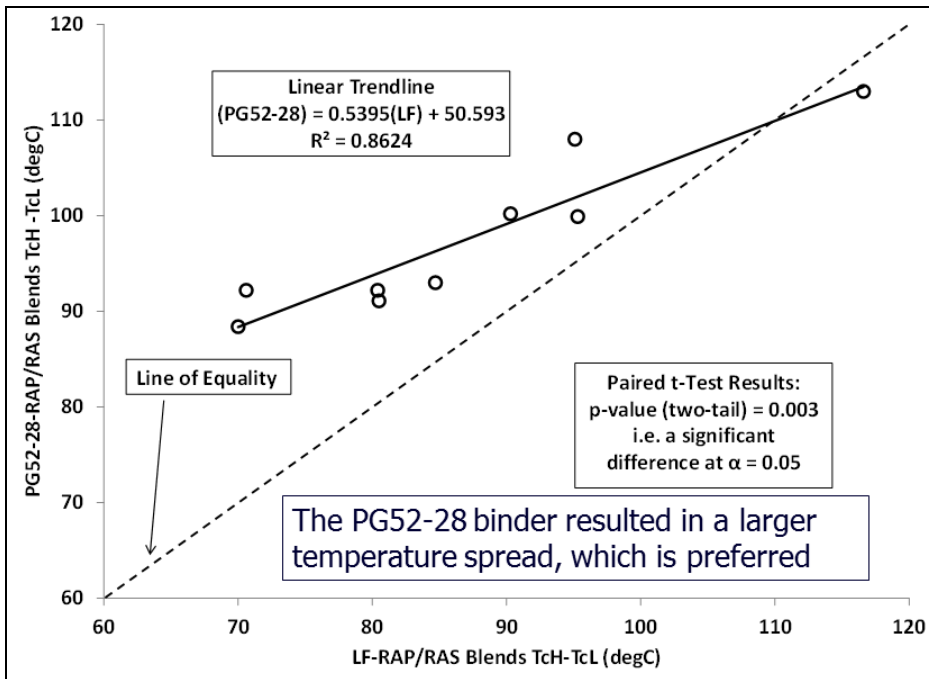
FIGURE 7: $T_cH – T_cL$ Correlation (LF vs. PG52-28)

As indicated in Figures 5 – 7, the PG52-28-RAP/RAS blends generally had higher (warmer) T_cH values, lower (cooler) T_cL values, and therefore, generally larger absolute temperature spreads than the proportionally-identical LF-RAP/RAS blends; i.e. a wider range means better overall performance. Although these results were discouraging, it should be understood that even though the LF did not favorably compare in a direct manner to the PG52-28 binder, the LF material

could still prove to be useful in a different type of FPM application. Nonetheless, these results focused the remaining effort in the project on the RR.   Figures 8 – 10 show the same correlations for the RR vs. CycL comparison.
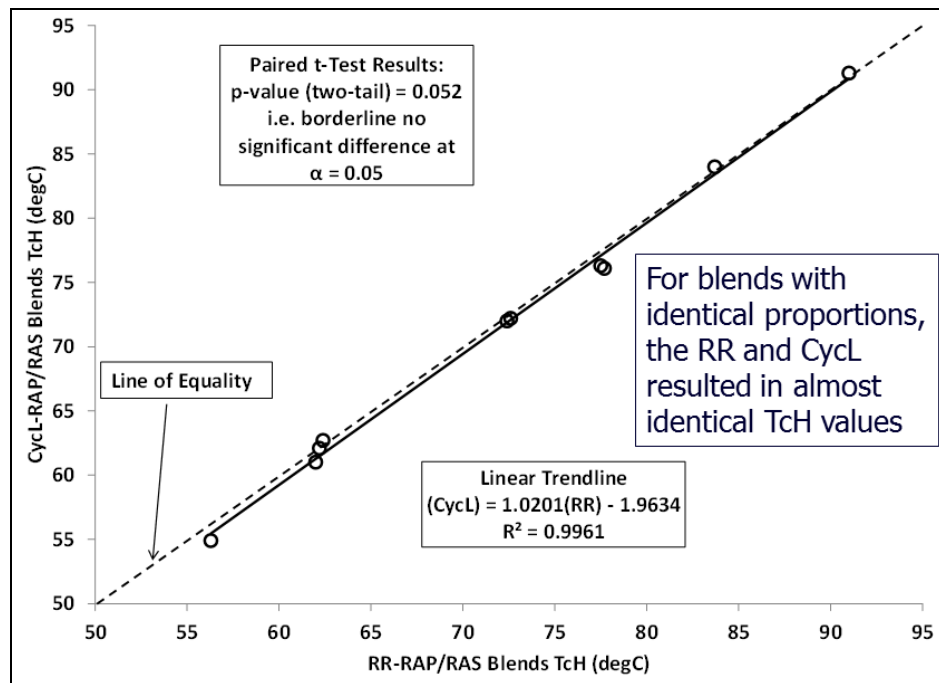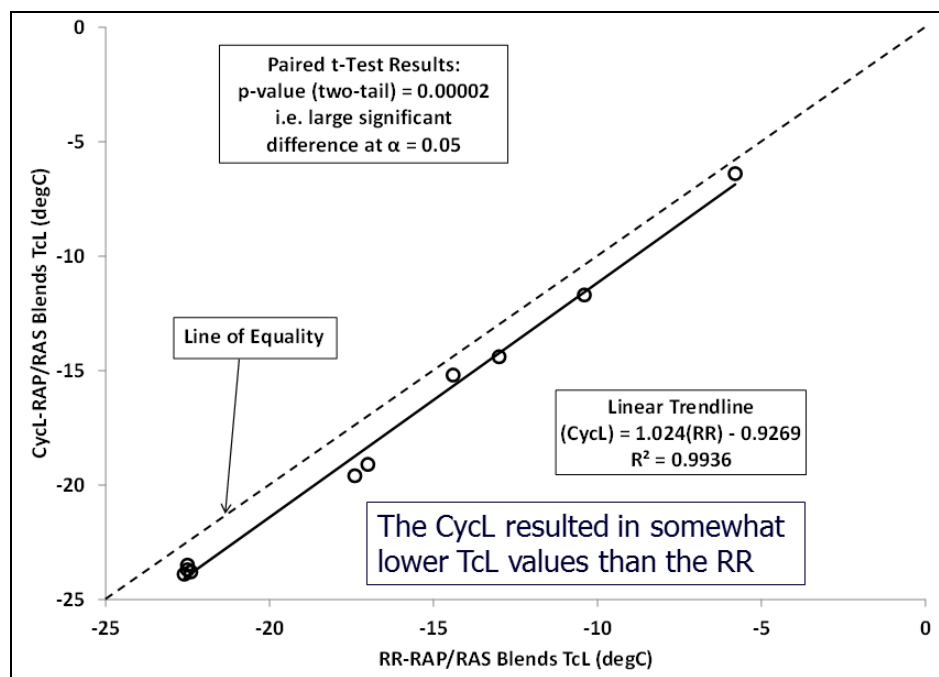

FIGURE 8: T_cH Correlation (RR vs. CycL)

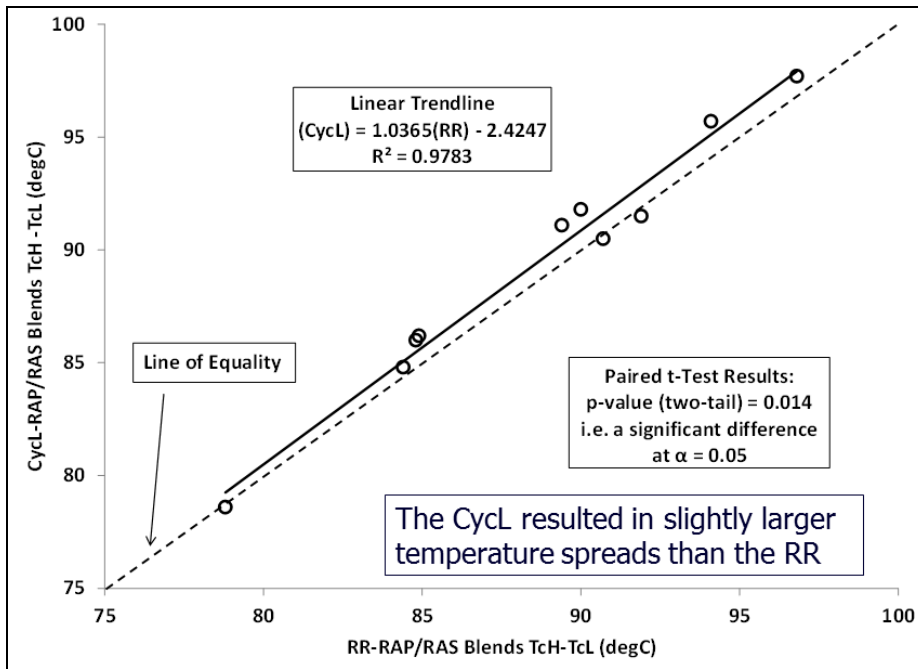**FIGURE 9: T_cL Correlation (RR vs. CycL)**

FIGURE 10: T$_c$H – T$_c$L Correlation (RR vs. CycL)

The plots in Figures 8 – 10 clearly show that the RR vs. CycL direct comparisons are much more favorable than the LF vs. PG52-28. The T$_c$H correlation is almost perfect but slightly favors the RR. The T$_c$L correlation definitely favors the CycL but the bias is only a degree or two on average. Most importantly, the absolute temperature spread correlation is significantly different at an alpha value of 0.05, but, again is only slightly biased toward the CycL, and not for every blend. These results demonstrated that the RR could be used in a practical sense as a recycling agent, and prompted the move to start FPM design, production, and testing.

Response Surface Models (RSMs): Generation and Optimization

Generation of the response surface models (RSMs) was performed using regression analysis procedures within DesignExpert using the data in Tables 6 – 9. To reiterate, two (2) RSMs were generated per table: one for T$_c$H and one for T$_c$L. As the focus was now only on the RR, only the RR RSMs were necessary to utilize and are given in Equations (Eqs.) 1 and 2 below, where the independent variables (RAP1, etc.) are the decimal fractions of the proportions in a given blend.

$$
\begin{aligned}
TcH = {} & 87.621(RAP1) + 94.333(RAP2) + 143.65(RAS) + 13.118(RR) + 4.678(RAP1 \times RAP2) \\
& - 45.586(RAP1 \times RR) - 55.897(RAP2 \times RR) - 118.04(RAS \times RR)
\end{aligned}
\tag{1}
$$

$$
\begin{aligned}
TcL = {} & -6.3091(RAP1) - 1.4086(RAP2) + 27.029(RAS) - 1.9789(RR) - 25.935(RAP1 \times RAS) \\
& - 83.281(RAP1 \times RR) - 108.97(RAP2 \times RR) - 152.18(RAS \times RR)
\end{aligned}
\tag{2}
$$

24

Before beginning the FPM design procedure, a particular blend had to be developed that would result in a desired grade of binder. To keep things as simple as possible, the DesignExpert optimization process was utilized to determine sets of component proportions (or solutions) that would generate a blend meeting the specifications for a PG64-22 binder. The solutions were based on the RR T_cH and T_cL RSMs shown in Eqs. 1 and 2.

If one should choose to use Eqs. 1 and 2 directly, the component proportions (independent variable values) should be in the form of decimal percentages, and the sum of those percentages must be 1.00 (i.e. 100%). For example, if one wanted to estimate T_cH for a blend that had 70% RAP1, 0% RAP2, 5% RAS, and 25% RR (all percentages sum to 100%), one would input 0.70 everywhere RAP1 appears in Eq. 1, 0.00 everywhere RAP2 appears in Eq. 1, 0.05 everywhere RAS appears in Eq. 1, and 0.25 everywhere RR appears in Eq. 1. The same procedure would be used for estimating T_cL, Eq. 2. Of course, it is important to remember that the regression coefficients on the main effects terms and the interaction terms are specific to the materials used in this project.

Figure 11 shows the DesignExpert optimization output for producing a blend estimated to meet specifications for a PG64-22 binder.

The "optimization" constraints on the components (i.e. the RAPs, RAS, and RR proportion limits) shown in Figure 11 were selected to be the same as those used in the design proportion generation at the very beginning of the binder-blending mixture experiments (discussed at the beginning of the Binder-Blending Mixture Experiments section). This selection utilizes the entire data space created during the testing of the various design blends. The "response" constraints shown in Figure 11 were chosen such that estimated T_cH would be greater than or equal to 67.5°C and the T_cL would be less than or equal to -20.2°C. The "weight" and "importance" of each constraint setting could also be adjusted, but were left at their default values for this project.

Constraints						
Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:RAP1	is in range	0	1	1	1	3
B:RAP2	is in range	0	1	1	1	3
C:RAS	is in range	0	0.15	1	1	3
D:RR	is in range	0	0.3	1	1	3
TcH	is in range	67.5		1	1	3
TcL	is in range		-20.2	1	1	3

Solutions							
Number	RAP1	RAP2	RAS	RR	TcH	TcL	Desirability
1	*0.424*	*0.178*	*0.150*	*0.248*	*67.5*	*-20.2*	*1.000* *Selected*
2	0.518	0.090	0.150	0.243	67.5	-20.2	1.000
3	0.463	0.143	0.148	0.245	67.5	-20.2	1.000
4	0.487	0.119	0.150	0.244	67.5	-20.2	1.000

FIGURE 11: Optimization Solutions to Satisfy PG64-22 Binder T_cH and T_cL Specifications

At this point, it is necessary to understand that there is a 10°C offset on the T_cL value when actually identifying a PG binder. During development of the standard BBR test procedure (AASHTO T 313), a time/temperature superposition phenomenon was observed that was adopted in the test procedure. Researchers noticed that if the test temperature was

increased 10°C (thereby decreasing the stiffness) and the loading time was decreased from two hours to 60 seconds, they obtained similar deflection results (*48*). So, in order for a binder to meet the specification for, say, a PG64-22 binder, the T_cH must be greater than or equal to 64°C (thus the estimated T_cH response constraint of 67.5°C, which allows for a 3.5°C margin of estimation error), and the T_cL must be less than or equal to -12°C. Remember that the T_cL is, by specification, to be determined on PAV-aged binder. Therefore, selection of -20.2°C as the upper threshold T_cL temperature for optimization purposes was an educated guess knowing that the T_cL RSMs were based on RTFO-aged blends, which are expected to be not as stiff, generally speaking, as PAV-aged blends.

Solution #1 in Figure 11 is only one of four solutions found in the optimization process that has the highest possible desirability rating of 1.000, and it is highlighted because it was selected for beginning FPM design. Based on discussion in the previous paragraph, the estimated "true" grade of the Solution #1 blend should be a PG67.5-30.2, again remembering that the lower number is based on testing RTFO-aged blends.

The next step was to physically build the Solution #1 blend, to be referred to as the RR5 blend throughout the remainder of this report, and verify that it did indeed meet the specifications for a PG64-22 binder. Binder grade verification testing, as described in the Binder-Blending Mixture Experiments section, was performed on the RR5 blend and its actual true grade was determined to be a PG65.5-24.3 based on T_cH, T_cL, and T_cInt testing required in M 320 (*45*). MSCR testing was performed per TP-70 (*46*) and the RR5 blend met the PG64-22 Standard Grade specifications set forth in MP-19 (*47*). The rotational viscosity of the original (non-aged) RR5 blend at 135°C was 390 centipoise or 0.39 Pa·sec, well below the maximum limit of 3 Pa·sec. But the RR5 blend did not meet the mass change specification. Mass change was -3.1% and the maximum allowable change is ±1.0%. Whether or not failing to meet the mass change specification is a significant issue is not clear at this time. Flash point is yet to be determined but it is assumed that it will easily meet the minimum of 230°C based on the high RAP/RAS content and the fact that the RR pure blend (not-blended with other materials) flash point was 213°C. Now knowing that the RR5 blend met almost all PG64-22 specifications, FPM design was initiated.

Flexible Pavement Mixture (FPM) Design, Production, and Testing (Task 3)

FPM Design

Using the verified RR5 blend discussed in the previous section, a spreadsheet was developed to control the blended-binder grade and the aggregate gradation of the FPM. Figure 12 shows a major portion of the spreadsheet. The goals of the FPM design process were to achieve a blend of reclaimed and virgin materials (aggregates and binder, which includes the RR) that will 1) achieve the desired binder grade (e.g. PG64-22), 2) meet gradation specifications, and 3) meet FPM volumetric criteria (% air voids, % voids in the mineral aggregate or VMA, and % voids filled with asphalt or VFA).

Aggregate Percentages

	RAP1	RAP2	RAS	1/2" Clean	3/8" Clean	MS	SUM chk	% Reclaim. Aggregate
	36.89%	13.43%	2.80%	0.00%	46.88%	0.00%	100.00%	53.12%
Pbr	4.76%	5.45%	22.79%					

Aggregate Batch Weights (grams)

		RAP1	RAP2	RAS	1/2" Clean	3/8" Clean	MS	SUM
Agg Wt. (g)	12200	RAP1	RAP2	RAS	1/2" Clean	3/8" Clean	MS	SUM
(2 gyro, 1 Rice)		4500.6	1638.5	341.6	0.0	5719.4	0.0	12200.0
Reclaimed Binder Weights (g)		224.9	94.4	100.8				
Total Reclaimed binder (g)		420.2						

RAP/RAS-Aggregate-Added Binder Batch Weights (grams) — **Added Binder (g)**

Mix ID		RAP1	RAP2	RAS	1/2" Clean	3/8" Clean	MS	SUM	RR	PG64-22
RR5M T25-RR(6")(2)		4725.5	1732.9	442.4	0.0	5719.4	0.0	12620.2	125.0	0.0

	RAP1		RAP2	
	R1/4"	P1/4"	R1/4"	P1/4"
%/fraction	39.8	60.2	34.2	65.8
Batch wt.	1881.3	2844.2	593.4	1139.5

- - - - - - - - - - - - - - -

Target %s of Blended Binder:	RAP1	RAP2	RAS	RR	NOTE: Based on only RTFO-aging, no PAV-aging.
Design Expert Results-->	42.4%	17.8%	15.0%	24.8%	Estimated True Grade = PG67.5-30.2
	56.38%	23.67%	19.95%		Actual True Grade = PG65.5-24.3
					This is based on PAV-aging

Blue highlighted proportions (above & below) should be about equal per reclaimed material.

% Binder Available for Blending

RAP1	RAP2	RAS	% of Blended binder			SUM			Total	Total		
100.0%	100.0%	100.0%	53.53%	22.48%	24.00%	100.00%	Added Binder (g)		Added	Mix	%RR	%PG64-22
95.0%	95.0%	75.0%	56.38%	23.67%	19.95%	100.00%	RR	PG64-22	Binder (g)	Weight (g)	by mix wt.	by mix wt.
Blended Binder weight (g)			213.7	89.7	75.6	379.0	125.0		125.0	12745.2	0.98%	0.00%
										%VB	Pbr by	Pbr by
	% of Total Binder =		42.40	17.80	15.00		24.80	0.00		by mix wt.	binder wt.	mix wt.
	Pb =		4.28%							0.98%	77.07%	3.30%
	Weighted Gb =		1.029									

Fractional Gradations

1/2" Clean	3/8" Clean	MS	RAP1	RAP2	RAS	1/2" Clean	3/8" Clean	MS	Sieve size	Combined Gradation	SP125 Spec	BP-1 Spec
% Pass	% Pass	% Pass	% Pass	% Pass	% Pass	% Pass	% Pass	% Pass		% Pass	% Pass	% Pass
100.0	100.0	100.0	36.9	13.4	2.8	0.0	46.9	0.0	3/4"	100.0	100 min	100 min
83.0	100.0	100.0	35.6	13.4	2.8	0.0	46.9	0.0	1/2"	98.7	90-100	85-100
38.6	100.0	100.0	33.5	13.2	2.8	0.0	46.9	0.0	3/8"	96.4	90 max	
4.6	44.3	100.0	25.7	10.0	2.8	0.0	20.8	0.0	#4	59.3		50-70
4.0	12.2	76.3	18.7	6.6	2.7	0.0	5.7	0.0	#8	33.8	28-58	30-55
3.9	6.8	47.2	15.1	4.8	2.3	0.0	3.2	0.0	#16	25.3		
3.7	5.6	29.5	12.3	3.6	1.6	0.0	2.6	0.0	#30	20.2		10-30
3.5	5.0	18.5	9.5	2.5	1.4	0.0	2.3	0.0	#50	15.7		
3.0	4.3	13.0	5.8	1.7	1.3	0.0	2.0	0.0	#100	10.7		
2.3	3.4	9.6	3.5	1.3	1.0	0.0	1.6	0.0	#200	7.4	2-10	5-12

FIGURE 12: FPM Mix Design Spreadsheet

The first item to point out in Figure 12 is the pink highlighted cells marked as "DesignExpert Results." These are input cells for any verified binder blend component proportions. In this case, it is the component proportions for the verified RR5 blend discussed in the previous section, 42.4% RAP1, 17.8% RAP2, 15.0% RAS, and 24.8% RR (or CycL for FPM comparative analyses; to be discussed later).

The next step in using the spreadsheet is to decide the "% Binder Available for Blending" percentages (green cells, center-left). It cannot be overstated how much of an issue this is in the FPM paving industry today. The question of how much of the binder in the RAP/RAS is actually blending with virgin binder and contributing to the performance of the FPM is being heavily investigated at this time. Detailed discussion of this matter will not be performed in this report due to report-length limitations. Suffice it to say that based on the literature and engineering judgment, the actual percentage of binder available for blending is less than 100% (complete binder availability) and much more than 0% (the "black

rock" scenario). Therefore, based on the literature and many conversations with industry practitioners and experts, the RAP and RAS binder availability percentages were set at 95% and 75%, respectively.

The next step is to adjust the purple highlighted cells at the very top so that the light-blue highlighted cells directly above one another in the reclaimed materials columns are approximately equal per reclaimed material. This step adjusts the proportions of the reclaimed materials to keep the blended-binder grade at the desired level. At the same time, it changes the combined aggregate gradation and the reclaimed binder content.

The next step is to adjust the virgin aggregate percentages to fine-tune the combined gradation. The aggregate percentages, reclaimed and virgin, must sum to 100%.

Finally, the total binder content as a percentage of the mix (P_b) can be adjusted upward by adding, in this case, some PG64-22 petroleum-based binder, or, if P_b is too high initially, the proportions of reclaimed materials can be lowered but in a manner that keeps the blended-binder grade at its desired level.

There is quite a bit of flexibility in this design process. There is choice in the binder blend proportions (DesignExpert solutions) that can produce a desired binder grade, the % Binder Available for Blending percentages can be changed if so desired, the number and gradation of the different virgin aggregates can be adjusted, the relative proportion of the reclaimed materials to the virgin aggregates can be adjusted, and the reclaimed materials could be fractionated to provide much more control of the gradation.

Following specification-verification of the RR5 blend, all effort has been focused on producing a FPM that meets, preferably, MoDOT Superpave specifications (i.e. SP125), or alternatively, plant mix bituminous pavement/base specifications (i.e. BP-1). However, many attempts to use the RR5 blend in a SP125 mix failed to produce a FPM meeting the volumetric and dust-to-effective binder (D/P_{be}) specifications; there are just too many fines in the RAPs. It seems that the only way to achieve a high-RAP/RAS FPM that meets Superpave specifications would be to fractionate the RAPs into two or, more probably, three (or more) fractions thus allowing for much more gradation control. Of course, this would require much more characterization testing of the fractionated RAPs (FRAPs). Based on the failure to produce a SP125 FPM using the RR5 blend and high percentages of RAP/RAS, the focus shifted to producing a FPM using the RR5 blend that would meet BP-1 specifications.

It is important to point out that for all of the preliminary design, production, and testing of FPMs, CycL was used in lieu of the RR because of the limited amount of RR on hand.

MoDOT requires the use of a modified Marshall FPM design procedure for BP mixes but allows for the use of six inch diameter, gyratory-compacted specimens for volumetric determination, when applicable. Once a design gradation is determined, at least three total binder contents (at a maximum separation of 0.5%) are to be evaluated.

The FPM in Figure 12 was used as the basic RR5 blend FPM design. The combined gradation meets the BP-1 specification, and there is *no* virgin, petroleum-based binder (i.e. PG64-22) added to the mixture; a feature that is highly relevant to the project concept. However, after many trial batches (again, using CycL in lieu of the RR), it became apparent that the RR5 blend was not conducive to 1) producing a BP-1 mix with no added virgin petroleum-based binder, and 2) satisfying volumetric specifications. A total of three total binder contents were evaluated. The lowest binder content was satisfied by the basic RR5 blend FPM design, but the intermediate and highest total binder content FPMs had some PG64-22 added to the basic RR5 blend FPM. For each FPM, sufficient mix was generated to produce a theoretical maximum specific gravity (G_{mm}) specimen and two, six inch diameter gyratory-compacted specimens. All specimens

were treated the same throughout the production process and each gyratory specimen was compacted using the MoDOT specified 35 gyrations. The results are shown in Table 10.

TABLE 10: RR5 FPM Design Results

Property Binder Level	P_b (%)**	PG64-22 (%)**	P_{be} (%)**	D/P_{be}	Specimen Height (mm)*	Air Voids (%)*	VMA (%)	VFA (%)
Low	4.28	0.00	3.64	2.03	117.8	5.83	14.15	58.75
Intermediate	4.58	0.31	3.94	1.87	116.2	4.60	13.70	66.40
High	4.88	0.63	4.25	1.74	114.8	3.47	13.43	74.13
BP-1 Specification				***	110 – 120	3.5	≥13.5	60 – 80

* Based on the average of two specimens
**Based on mix weight
***No MoDOT specification; BP-1 mix designers strive for D/P_{be} of 1.5 – 2.0

As can be seen in Table 10, a P_b of 4.88% should result in a FPM with the desired level of air voids and VFA. However, VMA would be borderline out-of-specification, and VMA could be argued to be the most important volumetric property to use for prioritizing FPM design selection. Many designers purposely select designs with VMA levels 0.5% higher than the required minimum. This is especially true when there is RAP/RAS in the FPM because the use of the effective specific gravity (G_{se}) of the RAP/RAS as a substitute for the RAP/RAS aggregate bulk specific gravity (G_{sb}) artificially inflates the calculated VMA of the FPM, and MoDOT requires the use of G_{se} in RAP/RAS FPM designs. Therefore, because the RAP/RAS content was so high for the RR5 FPM, 4.50% was chosen as the design P_b content as it should produce a VMA of about 13.8% and a VFA that is still within specification limits. However, it was expected that air void levels would be higher than desired but still within a generally acceptable level of $4 \pm 1\%$.

Using the Figure 12 design with an additional 30 grams of PG64-22 (i.e. P_b of 4.50%), four batches of FPM were ultimately produced. Prior to batching the aggregates, the RAPs were split (fractionated) on the ¼ inch screen in an effort to better control the gradation during batching. The percentages of the -¼ inch (P1/4) and +¼ inch (R1/4) for each RAP were determined and, during batching of the RAPs, the P1/4 and R1/4 fractions were recombined at the same percentages. This was done to assure that the RAP binder properties were not changed; i.e. without knowing each FRAP's binder content and grade, recombining the FRAPs in proportions other than the original percentages could have affected the total reclaimed binder content and/or blended binder grade.

In a continuing effort to minimize variability in the FPM batching process, sufficient amounts of each of the six aggregates (the four FRAPS, the RAS, and the ⅜ inch clean) were systematically reduced such that each half of the last split was used to build one FPM batch; one half for a RR-based FPM and the other half for a CycL-based FPM. This process was repeated twice resulting in two aggregate batches, or four FPM batches. The CycL for both CycL batches came from the same 5-gallon can while the RR for each RR batch came from two separately extracted/recovered (replicate) samples.

For each of the four FPM batches, sufficient mix was generated to produce a theoretical maximum specific gravity (G_{mm}) specimen and two, six inch diameter gyratory-compacted specimens. All specimens were treated the same throughout the production process and all eight gyratory specimens were compacted using 35 gyrations. The results are shown in Table 11.

TABLE 11: RR and CycL FPM Volumetric Comparison

Property	Specimen Height (mm)		Air Voids (%)		VMA (%)		VFA (%)	
Recycling Agent	RR	CycL	RR	CycL	RR	CycL	RR	CycL
Batch1-Spec1	116.9	115.0	5.28	3.90				
Batch1-Spec2	116.4	115.2	4.99	4.08				
Batch1 Mean	116.7	115.1	5.13	3.99	14.2	13.4	63.9	70.1
Batch2-Spec1	114.1	114.5	3.29	3.53				
Batch2-Spec2	114.2	114.6	3.38	3.67				
Batch2 Mean	114.2	114.6	3.33	3.60	12.6	13.0	73.5	72.3
Overall Mean			4.24	3.80	13.4	13.2	68.7	71.2
BP-1 Specification	110 – 120		3.5		≥13.5		60 – 80	

The batch-to-batch variability of the RR-based FPMs is high and is speculated to be a function of differences between the two RR replicate samples used for FPM batching. It seems that the RR can auto-oxidize over time; if exposed to air for a prolonged period of time, solids resembling fat globules eventually form and settle to the bottom of the storage container. This segregation was not sufficiently remedied before obtaining the RR from the container for mixing Batch2. The RR for Batch2 was noticeably less viscous as it was placed into the mixing bucket and, not surprisingly, reduced the overall binder blend viscosity relative to Batch1. This reduced viscosity reduced the resistance to compaction and resulted in a relatively shorter specimen with a considerably lower VMA and % air voids.

Setting the variability issue aside, the basic FPM design resulted in a fair mix; % air voids were slightly high but fell in the generally acceptable range of 4 ± 1%, VMA was slightly low, but VFA met specification. This FPM utilized 53% reclaimed aggregate (from the RAPs and RAS) and, therefore, 47% virgin aggregate. More importantly, and in relation to the project concept, only 0.23% of the total FPM mass (or 5.62% of total binder mass) was virgin PG64-22 binder. The rest of the binder was RAP/RAS binder and RR.

Hamburg Wheel-Track Testing

Hamburg wheel-track testing of the FPM was performed using the same FPM design reflected in the Table 11 results except specimen mass and thickness was controlled to produce 6 inch diameter specimens with a % air voids of 7±1% (*49*). The batching procedure was the same as in FPM design and produced four Superpave gyratory-compacted (SGC) specimens per recycling agent. A Hamburg specimen was produced by cutting two 62 mm thick SGC specimens in such a manner that when butted against one another resulted in a continuous wheel path approximately 10 inches long. Two Hamburg specimens were submerged in 50°C water and subjected to 20,000 wheel passes, or however many passes that produced a pre-determined, maximum impression depth based on the applicable specification (the software default of 14 mm was used for this study). A full 20,000 wheel passes takes about 7 hours to complete. Figure 13 shows the Hamburg test results.
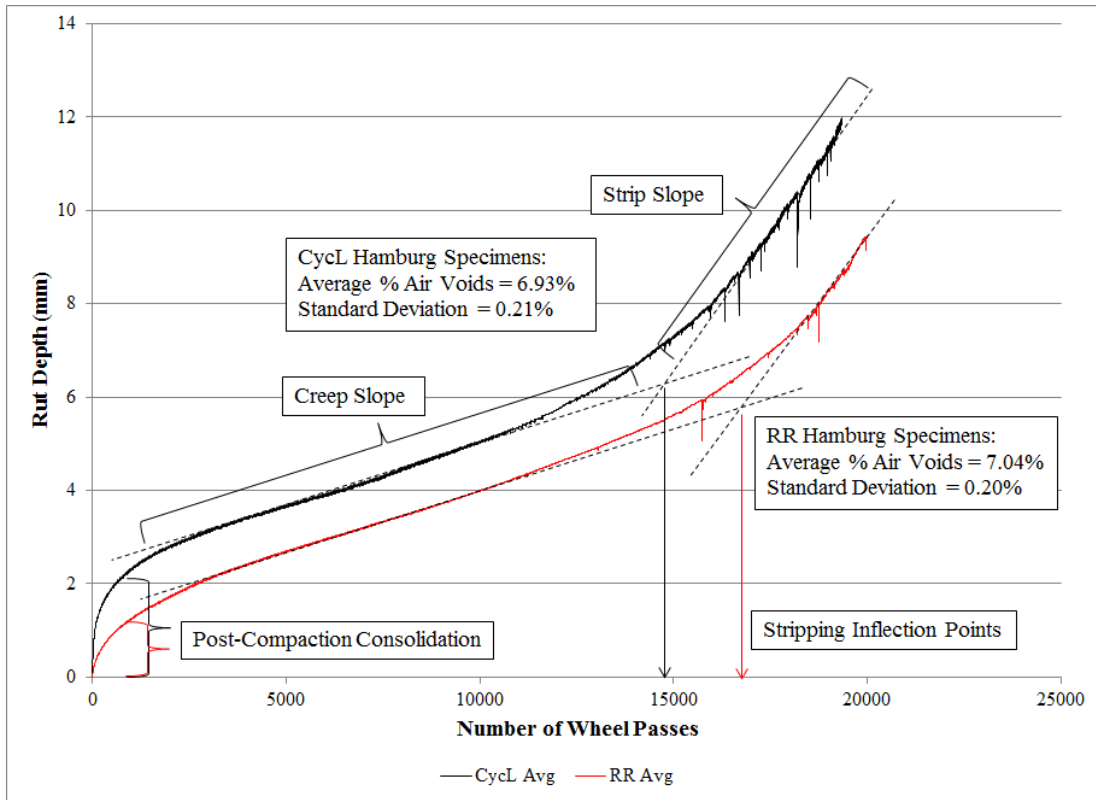
FIGURE 13: RR vs. CycL FPM-Hamburg Wheel-Track Testing Comparison

Hamburg wheel-track testing is a good indicator of rutting and stripping (moisture damage) potential of a FPM. There are four basic parameters identified in Figure 13: post-compaction consolidation, creep slope, strip slope, and stripping inflection point. Post-compaction consolidation is usually taken at 1000 wheel passes and is considered to be the result of the wheel load densifying the mixture. Creep slope is a measure of the rutting susceptibility due to gradation, binder stiffness, particle shape, etc., but not moisture damage. The stripping inflection point and strip slope are measures of moisture damage. Where the strip slope is a measure of accumulated deformation due to moisture damage, the stripping inflection point is a way to identify when the mixture performance becomes mostly a function of moisture damage. The Colorado Department of Transportation reports that a stripping inflection point occurring before 10,000 wheel passes indicates a stripping susceptible FPM (*50*).

The test data from two Hamburg specimens was averaged to produce the curves shown in Figure 13. The test method recommends a void content of 7 ± 1% for the test specimens. The average voids content (based on four SGC specimens) for each mix is indicated on Figure 13 and shows that the voids compare very closely with the RR mix having slightly higher voids. Variability among the SGC specimen voids content is essentially the same for each mix.

In making conclusions about the results shown in Figure 13, one must consider that the short-term aged viscosity of the pure RR is slightly higher than the short-term aged viscosity of the pure CycL. The increased post-compaction consolidation of the CycL FPM relative to the RR FPM could be due to this viscosity differential. All other major properties of the two FPMs (aggregate gradation, particle shape and geology, binder content, and volumetrics) are, however, essentially the same. The creep and strip slopes of both FPMs are parallel indicating that the rates of deformation due to non-moisture and moisture-induced damage are identical. However, the locations of the stripping

inflection points indicate that the RR FPM performed somewhat better than the CycL FPM insofar as the onset of moisture damage is concerned. Therefore, because of the slight short-term aged viscosity differential between the pure RR and the pure CycL, one can only conclude that the RR FPM performed "as well as" the CycL FPM in the Hamburg tests. Precision statements for this test method have not been adopted at this time.

Tensile Strength Ratio (TSR) Testing

The Tensile Strength Ratio (TSR) test, arguably the most widely used test for evaluating the stripping potential (moisture-susceptibility) of FPMs, was performed on RR and CycL mixes, again, for comparative purposes (*51*). The same process and FPM design used to produce the Hamburg SGC specimens was used for producing the TSR specimens. Six, 95 mm thick, 6 inch diameter SGC specimens per recycling agent were produced with a specified % air voids of 7 ± 0.5% as the target. After determining the actual % air voids for each SGC specimen, the six are grouped into two groups of three specimens each such that the average % air voids of each group is approximately equal. One group of three specimens is designated as the unconditioned (dry) set and the other group of three specimens is designated as the conditioned (wet). Conditioning requires 1) the vacuum saturation of the specimens to a level of 70 – 80% saturation, 2) the storage of the partially – saturated specimens in a freezer for at least 16 hours, 3) the immediate submergence of the frozen specimens into a 60°C water bath for 24 hours, 4) the transfer of the specimens to a room temperature bath for 2 hours, then 5) the determination of the indirect tensile strength of each specimen at room temperature. The dry set specimens are kept dry by placing them in watertight plastic bags, then they are submerged (still in the bag) in the room temperature bath for 2 hours prior to indirect tensile strength (ITS) determination. TSR is calculated as the ratio of the average wet ITS to the average dry ITS. TSR is usually expressed as a percentage, but not always, and is sometimes referred to as "retained strength." Table 12 shows the TSR test results.

TABLE 12: TSR Test Results

TSR Specimen Condition	TSR Specimen Number	RR				CycL			
		%Voids	%Voids(avg)	ITS (psi)	ITS (avg)	%Voids	%Voids(avg)	ITS (psi)	ITS (avg)
Dry	1	7.57		124		7.22		122	
Dry	2	7.81		123		7.29		122	
Dry	3	7.52	7.63	123	123	7.28	7.27	124	123
Wet	1	7.34		79		7.28		91	
Wet	2	8.04		77		7.41		90	
Wet	3	7.57	7.65	80	79	7.14	7.28	93	91
				TSR (%)	63.9			TSR (%)	74.3

The test results indicate that the RR FPM is more prone to stripping than the CycL FPM; a difference in the TSRs of 10% seems pretty conclusive in this regard. However, one should be aware that the TSR test is highly variable. The most recent study on developing precision statements for the TSR test report a single-operator standard deviation (i.e. 1s) of 3.3% and the single-operator acceptable range of two results (i.e. d2s) as 9.3% (*52*). Although the precision limits are not officially adopted by AASHTO yet, the report verifies what has been widely recognized for some time now; the TSR test variability is problematic and a better method of evaluating the moisture-susceptibility of a FPM is greatly needed. The

Hamburg Wheel-Track Test is increasingly the preferred alternative to the TSR in assessing stripping potential (*53, 54*), but as discussed earlier, there are no AASHTO-adopted precision statements for the Hamburg test at this time.

Table 12 also shows that the RR TSR specimen average % air voids for both the wet and dry sets were slightly out of specification on the high side, and were about 0.35% higher than the average % air voids of the CycL TSR specimens. One could argue that this higher void content allowed for greater penetration of the water into the RR TSR wet specimens and resulted in greater moisture damage. However, an observation made during the RR TSR hot water bath conditioning procedure may better explain the lower TSR for the RR FPM. When the hot water bath lid was raised just prior to transferring the specimens to the room temperature bath, the odor of the RR was unmistakable; i.e. it is speculated that there was some dissolution of the RR by the 60°C water during the 24 hour submergence period. This observation was not totally unexpected. One must remember that the RR was extracted from the bulk rubber using acetone, but the rubber was extracted from the guayule shrub using a water-based process. The issue of potential water solubility of the RR under certain conditions was always a concern. It may be instructive that the 50°C water temperature during the 7 hour long Hamburg test did not seem to negatively impact the results whereas the 60°C water temperature during the 24 hour TSR conditioning procedure may have had the opposite effect.

Low-Temperature Flexural Creep Stiffness Testing

A relatively new, and as yet un-adopted, test was used to determine low-temperature flexural creep stiffness of the RR and CycL FPMs. Two documents were used as guidance in this exercise: a Utah DOT research report (*55*), and a NCHRP-IDEA report (*56*). The test is essentially the same used to determine the low critical temperature of binders with two basic exceptions: the test specimens are FPM beams instead of binder beams, and the creep load is larger. The bending beam rheometer (BBR) may have to be retrofitted with a larger capacity load cell in order to accommodate a larger creep load. The FPM beams are of the same dimensions as the binder beams (6.35 mm thick, 12.70 mm wide, and 127 m long) and, for this study, were cut from the center of the SGC volumetric specimens.

Eight FPM beams were cut from each of the eight SGC volumetric specimens resulting in 32 RR FPM beams and 32 CycL FPM beams. A masonry saw was used to cut a 20 mm thick disk from the center of each of the volumetric specimens, then a wet tile saw was used to reduce each 20 mm thick, six inch diameter disk to eight FPM beams of the proper dimensions. The cutting and subsequent determination of the actual dimensions of the FPM beams were done at the Missouri University of Science & Technology (S&T) while the BBR testing was performed at the MoDOT Central Laboratory. Regarding test temperatures, the Utah report draft protocol states, "For quality control purposes the single test temperature shall be 10 ºC above the specified binder grade used in the mixture. For performance prediction at least 3 temperatures shall be used at 6 ºC intervals. The test temperatures of 4 ºC, 10 ºC, and 16 ºC above the specified binder grade used in the mixtures have been successfully used. Other temperatures can also be used depending on the project requirements." For this investigation, testing was performed at -12°C to meet the quality control criteria discussed above, and -18°C to be able to do a temperature-dependency analysis. Because the beam specimens can only be tested once, four of the beams cut from each of the eight volumetric specimens were tested at -12°C and the other four were tested at -18°C. Test results of interest for this comparative analysis were the stiffness and the m-value at 60 seconds of creep load which were obtained from the BBR software output. The results are shown graphically in Figures 14 and 15. Note that

"R" identifies the RR FPM and "C" identifies the CycL FPM. The numbers "1&2" mean that all valid data from the testing of both batches of each FPM are included in the trendline determination.
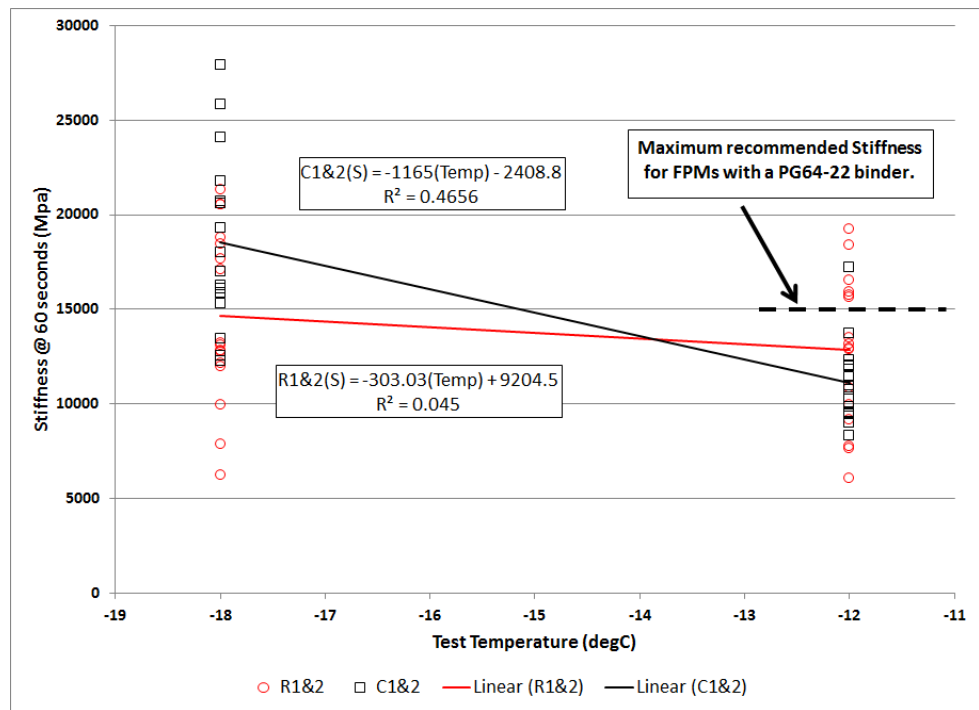

FIGURE 14: RR vs. CycL: Low-Temperature Flexural Creep Tests: Stiffness at 60 seconds

There are only 63 data points reflected in Figures 14 and 15. One beam test was discarded due to testing irregularities. The Utah DOT report recommends testing five beams per treatment combination and if the coefficient of variation (1s%) for those five repeated measurements is greater than 15%, one should check for an outlier, remove it if it exists, then recalculate 1s% for the four remaining beams. If 1s% is still greater than 15%, the entire test is invalid. If this precision recommendation had been applied to the data in this investigation, a majority portion would have been invalid. Personnel at the MoDOT Central Lab indicated that test data they have been collecting for their purposes also sometimes violates this 15% 1s% level. Therefore, this recommended precision statement was disregarded in this investigation. It is also important to note that the NCHRP-IDEA report concluded that differences in % air voids have no significant effect on creep stiffness of FPMs at low temperatures. No attempt was made during this exercise to verify this conclusion.

Figure 14 shows trends of increasing stiffness variability (at 60 seconds of creep load) as the temperature decreases, higher variability in the RR stiffness data relative to the CycL data as measured by R^2, and interestingly, a flatter stiffness – temperature trendline slope for the RR FPM. A check on the flatter RR FPM stiffness – temperature slope was performed by fitting trendlines individually to each set of FPM batch data. The result corroborated the trend shown in Figure 14.

The Utah report also has recommended maximum stiffness and minimum m-value limits. The report states, "The average stiffness of the mixture at 60-seconds and at a temperature of 10 ºC above the performance grade of the binder shall not exceed 15,000 MPa; the average m-value at the same loading time and temperature shall not *exceed* 0.12." The word, "exceed" is bolded and italicized to make the point that this could probably be worded better to indicate that 0.12 is

a recommended "minimum" value. As indicated in Figure 14, both the RR and CycL FPMs meet the maximum stiffness recommendation. Additionally, even though the RR trendline is slightly higher than the CycL trendline at -12°C, a two-sided t-test (blocked across the different FPM batches) showed that the 60 second creep stiffness of the RR and CycL FPMs are not significantly different, based on a significance level of 5% (i.e. alpha=0.05).
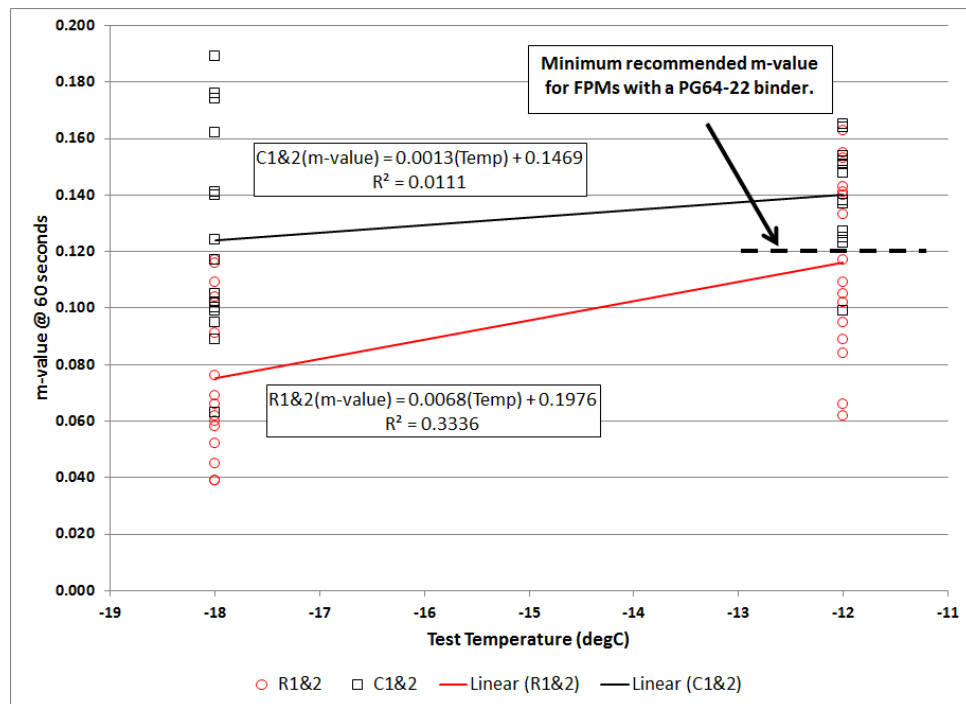

FIGURE 15: RR vs. CycL: Low-Temperature Flexural Creep Tests: m-value at 60 seconds

Although stiffness is an important and specified parameter to consider when evaluating the low temperature cracking potential of a FPM, the m-value is usually the parameter that controls when checking both parameters against the specifications, for example, in binder testing. Remember that the stiffness (or modulus) is a measure of a beam's resistance to deflection (strain), whereas the m-value is a measure of the rate-of-change of stiffness with time; i.e. as the temperature decreases, the thermal shrinkage stresses are dissipated quicker as the m-value increases (*48*).

Figure 15 shows trends of increasing m-value variability (at 60 seconds of creep load) as the temperature decreases (the same trend as stiffness), and higher variability in the CycL m-value data relative to the RR data as measured by R^2 (opposite of the stiffness trend). The trendline slopes are approximately the same, but the relative position of the CycL trendline to the RR trendline indicates that the CycL FPM has superior stress relaxation properties than the RR FPM. Again, as with all of the other comparative analyses presented in this report, an analytical result such as this does not necessarily mean that the RR cannot be used successfully as a recycling agent. For example, as shown in Figure 15, even though the RR FPM m-value trendline at -12°C is slightly below the recommended minimum, this only means that the blended binder proportions of the mix design need adjustment to raise the m-value. A two-sided, blocked, t-test was also performed on the -12°C m-value data and showed that the RR FPM and CycL FPM 60 second m-values are significantly different at a significance level of 5%, but not at a significance level of 1%.

PLANS FOR IMPLEMENTATION

The interest in the guayule plant as a renewable, domestic resource for natural rubber, resin, and biomass is growing (*57, 58, 59*). The Yulex Corporation has explicitly stated their desire to expand on the research in this study and continue investigations into using guayule-based materials in FPM applications (*18*). Their support will be requested in taking the next step to pursue a line of investigation into guayule plant oils as FPM binder modifiers. Preliminary work has already been undertaken at Missouri S&T. The RR was separated into a hexane-soluble (non-polar) "oil" phase and a methanol-soluble (polar) "polyphenol" phase using a liquid-liquid partitioning process (*60*). The de-solventized oil fraction still experienced significant mass loss upon short-term aging at 163°C, but it was not as great as that experienced by the RR. More significantly, the oil does not auto-oxidize (i.e. no formation of oxidation products with prolonged exposure to air), the probability is very low of it being water-soluble at any temperature or at any proportion within a FPM, and the viscosity is much lower than the RR.

The Missouri Department of Transportation agreed at the beginning of this project to support a practical field application of the concept. The original letter of support is on file and available upon request. Very recently, MoDOT representatives reiterated their interest in pursuing a pilot project in which the RR, or maybe another laboratory verified guayule-based material (e.g. the guayule oil), would be used in a state-approved FPM on a pavement project of their choosing.

At the earliest opportunity, a Type 2, NCHRP-IDEA project proposal to implement the findings in this study on a practical level will be developed and submitted.

CONCLUSIONS

Results of binder-blending mixture experiments were useful for comparative analyses and necessary for FPM design. The experiments required creating many different blends of the RAP/RAS binders with the acetone-extracted guayule rubber resin (RR) and the hexane-extract from the waste-stream guayule leaves and attached stems (LF), recreating those same blends but substituting the RR and LF with the appropriate petroleum-based binders, testing each blend, and then generating response surface models (RSMs) using those test results. Conclusions from the binder-blending experiments are as follows:

- Clay-gel chromatography testing verified that the pentane-soluble portion of the RR and LF pure blends met recycling agent specifications. Both contain polar and aromatic compounds, but less than 25% saturates by mass.
- The RR and LF pure blends are less temperature-susceptible than the petroleum-based binders; i.e. they exhibit a smaller change in viscosity for a given change in temperature, which is desirable.
- The RR and LF pure blends (not blended with petroleum-based binder; i.e. 100% guayule-based material) suffer significant mass loss upon short-term aging at 163°C. Additionally, the RR can auto-oxidize at room temperature if exposed to the air for prolonged periods of time, and may dissolve if exposed to hot water for prolonged periods of time.

- The absolute temperature spreads ($T_cH - T_cL$) for the LF-RAP/RAS blends were, to a large degree, significantly different than the proportionally-identical PG52-28-RAP/RAS blends. Eight of the nine LF-RAP/RAS blends in the analysis had, on average, a 12.3°C smaller temperature spread than the proportionally-identical PG52-28-RAP/RAS blends. This finding turned the remaining investigative effort solely onto the RR.

- Statistical analyses showed that the RR-RAP/RAS blends did perform the same as proportionally-identical CycL-RAP/RAS blends in terms of high-temperature stiffness (as measured by the high critical temperature, T_cH), but did not perform exactly the same in terms of cold-temperature cracking resistance (as measured by the low critical temperature, T_cL). In terms more familiar to the FPM designer, the difference between the absolute temperature spread ($T_cH - T_cL$) for the RR-RAP/RAS blends relative to proportionally-identical CycL-RAP/RAS blends was small, but statistically different. Of the ten blends in the analyses, seven of the CycL-RAP/RAS blends had the larger absolute temperature spread (an average of 1.1°C) while three of the RR-RAP/RAS blends had a slightly larger temperature spread (an average of 0.3°C). So, although the RR-RAP/RAS blends did not perform *exactly* as the CycL-RAP/RAS blends in terms of T_cH and T_cL, this does not mean the RR cannot be used as a recycling agent.

- A RR-RAP/RAS blend estimated by the RSM to meet PG64-22 binder specifications was produced and verification testing (except flash point) was performed. The flash point of the RR-RAP/RAS blend was assumed to be within specification based on successful flash point testing of the RR pure blend (i.e. not blended with other materials). The RR-RAP/RAS blend met all other PG64-22 specifications except mass change. The maximum allowable mass change for a PG binder is ±1.0% but the blend experienced a mass loss of 3.1%.

Using this binder blend, a RR-based FPM was designed, produced, and tested that 1) met the gradation specification for a Missouri Department of Transportation (MoDOT) moderate-quality FPM, but 2) did not quite meet all of the volumetric requirements. This FPM utilized 53% reclaimed aggregate (from the RAPs and RAS) and, therefore, 47% virgin aggregate. More importantly, and in relation to the project concept, only 0.23% of the total FPM mass (or 5.62% of total binder mass) was virgin, PG64-22 binder. The rest of the binder was RAP/RAS binder and RR. A CycL-based FPM using the same design proportions was also produced and tested for comparative analyses. Conclusions from the FPM testing are as follows:

- Standard Hamburg Wheel-Track testing, which specifies full submersion of the specimens in 50°C water during the approximately 7 hours of rut testing, was performed. The results indicated that the RR-based FPM performed as well as the CycL-based FPM in regard to rutting and stripping (moisture damage) resistance.

- Standard Tensile Strength Ratio (TSR) testing, which specifies a 24 hour full submersion of the specimens in 60°C water, was performed. The results of this moisture-susceptibility test, however, indicated that the RR-based FPM may be more prone to stripping than the CycL-based FPM.

- A (currently) non-standard, but promising test protocol for determining low-temperature, FPM flexural creep stiffness was performed on the RR- and CycL-based FPMs. At -12°C, statistical analyses showed that the creep stiffness of the RR-based FPM was not significantly different than the CycL-based FPM. However, the m-value of the RR-based FPM was statistically different (lower) than the CycL-based FPM m-value which indicates a somewhat higher cold-temperature cracking potential for the RR-based FPM relative to the CycL-based FPM.

Based on the test results, the RR produced in this study could, in a practical sense, be used as a recycling agent. FPM-production and paving industry representatives have shown interest in the results of this study and the future viability of

guayule-based materials. The Yulex® Corporation, the only U.S. company currently cultivating and processing the guayule plant for commercial purposes, recently indicated to the project researchers that a 10,000 ton/year rubber production facility is planned for the southwestern U.S. by 2014, with multiple facilities soon thereafter. Yulex also indicated that they are still in the economic modeling phase for guayule resins (one of the three basic products that can be obtained from the guayule shrub), and if resin were to be produced for high-volume (i.e. FPM) applications, procedures would be developed to extract the resin directly from the plant fiber and not from the rubber product (*18*). This would greatly increase the resin production as the plant resin yield is 2 – 3 tons/acre/year, and there are about 124 million acres of land in the U.S., mostly in the southwest, environmentally suitable for guayule agricultural practices. Therefore, resin production could reach 200 – 300 million tons/year in the U.S. alone.

The National Asphalt Pavement Association reported that about 3.2 million tons of reclaimed RAP and RAS binder were utilized in the U.S. in 2010, and the usage is projected to increase every year (*61*). If all 3.2 million tons of that reclaimed RAP/RAS binder had been softened with, say, 25% RR by weight of the blended binder, only 1.1 million tons of RR would have been required. So, even with a very large increase in the amount of RAP/RAS usage in the U.S. in the coming years, which is being aggressively promoted, the availability of sufficient quantities of resin seems quite possible. With availability deemed probable, price is the only other controlling factor. If the guayule producers (Yulex, and soon the tire companies, Bridgestone and Cooper (*58, 59*)) can hold the price of the resin engineered for FPM applications to something less than about $0.70 per pound, they can compete with not only petroleum-based products similar to Cyclogen L (*62*), but also bio-based recycling agents currently in use in the paving industry (e.g. Hydrogreen® (*63, 64*)).

So, although the economic viability of guayule products for FPM applications is difficult to determine at this point, the potential availability of guayule in the U.S. as a feedstock is substantial. This project was a first step and it verified that the guayule shrub, a perennial industrial plant, holds great potential as a renewable, domestic source of FPM binder additives such as resins, oils, and polymers.

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