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Rheology and performance evaluation of Polyoctenamer as Asphalt Rubber modifier in Hot Mix Asphalt

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**Rheology and performance evaluation of Polyoctenamer
as Asphalt Rubber modifier in Hot Mix Asphalt**

by

Ka Lai Nieve Ng Puga

A thesis submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Civil Engineering (Civil Engineering Materials)

Program of Study Committee:
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Iowa State University

Ames, Iowa

2013

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ABSTRACT

The high amount of scrap tires that are generated annually in the United States are stored in stockpiles, landfills and dumps all over the United States. Tires are mostly composed of rubber; and they can be recycled to obtain two types of ground tire rubber (GTR), ambient and cryogenic. Tire recycling can help reduce the accumulations of scrap tires, while the GTR can be used in many applications in different industries.

The asphalt industry has used GTR in highway pavement construction since the 1960's. GTR can be blended with any conventional asphalt binder to produce asphalt rubber binders, that due to the elastomeric properties of the GTR, will have better performance at high and intermediate temperatures than conventional binders. However, one of the challenges of asphalt rubber technology is its high viscosity, which increases its mixing and compaction temperatures when compared to conventional asphalts.

The objective of this research is to characterize the effects of the binder additive polyoctenamer (PO) on the rheological properties of laboratory-produced asphalt rubber binders with a base asphalt of PG46-34 and two different types of ground tire rubber (ambient and cryogenic); also, to see the effects of PO in the characterization of the performance of asphalt rubber mixtures.

The laboratory-produced binders were evaluated following the Superpave binder specification and testing procedures. Densities, viscosities, complex modulus (G^*), mass losses, creep stiffness were obtained from the binders. The statistical analysis performed on the binders demonstrated that the addition of PO improves the viscosities of asphalt rubber thereby obtaining a reduction in the estimated mixing and compaction temperatures. The binder grading demonstrates that PO does not affect negatively the

final performance grading for high, intermediate and low temperature. Further, the construction of the master curves showed that asphalt rubber binders with and without PO have similar stiffness performance.

The performances for the dynamic modulus, flow number and indirect tensile strength ratio test of the laboratory-produced mixtures with and without PO were not statistically different. Accordingly, the mixes' master curves showed no difference. Overall, the addition of PO does not negatively affect the performance of asphalt rubber mixes.

CHAPTER 1. INTRODUCTION

Background

The use of asphalt rubber binders to produce asphalt rubber mixtures is very common in geographic areas where rutting performance is a problem. In general, one of the difficulties of applying asphalt rubber technology is the high temperatures required for mixing and compaction of the mentioned mixtures when compared with conventional asphalt mixtures, due to the high viscosities encountered in asphalt rubber binders. The high temperatures used to produce the asphalt rubber binders and mixes carries an associated energy cost.

Therefore, the addition of chemical modifiers to asphalt rubber binders is used to improve the rheological properties of these binders to help reduce the mixing and compaction temperatures of their mixes and in turn reduce the energy cost of producing these asphalt rubber mixes. However, whenever a modification of binders is made, a comprehensive study on the real effects of the modifiers on the binder's rheological performance and on the mixes' performance is needed.

Problem Statement

Asphalt rubber binder is known for having higher viscosities when compared to conventional asphalt binders at a certain temperature. This causes the increase in mixing and compacting temperatures of asphalt mixtures to obtain the desirable viscosity required by the standard specifications to perform these activities.

It is believed that the addition of a binder additive like polyoctenamer (PO) will help reduce the viscosity of asphalt rubber binder without negatively affecting its

rheological properties. Likewise, it is thought that PO will not affect the performance of the asphalt rubber mixtures, but it will help decrease the mixing and compacting temperatures generally used in the asphalt rubber technology.

Objectives

The objectives of this study are to determine if PO affects the rheological properties of asphalt rubber binders produced with a base asphalt of PG46-34 and two different types of ground tire rubber (ambient GTR and cryogenic GTR). Also it will be determined if the performance of the asphalt rubber mixtures are influenced by the addition of PO.

Methodology

The experimental plan carried out in this study uses four laboratory-produced asphalt rubber binders following the procedure described in Chapter 3, and four laboratory-produced asphalt rubber mixes with the mentioned binders following the SuperPave mix design procedure.

The laboratory-produced binders were tested following the SuperPave performance graded asphalt binder specifications and testing procedures. Some of the tests performed to characterize the binders are density by means of a pycnometer test; dynamic shear rheometer tests on unaged, rolling thin film oven (RTFO) aged and pressurized aging vessel (PAV) aged materials to determine the performance grade; mass loss determination on the RTFO aged materials; and bending beam rheometer tests on the PAV aged materials. From the results of the dynamic shear rheometer, master curves for unaged, RTFO and PAV aged materials were constructed.

The performance of the laboratory-produced asphalt rubber mixes were then tested for their stiffness performance through the dynamic modulus testing, their rutting characterization by means of the flow number test and their moisture-susceptibility evaluated using the tensile strength ratio test. Master curves from the results of the dynamic modulus test were built for the four types of asphalt rubber mixtures.

Hypothesis

The following are the hypothesis used to evaluate statistically the results of the experimental plan presented in Chapter 3.

- The addition of PO to asphalt rubber binders influences the viscosity of the binders.
- The addition of PO to asphalt rubber binders reduces the compaction and mixing temperatures of their mixes
- The addition of PO to asphalt rubber mixes affects the high temperature performance of the mixes.
- The addition of PO to asphalt rubber mixes impacts the intermediate temperature performance of the mixes.
- The addition of PO influences to asphalt rubber mixes the low temperature performance of the mixes
- The addition of PO affects the rutting performance of asphalt rubber mixes.
- The addition of PO has an effect in the tensile strength ratio of asphalt rubber mixes

Organization

This thesis is divided in five chapters, including this Chapter 1 which provides a background on Asphalt Rubber, the problem statement, objectives, methodology and hypotheses in this study. Chapter 2 presents a literature review on Asphalt Rubber and PO. Chapter 3 describes the experimental plan and the properties of the materials, as well as the testing procedures followed to perform the characterization of the asphalt

rubber binders and mixtures developed. In Chapter 4 the results obtained for each testing are presented; and statistical analyses are performed and discussed accordingly. Finally, Chapter 5 presents a summary of the findings, conclusions and recommendations for further investigation on this topic.

CHAPTER 2. LITERATURE REVIEW

Asphalt cement

Asphalt is considered a bituminous material. It is a dark brown to black cementitious material that can be found naturally or it can be produced through petroleum (crude oils) distillation. The main producers of asphalt from crude oils in the world are countries like Mexico, Venezuela, Canada and the Middle East. In the United States, the main crude oil sources come from the Gulf Coast, Southwest, Rocky Mountain, West Coast regions and the North side of Alaska (Roberts, 2009).

The distillation process of the crude petroleum consists in the separation of the crude into various fractions that have different boiling ranges. The petroleum is heated in a large furnace at temperatures about 650°F (343°C) and vaporized. The vapors are then condensed in a distillation column where the lightest components rise and are cooled and extracted as gasoline, naphtha, kerosene and light gas oil. The heavier fractions or residues of this distillation are fed to a vacuum distillation unit and heavier gas oils are obtained. The residue of this vacuum distillation is then known as steam refined asphalt cement (Roberts, 2009).

The chemical composition of distilled asphalt consists of different fractions, known as SARA fractions. SARA stands for saturates, aromatics, resins and asphaltenes. The saturates of the asphalt represent around 5-15 weight percentage of the total amount of the asphalt. The aromatics part, also known as naphtene aromatics, constitute together with the asphaltenes most of the asphalt with around 30-45 weight percentage of the asphalt. Resins are polar aromatics that can make up to 30-45 by weight percentage of the asphalt, whereas the asphaltenes constitutes between 5 and 20 percent of asphalt.

Asphaltenes are known to be the insoluble part of asphalt in n-heptane, meanwhile saturates, aromatics and resins are grouped together into Maltenes, and they represent the soluble part of asphalt in n-heptane. Figure 1 presents a graphical depiction of the SARA fractions in asphalt (Lesueur, 2009).

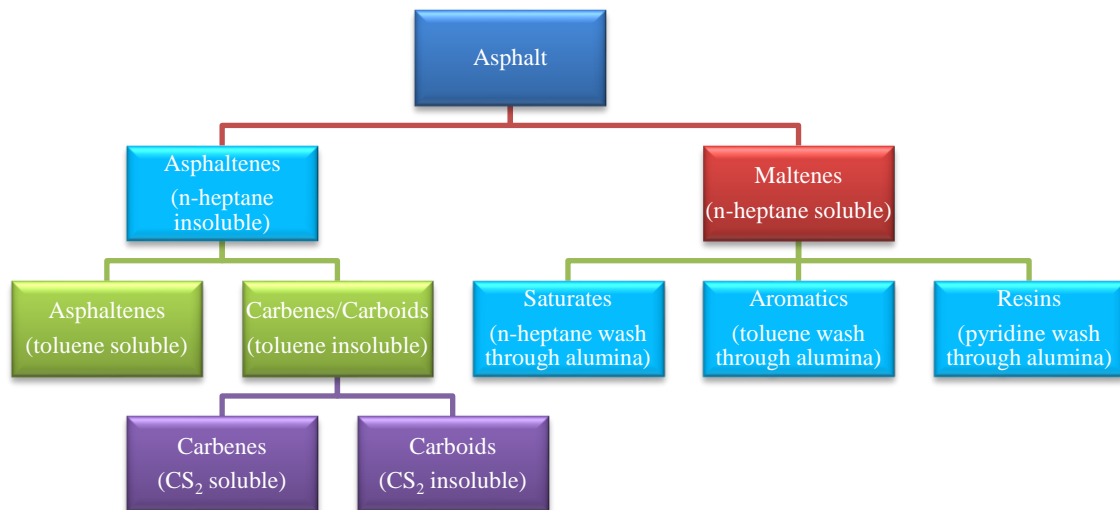


Figure 1. Chemical fractions of Asphalt (SARA fractions) (Lesueur, 2009)

Depending on the source of the crude-oil petroleum and the distillation process techniques applied, the composition of the asphalt cement will vary, thus its intrinsic properties will be different. The asphalt composition will affect its softening point, viscosity, shear susceptibility and complex (stress-strain) modulus. The asphaltene content will influence the softening point of the asphalt in a linear fashion; this means that the softening point will increase as asphaltenes increase (Oyekunle, 2007). Meanwhile, saturates and naphthene-aromatics have low softening points compared to polar-aromatics and asphaltenes which have high softening points (Corbett, 1970).

Various methods have been developed to characterize the properties of asphalt cements, the preferred method to characterize the asphalt cements today in the United

States is the performance based grade method developed by the Strategic Highway Research Program (SHRP) from 1987-1992 and better known as Superpave binder specifications.

The asphalt cement properties are characterized through the performance of the asphalt at high, intermediate and low temperatures under the Superpave binder specifications. The performance at high temperatures are related to the rutting resistance of the asphalt cement, the performance at intermediate temperatures are more related to fatigue cracking and the low temperatures performance to thermal cracking. The performance grade designation consists of a “PGXX-ZZ” designation, where “PG” stands for performance grade, “XX” is a number that corresponds to the high temperature performance grade and “ZZ” is the number related to the minimum low temperature grade.

Ground Tire Rubber

The generation of scrap tires in the US in 2009 was estimated to be more than 300 million tires, which represents approximately one tire per person (Rubber Manufacturers Association, 2009). Iowans generate around 3 million scrap tires annually according to the Iowa Department of Natural Resources website in 2013.

Modern tires are made up of many different components (Figure 2). The main components of tires are “vulcanized rubber, rubber filler, rubberized fabric, steel cord, fillers like carbon black or silica gel, sulfur, zinc oxide, processing oil, fabric belts, steel wire, reinforced rubber beads and many other additives”. Table 1 presents the typical

weight distribution of the components of a tire and it shows that tires are mostly composed of rubbers (Unapumnuk, 2006).

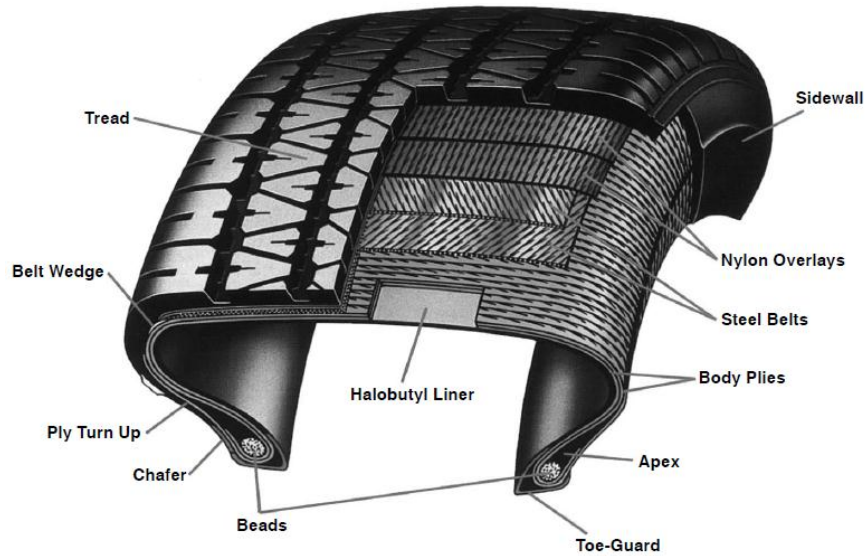


Figure 2. Cross section of a high-performance tire (Mark, 2005)

Table 1. Typical weight distribution of the various components of a tire

| Tire components | Percentage |
|---------------------------|-------------------|
| Natural rubber | 15-19 |
| Carbon black | 24-28 |
| Synthetic rubber | 25-29 |
| Steel cords | 9-13 |
| Textiles cords | 9-13 |
| Chemical additives | 14-15 |

From Unapumnuk, (2005)

Rubber is a type of elastomer, and as any elastomer it can go under large elastic deformations and return to its original shape. The ASTM D 6814 (2002) defines rubber as a natural or synthetic elastomer that can be chemically cross-linked/vulcanized to enhance its useful properties. Cross-linked rubber forms a strong three-dimensional chemical network. Rubber will swell in the presence of a solvent, but it will not dissolve and cannot be reprocessed by simply heating it (Hamed, 1992).

The amount of rubber that composes scrap tires makes them a potential source of raw material for the rubber industry. Moreover, landfills and legislation are requiring more economical and environmental friendly ways to dispose of scrap tires. There are many technologies to recover the rubber from scrap tires. Some of the methods that these technologies apply include retreading, reclaiming, grinding, pulverization, microwave and ultrasonic processes, pyrolysis, and incineration. The recycled rubber is generally known as ground tire rubber (GTR) (Isayev, 2005).

Two types of ground tire rubber can be obtained from scrap tire recycling. These are ambient ground tire rubber (ambGTR) and cryogenic ground tire rubber (cryoGTR). The processes from where these are obtained are different. Figure 3 shows the two types of ground tire rubbers that can be obtained from processing scrap tires.

Ambient GTR is obtained by the grinding of the ground tire rubber at or above ambient temperature, without the use of any cooling system to make the rubber brittle, through either cracker mills or a granulator. If the ambient GTR is ground using the granulator process, the rubber particles will tend to have a cut surface shape and rough texture. Meanwhile, if the ambient GTR is ground using cracker mills, its particles will

be long and narrow in shape with a high surface area (Recycling Research Institute, 2006).

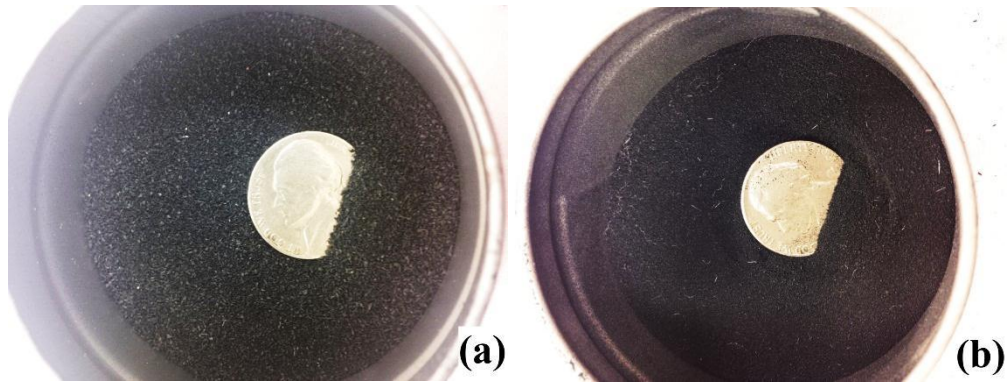


Figure 3. Types of GTR (a) Ambient GTR (b) Cryogenic GTR

Figure 4 shows the schematic of an ambient rubber processing plant, where the tires are fed into a shredder, which will reduce the tire into two inches size chips. Then the chips go into a granulator that makes them smaller than 3 /8 inches, at this stage most of the steel and fiber that compose the tire are freed. The steel is then removed through magnets and the fiber is shaken out or wind sifted. When the rubber is cleaner, it goes through finer grinding processes depending on the size desired, most of the mesh sizes range from 10 to 30. The usual equipment used to perform this fine grinding are: secondary granulators, high speed rotary mills, extruders or screw presses and cracker mills (Reshner, 2006).

Whereas, cryogenic GTR is obtained through a process where the scrap tire rubber is frozen using liquid nitrogen or other frozen method to a temperature below the glass transition temperature of the rubber to make it brittle like glass, and then the rubber is put in a hammermill and reduced to the desired particle size (Reschner, 2006).

Example of an Ambient Scrap Tire Recycling System

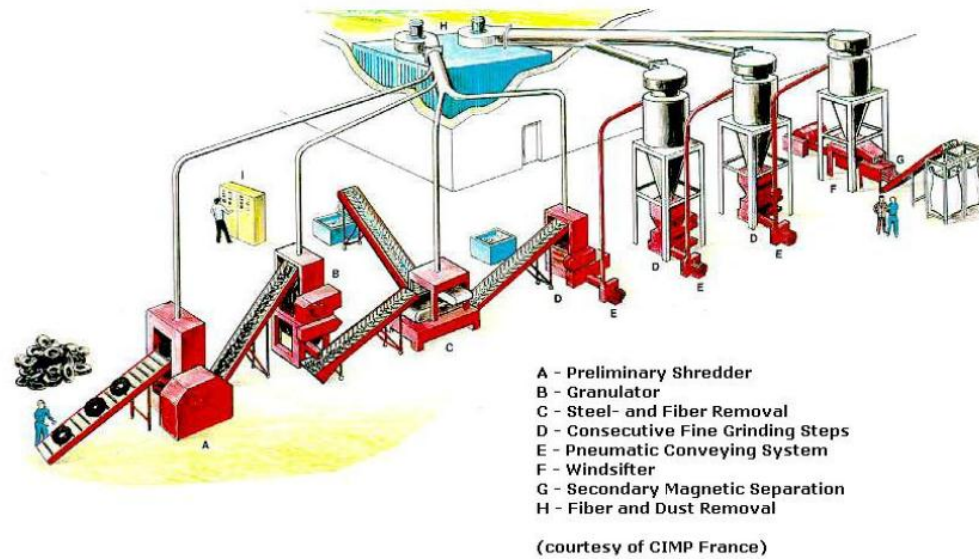
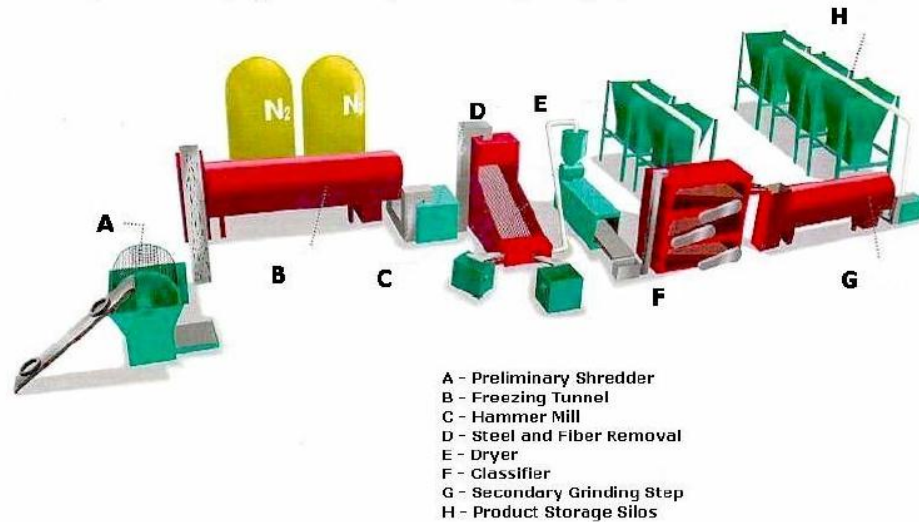


Figure 4. Ambient scrap tire processing plant schematics (Reschner, 2006)

Figure 5 represents the schematics of a cryogenic scrap tire processing plant, in which the tires are first fed to a shredder, like the first shredder found for the ambient scrap tire processing plant, reducing the tire rubber to two inch sized chips. These chips are then cooled to very low temperatures, approximately -120°C through a funnel system that has freezing elements like nitrogen. After being frozen, the rubber is then shattered with a hammermill system, then the steel and fibers are removed through magnets, aspiration and screening. Next, the rubber is dried and sieved into different particles sizes. The rubber particles obtained from the cryogenic method are even in size and smooth, with a low surface area. (Reschner, 2006).

Example of a Cryogenic Scrap Tire Recycling System



(Courtesy of RTI, Canada)

Figure 5. Cryogenic scrap tire processing plant schematics (Reschner, 2006)

Ground tire rubbers are usually used as an asphalt binder modifier due to its elastomeric properties which improves the performance of asphalt mixtures and at the same time it contributes to the reduction of the accumulation of scrap tires in landfills.

Asphalt rubber mixtures and asphalt rubber binders

Asphalt rubber started to be used as a binder in chip seal and dense and open graded asphalt concrete construction. The asphalt-rubber chip seal, or seal coat, is known as “asphalt-rubber interlayer”, which is placed beneath an asphalt concrete overlay, and it is intended to reduced reflection cracking in overlays. The hot-mix asphalt concrete made with asphalt-rubber binder is known by “asphalt-rubber concrete” in dense-graded mixes and “asphalt-rubber friction course” in open-graded mixes. (Shuler, 1986)

The early applications of asphalt rubber can be categorized as asphalt rubber concrete (ARC), open graded friction courses, stress absorbing membranes (SAM's), stress absorbing membrane interlayers (SAMI's), cape seals, three layer systems and waterproof membranes (FHWA, 2008). Figure 6 illustrates some cross-sections of the aforementioned applications of asphalt rubber technology.

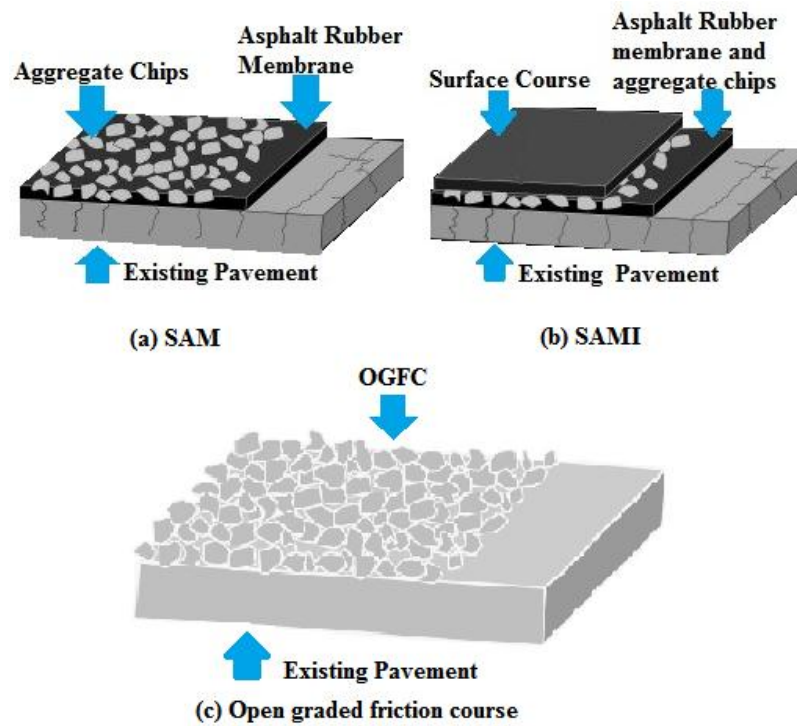


Figure 6. Different asphalt rubber applications

(Adopted from ARTS)

Asphalt rubber mixtures are highly resistant to oxidation and cracking due to the presence of the antioxidants of the carbon black in the rubber, high viscosities of asphalt rubber binders help in the rutting resistance of the mixture, while the elastic properties of rubber help to the resistance to reflective and thermal cracking of the pavement.

The production of asphalt rubber mixtures can be made through two processes. These are the wet process and the dry process. In the first process the crumb rubber is blended into the asphalt binder prior to the production of the mixes, whereas, in the second process the rubber is added to the aggregates before mixing it with the asphalt binder.

Use of asphalt rubber in the United States

Charles H. McDonald developed the wet process method in the mid-1960's. He developed commercial binder systems in conjunction with Atlos Rubber, Arizona Department of Transportation (Arizona DOT), and Sahuaro Petroleum and Asphalt Company. By mid-1970's, Arizona Refining Company (ARCO) also developed an asphalt rubber system. (Caltrans, 2002)

Arizona DOT carried several comprehensive researches on asphalt rubber between the mid-1970's and early 1980's, where they established that the rubber type, rubber gradations, rubber concentration, asphalt type, asphalt concentration, extender oils, reaction times and temperatures influenced the properties of the asphalt rubber binders (Caltrans, 2002). The common use of asphalt rubber binder during those years was as chip seals. However, by the beginning of 1990's one-inch thick asphalt-rubber mix overlays were preferred over the chip seals because the overlays provided smoother riding surface and produced less traffic noise. Both, the chip seals and the asphalt-rubber overlays provided retardation on the reflection of fatigue cracking and thermal cracking.

California Department of Transportation (CalTrans) started evaluating asphalt rubber as spray applications (chip seals, interlayers and cape seals) in 1970's and as hot

mix asphalt (dense-graded, open-graded, and gap-graded) in 1980's using the wet-process. CalTrans has reported that the use of asphalt rubber mixtures usually exhibits less distress, requires less maintenance and handles more deflections than regular dense-graded asphalt concrete and at least forty cities in California have asphalt rubber pavements (Caltrans, 2002).

Texas Department of Transportation (Texas DOT) also started using asphalt rubber in these applications around the same years as Caltrans. The most used application in Texas for asphalt rubber is in chip seals, since after many years of use of the technology they have concluded that asphalt rubber chip seals improve the resistance to fatigue cracking and raveling and at the same time the cost of placing if almost have of the cost of repaving (Estakhri et al, 1992). Dense-graded asphalt rubber hot mixes by the wet-process are also used by Texas DOT.

In 1979, Minnesota Department of Transportation constructed at least six asphalt rubber projects using the wet-process. These projects involved one dense-grade overlay, two SAM's and three SAMI's; however the results obtained for the SAM's were not encouraging, one was a disaster and the other a success. In the other projects the improvement on the resistance to reflective cracking was not considered enough to overcome the cost related to the technology.

In 1980's Kansas Department of Transportation (Kansas DOT) built five projects using asphalt rubber as interlayers, from those five projects only one presented better performance than the control mixes in the reduction of reflective cracking, whereas the others performed the same as the control mixes, thus Kansas DOT decided that the extra cost involved in asphalt rubber interlayer did not justify its use.

Between 1989 and 1990, Florida Department of Transportation (Florida DOT) constructed three asphalt-rubber demonstration projects using the Florida wet-process technology; these projects were one-dense graded and two open-graded friction courses.

In 1990, Iowa Department of Transportation (Iowa DOT) started studying laboratory asphalt rubber mixes through the wet-process. Between the years of 1991 and 1992, Iowa DOT constructed five projects using asphalt rubber binder in the pavements as chip seals, surface overlays and intermediate layers; these projects were built in Muscatine, Dubuque, Plymouth, and Black Hawk Counties. In 1992, Iowa DOT built two asphalt rubber overlay test sections in Webster County. In all these test projects the asphalt rubber pavements performance was better than conventional asphalt pavements in rutting, fatigue cracking, reflective cracking and better winter maintenance.

The Federal Highway Administration started several research studies about asphalt rubber in 1992, due to a federal government mandate to reduce the number of used tire stockpiles in the Intermodal Surface Transportation Efficiency Act (ISTEA) in 1991. The first phase of these research studies was carried by the University of Florida, where the common practices of that time were summarized and identification of research needs for a second phase were also established. The second phase was developed by Oregon State University in 1994 and concluded 1999, where guidelines for thickness design and construction and quality control were established, as well as long-term performance of mixes containing crumb rubber and the possibility of recycling mixes containing crumb rubber. The Western Research Institute (WRI) carried an evaluation study of asphalt rubber on the effect of the asphalt composition and time and temperature of reaction. The National Cooperative Highway Research Program (NCHRP) in 1994

synthesized the state of practice of asphalt rubber including all processes containing crumb rubber. However, before these studies were finalized the federal mandate on the use of recycled tires in asphalt pavement was revoked by the National Highway System (NHS) Designation act in 1995, but none the less the mentioned Act recommended in one of its sections that further research and development of tests and specifications for use of asphalt rubber in conformance with the SuperPave performance-based specifications should be done (FHWA, 2008).

Production of asphalt rubber mixtures and binders

The use of rubber in hot mix asphalt (HMA) is intended to improve the performance of HMA at high service temperatures by increasing its stiffness; also, to modify its performance at intermediate temperatures by increasing its elastic properties, thus improving its resistance to fatigue cracking.

Dry-process

A brief description of the dry process will be given in this section, since this technology was not used in during the course of this research. In the dry process the ground tire rubber is added to the aggregates in a 1-3 percentage by weight of aggregate. The usual aggregate gradation used in this method is a gap-graded gradation so the rubber particles can fit into the aggregate matrix. Coarse ground tire rubber of sizes about 2 mm to 4 mm are generally use in the dry-process. The dry process was developed in 1960's by the Swedish Company, EnviroTire, and it was commercialize under the name of PlusRide. A generic dry-process technology was then developed in the United States around 1980's and 1990's where the amount of ground tire rubber does not exceed the

2% by weight of aggregates, and it was used in experimental pavement sections by states like Florida, New York and Oregon (FHWA, 2008).

The Cold Regions Research and Engineering Laboratory (CRREL) of the U.S. Army Corps of Engineers evaluated the ice-bonding characteristics of several asphalt paving materials including the ones having rubber, like the PlusRide, as part of the Strategic Highway Research Program. During this evaluation the CRREL developed a new technology called the chunk rubber asphalt concrete, where a narrow gradation of aggregates is used, between 4.5 mm and 12mm aggregate size, and larger maximum sizes of crumb rubber than the ones used in PlusRide technology (Heitzman, 1992)

The asphalt rubber mixtures using the dry process can be produced by either batch or drum-dryer plants. The mixtures should be produced at 149°C – 177°C (300°F – 350°F). Laydown temperatures should be at least 121°C (250°F) and continuous compaction with the finishing roller is need until a temperature of at least 60°C (140°F) is reached to avoid swelling of the rubber particles (FHWA, 2008).

Wet-process

The first technology to apply the wet process was developed by Charles H. McDonald and was known as “McDonald process”. In the wet process, when the rubber is blended with the asphalt at high temperatures a non-chemical interaction occurs. Some components of the asphalt migrate due to diffusion into the rubber making it swell, becoming a gel-like material. The components of the asphalt that causes swelling on the rubber are the aromatic oils of the asphalt that form part of the maltenes fraction of the asphalt composition (Figure 7) (Heitzman, 1992).

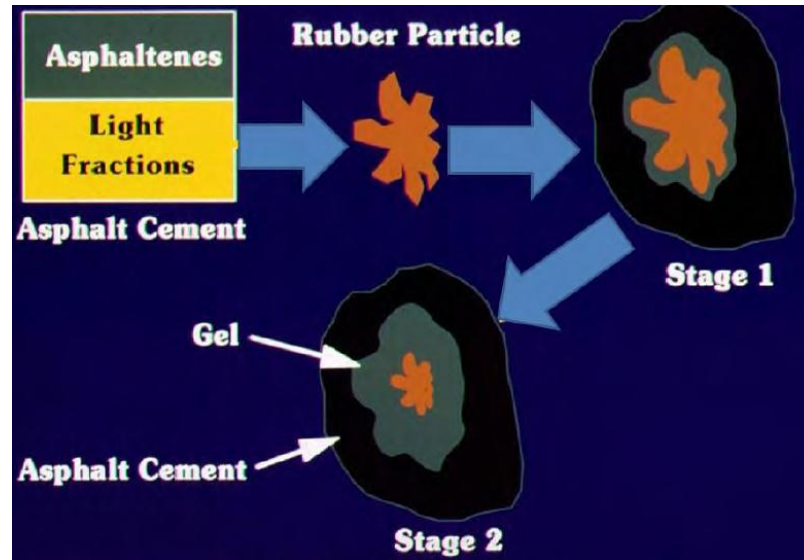


Figure 7. Depiction of reaction stages of asphalt and rubber
(RPA, 2011)

The state of Florida also developed the continuous blend using an 80 mesh for the ground tire rubber in 1980's. The differences between the McDonald's method and Florida's methods are in the percentage of ground tire rubber used, 8-10% in the Florida's method, versus 15-26% for McDonald's method; the size of the ground tire particles; the lower temperatures at which the blend is performed and the shorter reaction time in the Florida's method.

The amount of swelling of the rubber will depend on the particle's shape, surface area, type and amount of the rubber, type of asphalt, type and amount of shear mixing, blending temperature and time of interaction between the rubber and asphalt. The swelling of the rubber will increase the viscosity of the asphalt binder (Rahman, 2004).

Typical blending temperatures for asphalt rubber range between 160°C-205°C (320°F-400°F) for a minimum blending duration of 45 minutes. Higher temperatures than the aforementioned can lead to rubber depolymerization affecting its physical

properties (Hicks and Epps, 2000). Also higher temperatures will lead to excess of fumes and/or smoke (Hicks, 2002).

Addition of petroleum distillates or extender oils or other modifiers are added to the blend to reduce the viscosity and facilitate spray applications and promote workability.

Three categories of blending rubber and asphalt are the batch blending, continuous blending and terminal blending. The batch blending consists of the addition of the batches of rubber as it is mixed with the asphalt during the production of asphalt rubber. Continuous blending refers to the application of the wet process in a continuous production system developed by Florida in 1980's has mentioned before, whereas terminal blending is performed at the asphalt supply terminals using either the batch method or the continuous blending, one of its advantages is being able to store the asphalt rubber binder for extended periods of time, when compared to the other two methods (Heitzman, 1992 and FHWA, 2008).

The typical mixing temperature ranges for asphalt rubber mixes are: 163-191°C (325°F-375°F) for dense-graded asphalt rubber mixes and 135-163°C (275°F-325°F) for open-graded asphalt rubber mixes (Roberts, 2009).

Some of the limitations that asphalt rubber mixtures have presented are raveling and flushing, related to construction quality control; fatigue and reflection cracking when the correct thickness as not had been used; and tackiness of the asphalt rubber (Hicks, 2002).

On-site blending is considered the most efficient and economical way of combining ground tire rubber and asphalt. The on-site blending equipment must have the right components to successfully measure the right amount of rubber and asphalt to accommodate the needs of the in-site project (Figure 8 and Figure 9).



Figure 8. Example of on-site asphalt rubber blending plant
(CEI Enterprises, 2008)

Heated blending tanks are required to have agitation systems to keep the asphalt rubber blend homogenized until it is pumped to the hot plant, since depending on the specific gravity of the rubber and asphalt, the rubber particles can float on top of the tank or settle to the bottom of it. Screw auger systems are the most efficient way of agitation in horizontal blending tanks (Figure 10); these types of tanks are preferred due to the high surface area of material that provides better agitation with the screw auger system (RPA, 2011).



Figure 9. Example of asphalt rubber reaction tank
(CEI Enterprises, 2008)

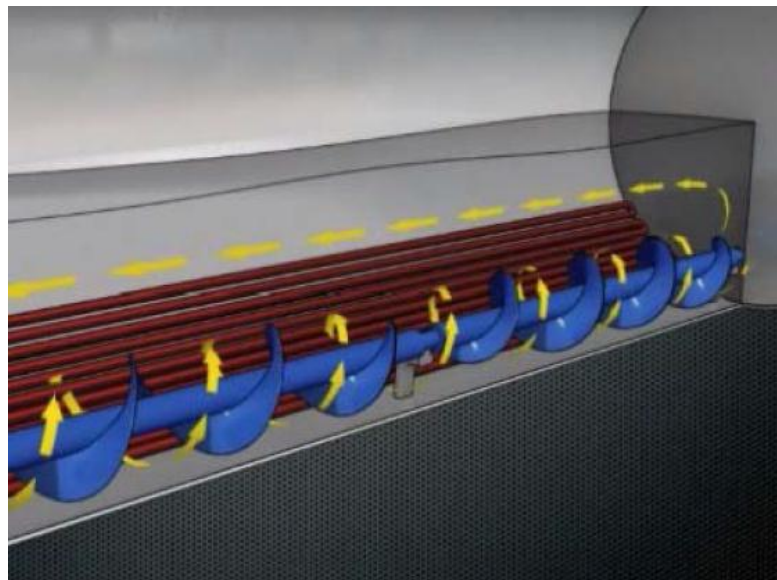


Figure 10. Depiction of auger system inside a horizontal blending tank
(RPA, 2011)

Asphalt rubber blending plants should consist of at least five main parts, being these the ingredient indicators, liquid asphalt meters for measurement and proportioning, crumb rubber hopper equipped with scales and meters, asphalt rubber binder blending equipment, asphalt rubber binder storage with internal agitation system, temperature control and metering heaters, heat exchangers, additive systems, mixing tank and asphalt rubber reaction tank.

In order to start the asphalt rubber mix production, special heavy-duty pumps, like the one showed in Figure 11 are attached from the asphalt rubber binder production equipment to asphalt cement plants, like a drum plants. The placing of asphalt rubber would vary depending on the application that it is being used for, but generally, its laydown temperature should not be less than 121°C (250°F) and conventional laydown machinery it is used and immediate rolling with a steel wheel roller is required. The use of rubber tire rollers is prohibited, since the asphalt rubber tends to build up on the roller tires (Way, 2011).

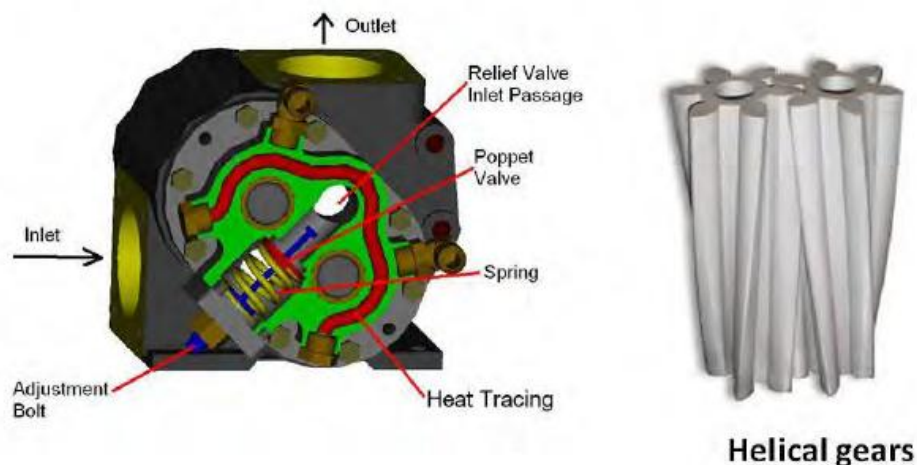


Figure 11. Special asphalt rubber pump with special heat tracing and relief valve
(Way, 2011)

Polyoctenamer (PO)

Polyoctenamer (PO) is a solid and opaque polymer, obtained from the cyclooctene monomer that is synthesized from 1,3-butadiene via 1,5-cyclooctadiene. The polymerization of the cyclooctadiene is achieved through a metathesis reaction, producing two types of macromolecules, linear and cyclic. The cyclic part of the macromolecules has a crystalline structure that exhibits low viscosity above its melting point. The cyclic part also contains a high amount of double bonds that can serve as cross-linking points and makes a rubbery polymer (Burns, 2000).

The level of crystallinity of PO will depend upon the cis/trans ratio of double bonds; this ratio is controlled by the polymerization conditions; thus the more trans-contents, the higher the crystallinity. Two degrees of crystallinity are usually obtained, one with a trans-content of 80% (cis-content of 20%), and the other with a trans-content of 60% (cis-content of 40%). The melting point of the former is about 54°C (129°F) and for the latter is about 30°C. PO is thermally stable to 271°C (520°F) (Burns, 2000).

The molecular formula of polyoctenamer is $-(C_4H_7=C_4H_7)-_n$ and its synthesis is shown in Figure 12.

PO is used in the asphalt industry to improve the tackiness of asphalt rubber. Its macrocyclic molecules when added to asphalt rubber will lower the initial viscosity during the initial mixing operation due to its crosslinking of the sulfur associated with the asphaltenes and maltenes in the asphalt and the sulfur in the surface of the ground tire rubber.

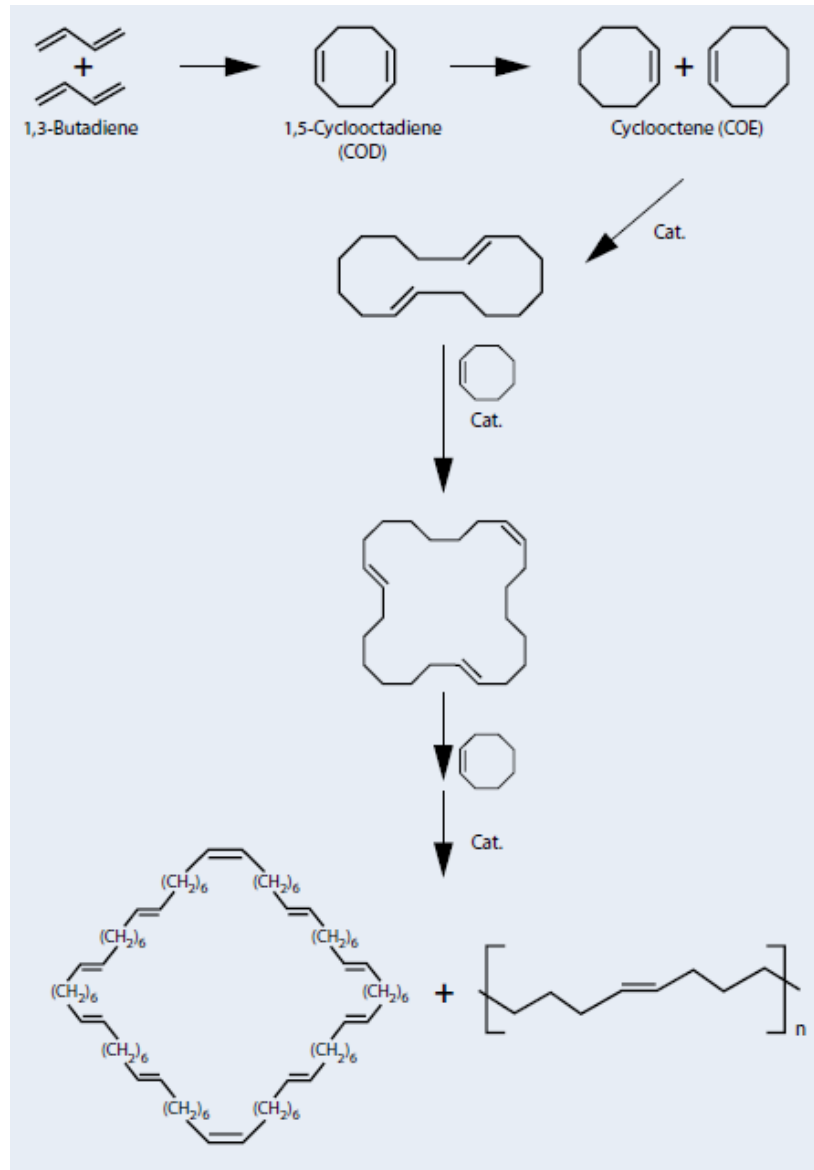


Figure 12. Synthesis of trans-polyoctenamer

As the polymerization spreads it will prevent the sinking of the rubber particles by increasing the viscosity. According to Rubber Asphalt Solutions, LLC (2010), this polymer chemically bonds to the ground tire rubber of the asphalt during its blending, it binds chemically to the aggregate reducing the stripping of the mixtures and will convert the thermoplastic asphalt to a thermoset polymer, that can help reduce cracking

and rutting. Other advantages claimed are easier, faster and more uniform mixing, faster paving, a superior surface finish, application at low road-surface temperatures, long service life, elimination of terminal blending and lower cost per mile.

The polyoctenamer is added in a dry particulate form to the molten asphalt cement at a temperature of about 163°C (325° F), although higher temperatures are allowed, the mixture being stirred or otherwise agitated until the polyoctenamer is dissolved and thoroughly mixed. The crumb rubber can be added to the hot asphalt cement with the polyoctenamer pellets (Figure 13) or after the polyoctenamer pellets have been dispersed and before or after they have been melted and mixed. The recommended dosage is 4.5% by weight of GTR (Burns, 2000).



Figure 13. Polyoctenamer pellets 80% crystalline

Many field trials were performed between the years of 1998 to 2003 throughout Canada and United States. Field trials were located in states like Arizona, New Jersey, Pennsylvania, Illinois, Ohio and Nebraska in United States; and in Ontario, Canada. All these field trials are performing as expected (Burns, 2004). Most of this research has

been done to study asphalt rubber modified with polyoctenamer used stiff binders with the following grades: PG58-28, PG64-22, PG70-28, PG76-28 and PG82-28.

CHAPTER 3. EXPERIMENTAL PLAN AND TESTING METHODS

Binders

Four types of binders were produced using a PG46-34 base asphalt binder from Flint Hills Resources, LP; two types of ground tire rubber (GTR), ambient GTR provided by Seneca Petroleum Company, Inc and cryogenic GTR from Lehigh Technologies, at 12% by weight of base asphalt and 4.5% of polyoctenamer (PO) by weight of GTR. The laboratory produced binders were given identification names to differentiate them. AMB stands for the asphalt rubber only containing AMBIent GTR, whereas CRYO is the asphalt rubber produced with CRYOgenic GTR. The AV is the Ambient GTR modified with PO and CV is the Cryogenic GTR modified with PO. Table 2 presents a matrix with the four types of binders developed and evaluated.

Table 2. Matrix of binders developed

| Base Asphalt PG46-34 | Rubber Type | | PO | |
|-------------------------|--------------------|----------------------|------|----|
| | 12% Ambient GTR | 12% Cryogenic GTR | 4.5% | 0% |
| AMB | ✓ | | | ✓ |
| CRYO | | ✓ | | ✓ |
| AV | ✓ | | ✓ | |
| CV | | ✓ | ✓ | |

The same procedure was used to produce the four types of binders. The following procedure was followed in the laboratory production of the binders.

- The base asphalt PG46-34 was preheated at 180°C.
- The base asphalt PG46-34 was placed in the shear mixer and stirred at a speed of 1000 rpm.

- The percentage of GTR by weight of base asphalt was slowly added into the heated asphalt; if the binder being prepared had PO, the amount of PO by weight of GTR was added too.
- After adding the GTR and PO, the speed of the shear mixer was increased to 3000 rpm.
- Because of the addition of GTR and PO a drop in the temperature occurred, thus the blend duration began when the temperature reached 180°C again and then the blending was maintained for an additional hour more; until then the blend was finished.
- Samples for unaged binder DSR testing and RTFO testing were taken right away after blending.

The four types of binders produced were then characterize and graded following the SuperPave binder grading specifications. Table 3 summarizes the experimental plan for characterizing the binders.

Table 3. Testing experimental plan for binder properties

| Binder Type | Test Method | | | | | | |
|-------------|-------------|----|------------|---------------|--------------|----------|-----|
| | Density | RV | DSR Unaged | DSR RTFO-Aged | DSR PAV-Aged | BBR | |
| | | | Gap | Gap | Gap | Temp °C. | |
| | | | 1mm | 1mm | 2mm | -24 | -30 |
| AMB | XXX | XX | XX | XX | XX | XX | XX |
| CRYO | XXX | XX | XX | XX | XX | XX | XX |
| AV | XXX | XX | XX | XX | XX | XX | XX |
| CV | XXX | XX | XX | XX | XX | XX | XX |

*where “X” represents one sample and the number of X’s within each cell represents sample size.

Density testing

The density of a material is defined as its mass per its unit volume; the densities are usually reported in units of kg/m³. The densities of the binders were determined following the standard test method for density of semi-solid bituminous materials

(Pycnometer method) describe in ASTM D70-97. In the procedure calibrated pycnometers are empty weighed with their stoppers, then completely filled with distilled water at the testing temperature, in this case 25°C and reweighed, both weights are recorded. Then, binder is poured into the pycnometer until filling three quarters of its volume, taking care of not having binder sticking to the walls of the pycnometer. The pycnometer is then allowed to cool down to the testing temperature (25°C) and when this temperature is reached the partially filled pycnometer its weight is recorded. After this, the partially filled pycnometer is then completely filled with distilled water at the testing temperature and the new weight is taken. The densities of the binder are then calculated using the following equation:

$$Density = \frac{C - A}{(B - A) - (D - C)} \cdot W_T$$

where:

A = weight of pycnometer with stopper,

B = weight of pycnometer completely filled with water,

C = weight of partially filled pycnometer with binder,

D = weight of completely filled pycnometer with binder and water,

W_T = density of water at testing temperature, 997 kg/m³ at 25 °C.

The densities of the binders are required during the SuperPave mix design procedure to properly determine the volumetrics properties of the mixes.

Viscosity testing

Viscosity is the resistance to flow of a liquid and it is usually defined as the ratio between the applied shear stress and the rate of shear, and its unit of measurement is Pascal second (Pa·s). The viscosities of the laboratory-produced asphalt rubber binders were measured using a Brookfield Rotational Viscometer and with the help of a temperature-controlled thermal cell to maintain the testing temperatures. The procedure followed is the outlined in the standard method for viscosity determination of asphalt at elevated temperatures using a rotational viscometer of ASTM D4402 (2002).

The measurement of the binders' viscosities are automatically done by the Brookfield Rotational Viscometer at the set rotational speeds and testing temperatures. For this experiment the testing temperatures used were 180°C, 185°C and 190°C. Three rotational speeds are usually used during this test; these are 10, 20 and 50 rpm. For each testing temperature a waiting time of about fifteen minutes is necessary to reach temperature equilibrium in the sample. When the viscosity has stabilized at each rotational testing speed, three viscosity readings are taken within one minute apart from each reading. At least three minutes of wait time is required when changing the rotational speed to start taking the viscosity readings.

Two samples were tested per each asphalt rubber binder and each of the tested samples weighted ten grams. Although a spindle number 27 is more common to be used for conventional asphalt binders testing, when this spindle number was tried an error was displayed in the Brookfield Rotational Viscometer, thus it was decided to change the spindle number to a lower number. The spindle size utilized then during the testing of the four types of asphalt rubber binders was a spindle number 21, and the change in

settings in the viscometer was made to take into account the new spindle number so a proper viscosity readings were obtained.

Dynamic Shear Rheometer (DSR)

The four asphalt rubber binders were tested in a dynamic shear rheometer. Two samples of unaged, RTFO aged and PAV aged materials were tested following the standard method of test for determining the rheological properties of asphalt binder using a dynamic shear rheometer (DSR) established in AASHTO T315 (2010) (Figure 14).



Figure 14. TA dynamic shear rheometer

The samples are prepared by pouring the asphalt rubber binders into silicone molds with the appropriate geometry for the type of material to be tested. The geometry of the samples of the unaged and RTFO aged materials is 25 mm in diameter, and the geometry of the PAV aged materials is 8 mm in diameter. The gap established by the standard to be used in the rheometer for sample testing is 1 mm for the unaged and RTFO aged materials, and 2 mm for PAV aged materials.

The complex shear modulus (G^*) and the phase angle (δ) of the samples are measured in the DSR. G^* is considered the total resistance of the binder to deformation when sheared at a certain frequency and temperature. Two components make the complex shear modulus, these are the storage modulus (G') and the loss modulus (G''); the first modulus is related to the elastic properties of the material, whereas, the second modulus relates to the viscous properties of the material. The phase angle is then the angle between the storage modulus (G') and the resultant complex shear modulus (G^*), the higher the phase angle the more viscous-like the material will behave; likewise the lower the phase angle the more elastic-like the material will behave.

The performance-graded asphalt binder specification uses the values of G^* and δ to determine the performance grade of the binders. The unaged and RTFO materials are related to the performance of the binders at their maximum design temperature for rutting. The criteria to grade the unaged materials is that $G^*/\sin \delta$ must be at least 1 kPa at 10 rad/s frequency for the testing temperature, ranging from 46°C to 82°C. For the RTFO aged materials, this criteria requires the $G^*/\sin \delta$ to be minimum 2.2 kPa at 10 rad/s frequency for the same testing temperatures used for the unaged materials. Meanwhile, PAV aged materials are tested at intermediate temperatures (between 40°C to 4°C) to estimate their fatigue cracking performance, they must have a $G^* \times \sin \delta$ maximum value of 5000 kPa at the same testing frequency rate of 10 rad/s used for unaged and RTFO aged testing.

The RTFO aged materials were obtained after the unaged asphalt rubber binders went through the aging process of the Rolling Thin Film Oven (RTFO), which simulates the aging of the binder at its early stages just after mixing and placement and before long-

term aging begins (Figure 15). The standard method that describes the test procedure followed in this study is AASHTO T240 (2010), which establishes the aging temperature to be 163°C (325°F) and the test duration of 85 minutes.



Figure 15. Rolling thin film oven

The PAV aged materials were procured from subjecting the RTFO aged materials to an aging process in the Pressurized Aging Vessel (PAV), in Figure 16 at a temperature of 100°C for 20 hours at a pressure of 2.1 MPa and degassed for 30 minutes in a vacuum oven at 170°C (Figure 17). The PAV aging simulates the in-service long-term aging of the binders for 8-12 years, and the standard practice followed in this study is outlined in AASHTO R28 standard (2010).



Figure 16. Pressurized aging vessel



Figure 17. Vacuum oven

Mass Loss

After the asphalt rubber binders are subjected to short-term aging in the RTFO, the original unaged weight of the binders is compared to the RTFO aged binders' weight to see if there is excessive mass loss or mass gain after the aging process. The

performance-graded asphalt binder specification established in AASHTO M320 (2010) requires a maximum change in mass either positive or negative of one percent.

Bending Beam Rheometer (BBR)

The thermal cracking performance of the asphalt rubber at low temperatures were evaluated according with the standard method of test for determining the flexural creep stiffness of asphalt binder using the Bending Beam Rheometer (BBR), AASHTO T313 (2000) (Figure 18). The testing temperatures for the BBR test are ten degrees higher than the performance grade at low temperatures; this is because the principle of time-temperature superposition is applied for the test, allowing the test to be run in shorter times at an elevated temperature rather the two hours that it would last if the test was run at the low temperature performance grade.



Figure 18. Bending beam rheometer

Two specimens of each asphalt rubber binder per testing temperature were prepared following the guidelines in the aforementioned standard. To determine the two testing temperatures a trial test was performed using two extra samples of one of the asphalt rubber binders; after the trial, the testing temperatures were determined to be minus 24°C and minus 30°C.

Two parameters are measured during the BBR testing; these are the creep stiffness (S) in units of MPa and the m-value, which is the slope of the logarithm of the stiffness curve and logarithm of the time. The total time duration of the test is 240 seconds, and the criteria to grade the asphalt is to look the values for the S and m-value at 60 seconds, where they need to be maximum 300 MPa and minimum 0.300, respectively.

Binders Master Curves

For the construction of binders master curves, frequency sweep tests in the DSR are performed at different testing temperatures. The geometry of the samples used for the three types of materials for each asphalt rubber binder (unaged, RTFO aged and PAV aged) was 25 mm diameter samples and 1 mm gap. Six testing temperatures and thirty one frequencies were used to test the asphalt rubber binders, their values are tabulated in Table 4.

Table 4. Parameters used for frequency sweeps

| Parameter | Values |
|-------------------------|--|
| Testing Temperature, °C | 20, 30, 46, 58, 70 and 82 |
| Frequency, Hz | 0.1, 0.1259, 0.1585, 0.1995, 0.2512, 0.3162, 0.3981, 0.5012, 0.631, 0.7943 1, 1.259, 1.585, 1.995, 2.512, 3.162, 3.981, 5.012, 6.31, 7.943 10, 12.59, 15.85, 19.95, 25.12, 31.62, 39.81, 50.12, 63.1, 79.43, 100 |

The time-temperature superposition principle was then used to construct the master curves, by finding the appropriate shifting factors and use them to multiply the testing frequencies to get the new shifted frequencies. The model used to obtain the shifting factors was the Williams-Landel-Ferry (WLF) equation, described as follows:

$$\log a_T = \frac{-C_1(T - T_{ref})}{C_2 + (T - T_{ref})}$$

where:

a_T = shift factor,

C_1 and C_2 = empirical constants related to the material,

T = Temperature in K,

T_{ref} = Reference temperature in K.

The data is at first manually shifted to find the appropriate shifting factors for each frequency that will allow the overlapping of the data. The WLF equation is then fitted to those shift factors to obtain the empirical constants C_1 and C_2 and determine how well the model fits the shifting factors obtained. The reference temperature selected to construct the master curves for the asphalt rubber binders was 20°C.

The Christensen-Anderson-Marasteanu (CAM) model is the most widely accepted to represent the time and temperature dependence of asphalt binders. The CAM model is defined as follows:

$$|G^*(\omega)| = G_g \left[1 + \left(\frac{\omega_c}{\omega} \right)^v \right]^{-\frac{w}{v}}$$

where:

$|G^*(\omega)|$ = absolute value of complex modulus as a function of frequency ω , Pa,

G_g = glassy modulus ($\log[G_g]$ is considered fixed at 1E9 Pa),

ω_c, ν, w = model fitting parameters.

After the data was shifted, the CAM model was used to get the model parameters and see how well this model fitted the data.

Mixtures

A coarse-graded aggregate mix gradation with a 19mm nominal maximum aggregate size was used to prepare the asphalt rubber mixtures; the gradation is presented in Table 5. Five types of aggregates were used in the asphalt rubber mixtures: 3/4" limestone, 3/8" limestone, quartzite, manufactured sand and natural sand. The limestone aggregates were from Martin Marietta Aggregates, the quartzite and manufactured sand were obtained from Manatts and the natural sand from Hallet Materials, all local aggregates from Ames, Iowa. Hydrated lime from Voluntary Purchasing Groups, Inc. was used to simulate the breakdown of limestone during handling and mixing in the field. Figure 19 illustrates the 0.45 power chart of the 19.0 mm mix gradation used in this study.

Blending of the binders was performed right before mixing was planned to be executed. It was procured to blend enough binder to have enough of the same batch of binder to prepare all the specimens for each mixture test.

Table 5. Aggregates Gradation

| Aggregate | | 3/4" LS | Quartzite | 3/8" LS | Man Sand | Nat Sand | Hydrated Lime |
|----------------------|----------------|-----------|-----------|-----------|-----------|-----------|---------------|
| | % Used | 25% | 30% | 12% | 18% | 14% | 1% |
| Sieve ^{.45} | U.S. Sieve, mm | % Passing | % Passing | % Passing | % Passing | % Passing | % Passing |
| 5.11 | 37.5 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| 4.26 | 25 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| 3.76 | 19 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| 3.12 | 12.5 | 36.3 | 99.6 | 100.0 | 100.0 | 100.0 | 100.0 |
| 2.75 | 9.5 | 15.8 | 84.3 | 99.8 | 100.0 | 100.0 | 100.0 |
| 2.02 | 4.75 | 1.6 | 14.8 | 70.7 | 94.4 | 97.9 | 100.0 |
| 1.47 | 2.36 | 0.8 | 2.9 | 19.4 | 63.6 | 87.2 | 100.0 |
| 1.08 | 1.18 | 0.7 | 2.0 | 7.9 | 37.5 | 72.0 | 100.0 |
| 0.79 | 0.6 | 0.6 | 1.7 | 5.9 | 19.7 | 43.1 | 100.0 |
| 0.58 | 0.3 | 0.6 | 1.4 | 5.3 | 8.7 | 12.6 | 100.0 |
| 0.43 | 0.15 | 0.5 | 1.1 | 4.9 | 4.5 | 1.5 | 99.0 |
| 0.31 | 0.075 | 0.5 | 0.8 | 4.5 | 3.5 | 0.7 | 98.0 |

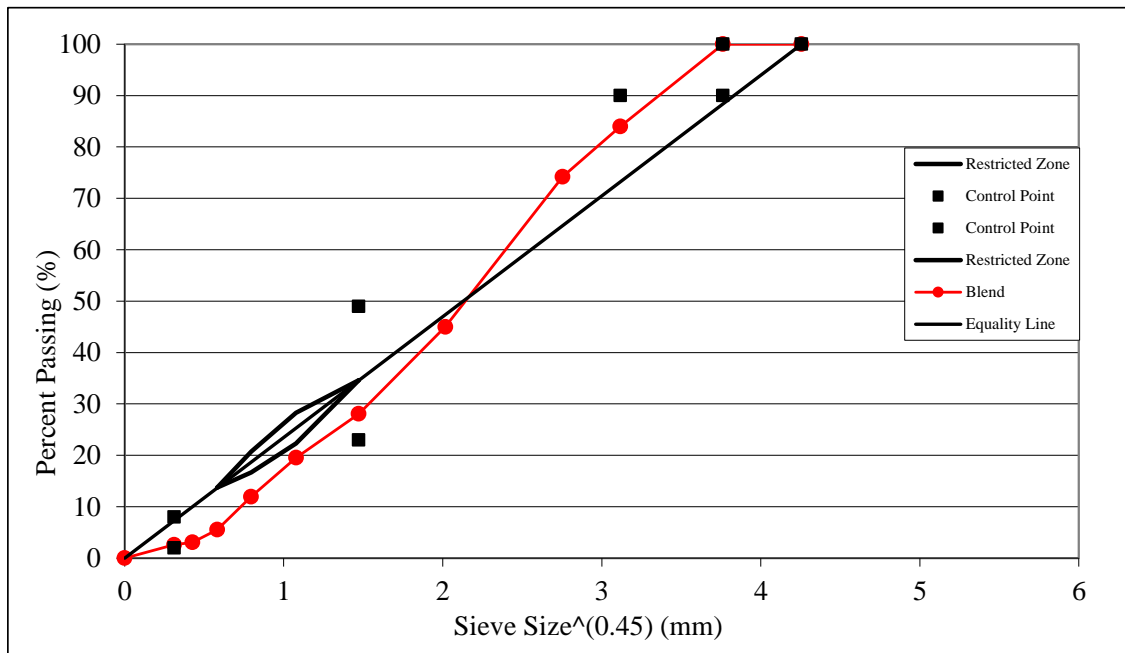


Figure 19. 0.45 power chart for 19.0 mm NMAS particle size distribution

The mixing temperature was set to be 180°C and the compacting temperature was 165°C. These temperatures were selected based upon past experience since the results of the testing of the binder viscosities did not yield reliable results, as it will be discussed in Chapter 4. The temperatures chosen to mix and compact are consistent with field practices when asphalt rubber is being produced (Roberts, 2009). The curing time was changed from the usual two hours that is used as standard practice to three hours of curing, due to the higher variability in the pre-trials for the Volumetric Mix Design.

The Volumetric Mix Design was performed in order to obtain the optimum binder content for the mix gradation; samples were compacted at 4.0 ± 0.5 percent air void content and tested. The optimum binder content was found to be 5.6 percent. With this binder content, four different types of asphalt mixtures were mixed and compacted at 7.0 ± 0.5 percent air void content. The SuperPave test procedures were followed to evaluate the performance of the four mixes.

The experimental plan followed to evaluate the performance of the four different asphalt rubber mixes is presented in Table 6. It should be noted that since the Dynamic Modulus (E^*) test is a non-destructive test, the same specimens are tested at the different testing temperatures. Also, after the E^* testing is finalized, the same specimens are used for the Flow Number test, which is a destructive test.

Table 6. Testing experimental plan for mixes performance

| Mix Type | Test | | | | | |
|-------------|---------------------------|--------|--------|-------------|------------------------------|---------------|
| | Dynamic Modulus (E^*) | | | Flow Number | Tensile Strength Ratio (TSR) | |
| | 4°C | 21°C | 37°C | 37°C | Conditioned | Unconditioned |
| AMB | XXX XX | XXX XX | XXX XX | XXX XX | XXX | XXX |
| CRYO | XXX XX | XXX XX | XXX XX | XXX XX | XXX | XXX |
| AV | XXX XX | XXX XX | XXX XX | XXX XX | XXX | XXX |
| CV | XXX XX | XXX XX | XXX XX | XXX XX | XXX | XXX |

*where “X” represents one specimen and the number of X’s within each cell represents sample size.

Dynamic Modulus (E^) test*

The dynamic modulus (E^*) describes the frequency-stiffness relationship of the asphalt mixtures. It is defined as the absolute ratio between the peak to peak stress amplitude and the peak to peak strain amplitude from the application of sinusoidal loads to the asphalt mixture. Along with the E^* , another property that is also measured during dynamic modulus testing is the phase angle (ϕ).

The Standard Method Test for determining Dynamic Modulus of Hot Mix Asphalt (AASHTO TP 62, 2010) with some modifications was followed to test the E^* of the asphalt rubber mixes. The modifications made were as the ones reported by Li and Williams (2012). The Dynamic Modulus testing was performed at three testing temperatures (4°C, 21°C and 37°C) and for nine frequencies (25, 20, 10, 5, 2, 1, 0.5, 0.2 and 0.1 Hz) at each temperature with a Universal Testing Machine (UTM-25) shown in Figure 20 and the stresses and strains responses were capture and digitally saved by a data acquisition system.



Figure 20. Universal testing machine (UTM-25) and data acquisition system

Five samples (100 mm diameter by 150 ± 2.5 mm height) for each mix type were mixed and compacted at the mixing and compaction temperatures (180°C and 165°C , respectively), the five samples had percent air voids ranging between 6.5 to 7.0 percent. Figure 21 shows the setup for the dynamic modulus testing.

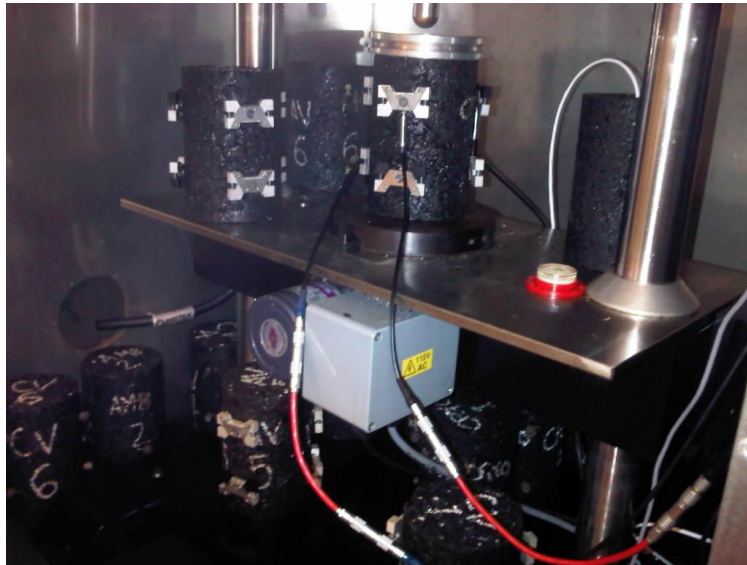


Figure 21. Dynamic modulus testing setup

Mixtures Master Curves

Similarly to the binder data obtained from the DSR, master curves can be built from the data obtained from the dynamic modulus testing (E^*) using the sigmoid function described as follows:

$$\log|E^*| = \delta \frac{(\alpha)}{1 + e^{\beta + \gamma \log f_r}}$$

where:

$|E^*|$ = Dynamic modulus,

α , β , δ and γ = fitting parameters,

f_r = reduced frequency.

The standard practice for developing dynamic modulus master curves for hot mix asphalt described in AASHTO PP62 (2010) was followed to construct the asphalt rubber mixtures master curves. The reference temperature was 21°C. The shifting factors were calculated and the second-order polynomial equation was applied to fit the master curve.

Flow Number test

The unconfined flow number test also known as repeated load permanent deformation (RLPD) simulates driving a heavy vehicle repeatedly over a pavement structure. Two outputs are obtained from the flow number test, these are the number of load cycles the pavement can tolerate before it flows and the permanent strain at which this happens.

The Flow Number test was chosen to be run at a temperature of 37°C, because the maximum annual average temperature (MAAT) for Central Iowa is 47.9°F (8.83°C) with a standard deviation of 1.6°F (-16.9°C), which gives a MAAT design of 50.53°F (10.3°C), that turns into an effective temperature of 37°C. The stress level used to perform the test was 600 kPa (87 psi) with a contact stress of 30 kPa (4.4 psi). Figure 22 presents a sample before and after being tested for flow number.



Figure 22. Flow number sample before and after testing

Tensile Strength Ratio

The moisture susceptibility of the compacted asphalt rubber mixtures was evaluated through the means of the standard method test for resistance of compacted hot mix asphalt to moisture-induced damage AASHTO T283-07 (2010). Two subsets of three specimens were tested for each asphalt rubber mixture, one subset moisture-conditioned and the other not-conditioned. The geometry of the samples tested were 100 mm of diameter by 63.5 ± 2.5 mm thick. The moisture conditioning of the moisture-

conditioned subsets consisted on partially vacuum saturate the specimens to a saturation degree between 70 and 80 percent, then the specimens were subjected to freezing temperatures for not less than 16 hours, and then submerged in a water bath at 60°C for 24 hours. Both subsets, conditioned and not-conditioned were submerged in a water bath at 25°C for two hours before being tested (Figure 23 and Figure 24).



Figure 23. Two hour moisture conditioning

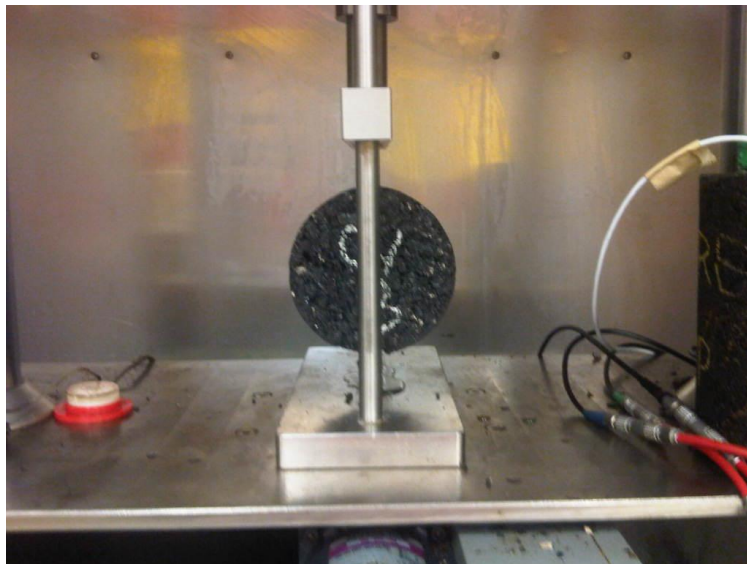


Figure 24. Indirect tensile strength test setup

CHAPTER 4. RESULTS AND DISCUSSION

This chapter will present and discuss the results obtained from the testing that were carried out according to the experimental plans presented in Chapter 3. The following sections will report first the binder testing results, which include viscosity, rheology, mass loss, thermal cracking and master curves of the four asphalt rubber binders that are the subject of this study. The mix performance testing will be reported later in this chapter, which includes dynamic modulus, master curves, flow number and indirect tensile strength ratio.

Binders Testing Results

Densities

The densities of the asphalt rubber binders were determined by means of the Pycnometer method described in the standard method ASTM D70-97 at 25°C. The average densities obtained for each binder type are summarized in Table 7. The densities obtained for the laboratory-produced asphalt rubber binders range from 1021 kg/m³ to 1030 kg/m³, these densities are higher than for conventional asphalts densities at 25°C, which usually have a range between 1007 kg/m³ to 1017 kg/m³. It appears that the addition of PO to asphalt rubber binders increases the density of the binders.

Table 7. Asphalt rubber binders densities

| Binder Type | Density (kg/m³) |
|--------------------|-----------------------------------|
| AMB | 1021 |
| AV | 1028 |
| CRYO | 1029 |
| CV | 1030 |

Viscosity

The viscosities of the asphalt rubber binders prepared were evaluated by means of the rotational viscometer using a Brookfield viscometer. The average viscosities for each asphalt rubber binder at each testing temperature are summarized in Table 8.

Table 8. Average viscosity of the asphalt rubber binders

| Test Temp. (°C) | Type of Binder | Average Viscosity, (Pa*s) | | |
|-----------------|----------------|---------------------------|--------|--------|
| | | @ Velocity | | |
| | | 10 rpm | 20 rpm | 50 rpm |
| 180 °C | AMB | 0.621 | 0.540 | 0.480 |
| | AV | 0.533 | 0.465 | 0.406 |
| | CRYO | 0.688 | 0.527 | 0.390 |
| | CV | 0.525 | 0.410 | 0.318 |
| 185 °C | AMB | 0.613 | 0.544 | 0.449 |
| | AV | 0.479 | 0.446 | 0.400 |
| | CRYO | 0.579 | 0.483 | 0.396 |
| | CV | 0.425 | 0.356 | 0.290 |
| 190 °C | AMB | 0.608 | 0.529 | 0.427 |
| | AV | 0.475 | 0.450 | 0.392 |
| | CRYO | 0.625 | 0.490 | 0.363 |
| | CV | 0.425 | 0.346 | 0.283 |

The standard practice to determine the viscosity of asphalt binders at different temperatures is to benchmark their mixing and compaction temperatures. The typical viscosity ranges are 0.17 ± 0.02 Pa*s for the mixing temperature and 0.28 ± 0.03 Pa*s for the compaction temperature for a set viscometer speed of 20 rpm. As Figures 19 to 21 show that these viscosities ranges were not reached for any of the four asphalt rubber binders, even at a testing temperature of 190°C for a speed of 20 rpm. Therefore, the decision to choose the mixing and compaction temperatures to be 180°C and 165°C was made based upon previous experience.

The very high viscosities encountered for the four types of asphalt rubber binders are due to the presence of the rubber particles in the binder. It should be noted that rubber does not melt in asphalt; instead it is a particle in suspension in the binder. Therefore it can be assumed that the particle effect of the rubber affects the readings of the rotational viscometer, thus higher viscosities values will be obtained than what truly is the viscosity of the binder without the particle effect of the rubber.

Even though the targeted viscosities were not obtained at the required speed to determine the mixing and compaction temperatures, the trend followed by the viscosities of the binders is consistent with what is expected to be the behavior of asphalt binders, at higher temperatures and higher rotational speed the viscosities are lower.

Also, as seen in Figure 29, the ambient GTR binder had the highest viscosity compared to the rest of the asphalt rubber binders; this phenomenon was expected since the ambient GTR particles are bigger in size distribution than the cryogenic GTR.

It should be noted that the addition of PO to the asphalt rubber seemed to help improve the viscosities of the asphalt rubber by reducing them; this was observed for both types of rubber. Of the four asphalt rubber binders, the cryogenic GTR and PO binder showed the less viscous behavior. Thus, it can be suspected that PO has greater influence in reducing the viscosity of cryogenic GTR binders than ambient GTR binders. The reduction on the viscosity of the asphalt rubber binders can be helpful in reducing the mixing and compaction temperatures. However, to be able to reduce the mixing and compaction temperatures, the reduction in viscosity must be significant enough to obtain the ideal range of viscosities needed.

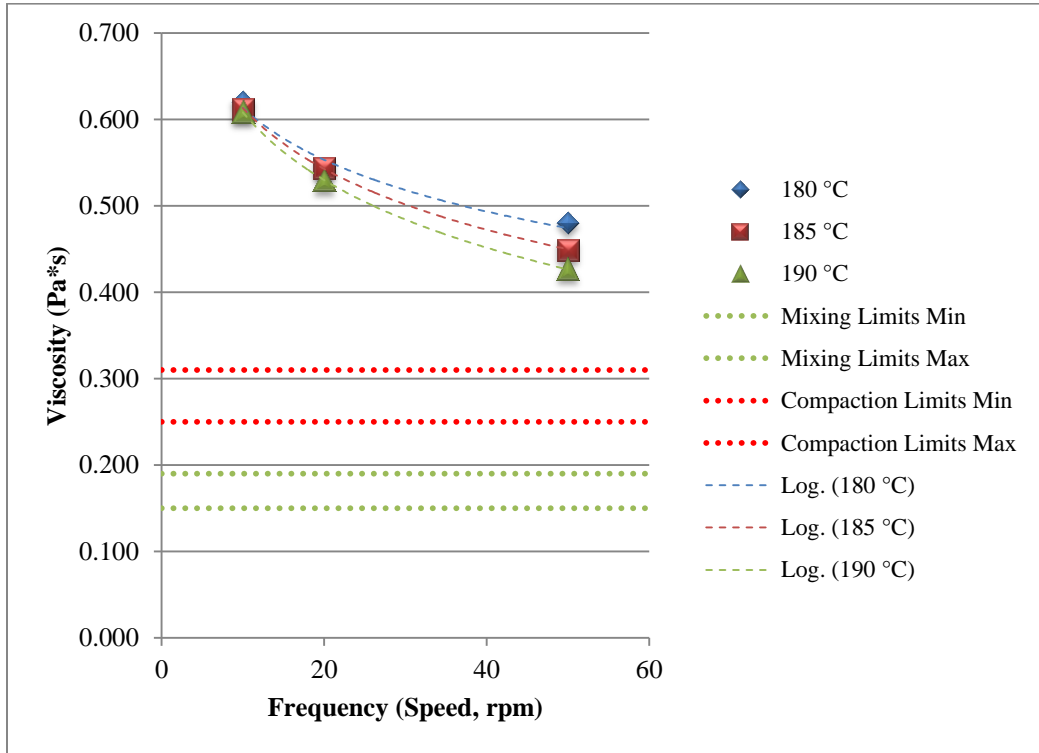


Figure 25. Average viscosities of AMB binder

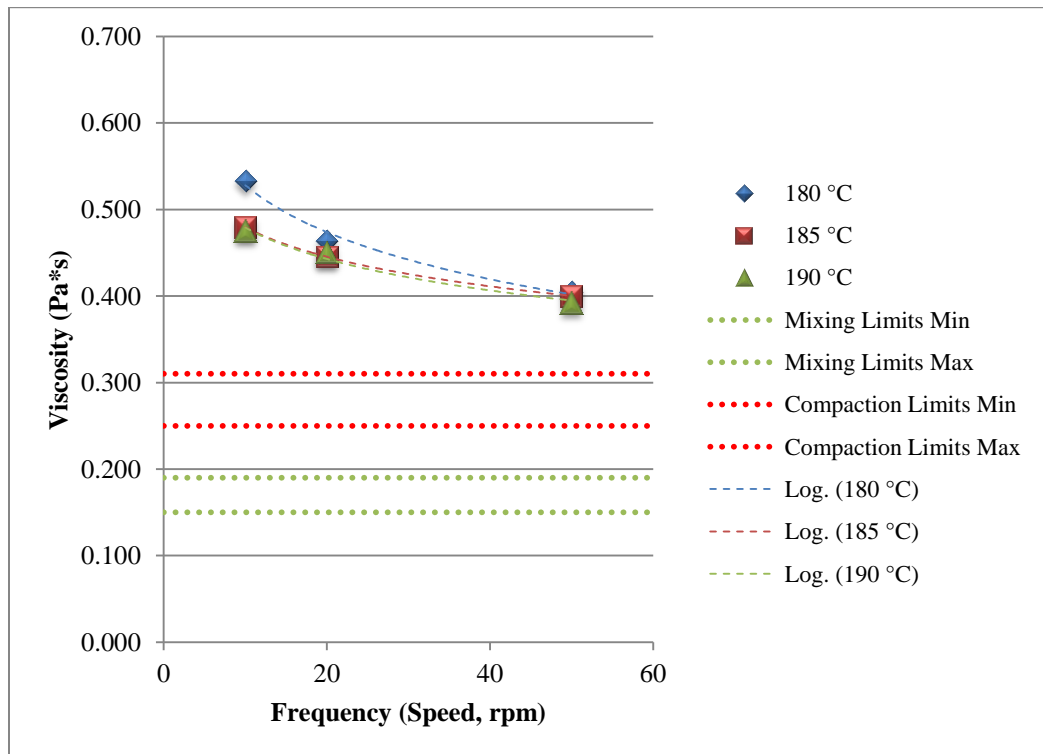


Figure 26. Average viscosities of AV binder

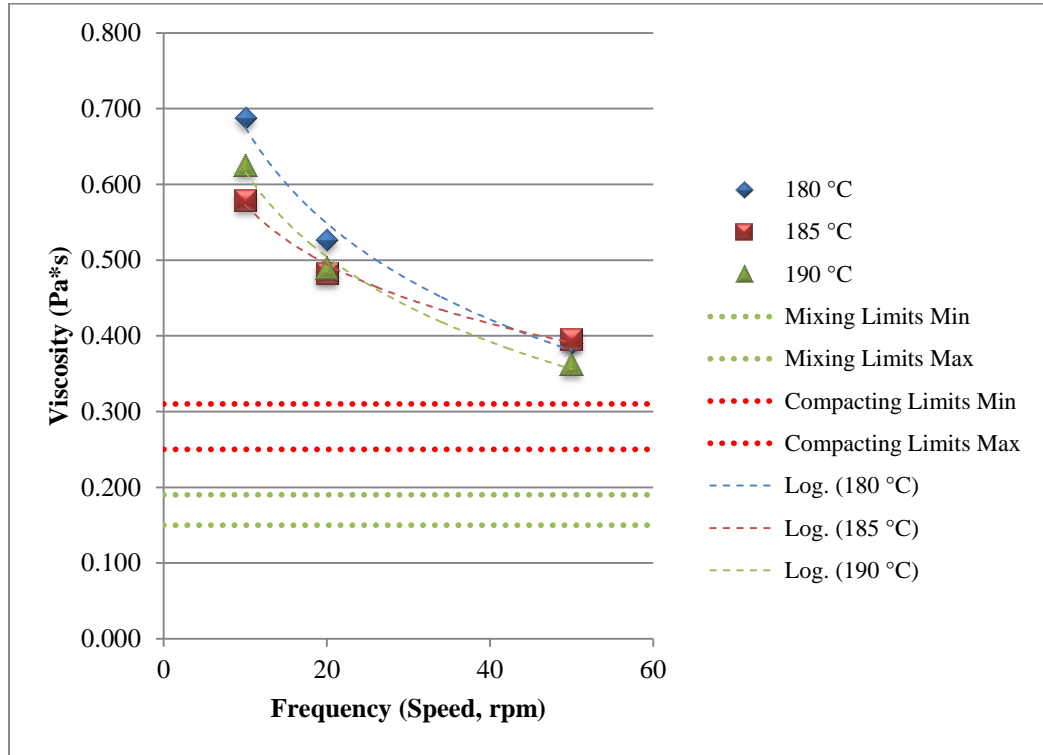


Figure 27. Average viscosities of CRYO binder

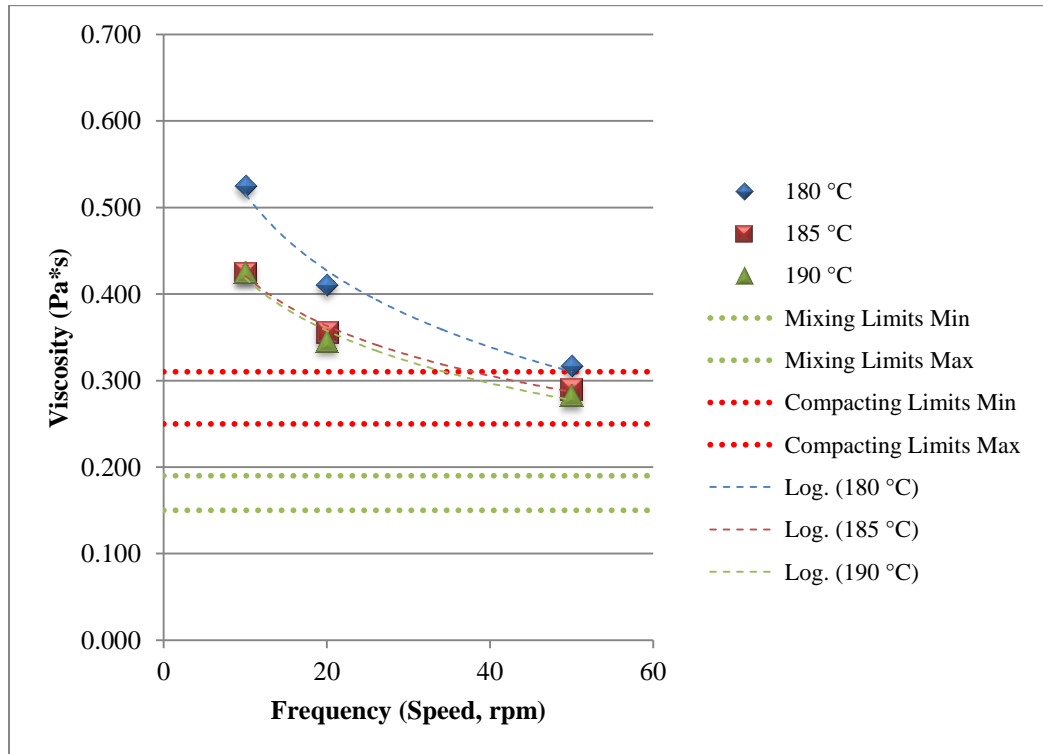


Figure 28. Average viscosities of CV binder

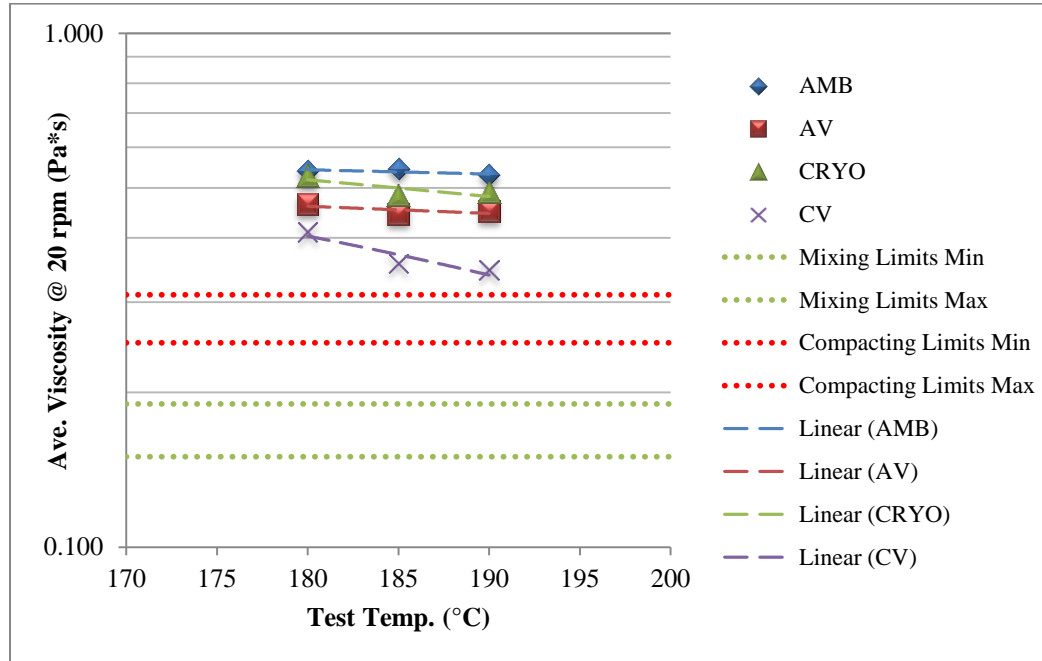


Figure 29. Comparison of viscosities at a testing rotational speed of 20rpm

One must keep in mind that the viscosities reported have the particle effect of the rubber, therefore a new method or better way to measure the true viscosity of the binder should be implemented for asphalt rubber binders.

The statistical analysis performed to look at the effects on the viscosities of the four asphalt rubber binders is summarized in Table 9. It was found that the differences between binder types is statistically significant, thus a least square means comparison was made, shown in Table 10. The results of the comparison between binder types are that ambient GTR binder and cryogenic GTR binder had no statistical significant difference in their viscosities, however, the two asphalt rubber binders with PO were found to be different from each other and from their similar asphalt rubber binder without PO.

Table 9. Binder viscosities ANOVA table

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|------------------------------|----|----------------|-------------|----------|----------|
| Binder Type | 3 | 263981.97 | 87994.0 | 62.8434 | <.0001* |
| Temperature (°C) | 2 | 24330.81 | 12165.4 | 8.6883 | 0.0006* |
| Speed (rpm) | 2 | 334731.38 | 167365.7 | 119.5290 | <.0001* |
| Binder Type*Temperature (°C) | 6 | 5391.26 | 898.5 | 0.6417 | 0.6963 |
| Binder Type*Speed (rpm) | 6 | 35152.03 | 5858.7 | 4.1841 | 0.0018* |
| Temperature (°C)*Speed (rpm) | 4 | 6279.09 | 1569.8 | 1.1211 | 0.3576 |
| Error | 48 | 67210.07 | 1400.2 | | |
| C. Total | 71 | 737076.61 | | | |

*statistically significant at $\alpha < 0.05$

Table 10. Binder viscosity least square means differences due to binder type

| Q | α | | | |
|---------|----------|---|---|---------------|
| 2.66137 | 0.050 | | | |
| Level | | | | Least Sq Mean |
| AMB | A | | | 534.44444 |
| CRYO | A | | | 504.44444 |
| AV | | B | | 449.49074 |
| CV | | | C | 375.27778 |

Levels not connected by same letter are significantly different.

To better understand the trends and relationships between the binder types, a least square means plot is shown in Figure 30. From this plot one can observe that the cryogenic GTR binder had the lowest mean average viscosity from the four types of asphalt rubber binders, and that the mean average viscosities for the asphalt rubber binders with PO were lower than the ones without PO. Thus it can be inferred that PO will decrease the viscosity of asphalt rubber binders containing the PG46-34 base asphalt.

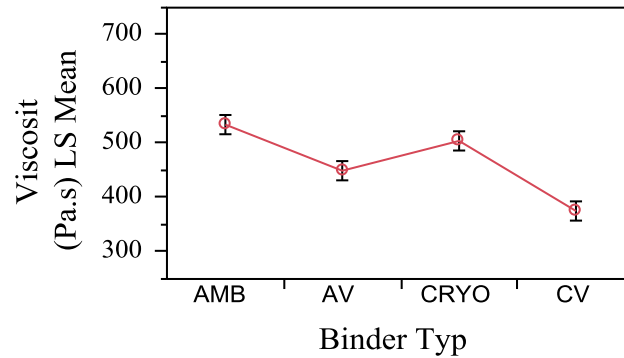


Figure 30. Binder type least square means plot

From the analysis of variance analysis (ANOVA) it was also found that there are significant differences for the testing temperature and the testing rotational speed. Although these were expected, when looking at the testing temperatures, one can see in the least square means comparison (Table 11) that the testing temperatures of 185°C and 190°C had no statistical significant difference. This can be better seen in the least square means plot presented in Figure 31. A possible explanation to this phenomenon is the high testing temperatures and the type of base asphalt binder used, which is a PG 46-34 and known for being a softer binder. This might indicate that the binder's viscosity will no decrease any further if the testing temperature increases beyond 185°C.

Table 11. Binder viscosities least square means due to testing temperature

| Q | α | | |
|------------|----------|---|---------------|
| 2.41849 | 0.050 | | |
| Level (°C) | | | Least Sq Mean |
| 180 | A | | 491.80556 |
| 185 | | B | 455.00000 |
| 190 | | B | 450.93750 |

Levels not connected by same letter are significantly different.

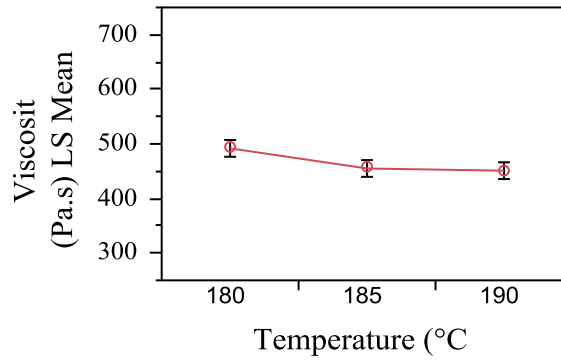


Figure 31. Testing temperature least square means plot

Table 12 and Figure 32 present the least square means comparisons of the binder viscosities due to testing rotational speed factor. It is observed that the viscosities for each level of this factor are statistically different between them as expected. The higher the testing rotational speed the lower the viscosity of the binder, due to the high shear stresses at which the binder is being subjected.

Table 12. Binder viscosities least square means due to testing rotational speed

| Q | α | | | |
|-------------|----------|---|---|---------------|
| 2.41849 | 0.050 | | | |
| Level (rpm) | | | | Least Sq Mean |
| 10 | A | | | 549.65278 |
| 20 | | B | | 465.45139 |
| 50 | | | C | 382.63889 |

Levels not connected by same letter are significantly different.

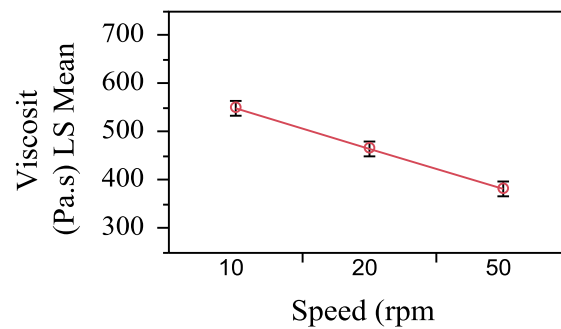


Figure 32. Testing speed least square means plot

The analysis of variance (ANOVA) table also shows that the interaction between the binder type and the testing rotational speed is statistically significant. Figure 33 presents the least square means plot for this interaction. The slopes of the viscosities for each type of binder at the testing rotational viscosities confirm that there is interaction between these two factors, because none of the slopes are parallel.

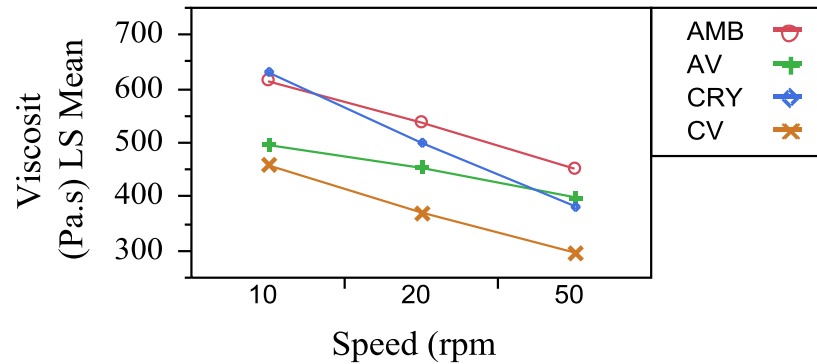


Figure 33. Binder type * speed least square means plot

Dynamic Shear Rheometer (DSR)

Each of the four binders was tested at high and low temperatures in the Dynamic Shear Rheometer. The rheological properties of unaged and RTFO aged samples of the four binders were evaluated for high temperatures. Meanwhile, the rheological properties of PAV aged samples were evaluated at low temperatures. From the results of the rheological properties, continuous grading of the four binders can be performed.

High Temperatures

The results obtained from the DSR testing at high temperatures are presented in Table 13. Results on the continuous grading for the base asphalt PG46-34 (presented

elsewhere) are also shown in aforementioned table as comparison on how ground tire rubber (GTR) can increase the binder's rheological properties at high temperatures.

From Table 13 it can be seen that unaged ambient GTR binder high continuous grading is 0.6 degrees higher compared to the unaged cryogenic GTR binder. When comparing the effect of PO on the unaged asphalt rubber blends, it can be seen that PO will increase the continuous grading for both ambient and cryogenic GTR blends.

Table 13. High temperature continuous grading of binders

| Blend Type | High Temperature Continuous Grading, °C | |
|-----------------|--|-----------|
| | Unaged | RTFO Aged |
| PG 46-34 | 50.9 | 53.0 |
| Amb | 66.7 | 67.0 |
| AV | 67.2 | 68.3 |
| Cryo | 66.1 | 66.7 |
| CV | 66.5 | 67.9 |

As for the RTFO aged material, it is expected that the resulting grading would be higher than for the unaged material, this trend is observed for all the binders. However, even though the binders with PO became stiffer after being RTFO aged, with respect to their respective unaged binder, the magnitude of how much stiffer they got is higher than the binders that did not have PO. These differences can be easily observed in Figure 34.

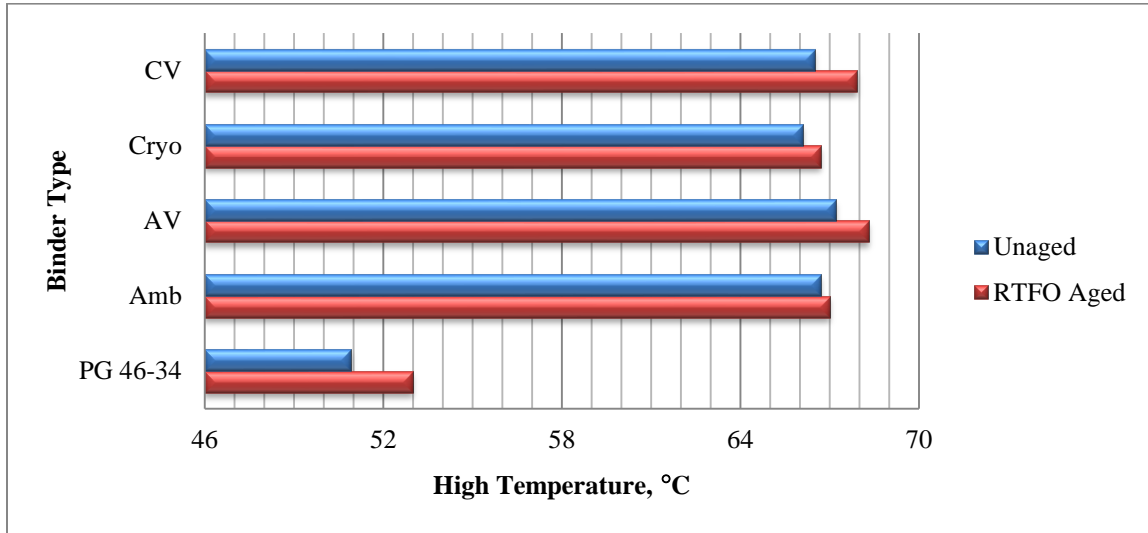


Figure 34. High temperature continuous grading

From Figure 34, it can also be observed how the two types of ground tire rubber increased the performance grading on the high temperature of the base asphalt, an increase in three grades, from being a PG46 to a PG64.

Intermediate Temperatures

The binders were tested in the dynamic shear rheometer (DSR) after being PAV aged at 100°C, at intermediate temperatures as required by the AASHTO M320 standard specification (2010) to be able to grade the binders. The results from the DSR are tabulated in Table 14. Due to equipment constraints all the binders were tested just from 16°C until 7°C, most of the samples did not fail at 7°C, except for two samples. Thus, the results were averaged and the failure temperatures for those samples that did not fail at 7°C were estimated. Likewise for high temperatures, Table 14 also presents the intermediate temperature grading for the PAV aged material of the base binder PG46-34

reported elsewhere, so it can be observed how GTR affects the intermediate temperature grading on the binders.

Table 14. Intermediate temperature continuous grading of PAV aged binders

| | Intermediate Temperature |
|-------------------|---------------------------------|
| | Continuous Grading, °C |
| Blend Type | PAV Aged |
| PG 46-34 | 9.0 |
| Amb | 5.7 |
| AV | 6.8 |
| Cryo | 6.4 |
| CV | 6.8 |

From Table 14, it can be seen how both GTRs improve the intermediate temperature grading of the binders by lowering the continuous performance grade. However, it seems that ambient GTR lowers the continuous grading a bit more than cryogenic GTR does. When looking at the effects of PO on the asphalt rubber blends, it is noticeable that it causes an increase on the intermediate continuous grading of the binders, however it does not affect the final performance grading because all the binders will have to be reported as being a 7°C PAV intermediate temperature grade. Figure 35 illustrates graphically the trends mentioned, it can also be observed how the GTR increases by one grade the intermediate temperature performance of the base binder PG46-34, that had an original PAV intermediate temperature grade of 10°C.

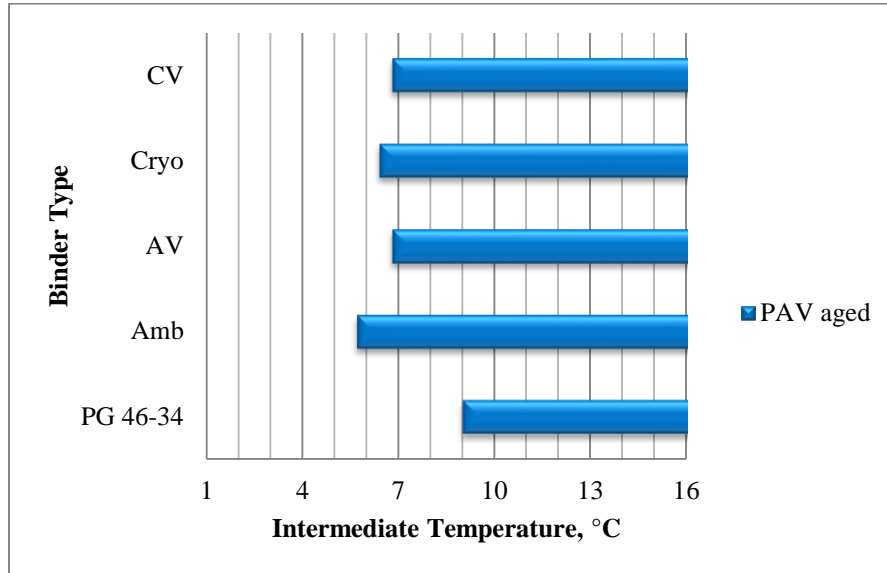


Figure 35. Intermediate temperature continuous grading for PAV Aged binders

Mass Loss

After performing the RTFO aging procedure, the mass loss of the four types of binder was computed, and these are tabulated in Table 15. The percentage mass losses obtained for the asphalt rubber binders with PO did not meet the specified mass loss criteria of no more than one percent of mass loss in AASHTO M320 standard specification for binder grading. The percentage mass loss of the base asphalt PG 46-34 reported elsewhere is presented as general reference.

Table 15. Summary of average percentage mass loss

| Binder Type | Average % Mass Loss |
|-----------------|---------------------|
| PG 46-34 | 0.43% |
| AMB | 0.86% |
| AV | 1.24% |
| CRYO | 0.81% |
| CV | 1.28% |

From the results presented in Table 15, it can be thought that PO increases the mass loss in the binders, this trend is observed in both types of GTR binders. Figure 36 illustrates graphically the mass loss percentage of the four types of RTFO aged asphalt rubber binders. However, it is believe that the high percentage of mass loss for the four types of binders is not completely due to the evaporation of the volatiles in the asphalt rubber, but due to swelling of the rubber while in the RTFO oven. This swelling can make the asphalt rubber film that coats the bottles during the test to be thicker and somehow making the binder to overflow the bottles, and thus binder is spilled inside the oven during the test. One can hypothesize that this swelling is increased when PO is added, promoting more spillage of the binder during the testing.

Bending Beam Rheometer (BBR)

The results obtained from the Bending Beam Rheometer (BBR) testing are summarized in Table 16. The continuous grading for low temperature of the base asphalt PG 46-34 was reported elsewhere, and it is just presented as a general reference.

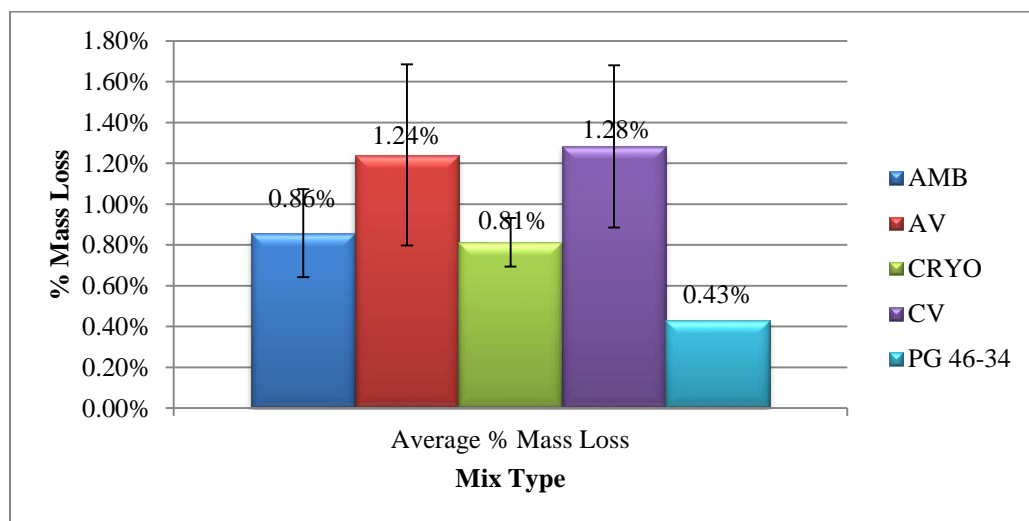


Figure 36. Average percentage mass loss

Table 16. Low Temperature continuous grading of binders

| | Low Temperature |
|-------------------|------------------------|
| | Continuous Grading, °C |
| Blend Type | BBR results |
| PG 46-34 | -36.2 |
| Amb | -38.9 |
| AV | -37.8 |
| Cryo | -37.3 |
| CV | -35.8 |

The two binders that contained PO graded lower when compared with their respective type of binder but without PO. The resistance to thermal cracking is somehow affected with the addition of PO to the asphalt rubber. However, the performance grade for the four types of asphalt rubber binders was not affected, remaining a -34 performance grade for creep stiffness. It is known that when binders are polymer modified the performance of the binder can improve on one side of the temperature (high or low), but for the other temperature (low or high) the performance typically diminishes. This situation was not observed for the binders in this study.

Figure 37 shows that the binder with ambient GTR had the better thermal cracking temperature performance of the four binders, and the binder with cryogenic GTR and PO had reduced thermal cracking temperature performance of the these binders.

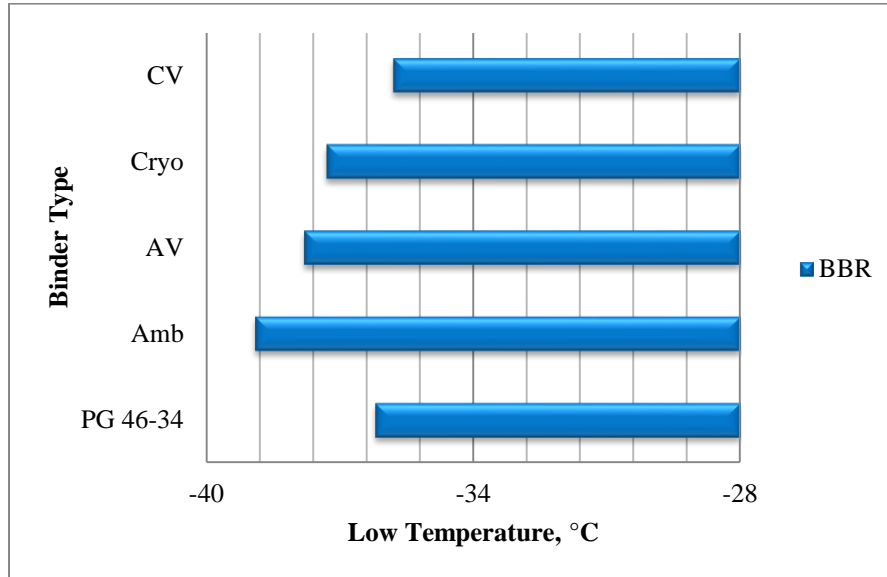


Figure 37. BBR low temperature continuous grading

Asphalt Binders Master Curves

Frequency sweeps were performed for each type of asphalt rubber binder through a DSR, the results obtained were used to construct the master curves of the unaged, RTFO aged and PAV aged binders for the rheological property of the complex modulus G^* . These master curves were all developed using a reference temperature of 20°C and are presented in Figures 36 through 38.

The four unaged asphalt rubber binders presented similar rheological performance for their complex modulus (G^*) at low, intermediate and high temperatures. However, it is noticeable that the cryogenic GTR binder presents somewhat lower complex modulus values compared to the other three asphalt rubber binders, this difference in stiffness is more noticeable at high temperatures. In contrast, the cryogenic GTR and PO binder behaves stiffer than its version without PO, the increase in stiffness can be due to the addition of PO. However, this trend was not observed for the asphalt rubber with

ambient GTR, since the master curves of both binders, with and without PO are one over the other. For RTFO aged materials, as expected the complex modulus for the four asphalt rubber binders increased, meaning that the binders got stiffer due to aging. The same trend observed for the unaged materials was observed for the RTFO aged materials, being cryogenic GTR softer than the rest of the asphalt rubber binders, which will impact negatively the performance of the binder for rutting resistance.

The trend observed in the unaged and RTFO aged materials, of the cryogenic GTR binder, being softer than the rest of the asphalt rubber binder is observed as well for the PAV aged materials. The complex modulus for the four asphalt rubber binders increased as expected due to the PAV aging.

One possible explanation on why PO seems to affect only the stiffness of the cryogenic asphalt rubber rather than both types of GTRs can be due to the difference in size distributions of the rubbers; cryogenic GTR is a finer material than ambient GTR which makes it easier for the PO to interact with it than with ambient GTR.

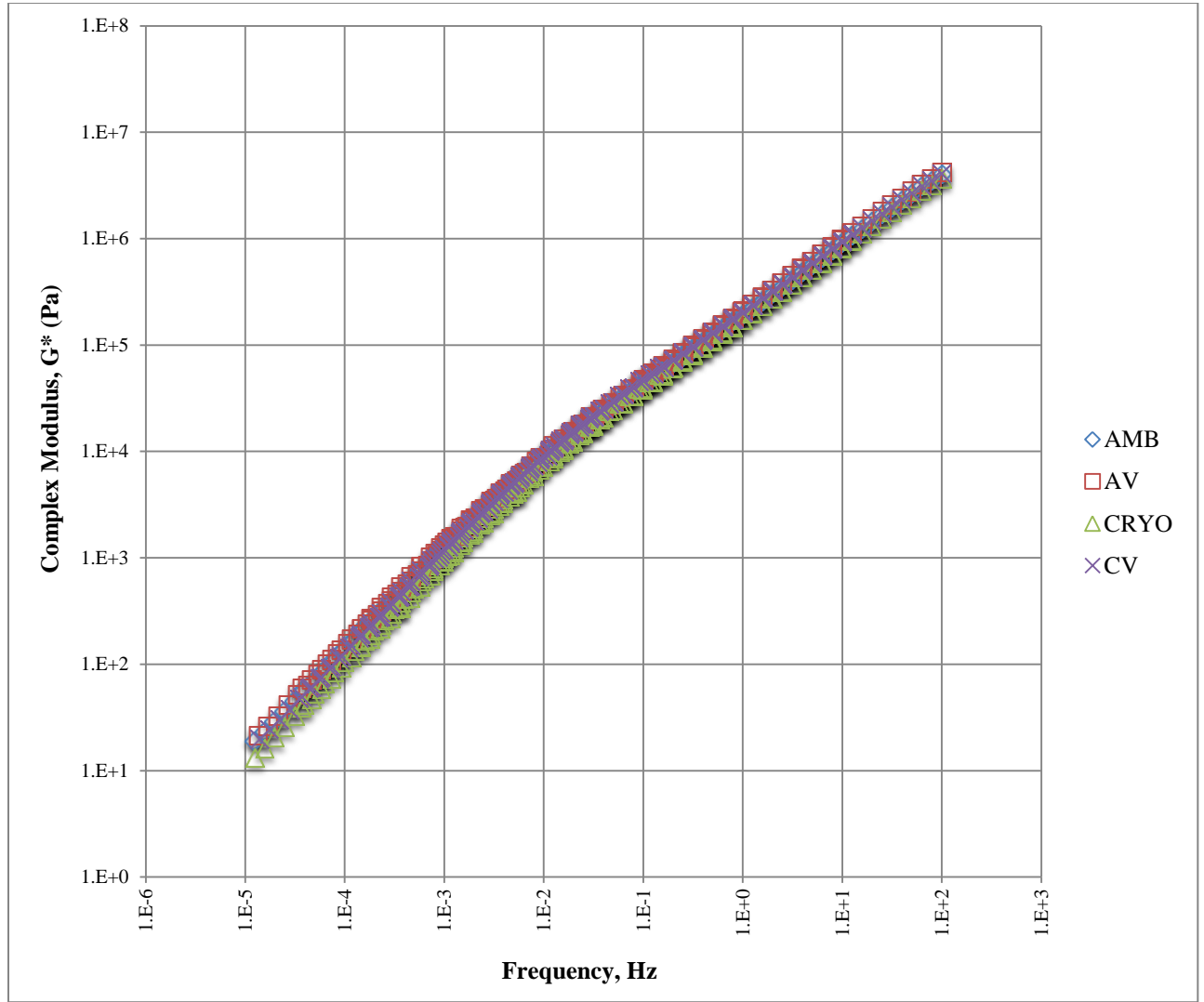


Figure 38. Master curves of Unaged asphalt rubber binders

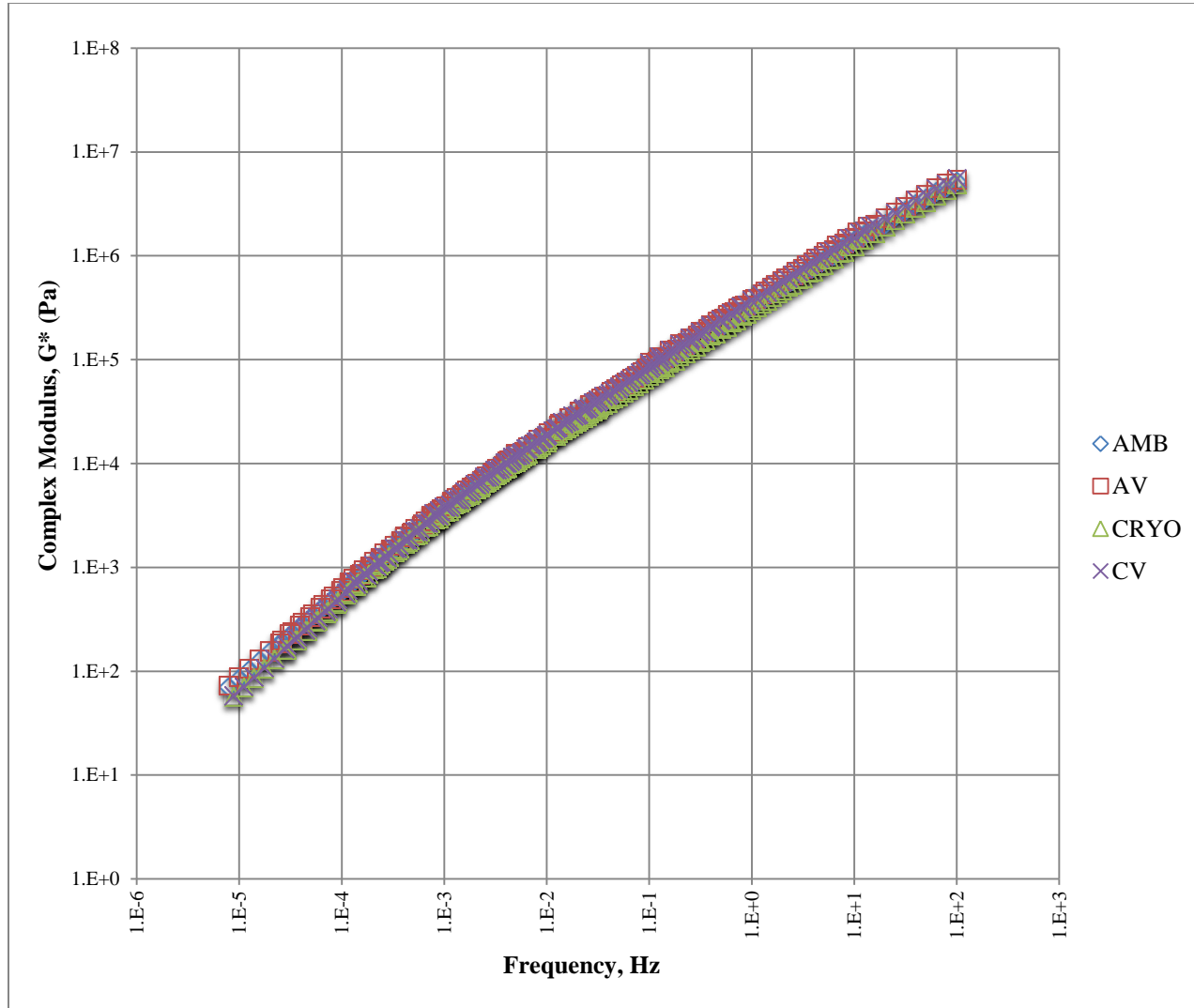


Figure 39. Master curves of RTFO asphalt rubber binders

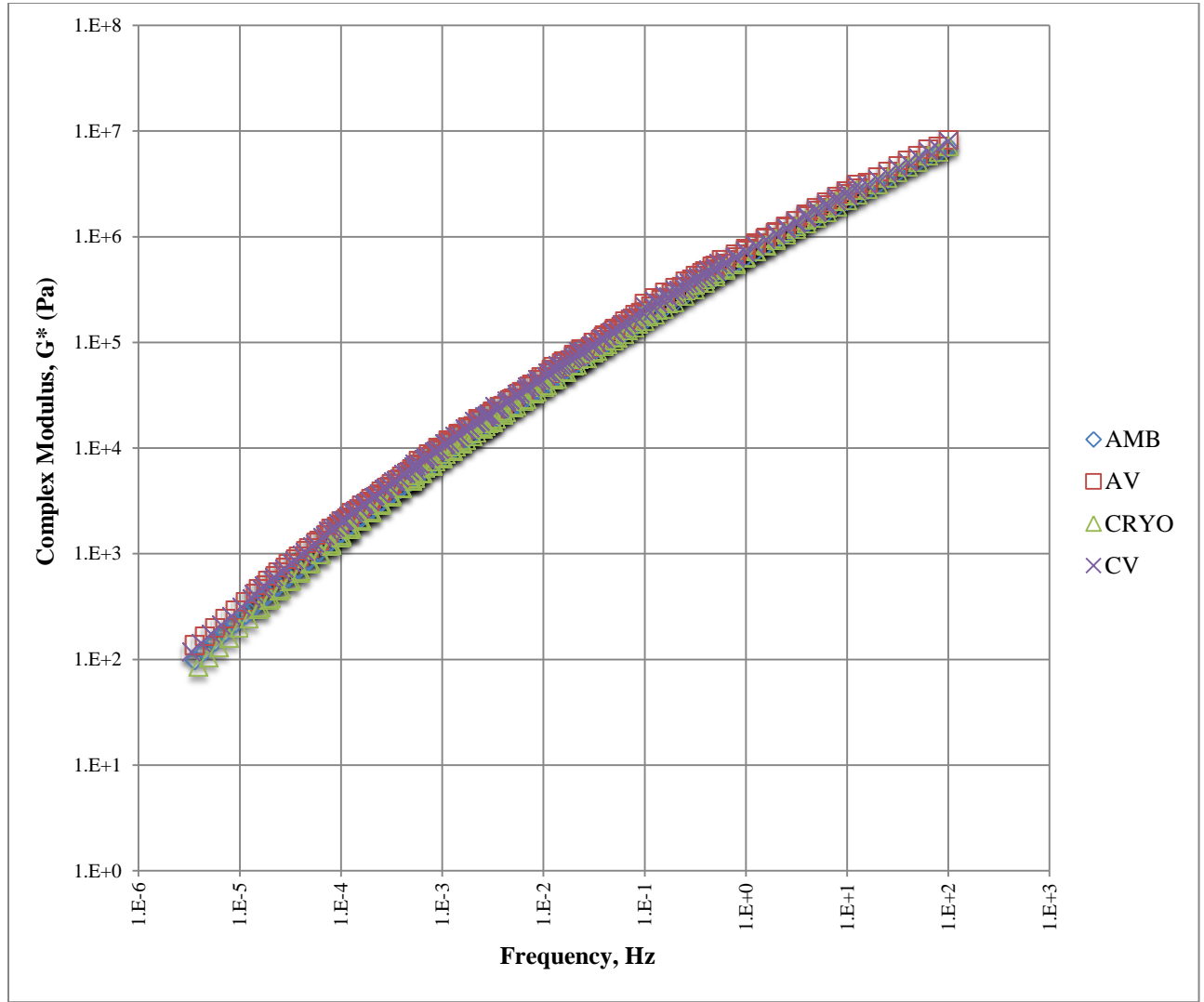


Figure 40. Master curves of PAV Aged asphalt rubber binders

Mixes Performance Testing Results

Dynamic Modulus (E^*)

The results of the dynamic modulus (E^*) for the four asphalt rubber mixtures are tabulated in Table A. 1 to Table A. 4 in Appendix A. From these results the master curves for the four types of asphalt rubber mixtures were constructed and are presented and discussed in the next section of this chapter.

The greyed values in the appendix tables were considered outliers and were not taken into consideration to build the master curves of the four asphalt rubber mixtures. The criteria used to determine if a value was considered to be an outlier, was to look up at the coefficient of variation, if it was higher than twelve percent, then the smaller value was not taken into consideration for the average used in the master curves construction.

This procedure was performed for the ambient GTR and PO mixture and the cryogenic GTR mixture. Thereby coefficients of variations lower than twelve percent were obtained by applying this method to the ambient GTR and PO mixture. However, for the cryogenic GTR mixtures the two smaller values were ignored for the average used to build its master curve, due to the high variability that these two values were introducing to the average. It should be noted that the averages and coefficient of variation reported in the appendix tables do take into consideration these values.

Asphalt Rubber Mixtures Master Curves

After taking care of the possible outliers in the data, the procedure described in the standard practice AASTHO PP62 (2010) was followed to construct the master curves of the asphalt rubber mixtures. The second-order polynomial equation described in

aforementioned standard practice was used to find the shifting factors to reduce the nine frequencies of the test. A reference temperature of 21°C was used to build the master curves, therefore the related frequencies of the temperatures 4°C and 37°C were shifted, to the right and to the left, respectively, to obtain the master curves.

Figures 33 to 36 illustrate the data before and after being shifted for the four types of asphalt rubber mixtures. For a better comparison Figure 45 presents the four master curves together, from this figure it can be seen that the behavior of the four asphalt rubber mixtures are very similar at low and intermediate temperatures, however it seems that for high temperatures the ambient GTR mixtures performs a little bit better than the other three asphalt rubber mixtures. To see if there is any statistically significant difference a statistical analysis was performed for each testing temperature at a certain frequency. This is, for the data at a testing temperature of 4°C which is located at the farther right side of the master curves and that is related to high frequencies and low temperatures, the analysis was performed at the 25 Hz frequency. As for the testing temperature of 21°C, which is the reference temperature and which values are located in the middle of the master curve, is related to the intermediate frequencies and temperatures, thus the data at the frequency of 10 Hz was chosen. For the data at a testing temperature of 37°C, that is located at the farther left side of the master curves belongs to the lower frequencies and high temperatures, thus the lower testing frequency of 0.1 Hz was pick to be analyzed.

Table 17 presents the results obtained from the ANOVA analysis, which showed that no significant difference was observed between the mix types at the determined frequency and at that specific testing temperature. Thus, it can be inferred that the four

asphalt rubber mixtures will have a similar performance at low, intermediate and high frequencies.

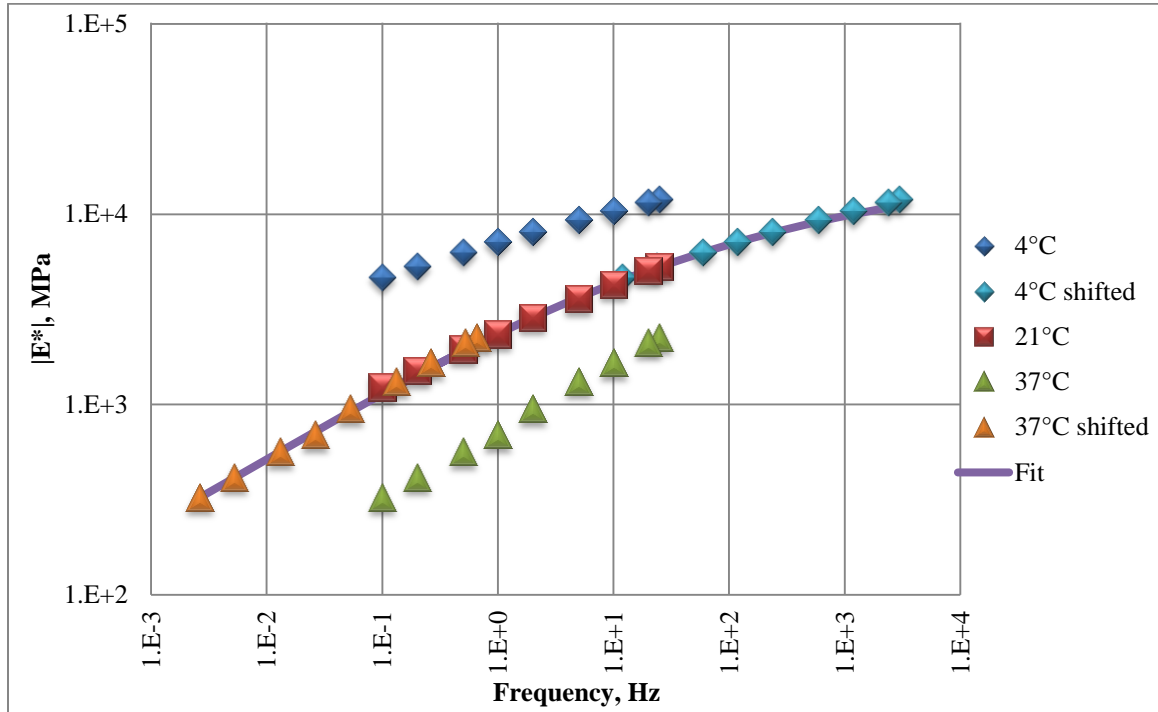


Figure 41. Master curve of AMB Mix

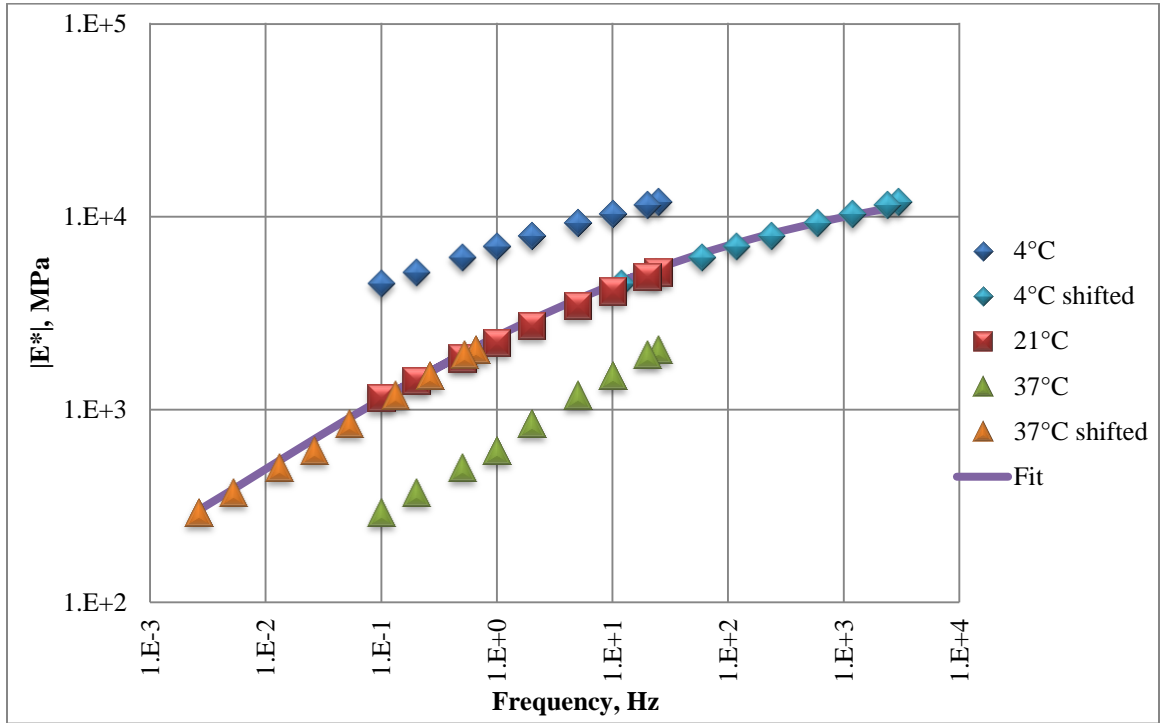


Figure 42. Master curve of AV Mix

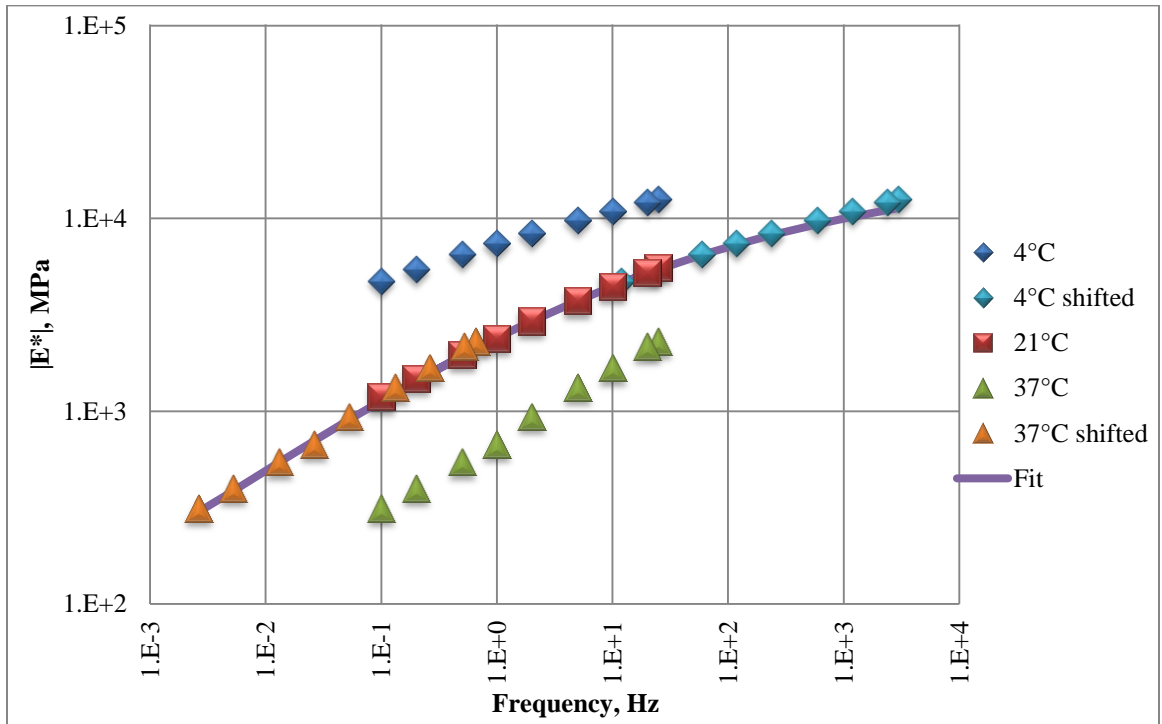


Figure 43. Master curve of CRYO Mix

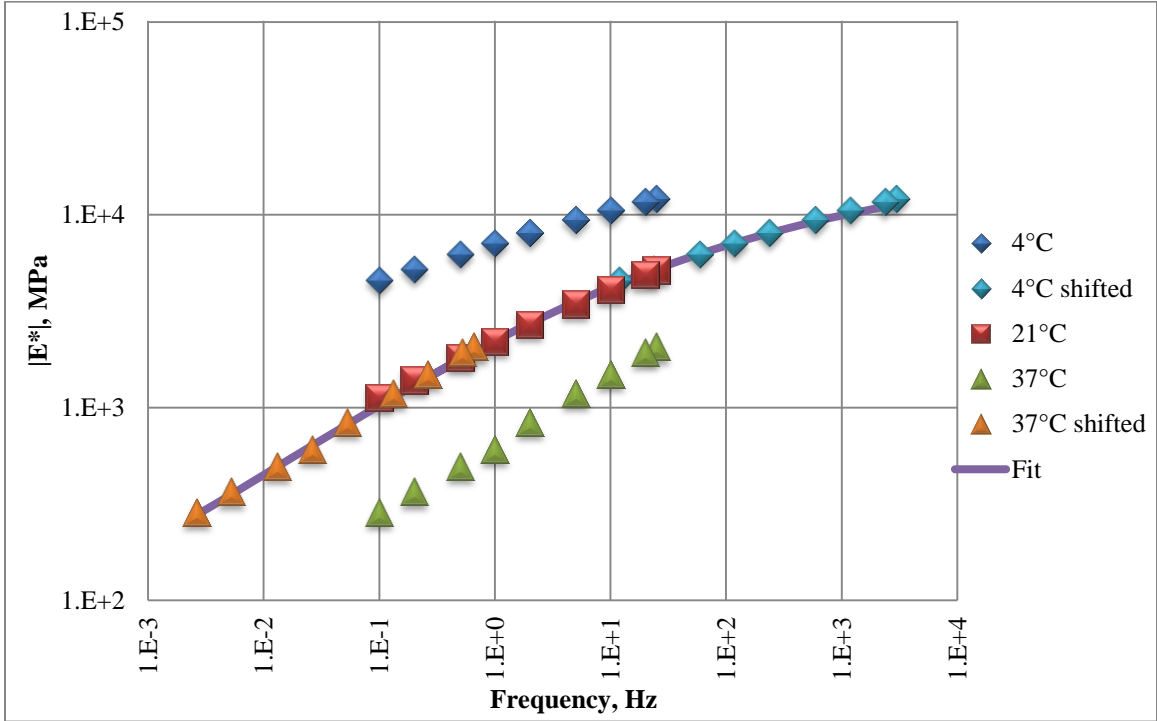


Figure 44. Master curve of CV Mix

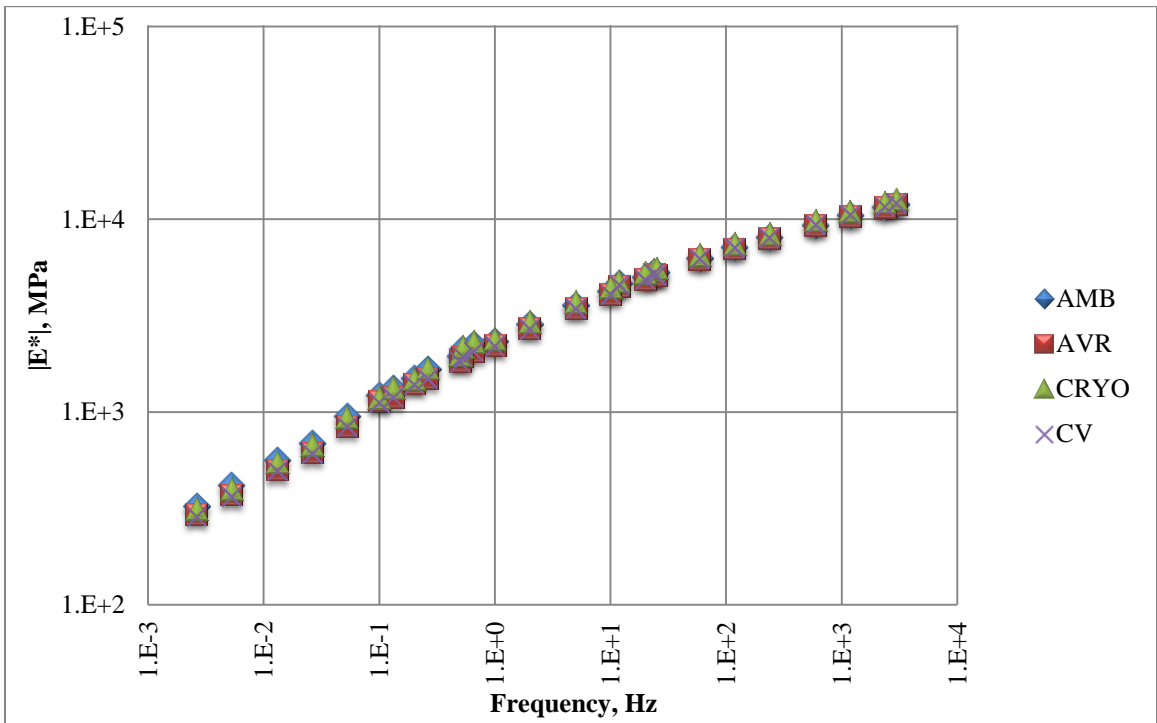


Figure 45. Comparison between master curves mixtures

Table 17. Dynamic modulus ANOVA table

| Dynamic modulus at 4°C for high frequency | | | | | |
|---|-----------|-----------------------|--------------------|----------------|--------------------|
| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
| Mix Type | 3 | 3167544 | 1055848 | 0.5074 | 0.6827 |
| Error | 16 | 33295947 | 2080997 | | |
| C. Total | 19 | 36463491 | | | |
| | | | | | |
| Dynamic modulus at 21°C for intermediate frequency | | | | | |
| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
| Mix Type | 3 | 505744.4 | 168581 | 0.5969 | 0.6261 |
| Error | 16 | 4518515.6 | 282407 | | |
| C. Total | 19 | 5024260.0 | | | |
| | | | | | |
| Dynamic modulus at 37°C for low frequency | | | | | |
| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
| Mix Type | 3 | 10397.106 | 3465.70 | 1.4216 | 0.2732 |
| Error | 16 | 39006.016 | 2437.88 | | |
| C. Total | 19 | 49403.122 | | | |

Flow Number (FN)

After performing the dynamic modulus test, the same specimens were then tested for flow number at a testing temperature of 37°C. The flow number test is an indicative of the rutting performance of the asphalt mixes. The higher the flow number, the better rutting performance the mix is supposed to have. The results obtained from the test are presented in Appendix B. It should be noted that one outlier for each mix type was not taken into consideration to obtain the average flow number of each mix; these values are shown in gray in Table B. 1, and were not included in the statistical analysis performed. Figure 46 shows the results obtained from the testing of the four asphalt rubber mixtures. Cryogenic GTR mixture had the lowest flow number from all the asphalt rubber mixtures, this result goes along with the results obtained from the DSR testing for at high temperatures and from the binder master curve. Likewise the high flow number obtained

for the ambient GTR mixtures relates with the results from the binder testing, having these higher stiffness when compared to cryogenic GTR mixtures.

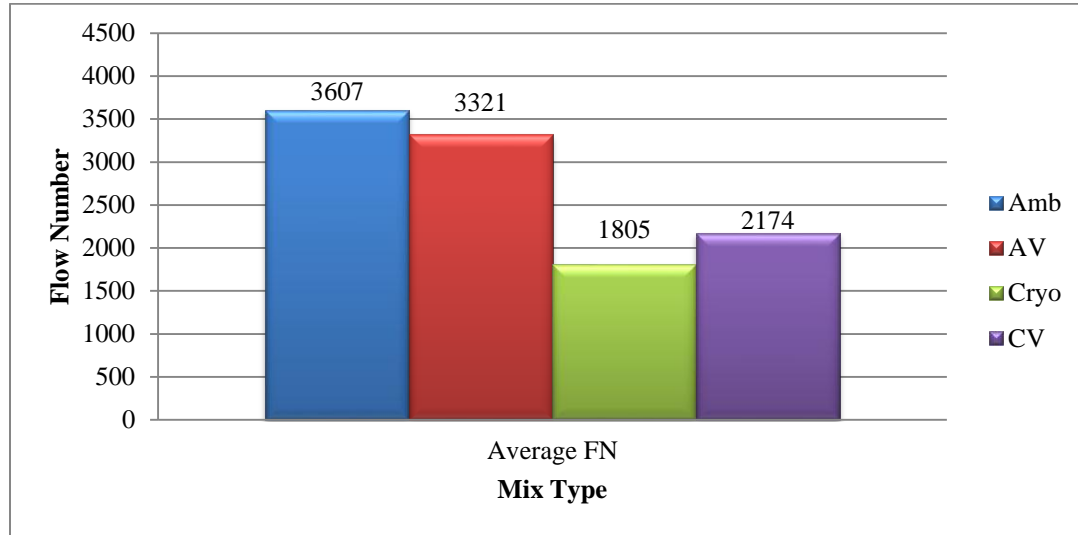


Figure 46. Average Flow Number

A statistical analysis was performed in order to see if these differences between flow numbers are statistically significant. Tables 17 and 18 summarize the statistics performed for the flow number results and show that there is a significant difference between the mix types due to the p-value of 0.0001, and that the mix types that are different amongst them are the mixes containing ambient GTR and cryogenic GTR, although no difference was found between the mixes with or without PO.

Table 18. Flow number ANOVA table

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|----------|----|----------------|-------------|---------|----------|
| Mix Type | 3 | 9133005 | 3044335 | 16.6908 | 0.0001* |
| Error | 12 | 2188757 | 182396 | | |
| C. Total | 15 | 11321762 | | | |

*statistically significant at $\alpha < 0.05$

Table 19. Flow number least square means differences

| q* | Alpha | | |
|--------------|--------------|---|-------------|
| 2.96880 | 0.05 | | |
| Level | | | Mean |
| AMB | A | | 3607.25 |
| AV | A | | 3320.50 |
| CV | | B | 2173.75 |
| CRYO | | B | 1805.00 |

Levels not connected by same letter are significantly different.

Tensile Strength Ratio (TSR)

The tensile strength ratio (TSR) of two sets of three samples each, one conditioned and the other non-conditioned, for each asphalt rubber mixture were obtained by means of the indirect tensile test. Figure 47 illustrates the results from the tensile strength ratio. It should be noted that the four ratios were higher than 1.0, in normal circumstances it is expected that the ratio of the conditioned versus non-conditioned samples would give a ratio lower than 1.0, being 0.80 the lower accepted boundary for reasonable performance, however this was not observed. Cryogenic GTR mixture presented the highest ratio from the set of asphalt rubber mixtures to be tested. In contrast with ambient GTR that had the lowest ratio from the mixtures tested, very much similar to the cryogenic GTR and PO mixture.

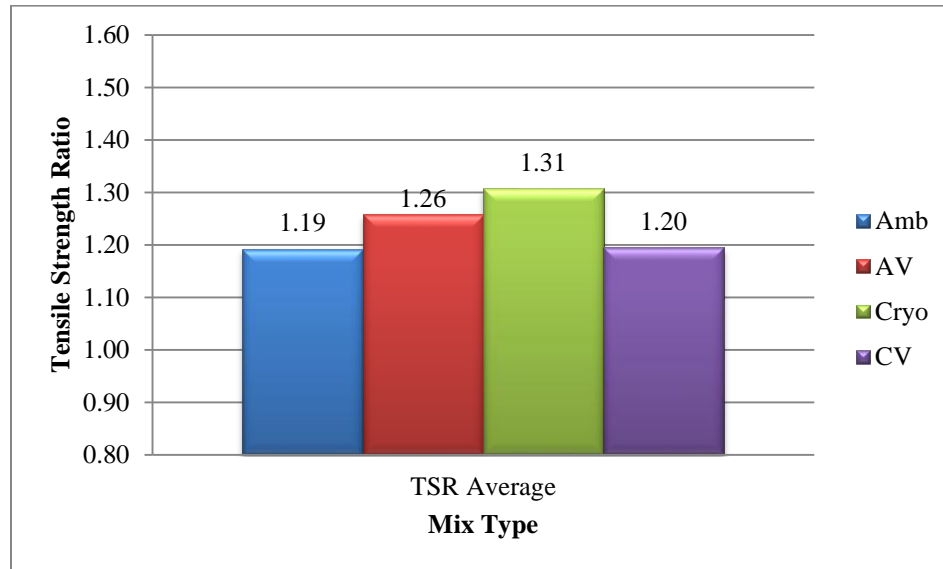


Figure 47. Average Tensile Strength Ratios

A statistical analysis was performed to determine if the differences in tensile strength ratio between mix types were significant, the ANOVA table is shown in Table 20, where it can be seen that the differences in tensile strength ratio are not statistically significant since the p-value obtained from the statistical analysis of 0.5197 is very high compared to the alpha value of 0.05 used in the statistical analysis.

Table 20. Tensile strength ratio ANOVA table

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|----------|----|----------------|-------------|---------|----------|
| Mix Type | 3 | 0.02608656 | 0.008696 | 0.8173 | 0.5197 |
| Error | 8 | 0.08511266 | 0.010639 | | |
| C. Total | 11 | 0.11119923 | | | |

The peak loads of the specimens were also analyzed statistically to see if there was a statistically significant difference between mixes and to see the effects of the treatment (conditioned versus dry specimens) had any influence in the peak loads. Table 21 presents the results of the ANOVA analysis, from where it can be inferred that there is no

statistical difference amongst the mix types due to the high p-value of 0.3175; however, the treatments (conditioned versus dry) presented a statistical significant difference, as shown by the p-value obtained of less than 0.0001. The least square means plot in Figure 48 shows that the conditioned specimens presented higher mean average on the peak load, than the dry (non-conditioned) specimens.

Table 21. Peak load ANOVA table

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|--------------------|----|----------------|-------------|---------|----------|
| Mix Type | 3 | 0.9537108 | 0.317904 | 1.2721 | 0.3175 |
| Treatment | 1 | 9.8880844 | 9.888084 | 39.5664 | <.0001* |
| Mix Type*Treatment | 3 | 0.3396968 | 0.113232 | 0.4531 | 0.7187 |
| Error | 16 | 3.998581 | 0.24991 | 6.3917 | |
| C. Total | 23 | 15.180073 | | | |

*statistically significant at $\alpha < 0.05$

It is hypothesized that there is an interaction between the rubber and the base asphalt when subjected to moisture conditioning; this can be due to the elastic properties of the rubber that can make the mixes in general to withstand more strains without failing and somehow when the rubber mixtures were subjected to moisture conditioning a rejuvenator reaction occurred to the mixes. However, it seems that the addition of PO does not influence this behavior, since no statistical differences were found between the mixes.

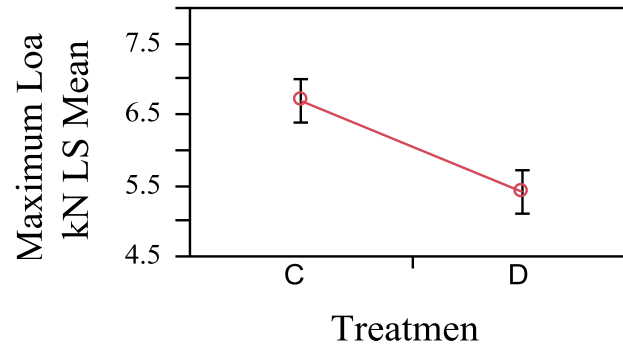


Figure 48. Treatment least square means plot for peak load

It should be mentioned that none to almost no stripping was observed during the inspection of the broken samples after the indirect tensile strength test, as it can be seen from Figure 49. The light spots in the figure are broken aggregates, most of the samples failed through the interface of the binder and the aggregates, but as said before no stripping was observed.

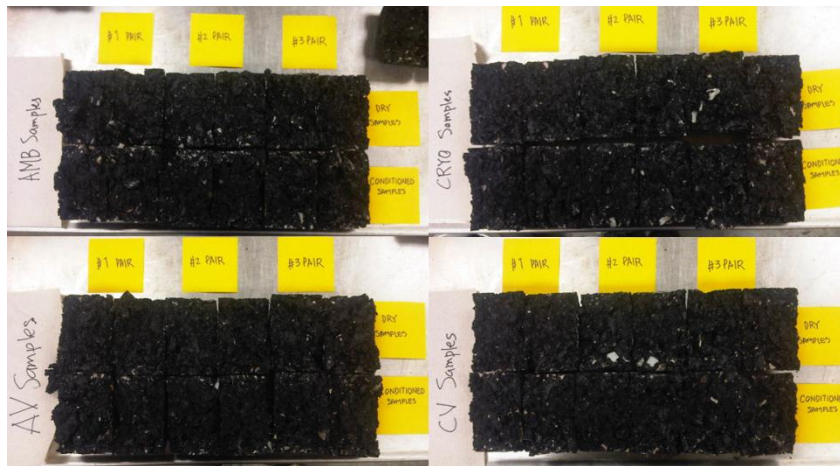


Figure 49. Indirect tensile strength specimens tested

CHAPTER 5. CONCLUSIONS AND RECOMMENDATIONS

This research has determined how the addition of 4.5 percentage of polyoctenamer (PO) by weight of ground tire rubber (GTR) influences the rheological properties and performance of asphalt rubber binders laboratory-produced with a base asphalt PG46-34 and two different ground tire rubbers at 12 percent by weight of asphalt, ambient GTR and cryogenic GTR. Likewise, the performance of their respective asphalt rubber mixtures, mixed and compacted in laboratory, was also evaluated. The aggregate gradation and binder content of the laboratory-produced asphalt rubber mixtures were kept the same throughout the course of this research.

Binders

The validation of the research hypothesis about the influence of the addition of PO to asphalt rubber binders over their viscosity was corroborated. The viscosities of the asphalt rubber binders containing PO were found statistically different from the asphalt rubber binders without PO for alpha equals to 0.05. It was also found that the viscosities of the two asphalt rubber binders with PO were statistically different between them, whereas the viscosities of the two asphalt rubber binders without PO were not statistically different between them.

Although the statistical analysis showed that the addition of PO will indeed decrease the viscosity of the asphalt rubber binders, the amount of reduction in viscosity was not enough to meet the criteria for the mixing and compaction viscosities ranges established in the performance-graded asphalt binder specification (SuperPave). However, it is believe that the high viscosity readings were due to the particle effect of the rubbers during the viscosity testing, leading to higher viscosity values than those that

would be obtained if only the liquid part was tested. Thus, it is recommended to reevaluate the viscosities of just the liquid part of the asphalt rubber binder to avoid having the particle effects of the rubber influencing the viscosity readings, and to see if the new viscosities meet the SuperPave binder specification criteria, and new mixing and compaction temperatures can be determine.

The final performance grading for high temperature performance of the four types of asphalt rubber binders were found to be the same for the high temperature, PG64, thus it is concluded that the addition of PO will not affect the final grading for high temperature of asphalt rubber binders. However, the predicted continuous grading of the asphalt rubber binders without PO showed lower performance grading than the predicted continuous grading of the asphalt rubber binders with PO, this difference being about half degree for the unaged materials and one degree for the RTFO aged materials.

The low temperatures for the final performance grading for the PAV aged of the four asphalt rubber binders in the DSR was determined to be 7°C. However, the predicted continuous grading revealed that the low temperature for the PAV aged materials of the asphalt rubber binder containing ambient GTR where around one degree different, having the binder with PO the higher temperature. This means that the addition of PO reduced the low temperature performance of the PAV aged materials by one degree. While the difference between the asphalt rubber binders containing cryogenic GTR was less than half degree, the same trend of the binder with PO diminishing the low temperature performance was found.

The mass loss results for the asphalt rubber binders with PO were higher than the criteria established in the performance-graded asphalt binder specification (more than 1.0

percent). This can be explained due to more swelling of the rubber particles during the aging process. It is suspected that PO interacts in some way with the rubber promoting more the swelling of the rubber particles, causing spillage of binder out of the RTFO bottles during the aging process. It is recommended to try with less amount of binder per bottle, instead of 35 grams, to confirm this hypothesis.

The final low temperature performance grading for the PAV aged materials in the BBR was found to be a PG -34 for the four types of asphalt rubber binders. However, for the continuous grading, the asphalt rubber binders without PO had better performance at low temperatures than the binders with PO; the differences were about one degree of temperature. Also, the asphalt rubber binders containing cryogenic GTR had lower performance compared with the asphalt rubber binders having ambient GTR.

The master curves obtained from the results of the testing for the four asphalt rubber binders showed that the behavior of the asphalt rubber binders is very similar between them. Being the binders containing ambient GTR somewhat stiffer than the binders with cryogenic GTR.

The binders study performed in this research did not include separation tests on the laboratory-produced binders, it is recommended to evaluate in future research how the addition of PO can influence over the stability of asphalt rubber binders.

Mixes

The results from the dynamic modulus testing were used to construct the mixtures master curves. The statistical analysis performed to test the research hypothesis revealed

that the addition of the PO did not influence the low, intermediate and high temperatures performance of the mixtures prepared with the four types of asphalt rubber binders.

The flow number test revealed that the rutting performances of the asphalt rubber mixes were not different between the mixes with the same type of ground tire rubber. Thus, statistically the performance of the asphalt rubber mixes containing PO is no different from the mixes without it. However, the statistical analysis showed that the type of ground tire rubber will influence the rutting performance; having the ambient GTR mixes better rutting performance than the cryogenic GTR mixes.

The tensile strength ratio was found not to be different for the four types of asphalt rubber mixes. Thus the addition of PO does not affect the moisture susceptibility of asphalt rubber mixes.

This study did not include fatigue cracking performance testing of the asphalt rubber mixes by means of the beam fatigue test; it is recommended that testing for fatigue performance should be done in the future to evaluate the influence of PO over the performance of the asphalt rubber mixtures.

It is also recommended to evaluate the fracture resistance of the asphalt rubber mixes by means of the semi-circular bend geometry (SCB) test and study the influence of PO over the asphalt rubber mixes at low temperatures.

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APPENDIX A. DYNAMIC MODULUS TEST RESULTS

Table A. 1. Dynamic modulus test results, E* AMB

| Temp | Frequency | Dynamic Modulus, E* (MPa) | | | | | Average | COV |
|-----------|-----------|---------------------------|----------|----------|----------|----------|---------|--------|
| | | AMB | | | | | | |
| °C | Hz | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 12534 | 11544 | 13507 | 11219 | 11005 | 11961.8 | 8.72% |
| | 20 | 12076 | 11145 | 13080 | 10848 | 10618 | 11553.4 | 8.81% |
| | 10 | 10928 | 10044 | 11870 | 9782 | 9572 | 10439.2 | 9.12% |
| | 5 | 9804 | 9007 | 10702 | 8759 | 8611 | 9376.6 | 9.30% |
| | 2 | 8439 | 7740 | 9257 | 7505 | 7467 | 8081.6 | 9.46% |
| | 1 | 7472 | 6847 | 8235 | 6621 | 6685 | 7172.0 | 9.52% |
| | 0.5 | 6574 | 6018 | 7264 | 5795 | 5948 | 6319.8 | 9.56% |
| | 0.2 | 5520 | 5059 | 6133 | 4841 | 5070 | 5324.6 | 9.67% |
| | 0.1 | 4813 | 4454 | 5374 | 4224 | 4497 | 4672.4 | 9.52% |
| 21 | | | | | | | | |
| | 25 | 5344 | 5153 | 5940 | 4953 | 5171 | 5312.2 | 7.10% |
| | 20 | 5073 | 4895 | 5646 | 4625 | 4930 | 5033.8 | 7.52% |
| | 10 | 4283 | 4134 | 4774 | 3871 | 4188 | 4250.0 | 7.77% |
| | 5 | 3622 | 3488 | 4041 | 3235 | 3578 | 3592.8 | 8.13% |
| | 2 | 2863 | 2756 | 3210 | 2555 | 2865 | 2849.8 | 8.34% |
| | 1 | 2358 | 2266 | 2646 | 2095 | 2356 | 2344.2 | 8.52% |
| | 0.5 | 1970 | 1882 | 2199 | 1709 | 1963 | 1944.6 | 9.10% |
| | 0.2 | 1535 | 1451 | 1686 | 1313 | 1515 | 1500.0 | 9.03% |
| | 0.1 | 1281 | 1190 | 1373 | 1068 | 1243 | 1231.0 | 9.18% |
| 37 | | | | | | | | |
| | 25 | 2259 | 2159 | 2557 | 2005 | 2310 | 2258.0 | 9.02% |
| | 20 | 2125 | 2028 | 2400 | 1888 | 2188 | 2125.8 | 8.96% |
| | 10 | 1662 | 1584 | 1875 | 1472 | 1707 | 1660.0 | 9.01% |
| | 5 | 1336 | 1262 | 1504 | 1173 | 1367 | 1328.4 | 9.29% |
| | 2 | 952 | 910.3 | 1082 | 836.4 | 971.8 | 950.5 | 9.47% |
| | 1 | 687.7 | 675.1 | 801.5 | 613.4 | 688.2 | 693.2 | 9.81% |
| | 0.5 | 563.5 | 554.6 | 653.9 | 495.5 | 555.1 | 564.5 | 10.08% |
| | 0.2 | 416.1 | 415.9 | 485.1 | 360.7 | 404.6 | 416.5 | 10.71% |
| | 0.1 | 321 | 334.9 | 383.3 | 278.8 | 308.9 | 325.4 | 11.81% |

Table A. 2. Dynamic modulus test results, E* AV

| Temp °C | Frequency Hz | Dynamic Modulus, E* (MPa) | | | | | Average | COV |
|------------|-----------------|---------------------------|-------------|-------------|-------------|-------------|---------|--------|
| | | AV | | | | | | |
| | | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 11616.0 | 12876.0 | 11989.0 | 11393.0 | 8600.0 | 11294.8 | 14.25% |
| | 20 | 11195.0 | 12470.0 | 11542.0 | 10939.0 | 8266.0 | 10882.4 | 14.46% |
| | 10 | 10087.0 | 11319.0 | 10356.0 | 9816.0 | 7357.0 | 9787.0 | 15.04% |
| | 5 | 9020.0 | 10198.0 | 9257.0 | 8756.0 | 6546.0 | 8755.4 | 15.41% |
| | 2 | 7722.0 | 8808.0 | 7920.0 | 7494.0 | 5590.0 | 7506.8 | 15.74% |
| | 1 | 6810.0 | 7805.0 | 6991.0 | 6613.0 | 4934.0 | 6630.6 | 15.85% |
| | 0.5 | 5973.0 | 6854.0 | 6110.0 | 5805.0 | 4322.0 | 5812.8 | 15.91% |
| | 0.2 | 4984.0 | 5739.0 | 5123.0 | 4849.0 | 3632.0 | 4865.4 | 15.80% |
| | 0.1 | 4360.0 | 4970.0 | 4499.0 | 4224.0 | 3185.0 | 4247.6 | 15.47% |
| 21 | | | | | | | | |
| | 25 | 4921.0 | 5660.0 | 5308.0 | 4718.0 | 3832.0 | 4887.8 | 14.17% |
| | 20 | 4661.0 | 5343.0 | 5043.0 | 4487.0 | 3620.0 | 4630.8 | 14.16% |
| | 10 | 3910.0 | 4503.0 | 4246.0 | 3757.0 | 3019.0 | 3887.0 | 14.55% |
| | 5 | 3281.0 | 3798.0 | 3585.0 | 3161.0 | 2539.0 | 3272.8 | 14.69% |
| | 2 | 2578.0 | 2990.0 | 2823.0 | 2481.0 | 1980.0 | 2570.4 | 15.02% |
| | 1 | 2116.0 | 2461.0 | 2311.0 | 2020.0 | 1590.0 | 2099.6 | 15.83% |
| | 0.5 | 1747.0 | 2037.0 | 1913.0 | 1667.0 | 1329.0 | 1738.6 | 15.55% |
| | 0.2 | 1331.0 | 1566.0 | 1460.0 | 1267.0 | 1018.0 | 1328.4 | 15.70% |
| | 0.1 | 1088.0 | 1291.0 | 1180.0 | 1032.0 | 826.8 | 1083.6 | 16.05% |
| 37 | | | | | | | | |
| | 25 | 2001.0 | 2152.0 | 2144.0 | 1934.0 | 1681.0 | 1982.4 | 9.72% |
| | 20 | 1882.0 | 2013.0 | 2025.0 | 1822.0 | 1569.0 | 1862.2 | 9.95% |
| | 10 | 1467.0 | 1575.0 | 1571.0 | 1403.0 | 1199.0 | 1443.0 | 10.71% |
| | 5 | 1168.0 | 1255.0 | 1245.0 | 1107.0 | 948.0 | 1144.6 | 10.95% |
| | 2 | 827.6 | 893.4 | 881.0 | 782.1 | 663.8 | 809.6 | 11.46% |
| | 1 | 607.1 | 653.6 | 645.6 | 556.6 | 468.7 | 586.3 | 12.99% |
| | 0.5 | 492.4 | 539.6 | 525.8 | 455.9 | 391.1 | 481.0 | 12.42% |
| | 0.2 | 359.3 | 408.3 | 389.7 | 333.6 | 293.5 | 356.9 | 12.75% |
| | 0.1 | 277.2 | 335.2 | 305.7 | 259.5 | 235.6 | 282.6 | 13.79% |

Table A. 3. Dynamic modulus test results, E* CRYO

| Temp | Frequency | Dynamic Modulus, E* (MPa) | | | | | Average | COV |
|-----------|-----------|---------------------------|----------|----------|----------|----------|---------|--------|
| | | CRYO | | | | | | |
| °C | Hz | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 9457.0 | 8703.0 | 12935.0 | 12474.0 | 12621.0 | 11238.0 | 17.75% |
| | 20 | 9104.0 | 8332.0 | 12437.0 | 12015.0 | 12175.0 | 10812.6 | 17.92% |
| | 10 | 8103.0 | 7368.0 | 11229.0 | 10809.0 | 10953.0 | 9692.4 | 18.69% |
| | 5 | 7177.0 | 6499.0 | 10019.0 | 9673.0 | 9789.0 | 8631.4 | 19.22% |
| | 2 | 6064.0 | 5479.0 | 8595.0 | 8283.0 | 8375.0 | 7359.2 | 19.95% |
| | 1 | 5304.0 | 4781.0 | 7575.0 | 7309.0 | 7389.0 | 6471.6 | 20.41% |
| | 0.5 | 4601.0 | 4155.0 | 6623.0 | 6410.0 | 6469.0 | 5651.6 | 20.81% |
| | 0.2 | 3778.0 | 3436.0 | 5533.0 | 5375.0 | 5395.0 | 4703.4 | 21.47% |
| | 0.1 | 3258.0 | 2992.0 | 4847.0 | 4719.0 | 4685.0 | 4100.2 | 21.88% |
| 21 | | | | | | | | |
| | 25 | 3872.0 | 3797.0 | 5680.0 | 5580.0 | 5341.0 | 4854.0 | 19.35% |
| | 20 | 3689.0 | 3589.0 | 5383.0 | 5300.0 | 5067.0 | 4605.6 | 19.34% |
| | 10 | 3054.0 | 2970.0 | 4523.0 | 4472.0 | 4270.0 | 3857.8 | 20.18% |
| | 5 | 2528.0 | 2473.0 | 3797.0 | 3767.0 | 3595.0 | 3232.0 | 20.81% |
| | 2 | 1938.0 | 1897.0 | 2986.0 | 2958.0 | 2825.0 | 2520.8 | 21.99% |
| | 1 | 1534.0 | 1515.0 | 2439.0 | 2414.0 | 2294.0 | 2039.2 | 23.20% |
| | 0.5 | 1248.0 | 1246.0 | 2009.0 | 1984.0 | 1883.0 | 1674.0 | 23.46% |
| | 0.2 | 914.0 | 936.6 | 1525.0 | 1495.0 | 1411.0 | 1256.3 | 24.29% |
| | 0.1 | 714.0 | 749.1 | 1226.0 | 1198.0 | 1121.0 | 1001.6 | 24.94% |
| 37 | | | | | | | | |
| | 25 | 1569.0 | 1547.0 | 2415.0 | 2299.0 | 2213.0 | 2008.6 | 20.79% |
| | 20 | 1450.0 | 1431.0 | 2275.0 | 2161.0 | 2071.0 | 1877.6 | 21.60% |
| | 10 | 1096.0 | 1098.0 | 1785.0 | 1666.0 | 1593.0 | 1447.6 | 22.61% |
| | 5 | 839.6 | 876.7 | 1431.0 | 1306.0 | 1250.0 | 1140.7 | 23.36% |
| | 2 | 571.3 | 589.7 | 1027.0 | 915.3 | 869.4 | 794.5 | 25.64% |
| | 1 | 397.7 | 401.7 | 758.1 | 655.2 | 613.3 | 565.2 | 28.31% |
| | 0.5 | 322.0 | 331.9 | 622.9 | 524.1 | 491.8 | 458.5 | 28.25% |
| | 0.2 | 230.8 | 246.6 | 466.5 | 377.9 | 354.3 | 335.2 | 29.14% |
| | 0.1 | 181.1 | 193.2 | 371.9 | 290.4 | 275.0 | 262.3 | 29.73% |

Table A. 4. Dynamic modulus test results, E* CV

| Temp °C | Frequency Hz | Dynamic Modulus, E* (MPa) | | | | | Average | COV |
|------------|-----------------|---------------------------|-------------|-------------|-------------|-------------|---------|--------|
| | | CV | | | | | | |
| | | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 12803.0 | 12825.0 | 11565.0 | 12506.0 | 11004.0 | 12140.6 | 6.72% |
| | 20 | 12331.0 | 12313.0 | 11243.0 | 12038.0 | 10581.0 | 11701.2 | 6.55% |
| | 10 | 11107.0 | 11077.0 | 10150.0 | 10863.0 | 9490.0 | 10537.4 | 6.66% |
| | 5 | 9951.0 | 9909.0 | 9099.0 | 9735.0 | 8427.0 | 9424.2 | 6.94% |
| | 2 | 8536.0 | 8504.0 | 7788.0 | 8360.0 | 7153.0 | 8068.2 | 7.36% |
| | 1 | 7536.0 | 7524.0 | 6864.0 | 7394.0 | 6256.0 | 7114.8 | 7.77% |
| | 0.5 | 6606.0 | 6604.0 | 6009.0 | 6508.0 | 5435.0 | 6232.4 | 8.18% |
| | 0.2 | 5533.0 | 5556.0 | 5013.0 | 5492.0 | 4514.0 | 5221.6 | 8.70% |
| | 0.1 | 4825.0 | 4877.0 | 4375.0 | 4834.0 | 3944.0 | 4571.0 | 8.88% |
| 21 | | | | | | | | |
| | 25 | 5433.0 | 5382.0 | 4896.0 | 5393.0 | 4600.0 | 5140.8 | 7.28% |
| | 20 | 5132.0 | 5102.0 | 4645.0 | 5109.0 | 4358.0 | 4869.2 | 7.21% |
| | 10 | 4316.0 | 4285.0 | 3882.0 | 4302.0 | 3641.0 | 4085.2 | 7.53% |
| | 5 | 3638.0 | 3608.0 | 3250.0 | 3630.0 | 3046.0 | 3434.4 | 7.90% |
| | 2 | 2870.0 | 2823.0 | 2540.0 | 2863.0 | 2374.0 | 2694.0 | 8.35% |
| | 1 | 2359.0 | 2297.0 | 2065.0 | 2343.0 | 1930.0 | 2198.8 | 8.70% |
| | 0.5 | 1962.0 | 1880.0 | 1709.0 | 1940.0 | 1585.0 | 1815.2 | 8.95% |
| | 0.2 | 1513.0 | 1416.0 | 1305.0 | 1482.0 | 1198.0 | 1382.8 | 9.44% |
| | 0.1 | 1246.0 | 1133.0 | 1069.0 | 1205.0 | 960.7 | 1122.7 | 10.08% |
| 37 | | | | | | | | |
| | 25 | 2288.0 | 2249.0 | 1952.0 | 2069.0 | 1783.0 | 2068.2 | 10.14% |
| | 20 | 2128.0 | 2102.0 | 1834.0 | 1941.0 | 1659.0 | 1932.8 | 10.07% |
| | 10 | 1660.0 | 1623.0 | 1409.0 | 1506.0 | 1276.0 | 1494.8 | 10.53% |
| | 5 | 1328.0 | 1278.0 | 1120.0 | 1199.0 | 1008.0 | 1186.6 | 10.73% |
| | 2 | 943.4 | 892.3 | 788.5 | 852.3 | 711.6 | 837.6 | 10.79% |
| | 1 | 688.3 | 641.8 | 573.6 | 622.4 | 511.4 | 607.5 | 11.14% |
| | 0.5 | 561.5 | 514.6 | 469.0 | 507.7 | 426.6 | 495.9 | 10.24% |
| | 0.2 | 415.0 | 372.6 | 344.7 | 377.7 | 320.2 | 366.0 | 9.79% |
| | 0.1 | 322.8 | 286.3 | 267.9 | 300.4 | 257.5 | 287.0 | 9.05% |

Table A. 5. Dynamic modulus test results, ϕ AMB

| Temp | Frequency | Phase Angle, ϕ (°) | | | | | Average | COV |
|-----------|-----------|-------------------------|----------|----------|----------|----------|---------|-------|
| | | AMB | | | | | | |
| °C | Hz | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 11.92 | 11.96 | 11.81 | 12.67 | 11.06 | 11.9 | 4.81% |
| | 20 | 12.25 | 12.36 | 12.17 | 13.1 | 11.44 | 12.3 | 4.82% |
| | 10 | 13.44 | 13.42 | 13.3 | 14.3 | 12.46 | 13.4 | 4.88% |
| | 5 | 14.55 | 14.5 | 14.46 | 15.49 | 13.47 | 14.5 | 4.93% |
| | 2 | 16.03 | 15.93 | 15.91 | 16.98 | 14.84 | 15.9 | 4.76% |
| | 1 | 17.19 | 17.07 | 17.03 | 18.23 | 15.99 | 17.1 | 4.64% |
| | 0.5 | 18.37 | 18.22 | 18.16 | 19.49 | 17.09 | 18.3 | 4.67% |
| | 0.2 | 19.75 | 19.56 | 19.5 | 20.92 | 18.41 | 19.6 | 4.55% |
| | 0.1 | 20.69 | 20.49 | 20.36 | 21.84 | 19.28 | 20.5 | 4.45% |
| 21 | | | | | | | | |
| | 25 | 21.91 | 21.26 | 21.64 | 22.58 | 20.64 | 21.6 | 3.35% |
| | 20 | 22.16 | 21.67 | 21.95 | 23 | 20.81 | 21.9 | 3.62% |
| | 10 | 23.39 | 22.98 | 23.21 | 24.24 | 21.97 | 23.2 | 3.53% |
| | 5 | 24.18 | 23.92 | 23.94 | 25.15 | 22.55 | 23.9 | 3.88% |
| | 2 | 25.42 | 25.38 | 25.1 | 26.32 | 23.72 | 25.2 | 3.73% |
| | 1 | 26.39 | 26.22 | 25.9 | 27.22 | 24.49 | 26.0 | 3.82% |
| | 0.5 | 26.59 | 26.6 | 26.1 | 27.73 | 24.63 | 26.3 | 4.26% |
| | 0.2 | 26.96 | 27.13 | 26.39 | 27.78 | 24.96 | 26.6 | 3.99% |
| | 0.1 | 26.82 | 27.12 | 26.36 | 27.7 | 24.97 | 26.6 | 3.87% |
| 37 | | | | | | | | |
| | 25 | 27.79 | 27.16 | 26.82 | 28.14 | 25.91 | 27.2 | 3.21% |
| | 20 | 27.45 | 26.73 | 26.32 | 27.76 | 25.25 | 26.7 | 3.71% |
| | 10 | 28.44 | 27.42 | 26.73 | 28.42 | 25.87 | 27.4 | 4.05% |
| | 5 | 27.92 | 26.97 | 26.2 | 27.89 | 25.49 | 26.9 | 3.95% |
| | 2 | 29.65 | 28.8 | 27.31 | 29.36 | 27.16 | 28.5 | 4.07% |
| | 1 | 30.97 | 29.66 | 27.49 | 30.08 | 28.07 | 29.3 | 4.93% |
| | 0.5 | 29.65 | 27.84 | 25.93 | 28.6 | 27.11 | 27.8 | 5.09% |
| | 0.2 | 28.46 | 26.56 | 24.82 | 28.02 | 26.48 | 26.9 | 5.36% |
| | 0.1 | 27.64 | 25.78 | 23.54 | 27.42 | 25.94 | 26.1 | 6.30% |

Table A. 6. Dynamic modulus test results, ϕ AV

| Temp °C | Frequency Hz | Phase Angle, ϕ (°) | | | | | Average | COV |
|------------|-----------------|-------------------------|-------------|-------------|-------------|-------------|---------|-------|
| | | AV | | | | | | |
| | | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 12.42 | 11.51 | 12.73 | 12.97 | 13.52 | 12.6 | 5.89% |
| | 20 | 12.87 | 11.94 | 13.19 | 13.29 | 13.89 | 13.0 | 5.49% |
| | 10 | 14.1 | 13.17 | 14.43 | 14.54 | 14.99 | 14.2 | 4.78% |
| | 5 | 15.28 | 14.34 | 15.58 | 15.69 | 15.94 | 15.4 | 4.04% |
| | 2 | 16.79 | 15.82 | 17.11 | 17.17 | 17.44 | 16.9 | 3.73% |
| | 1 | 18.01 | 16.97 | 18.27 | 18.27 | 18.64 | 18.0 | 3.52% |
| | 0.5 | 19.22 | 18.25 | 19.47 | 19.42 | 19.81 | 19.2 | 3.07% |
| | 0.2 | 20.62 | 19.67 | 20.77 | 20.72 | 21.15 | 20.6 | 2.67% |
| | 0.1 | 21.56 | 20.64 | 21.57 | 21.59 | 22.06 | 21.5 | 2.41% |
| 21 | | | | | | | | |
| | 25 | 22.47 | 20.99 | 22.31 | 23.32 | 23.12 | 22.4 | 4.08% |
| | 20 | 22.83 | 21.41 | 22.58 | 23.57 | 23.4 | 22.8 | 3.76% |
| | 10 | 24.17 | 22.73 | 23.78 | 24.87 | 24.7 | 24.1 | 3.56% |
| | 5 | 25 | 23.54 | 24.53 | 25.47 | 25.39 | 24.8 | 3.19% |
| | 2 | 26.48 | 24.93 | 25.77 | 26.73 | 26.87 | 26.2 | 3.08% |
| | 1 | 27.46 | 25.85 | 26.62 | 27.66 | 28.65 | 27.2 | 3.91% |
| | 0.5 | 27.72 | 26.17 | 26.81 | 27.82 | 28.07 | 27.3 | 2.93% |
| | 0.2 | 28.01 | 26.63 | 27.15 | 28.14 | 28.48 | 27.7 | 2.77% |
| | 0.1 | 28.1 | 26.61 | 27.2 | 28.06 | 28.62 | 27.7 | 2.89% |
| 37 | | | | | | | | |
| | 25 | 28.79 | 27.8 | 27.66 | 28.74 | 27.39 | 28.1 | 2.30% |
| | 20 | 28.39 | 27.52 | 27.12 | 28.4 | 27.38 | 27.8 | 2.14% |
| | 10 | 29.12 | 28.19 | 27.74 | 29.02 | 28.1 | 28.4 | 2.13% |
| | 5 | 28.52 | 27.71 | 27.24 | 28.37 | 27.43 | 27.9 | 2.04% |
| | 2 | 30.26 | 29.16 | 28.79 | 29.9 | 29.33 | 29.5 | 2.00% |
| | 1 | 30.92 | 30.05 | 29.38 | 30.47 | 30.21 | 30.2 | 1.88% |
| | 0.5 | 29.29 | 28.21 | 27.65 | 28.56 | 28.06 | 28.4 | 2.18% |
| | 0.2 | 28.2 | 26.93 | 26.67 | 27.38 | 26.64 | 27.2 | 2.40% |
| | 0.1 | 27.59 | 26.12 | 25.64 | 26.58 | 25.88 | 26.4 | 2.92% |

Table A. 7. Dynamic modulus test results, ϕ CRYO

| Temp °C | Frequency Hz | Phase Angle, ϕ (°) | | | | | Average | COV |
|------------|-----------------|-------------------------|-------------|-------------|-------------|-------------|---------|--------|
| | | CRYO | | | | | | |
| | | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 10.94 | 14.95 | 12.15 | 12.74 | 12.44 | 12.6 | 11.54% |
| | 20 | 11.09 | 15.38 | 12.73 | 13.21 | 12.98 | 13.1 | 11.72% |
| | 10 | 11.81 | 16.64 | 14.04 | 14.56 | 14.25 | 14.3 | 12.05% |
| | 5 | 12.66 | 17.78 | 15.34 | 15.81 | 15.45 | 15.4 | 11.85% |
| | 2 | 14.04 | 19.28 | 16.94 | 17.41 | 17 | 16.9 | 11.09% |
| | 1 | 15.37 | 20.4 | 18.19 | 18.59 | 18.17 | 18.1 | 9.93% |
| | 0.5 | 16.76 | 21.41 | 19.41 | 19.78 | 19.4 | 19.4 | 8.62% |
| | 0.2 | 18.43 | 22.61 | 20.78 | 21.16 | 20.82 | 20.8 | 7.23% |
| | 0.1 | 19.15 | 23.27 | 21.58 | 21.94 | 21.75 | 21.5 | 6.93% |
| 21 | | | | | | | | |
| | 25 | 24.44 | 23.83 | 22.42 | 22.65 | 22.44 | 23.2 | 3.99% |
| | 20 | 24.95 | 23.81 | 22.63 | 22.84 | 22.55 | 23.4 | 4.38% |
| | 10 | 26.81 | 24.99 | 23.96 | 24.11 | 23.83 | 24.7 | 5.02% |
| | 5 | 28.14 | 25.66 | 24.73 | 24.85 | 24.43 | 25.6 | 5.91% |
| | 2 | 29.43 | 26.92 | 25.92 | 26.14 | 25.67 | 26.8 | 5.72% |
| | 1 | 30.4 | 28.11 | 26.83 | 26.97 | 26.66 | 27.8 | 5.63% |
| | 0.5 | 30.48 | 27.95 | 27.01 | 27.14 | 26.81 | 27.9 | 5.44% |
| | 0.2 | 30.71 | 27.86 | 27.18 | 27.51 | 27.24 | 28.1 | 5.28% |
| | 0.1 | 30.65 | 27.8 | 26.88 | 27.61 | 27.46 | 28.1 | 5.26% |
| 37 | | | | | | | | |
| | 25 | 31.47 | 27.67 | 27.8 | 28.32 | 27.75 | 28.6 | 5.68% |
| | 20 | 31.15 | 27.53 | 27.45 | 27.63 | 27.14 | 28.2 | 5.93% |
| | 10 | 31.63 | 27.88 | 27.91 | 28.1 | 27.79 | 28.7 | 5.80% |
| | 5 | 31.27 | 26.61 | 27.26 | 27.37 | 27.17 | 27.9 | 6.75% |
| | 2 | 32.59 | 28.8 | 28.46 | 28.95 | 28.84 | 29.5 | 5.83% |
| | 1 | 37.38 | 30.18 | 28.81 | 29.59 | 29.39 | 31.1 | 11.46% |
| | 0.5 | 32.26 | 27.97 | 27.15 | 27.9 | 27.72 | 28.6 | 7.24% |
| | 0.2 | 31.41 | 26.4 | 25.7 | 26.89 | 26.48 | 27.4 | 8.38% |
| | 0.1 | 31.09 | 26 | 25.08 | 25.94 | 25.71 | 26.8 | 9.14% |

Table A. 8. Dynamic modulus test results, ϕ CV

| Temp °C | Frequency Hz | Phase Angle, ϕ (°) | | | | | Average | COV |
|------------|-----------------|-------------------------|-------------|-------------|-------------|-------------|---------|-------|
| | | CV | | | | | | |
| | | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | | |
| 4 | | | | | | | | |
| | 25 | 12.46 | 12.61 | 12.58 | 12.23 | 13.01 | 12.6 | 2.26% |
| | 20 | 12.84 | 13.05 | 13 | 12.7 | 13.52 | 13.0 | 2.39% |
| | 10 | 14.08 | 14.31 | 14.28 | 13.93 | 14.82 | 14.3 | 2.36% |
| | 5 | 15.25 | 15.54 | 15.47 | 15.11 | 16.09 | 15.5 | 2.43% |
| | 2 | 16.76 | 17.07 | 17.12 | 16.6 | 17.77 | 17.1 | 2.64% |
| | 1 | 17.92 | 18.28 | 18.35 | 17.78 | 19.13 | 18.3 | 2.88% |
| | 0.5 | 19.08 | 19.47 | 19.62 | 18.94 | 20.44 | 19.5 | 3.02% |
| | 0.2 | 20.43 | 20.82 | 21.09 | 20.3 | 21.91 | 20.9 | 3.07% |
| | 0.1 | 21.27 | 21.58 | 21.99 | 21.15 | 22.87 | 21.8 | 3.19% |
| 21 | | | | | | | | |
| | 25 | 22.28 | 22.73 | 23.03 | 22.22 | 23.32 | 22.7 | 2.09% |
| | 20 | 22.49 | 22.8 | 23.34 | 22.49 | 23.61 | 22.9 | 2.22% |
| | 10 | 23.75 | 24 | 24.71 | 23.73 | 24.97 | 24.2 | 2.36% |
| | 5 | 24.43 | 24.68 | 25.42 | 24.42 | 25.66 | 24.9 | 2.33% |
| | 2 | 25.67 | 26.01 | 26.79 | 25.72 | 27.06 | 26.3 | 2.43% |
| | 1 | 26.6 | 26.99 | 27.98 | 26.59 | 28.03 | 27.2 | 2.64% |
| | 0.5 | 26.79 | 27.23 | 28.04 | 26.73 | 28.28 | 27.4 | 2.60% |
| | 0.2 | 27.12 | 27.51 | 28.1 | 27.11 | 28.54 | 27.7 | 2.27% |
| | 0.1 | 26.96 | 27.42 | 27.93 | 27.12 | 28.53 | 27.6 | 2.32% |
| 37 | | | | | | | | |
| | 25 | 27.58 | 27.75 | 28.27 | 28.01 | 28.87 | 28.1 | 1.80% |
| | 20 | 27.19 | 27.18 | 28.13 | 27.75 | 28.56 | 27.8 | 2.16% |
| | 10 | 27.82 | 27.92 | 28.56 | 28.47 | 29.12 | 28.4 | 1.86% |
| | 5 | 27.35 | 27.44 | 27.69 | 27.75 | 28.33 | 27.7 | 1.38% |
| | 2 | 28.65 | 28.79 | 29.28 | 29.29 | 29.84 | 29.2 | 1.62% |
| | 1 | 29.52 | 29.3 | 29.9 | 29.53 | 30.49 | 29.7 | 1.57% |
| | 0.5 | 28.2 | 27.72 | 28.13 | 27.94 | 28.22 | 28.0 | 0.75% |
| | 0.2 | 27.17 | 26.69 | 26.7 | 26.74 | 26.57 | 26.8 | 0.86% |
| | 0.1 | 26.61 | 26.1 | 26.11 | 26.15 | 25.74 | 26.1 | 1.18% |

APPENDIX B. FLOW NUMBER TEST RESULTS

Table B. 1. Summary of flow number test results

| Mix Type | Strain Rate | | | | Flow Number | | | |
|-------------------|-------------|----------|----------|----------|-------------|------|------|------|
| | Amb | AV | Cryo | CV | Amb | AV | Cryo | CV |
| FN1 | 2.34E-06 | 2.80E-06 | 9.21E-06 | 1.53E-06 | 3681 | 3325 | 1489 | 2931 |
| FN2 | 2.60E-06 | 1.87E-06 | 6.60E-06 | 2.60E-06 | 3724 | 3786 | 1474 | 1855 |
| FN3 | 1.27E-06 | 2.94E-06 | 3.27E-06 | 4.80E-06 | 4089 | 3442 | 2195 | 1922 |
| FN4 | 2.87E-06 | 3.07E-06 | 3.00E-06 | 2.14E-06 | 2416 | 2320 | 2062 | 2732 |
| FN5 | 1.93E-06 | 3.54E-06 | 2.60E-06 | 3.20E-06 | 2935 | 2729 | 3567 | 2186 |
| Average FN | 2.43E-06 | 3.09E-06 | 2.96E-06 | 2.65E-06 | 3607 | 3321 | 1805 | 2174 |
| COV | 16% | 10% | 11% | 20% | 13% | 13% | 21% | 18% |

Note: Grayed numbers were considered outliers, thus they were not taken into consideration for the computation of the average flow number reported here nor for the coefficient of variation presented in this table.

APPENDIX C. TENSILE STRENGTH RATIO RESULTS

Table C. 1. Tensile Strength Ratio (TSR) results

| Treatment | Specimen # | Maximum Load, kN | St, kPa | Tensile Strength Ratio |
|------------------|-------------------|-------------------------|----------------|-------------------------------|
| D | AMB | 6.448 | 644.145 | 1.09 |
| C | AMB | 7.004 | 700.495 | |
| D | AMB | 4.727 | 473.533 | 1.20 |
| C | AMB | 5.700 | 569.362 | |
| D | AMB | 5.192 | 518.321 | 1.31 |
| C | AMB | 6.814 | 680.459 | |
| D | AV | 5.308 | 531.763 | 1.24 |
| C | AV | 6.576 | 658.517 | |
| D | AV | 4.981 | 499.659 | 1.18 |
| C | AV | 5.913 | 591.876 | |
| D | AV | 5.027 | 501.796 | 1.35 |
| C | AV | 6.792 | 679.328 | |
| D | CRYO | 5.760 | 573.197 | 1.16 |
| C | CRYO | 6.668 | 666.228 | |
| D | CRYO | 5.427 | 541.158 | 1.40 |
| C | CRYO | 7.582 | 757.946 | |
| D | CRYO | 5.232 | 521.442 | 1.37 |
| C | CRYO | 7.139 | 714.184 | |
| D | CV | 5.641 | 561.999 | 1.24 |
| C | CV | 7.021 | 698.501 | |
| D | CV | 5.912 | 589.336 | 1.11 |
| C | CV | 6.586 | 654.849 | |
| D | CV | 5.244 | 520.546 | 1.24 |
| C | CV | 6.509 | 644.675 | |