
**Innovations Deserving
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I **IDEA**

NCHRP IDEA Program

Producing a Sustainable, Bio-Based Alternative to Petroleum-Based Asphalt Binder

Final Report for
NCHRP IDEA Project 171

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NCHRP IDEA-171- Final Report

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Submitted by

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EXECUTIVE SUMMARY

Ground tire rubber, also called “crumb rubber”, has been commonly used in the United States for modification of asphalt binder. There have been several studies on improving the interaction and adhesion between rubber and the asphalt binder matrix to alleviate the phase separation and settling of the rubbery phase. Using crumb rubber (CR) as an asphalt modifier enhances the performance of asphalt mixture at high, intermediate, and low service temperatures. Commonly practiced modifications for application of rubber particles inside the asphalt include very fine grinding of rubber particles using a cryogenic method, application of a high shear rate and high temperature to expedite rubber-asphalt interactions, and use of chemical additives and stabilizers. The main idea of this project is to add bio-binder to crumb-rubber-modified (CRM) asphalt to enhance its low-temperature rheological properties while improving workability and reducing phase separation. The product produced by adding bio-binder to CRM asphalt is called Bio-Modified-Rubber (BMR) asphalt.

It should be noted that the modification of asphalt binder with crumb rubber (CR) is mainly controlled by the exchange equilibrium between the components of asphalt binder and CR. The light components of asphalt binder easily penetrate into rubber particles, while some polymer chains from CR move into the asphalt binder matrix. These phenomena increase the size of rubber particles (swelling) up to three times their original size, leading to a significant increase in the resulting asphalt's viscosity. Swelling and increased viscosity not only promote separation of the modified matrix into two distinguishable phases, but also increase the difficulty of pumping and application of rubberized asphalt. Promoting interactions between rubber polymers and asphalt aromatic compounds by adding bio-binder (BB) could alleviate the aforementioned issues while enhancing overall performance. Therefore, this NCHRP IDEA project investigates the effectiveness of treating rubber with an amide-enriched bio-binder to facilitate breakage of disulfide bonds in rubber polymers, while promoting interactions between free radicals and the amide groups of BB. Accordingly, the physiochemical and rheological properties of rubberized asphalt before and after bio-modification at both binder and mixture level were studied.

The results of the study showed that crossover temperatures of the BMR are consistently lower when compared to those of CRM specimens, even after short- and long-term aging indicating BMR is more ductile than CRM. In addition, bio-modification of rubber asphalt showed to increase the non-recoverable creep compliance (Jnr) and reduce the percent recovery. However, the inclusion of 15% bio-binder appears to show improved results compared to CRM for unaged binders as well as PAV aged binders with and without conditioning. Compared to the CRM sample, the results indicate that BMR has an improved ability to relax stress, making the stress accumulation lower than CRM. For the unaged samples, the CRM showed to have higher fracture energy results after both 0- and 12-hour conditioning at -18°C compared to the corresponding BMR samples. However, after PAV aging, the BMR showed to have significantly higher fracture energy results at both 0- and 12-hour conditioning. It was further observed that the introduction of bio-modified binder into crumb rubber in this study led to increased workability as shown by the compaction resistance ratio.

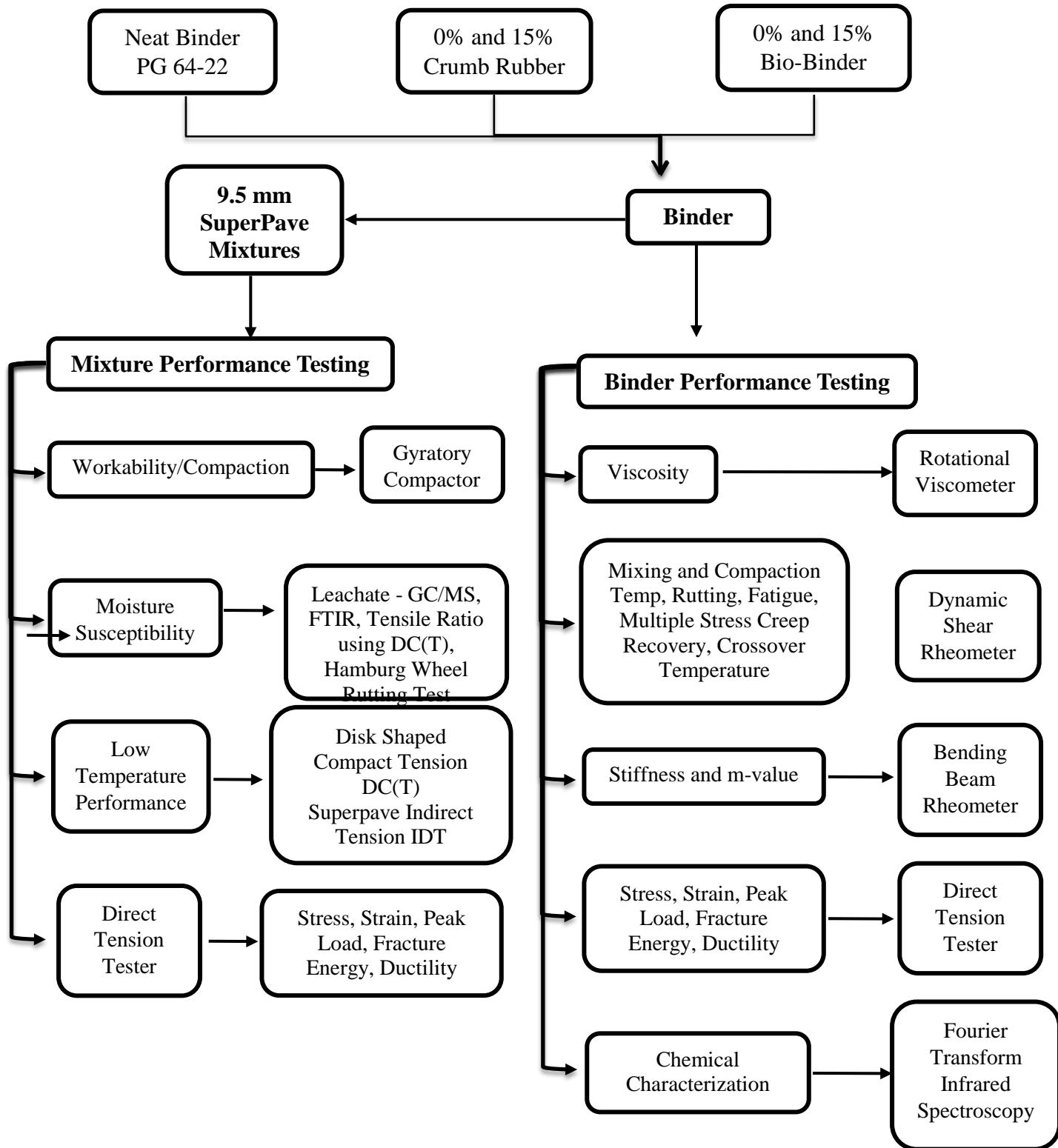
When examining samples after moisture conditioning, the introduction of bio-binder showed that CRM had the lowest tensile strength ratio, followed by the neat binder, while the BMR had the highest tensile strength ratio. None of the CRM and BMR showed any sign of stripping inflection. At low temperature, the results of the disk shape compact tension (DCT) test indicates that the BMR displayed a more ductile behavior than the CRM as evidenced by the larger area under the load-displacement curve for BMR than that of the CRM. At low temperature, the indirect tensile test (IDT) results showed the BMR samples have nearly similar creep compliance behavior to the neat samples. In addition, BMR asphalt showed enhanced workability by reducing the required compaction energy, while improving low-temperature cracking properties by increasing the fracture energy.

The results of study showed that application of BMR could be a promising method to promote the use of crumb rubber in asphalt while enhancing rubber-modified asphalts' low temperature properties and workability. In addition, surface activation of the rubber particles using an amide-enriched bio-modifier could enhance rubber-asphalt interactions while increasing its resistance to moisture damage.

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Schematic of Experimental Pla

INTRODUCTION AND BACKGROUND

There has been significant research on enhancing asphalt mixtures' service life under traffic loadings as well as environmental conditions. Asphalt mixture failures are categorized in five main groups, based on the type of pavement distress and the failure mechanisms: rutting, fatigue cracking, low-temperature cracking, moisture damage, and raveling. There have been several modification methods to improve asphalt properties and consequently increase pavement service life; rubber modification is an approach that has been used successfully by many states' departments of transportation.

The application of rubber in asphalt can enhance performance of asphalt mixture in a wide range of service temperatures: improved rutting resistance at high temperature, increased fatigue resistance at intermediate service temperature, and enhanced stress relaxation properties at low temperature. In addition, it has been reported that using crumb rubber in asphalt mixtures improves the skid resistance, reduces pavement noise, and reduces construction cost (McLeod et al. 2007). The effects of crumb rubber on the asphalt matrix can be divided in two categories: the physical (filler) effect, which is highly controlled by rubber particles' shape and size, and the polymer contribution effect, which is mainly controlled by rubber-asphalt interaction (Hosseinezhad et al. 2014; Bocoum et al. 2014). The latter is highly affected by environmental conditions and could vary with the asphalt composition and rubber surface treatments, both evolving during mixing time (Jeong et al. 2010).

It should be noted that the physical effect of rubber is the main contributor to the increase of rubberized asphalt viscosity; this in turn could make rubberized asphalt application problematic, due to the increased difficulty of pumping and the need for continuous agitation (Hosseinezhad et al. 2014). Such problems especially reduce asphalt workability when high percentages of CR are included (Thodesen et al. 2009).

The enhancement of the interactions between asphalt matrix and rubber could significantly affect the dissociation of rubber polymer chains, the so-called "dissolution" of Crumb Rubber Modifier (CRM), which consequently releases the backbone polymer of rubber. This phenomenon reduces the viscosity of asphalt, which typically occurs after the initial increase of viscosity due to rubber's physical effect (Macleod 1995). Therefore, this paper investigates application of a BB (an amide-based modifier made from thermochemical conversion of swine manure) to facilitate the dissolution of rubber polymers by facilitating the breakage of disulfide bonds in rubber polymers while improving workability and rheological properties.

Scrap Tires

A typical passenger tire contains 15% to 19% vulcanized natural rubber, 25% to 29% synthetic rubber, and other components such as carbon black. Carbon black is used to improve tire treads' rigidity and to minimize heat buildup in sidewalls (Shulman 2000).

Another important component is steel, which provides rigidity and flexibility to the carcass. Nylon, rayon, and polyester are also used as textile components (Rahman 2004). A common weight distribution is given in Table 1 (Unapumnu 2005). Figure 1 shows the different components of a typical passenger tire.

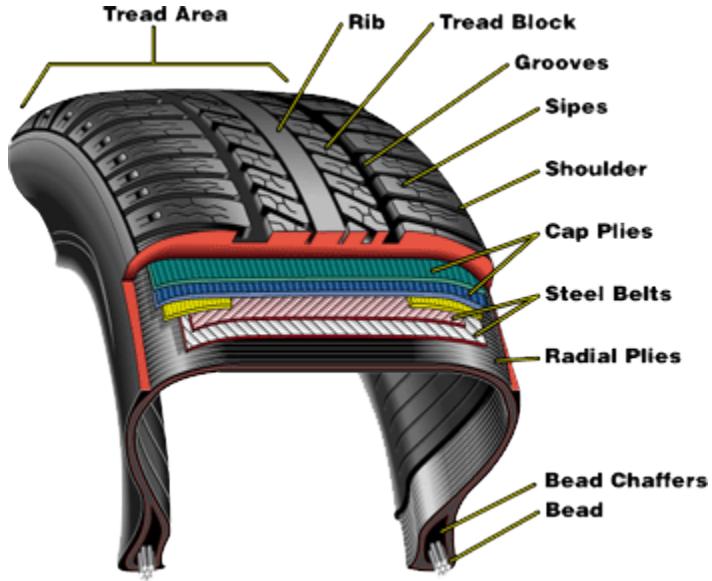


FIGURE 1. Cross section of a high-performance tire.

Table 1. Typical Weight Distribution of the Components of a Tire

Tire Components	Percentage
Natural rubber	15–19
Carbon black	24–28
Synthetic rubber	25–29
Steel cords	9–13
Textile cords	9–13
Chemical additives	14–15

Tire production is growing in proportion to demand around the world (Rubber Manufacturers Association 2009). In North America, 290 million old tires were generated in 2003 [(U.S. Environmental Protection Agency (EPA) 2013)]. Each year 33.5 million tires are retreaded, and an estimated 10 million tires are reused as second-hand tires. It is estimated that 7 percent of the discarded tires are presently being reprocessed into new products, and 11 percent are converted into energy. Nearly 78 percent of the discarded tires are being landfilled, stockpiled, or illegally dumped, with the remainder being exported (US EPA 2013). For many years, scrap tire recycling has drawn attention from both a value-added product perspective and an environmental protection perspective. Tires' main component of rubber is a rubber polymer known as styrene-butadiene-styrene, which can be used for many applications such as an asphalt modifier. Reuse and recycling of rubber can also address the negative environmental impact of disposed tires. According to tire experts, scrap tires disposed in landfills provide breeding sites for mosquitoes that can spread disease, and large tire piles often constitute fire hazards (US EPA 2013).

According to the Rubberized Asphalt Foundation, the largest tire pile in the nation is in Sycamore, Ohio. It has been estimated that this location has over eighty million tires being piled up during the past twenty years. The second

largest storage place is a location in Smithfield, Rhode Island, covering fourteen acres of land with over 10 million tires (Rubberized Asphalt Foundation 2010). Figure 2 is a picture of one of the world's largest tire dumps (EPA 2013).

Among technologies developed for the recycling of scrap tires, the most familiar and widely used is the grinding process. Most grinding mechanisms produce different particle sizes of crumb rubber, separated from steel wire. The end product of grinding tire is named ground tire rubber (GTR). GTR has been used mostly in the pavement industry since 1960 (Daryl et al. 2007). It was first implemented by Charles McDonald in Arizona (Shatanawi 2011a). A current estimate of the amount of ground tire used in civil engineering projects is 21 percent of the quantity generated annually (EPA 2013). In practice, GTR is produced using two different methods, resulting in ambient or cryogenic GTR. Ground tire can be used as a basic stock for further processes like pyrolysis and devulcanization. Another application is to use scrap tires as supplemental fuel for power plants (Barlaz et al. 1993).



FIGURE 2. One of the world's largest tire dumps at Hudson, Colorado (EPA).

The Rubber Manufacturers Association (RMA) estimates that about 290 million tires were generated in the U.S. in 2009 (Rubber Manufacturers Association 2009). It should be noted that 11 states generate 90% of these tires. As shown in Figure 3, about 60.9 million tires were recycled, 130.5 million were recovered for energy, and about 8.7 million were exported, leaving 26.1 million for landfilling, stockpiling, or illegal dumping.

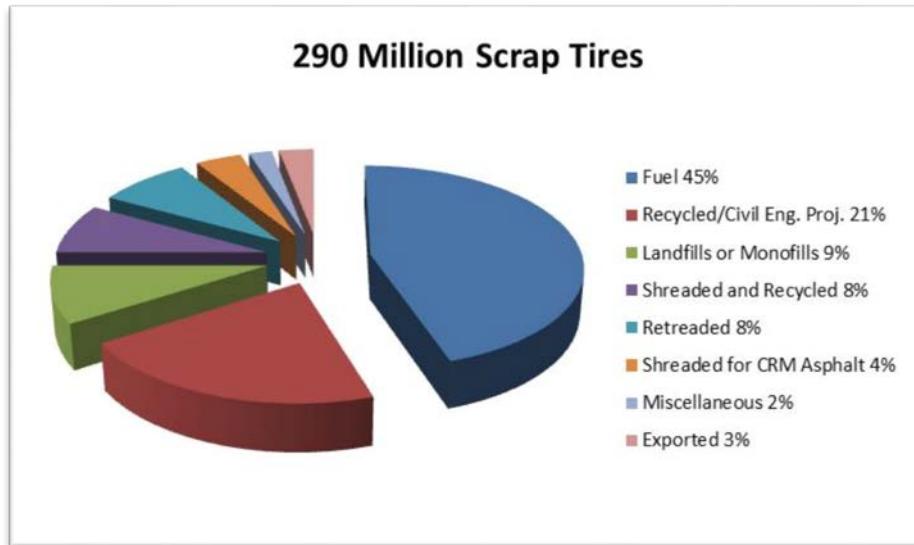


FIGURE 3. Current uses of scrap tires (US EPA 2013).

There are three commonly used recycling alternatives for scrap tires: use of whole tires, requiring no extensive processing; splitting or punching tires to make products; and finely grinding the tires to enable the manufacture of crumb rubber products. These are some applications for each alternative:

- Whole tire applications: artificial reefs and breakwaters, playground equipment, erosion control, highway crash barriers.
- Split or punched tire applications: floor mats, belts, gaskets, shoe soles, dock bumpers, seals, muffler hangers, shims, washers, and insulators.
- Shredded tire applications: lightweight road construction material, playground gravel substitutes, sludge composting, ground rubber applications, rubber and plastic products like molded floor mats, mud guards, carpet padding, plastic adhesives, rubber railroad crossings, additives for asphalt pavements.

All the tire recycling alternatives listed above are being used to some extent. However, the total usage of tires for recycling currently is estimated to be less than 8 percent of the annual generation (EPA 2013). According to the EPA, the markets for most of the above products may be increased, but even if the markets are increased to their fullest extent, the total usage appears to be small compared to the number of tires generated each year. Ground rubber applications hold the greatest promise. The tire recycling alternative with the highest potential to significantly reduce the scrap tire problem of the United States is use in asphalt highway construction (EPA 2013).

There are two types of processes for using crumb rubber in pavements. One application, called rubber-modified asphalt concrete, involves replacing some of the aggregate in the asphalt mixture with ground tires. The second application is called asphalt-rubber, defined by the American Society of Testing Materials (ASTM) as “a blend of asphalt cement, reclaimed tire rubber, and certain additives in which the rubber component is at least 15 percent by weight of the total blend and has reacted in the hot asphalt cement sufficiently to cause swelling of the rubber particles” (ASTM D-6114, 2014). Both systems are being evaluated by several state agencies as well as the federal government.

Tires can be reclaimed into virgin asphalt as CRM to produce recycled asphalt pavement (Kandhal 1992) or as an aggregate replacement in portland cement (Nehdi and Khan 2001). In addition, recent progress in rubber devulcanization facilitates recycling of scrap tires for use in new tires. Devulcanization has been reported as a highly effective method of recovering rubber polymer and transforming the structure of waste vulcanized rubber or elastomers for reuse as a virgin rubber substitute. Devulcanization can be performed mechanically or chemically using devulcanizing agents (Isayev 2005). In the chemical method of devulcanization, crumb rubber is mixed with a reagent in a simple reaction vessel. The reagent penetrates the crumb rubber and breaks the carbon-sulfur and sulfur-sulfur bonds that cross-link the linear polymers in the crumb rubber. The resulting mixture is conveyed to a filtration unit to separate it into a particulate solids stream and a liquid stream (Fan and Shafie 2013). The process can operate at low temperature under atmospheric pressure using a non-toxic and non-corrosive process (Fan and Shafie 2013).

Crumb Rubber

Crumb rubber is the name given to any material derived by reducing scrap tires or other rubber into uniform granules, removing all reinforcing materials (e.g., steel, fiber) and inert contaminants (e.g., dust, glass, rock) (Carlson and Zhu 1999). The crumb rubber is made by first processing the scrap tires to granules. The initial product consists of 2-inch chips of shredded scrap tires. The next step is to size the CRM into two different granulations: 3/4 inch and 1/4 inch. The final step is to grind the material to a size of 1 mm (10 mesh) or slightly larger, depending on the targeted application. Most crumb rubber particle sizes range between mesh #20 (1.2 mm) and #40 (0.42 mm), while some particles may be ground to a finer size such as #200 (0.075mm) (Way et al. 2011). Table 2 shows the

crumb rubber mesh size range for various applications. Table 3 shows common gradations for rubber particle sizes used in asphalt by several states (Carlson and Zhu 1999).

Table 2. Crumb Rubber Mesh Size by Market Category

Market	Mesh Sizes
Molded and Extruded Products	4 - 100 mesh
Asphalt Modification	16 - 40 mesh
Sport Surfacing	1/4" - 40 mesh
Automotive Products	10 - 40 mesh
Tires	80 - 100 mesh
Rubber and Plastic Blend	10 - 40 mesh
Construction	10 - 40 mesh

Table 3. Crumb Rubber Gradations for Asphalt-Rubber (Crumb Rubber Markets 1999)

Sieve (mm)	#8 (2.38)	#10 (2)	#16 (1.19)	#20 (0.841)	#30 (0.595)	#40 (0.4)	#50 (0.297)	#80 (0.177)	#100 (0.149)	#200 (0.074)
Arizona		100	75–100		25–100		0–45	0–0		0
California	100	95–100								0–3
Florida				100		85–100			50–30	

Tire-Derived Products

Scrap tires can be reclaimed in many different ways:

1. A steel mill can recover tires as a carbon source to replace coal or coke in steel manufacturing.
2. Tires can be bound together and used as barriers to reduce collision impact, control erosion, slow rainwater runoff, protect piers and marshes from wave action, or create sound barriers between roadways and residences.
3. Tires can be shredded to make tire-derived aggregate (TDA), which has many applications in civil engineering. To prepare TDA, tires go through a primary shredder that cuts the tires using knives that rotate at slow speed.
4. Tires can be chipped and shredded tires to make tire-derived fuel (TDF). To produce TDF-size shreds and chips, whole tires are reduced to nominal 2-inch pieces using one shredder or a series of shredders, screening equipment, and magnetic separation equipment.
5. Tires can be used to make crumb rubber; the manufacturing processes have been described in earlier sections. The finished product is mainly used in moldable products and paving projects. The resulting pavements are referred to as rubber-modified asphalt (RMA) or rubberized asphalt concrete (RAC).

Rubber-Modified Asphalt (RMA) Manufacturing Processes

Two main processes have been documented for incorporating reclaimed ground tire rubber in hot mix asphalt (HMA): the wet process and the dry process. In 1991, the Federal Highway Administration (FHWA) introduced standard terminology to improve the capability of agencies to communicate their experience when evaluating CRM processes. Figure 4 summarizes different processes and terminology.

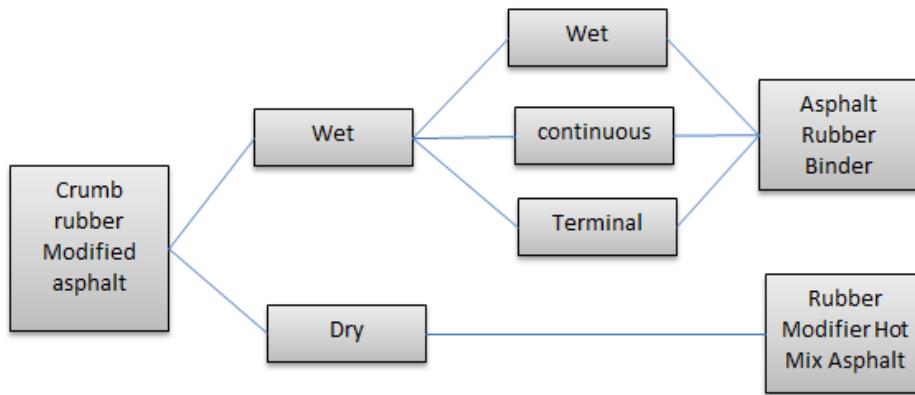


FIGURE 4. Crumb-rubber modified asphalt processes (FHWA 1994).

In the wet process, 18% to 25% of tire rubber is reacted with asphalt cement before the binder is added to the aggregate. Typically, the crumb-rubber weight is 18%, and the two base products are blended at temperatures from 330°F to 400°F (166°C to 204°C), at a reaction time ranging from 10–15 minutes up to two hours (FHWA 1997). The mixing temperature can range from 375°F to 435°F (190°C to 224°C), and the resulting blend should be kept at an elevated temperature of 375°F to 425°F (190°C to 218°C) for an indicated minimum period of time, usually 45 minutes (Caltrans 2006). The reaction is affected by the crumb rubber type and size, the temperature, the aromatic type of asphalt binder, and the type and amount of mechanical mixing (FHWA 1997).

When reclaimed tire rubber is mixed with asphalt in the wet process, the end products are referred to as asphalt-rubber binders, which are used in chip-seal coating as well as hot-mix asphalt paving. The asphalt-rubber application in chip-seal coating has been known as stress-absorbing membranes (SAM) (FHWA 1997). SAM covered with hot-mix asphalt is known as stress-absorbing membranes interlayer (SAMI).

The wet process is used to produce a large variety of crumb-rubber modified binders with different physical properties. The most important differences among various blends appear to be related to rotational viscosity of the resulting CRM-asphalt-cement blend at elevated temperature; the threshold is 1500 centipoises (cps) or 1.5 Pa.s at 375°F (190°C). The size of crumb-rubber particles and the tire-rubber content strongly affect the viscosity of the CRM-modified blend. CRM-modified binders with viscosities \geq 1500 cps at 375°F are assumed to require continuous agitation (Caltrans 2003).

The dry process is known as rubber-modified mixes, and is currently marketed in the United States under the trademark PlusRide. In this process, granulated rubber accounting for about 3% to 5% of the aggregate weight is added before incorporating asphalt binder (Roberts et al. 1989). The resulting product is referred to as rubber-modified asphalt concrete (RUMAC). Hot-mix asphalt paving (both dense-graded and open-graded) can be made using the dry process. The dry process is not usable in asphalt paving applications such as cold mix, chip seals, or surface treatments (FHWA 1997).

It has been reported that asphalt-rubber binders can enhance pavements' thermal cracking and high-temperature properties. In addition, it has been reported that asphalt-rubber mixes such as chip seals (SAM and SAMI) are effective in reducing reflective cracking in warmer climates (Massimo et al. 2012). Moreover, open-graded asphalt-rubber friction courses showed improved durability in warmer climates compared to conventional friction courses. Furthermore, a dense-graded asphalt-rubber layer has been shown to be effective for allowing paving at reduced layer thickness.

Terminal blends are classified as wet processes. In these processes, asphalt cement and crumb-rubber modified asphalt can be blended and stored for long periods of time. The processes use a fine gradation of crumb rubber extracted from 100% tire rubber, and currently use the PG grading specification system similar to polymer modified binders, with variety grading such as PG64-28TR, or PG70-22TR. These processes have been used since the 1980s in Florida, Texas, California, Colorado, Louisiana, Arizona, and Nevada (Shatanawi 2011b).

Performance History

Performance results show that adding rubber to hot-mix asphalt construction can reduce pavement rutting and increase its fatigue life. Asphalt-rubber approval systems have been primarily regional, depending on the beneficial experience gained during trial stages (Roberts et al. 1989). Several states have experienced the use of crumb rubber. The use of CRM in hot-mix asphalt increased significantly in the 1990s as a result of a government mandate; a 1992 statement indicated that 21 states use CRM in hot-mix pavement applications (Hicks and Epps 1999). According to the EPA, state departments of transportation are using large quantities of asphalt-rubber. States such as Arizona, Florida, California, Texas, and Nebraska are presently using large amounts of asphalt-rubber. South Carolina, New York and New Mexico are also studying rubberized asphalt (EPA 2013).

Arizona is a leading state in the use of asphalt-rubber. More than 14 million scrap tires have been used by the Arizona Department of Transportation (ADOT) on asphalt-rubber paving construction. It is currently estimated that three-fourths of Arizona's five million annually generated scrap tires are reclaimed in asphalt-rubber pavement construction. The number of asphalt-rubber projects has increased from one in 1988 to 54 in 2000. More than 3,000 lane-miles of streets in Phoenix, Arizona, were paved with seal coat using asphalt-rubber technology over a 20-year period (FHWA 1997). Use of one-inch thick asphalt-rubber overlay replaced the practice of chip seal in 1990. Good performance has been reported, such as reducing shrinkage cracks and alligator cracks (FHWA 1997). Even though both wet and dry processes have been used in Arizona, the most common process used in Arizona is the wet process, also called the McDonald Process.

In 1975, California started using asphalt-rubber as chip seals in laboratory experiments and small test patches, and results were promising (Caltrans 2003). The first California dry process rubber-modified asphalt concrete pavement included one percent ground rubber by mass added to the aggregate. The wet process has been extensively used; it was first implemented in pavement in California in 1980 (Caltrans 2003). Caltrans has built 17 wet-process coat installations (FHWA 1997) and has reported that rubberized asphalt concrete (RAC) out-performed conventional thicker dense-graded asphalt concrete (Caltrans 2003) by showing a lower extent of pavement distress (FHWA 1997).

In Texas, crumb rubber was first implemented in asphalt in 1976; from 1976 to 1981, 850 miles of asphalt-rubber seal coat were placed (Estakhri et al. 1990). More than 2,000 miles of asphalt-rubber chip seals (SAM's) have been built. Based on many years of experience using asphalt-rubber, the Texas DOT reported that asphalt rubber chip seal can improve resistance to alligator cracking and raveling, but resistance to shrinkage cracking may not be improved (FHWA 1997).

In 1988–1989, the Florida Department of Transportation began the use of crumb-rubber modified hot-mix asphalt (CRM-HMA). Their first trial included 5% by weight of rubber in dense-graded friction courses of 25 mm (1 in) thickness to enhance the resistance to shoving and rutting (FHWA 1995).

Rubberized asphalt matrix has been used since the sixties, when Charles McDonald employed a dry process to produce a rubberized asphalt mixture named Overflex™. Following that, rubberized asphalt continued to be explored by many researchers to better understand the interaction between rubber and asphalt when CR is added to asphalt pavements (Epps 1994). Rubberized asphalt has strong resistance to rutting (Shatanawi et al. 2008), and the rutting resistance of CR modified binders improves as the CR percentage increases (Shen et al. 2009). Moreover, adding CR can reduce penetration, temperature susceptibility, and ductility while increasing the softening point,

elastic recovery, and adhesion (Shen et al. 2009). Also, introducing specific additives to CR further improves its rheological properties (Yadollahi et al. 2010). Polyphosphoric acid (PPA) has been used as an additive to CR modified asphalt; it was found that increasing PPA content increases the values of the softening point while decreasing penetration grade (Yadollahi et al. 2010). Liu et al. (2014) used trans-polyoctenamer rubber (TOR) as an additive to CRM asphalt, and they concluded that TOR decreases the viscosity and increases the workability of CRM asphalt.

Wang et al. (2012) showed that application of CR to asphalt binder increases its viscosity significantly; to meet the Superpave maximum viscosity threshold of 3.00Pa.s, CRM asphalt needs to be heated to a higher temperature. In addition, to maintain constant viscosity, the mixing temperature has to be continuously increased as the percentage of rubber increases (Rodriguez et al. 2014). It has been shown that by introducing 15%, 20%, or 25% of CR to the base asphalt, viscosity increases significantly, to the extent that CRM asphalt's viscosity value no longer meets the threshold (Viscosity \leq 3.0 Pa.s at 135°C), so the temperature must be increased from the original 135°C to 147°C, 162°C, or 174°C, respectively, to maintain viscosity below the 3.0 Pa.s threshold (Yadollahi et al. 2010). Thus, mixing and compaction temperatures of rubberized asphalt are much higher than those of non-modified asphalt mixtures. In addition, due to the significant difference between the density of rubber compared to asphalt, rubberized asphalt tends to be prone to phase separation, which can be detrimental to rubberized asphalt's overall performance. Phase separation can cause further practical difficulties when applying rubberized asphalt, including the need for continuous mixing to prevent separation, as well as the use of higher production temperatures to maintain viscosity within the threshold. The latter can lead to additional problems with construction schedules, as well as an increase in fuel consumption used for heating purposes (Yadollahi et al., 2011). More importantly, high temperature and continuous mixing can damage the rubber structure, causing disintegration of rubber particles and leading to excessive asphalt aging (Wang et al. 2012). Rodríguez-Alloza et al. tested four different warm-mix additives to investigate their effect on high-temperature performance of 20% CRM asphalt. Their results showed that introducing these additives reduced the viscosity of CRM at high temperature (Rodriguez et al. 2014).

Crumb Rubber Leachate

Natural precipitation is the source of ground and surface water supplies when it runs off the surface of the ground into streams or soaks directly into the ground. Water that seeps into the ground travels downward due to gravity, until it reaches a depth where the soil and rock are saturated with water. As the water runs off across the surface of paved roads, parking lots, and building rooftops, it collects debris, chemicals, sediment, or other pollutants that could affect water quality if the runoff is discharged untreated.

Pavement is a mixture of aggregate and binder. The aggregate is typically from local sources and is not considered a human or ecological concern. However, the bitumen binder is composed of long-chain hydrocarbons that could be a concern. The Polycyclic aromatic hydrocarbons (PAHs) present in fuels and oils are the main environmental concern. PAHs can be present in the bitumen and have small molecular size. This means that bitumen straight out of the refinery should not contain any of these constituents. These compounds can be produced during asphalt mixture production or in the field (Mangiani et al. 2003). Previous studies found these PAHs leaching into the water: Anthracene, Benzo[a]pyrene, Pyrene, and Benz[a]anthracene (Brantley et al. 1999). Other polycyclic aromatic hydrocarbons have been found in roadway runoff and first flush storm water collection analysis in varying concentrations: Benzo(ghi)perylene, Chrysene, Coronene, Dibenz(ah)anthracene, Fluoranthene, Fluorene, Indeno(cd)pyrene, Naphtalene, Phenanthrene, and Pyrene (Krein et al. 2000).

Although the application of crumb rubber has many advantages for the asphalt industry, there is a potential problem with leachates of metals and organics (Ahmed et al. 1992). During wet seasons, there is a potential for leaching of the chemical constituents in crumb rubber and subsequent transporting of those compounds and metals to nearby surface and subsurface bodies of water. The leachates from highway CRMs are chemically complex; they are both combination of organics and inorganics in solution. Different aspects influence the release of specific

compounds from a CRM: the chemistry of the rubber, the pH, the redox (reduction/oxidation) conditions, soil sorption, and biological activity (Kriech et al. 2003; Azizian et al. 2013). Benzothiazoles are one of the rubber additives likely to be present in runoff, particularly benzothiazole itself (BTH), 2-hydroxybenzothiazole (BTOH), 2-mercaptopbenzothiazole (MBT) and benzothiazole-2-sulphonic acid (BTSA) (Mainprize et al. 2002). Other studies showed that benzothiazole, 2-hydroxybenzothiazole, and 2-(4-morpholino) benzothiazole are the major organic compounds that leach from rubber and asphalt containing 1–3% rubber (Reddy et al. 1997).

Swine-Manure-Derived Bio-Binder

In an effort to enhance rubber-asphalt interaction to improve the rheological properties of asphalt rubber and to improve the workability of rubberized asphalt, this project uses BB to promote rubber-surface activation via dissociation of rubber polymers. This BB is made through fractional distillation of bio-oils from swine manure. The efficiency of this process is reported to be approximately 70%, meaning a swine farm with 10,000 hogs per year could produce 5,000 barrels of crude bio-oil per year; the production cost of bio-oil from swine manure is estimated to be about \$0.54/gallon (cost of asphalt is about \$2/gallon), making bio-oil an economically viable modifier (Fini et al. 2012). The chemical characterization of bio-oil showed it has a significant amount of amide-based compounds, which can facilitate dissociation of rubber polymer chains, as well as bringing some fatty acids to the matrix (Hill et al. 2015). This could alleviate the dramatic increase in viscosity caused by the physical effects of rubber particles. As a result, the viscosity thresholds could be met at relatively lower temperatures. In addition, the presence of free radicals in rubber, as well as the amide groups in BB, could enhance asphalt-rubber interaction, alleviating the phase separation issue. This not only prevents disintegration of rubber particles by allowing for reduced mixing and compaction temperature, but also facilitates pumping and enhances asphalt rubber workability.

Feedstock

According to the EPA, pork is the most commonly consumed meat in the world, estimated to be 43% of world meat consumption. In addition to the meat, several valuable products or by-products come from swine, such as insulin for the regulation of diabetes, valves for human heart surgery, and gelatin for many food and non-food uses (U.S. EPA 2012). On the other hand, it is documented that more than 335 million tons of manure are generated annually in the United States; North Carolina produces 40 million tons, making the state the second largest producer of manure after Iowa (Fini et al. 2012). Swine manure management can be cumbersome; disposal of manure is usually handled by storing it in lagoons (Figure 5). This method has negative environmental consequences, such as leachate that can contaminate surface and ground water, and nauseous odors and gaseous emissions that degrade air quality. The originated gases such as hydrogen sulfide (H_2S), carbon dioxide (CO_2), ammonia (NH_3), and methane (CH_4) emitted from storage facilities can cause various diseases (Fini et al. 2012). To address these problems, Fini et al. (2012) performed extensive research on swine manure to repurpose the generated waste into useful pavement material. To do so, thermochemical liquefaction of swine manure is used to produce bio-oil, which undergoes further processing to produce the final product, Bio-binder (BB), as described in the following sections.



FIGURE 5. Swine manure storage lagoon at NCA&T farm.

Chemical Characterization

A study conducted by Fini and her team focused on evaluating the compatibility of BB with petroleum-asphalt binder, using elemental analysis to compare asphalt and bio-binder components, including saturates, aromatics, resins, and asphaltenes (SARA) fractions (Fini et al. 2011). The comparison of elemental analysis of bio-binder with a petroleum-based asphalt binder used in the Federal Highway Administration's SHRP program (AAD-1) is shown in Table 4. The chemical composition of bio-binder compared with AAD-1 is shown in Table 5.

Table 4. Comparison of SARA components of bio-binder and bituminous binder (Fini et al. 2011)

Adhesive Type	Saturate (Aliphatic) (wt%)	Naphthene Aromatics (wt%)	Polar Aromatic (Resin) (wt%)	Asphaltenes
Bio-binder (from swine manure)	2.48	1.67	45.87	43.39
AAD-1	8.6	41.3	25.1	20.5

Table 5. Chemical composition of bio-binder and bituminous binder (Fini et al. 2011)

Component Percent by weight	Bio-binder	AAD-1
Carbon (C)	72.58	81.6
Hydrogen (H)	9.76	10.8
Nitrogen (N)	4.47	0.77
Oxygen (O)	13.19	0.9

Bio-Modified Binder Previous Research

Oldham et al. (2014) investigated the use of bio-binder with wet-processed recycled asphalt shingles (RAS) particles at 5%, 15%, 30%, and 40% by weight. At 135°C, the viscosity of the binder was observed to increase as much as 117% from the neat binder; however, adding 10% Bio-binder was observed to result in a viscosity lower than the neat binder for up to 15% RAS. For 30% RAS, 10% BB decreased the viscosity 38% to within 0.2 Pa*s of the neat binder's viscosity. This shows that bio-binder is able to help alleviate the stiffening effects of stiff materials.

Oldham et al. (2012) also investigated the use of 5% bio-binder with 5%, 10%, and 15% wet-processed shredded scrap tires. At 15% scrap tires, 5% bio-binder was observed to decrease the binder viscosity 46% from 5.133 Pa.s to 2.75 Pa.s. This brought the binder viscosity below the Superpave 3.0 Pa.s maximum specification.

At the mixture level, Mogawer et al. (2012) studied the use of bio-binder with 40% RAP in asphalt mixtures while using PG 52-28 neat binder. The study found that the use of 5% BB was able to reduce the dynamic modulus (E^*) master curve closer to that of the neat binder specimens. The Overlay Tester (OT) also found that 5% BB was able to increase the cycles to failure of 40% RAP samples by 523 cycles, which was only a 48% reduction from the control specimens.

Hill et al. (2013) also investigated the low-temperature performance of bio-binder in the presence of high RAP percentages. With 15% and 45% RAP, there was a significant improvement in disk-shaped compact tension (DC(T)) test fracture energy, Superpave indirect tensile test (IDT) creep compliance, and the acoustic emission (AE) test, such that the samples with 15% RAP and 5% Bio-binder performed identically to the neat binder samples. In another study, Hill et al. (2015) found that increases in the percentage of bio-binder lead to increased improvement in low-temperature performance; however, increasing the mixing and compaction temperatures too high could increase evaporation effects that would negate the positive low-temperature performance associated with bio-binder modified samples. Therefore, for this study, the temperature at which the bio-binder modified samples are to be mixed and blended should take into consideration the possible evaporation effect.

EXPERIMENT PROCEDURE

Materials

This paper investigates the compatibility, fracture energy (using the disk-shaped compact tension test), and creep compliance of crumb-rubber modified asphalt mixtures (CRM) and bio-modified rubber mixtures (BMR). Bio-binder was added to the crumb-rubber modified asphalt to compensate for the increased energy needed for compaction of the CRM mixtures, and also to show the enhancement in fracture energy and creep compliance. Asphalt mixtures with neat asphalt, crumb-rubber modified asphalt, and bio-modified rubber asphalt with the same aggregate gradations and similar asphalt binder percentages were prepared and compacted with the Superpave gyratory compactor.

Crumb Rubber

Crumb rubber is the name given to any material derived by reducing scrap tires or other rubber into uniform granules, removing all reinforcing materials such as steel and fiber along with inert contaminants such as dust, glass, and rock (Carlson and Zhu 1999). The crumb rubber is made by first processing the scrap tires to granules. The initial product consists of 2-inch chips of shredded scrap tires. The next step is to size the CRM into two different granulations: 3/4 inch and 1/4 inch. The final step is to grind the material to a size of 1 mm (10mesh) or slightly larger, depending on the targeted application. Most crumb rubber particle sizes range between mesh #20 (1.2 mm) and #40 (0.42 mm), while some particles may be ground to a finer size such as #200 (0.075 mm) (Way et al. 2011).

Table 6 shows the crumb rubber mesh size range for various applications. Table 7 shows common gradations for rubber particle sizes used in asphalt by several states (Carlson and Zhu 1999).

Table 6. Crumb rubber mesh size by market category

Market	Mesh Sizes
Molded and Extruded Products	4 - 100 mesh
Asphalt Modification	16 - 40 mesh
Sport Surfacing	1/4" - 40 mesh
Automotive Products	10 - 40 mesh
Tires	80 - 100 mesh
Rubber and Plastic blend	10 - 40 mesh
Construction	10 - 40 mesh

Table 7. Crumb rubber gradations for asphalt-rubber (Crumb Rubber Markets 1999)

Sieves (mm)	#8 (2.38)	#10 (2)	#16 (1.19)	#20 (0.841)	#30 (0.595)	#40 (0.4)	#50 (0.297)	#80 (0.177)	#100 (0.149)	#200 (0.074)
Arizona		100	75– 100		25–100		0–45	0–10		0
California	100	95– 100								0–3
Florida				100		85–100			50–30	

The mechanical shredding at an ambient temperature produces crumb rubber modifier. Crumb Rubber Manufacturers located in Mesa, Arizona, provided the crumb rubber with a mesh size of 80-200. Table 8 shows the grading and characteristics of the crumb rubber particles. Figure 6 shows the CRM used in this study.

Table 8. Grading and characteristics of crumb rubber particles by mesh size

Sieve No.	mm	Weight Retained	Individual % Retained	Cumulative % Passing
80	0.177	42	41.50%	58.50%
100	0.149	13.7	13.60%	44.9%
120	0.125	16.5	16.3%	28.6%
140	0.105	13.9	13.7%	14.8%
170	0.088	7.2	7.1%	7.7%
200	0.074	7.8	7.7%	0.00%
Pan	N/A	3.1	0.00%	N/A
Total		104.2	100%	

Control Asphalt (Neat)

The control asphalt used in this study was PG64-22, which is the most commonly used virgin binder in North Carolina, South Carolina, Illinois, and Texas. It is used in low-volume secondary, primary, and interstate pavement construction and rehabilitation (Horan 2003). According to the material safety data sheet from the PG64-22 provider (acquired through Asphalt Associates Inc.), the melting temperature ranges from 110°F to 130°F; this is different from the heating point. Some state specifications require a maximum heating temperature of 177°C at hot-mix asphalt plants for PG64-22. The desired storage temperature ranges from 140°C to 168°C. The physical data of the base binder is presented in Table 9.

Table 9. Physical data of PG 64-22

Product name	PG 64-22
Chemical name	Petroleum asphalt
Chemical family	Petroleum hydrocarbon
Boiling point	> 900°F
Specific gravity	1.0–1.10
Vapor pressure (mm hg)	Not determined
Melting point	110 °F –130°F
Solubility in water	Negligible
Vapor density (air = 10)	Not determined
Evaporation rate (N-butyl)acetate = 1)	Not determined
pH	Neutral
Appearance and odor	Black viscous semisolid. Asphalt odor
Flash point and method	> 550°F

Bio-Binder

The bio-binder used in this study was produced in the lab through a newly developed thermochemical liquefaction process (Fini et al. 2011). The process (Figure 6) includes subjecting swine manure to a relatively high temperature (T) and pressure (P), for a specific residence time (RT): T = 340°C or 644°F, P = 17.92MPa or 2600 psi, and RT = 120mn. The process starts by mixing swine manure with water to make a slurry. Different percentages of water and manure have been investigated by the North Carolina A&T SIM LAB research group; the optimum mixing ratio that gives the maximum yield of bio-oil was determined to be 50% manure plus 50% water for a total weight of 2,900 g. A sample of mixed manure and water is shown in Figure 6. The aforementioned mix is transferred to the canister (part 1) of the reactor, an air pressure lift is used to lift the canister to be connected to the upper part (part 2), and the two parts are tightened together using a torque wrench set at 30 Nm. Two heating sleeves, 1,000 watts and 2,000 watts (part 3), are mounted to the canister. The temperature is then set to 340°C at the controller (part 4), and water is circulated through the reactor to maintain a moisture level at 20%. The reactor is heated to the set temperature (340°C), which is maintained at a constant level for 15 minutes. After the reaction is completed, the reactor is rapidly cooled to room temperature. The gas is then released from the canister, reducing the pressure in the canister to atmospheric pressure. The sticky residue is then collected from the aqueous solutions, and undergoes vacuum filtration to obtain the final product, bio-binder.

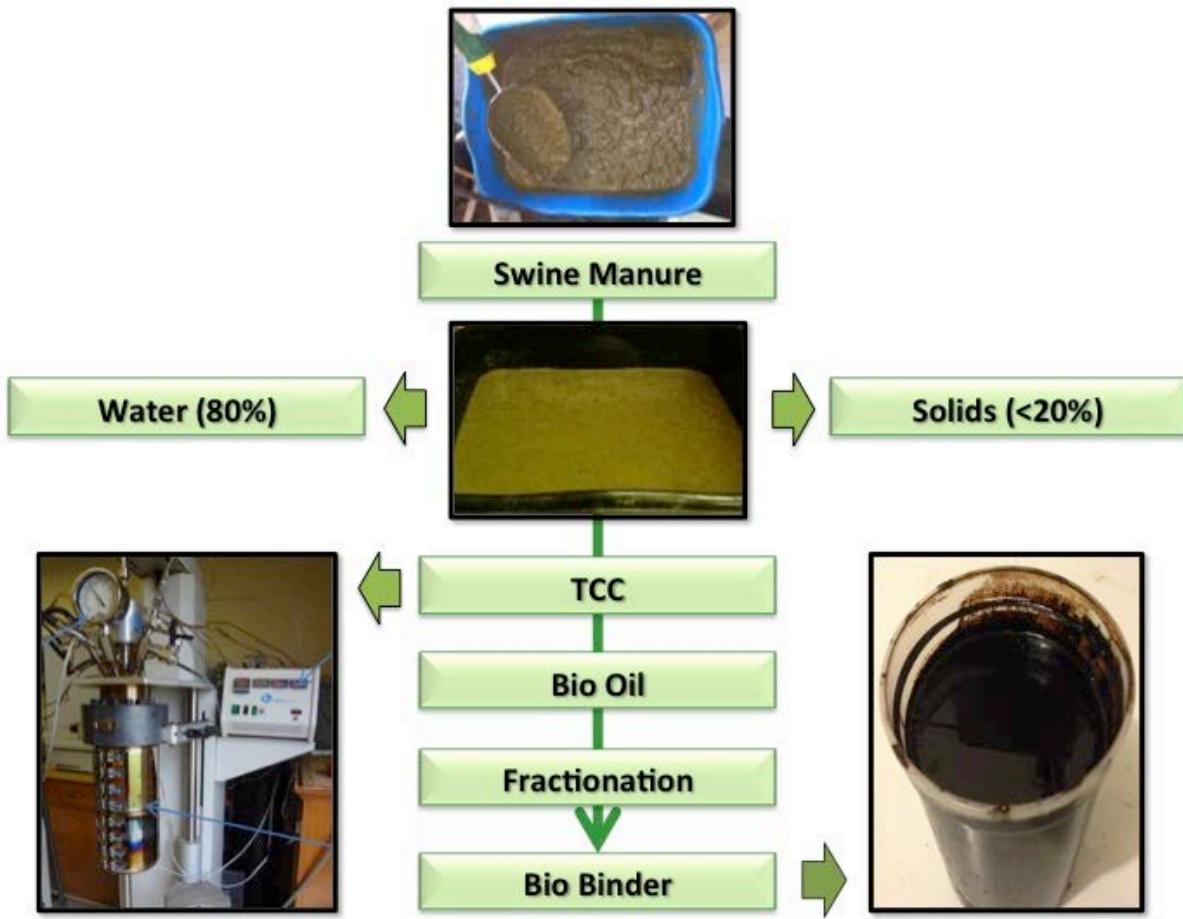


FIGURE 6. Bio-binder production diagram.

Binder Blending

The asphalt-rubber was produced in the laboratory, incorporating an ambient process CRM (provided by Crumb Rubber Manufacturers of Mesa, Arizona) at 15% by weight of asphalt binder into the PG 64-22. The blending of the CRM with the base asphalt binder (PG 64-22) was done mechanically, using a 3,000 rpm mixer equipped with an open mixing blade. This mixing condition matches the practices used in California to produce field mixtures (Caltrans 2003).

To prepare crumb rubber asphalt, the neat binder (PG 64-22) was preheated to 180°C. Then 500 grams of preheated asphalt was poured into a container, which was placed on a hot plate at 180°C. The CRM 80 mesh was gradually poured into the base binder PG64-22, the temperature was maintained at 180°C by a temperature control hot plate for 30 min, and the blending speed was held constant at 3,000 rpm. This mixing condition matches the practices used in California to produce crumb-rubber modified field mixtures (Caltrans 2003).

The same procedure was used to prepare the bio-modified rubber (BMR) specimens. The crumb-rubber modified binder was first blended with the neat binder at 180°C for 30 minutes at 3,000 rpm. Additionally, the CRM binder was preheated at 135°C before the addition of 15% bio-binder by weight. After the bio-binder was poured gradually, the contents were stirred for 30 minutes while the temperature was maintained using a temperature-controlled hot plate. The following abbreviations were used for all prepared specimens: Crumb-Rubber Modified and Bio-Modified Rubber were referred to as CRM and BMR, respectively, and Control Asphalt was referred to as Neat.

Asphalt Mix Design

Gradation

A 9.5-mm dense-graded asphalt mixture with the control asphalt (PG 64-22) was used to compare the properties of Neat, CRM, and BMR asphalt mixtures. Crushed aggregates were supplied by an NCDOT contractor, *Sharp Brothers Inc.* The aggregate and crushed sand were dried in an oven at $105 \pm 1^\circ\text{C}$ for 24 hours.

To separate different sizes of aggregate, sieves were placed in the order top to bottom of $\frac{3}{4}''$, $\frac{1}{2}''$, $\frac{3}{8}''$, #4, #8, #16, #30, #200, and the pan. One scoop (approximately 400 g) of the dried aggregate was placed on top of the sieve set after all sieves were stacked; the loaded stack was then placed in the sieve shaker for 30 min. The aggregate was then hand sieved for each sieve size (Figure 7).

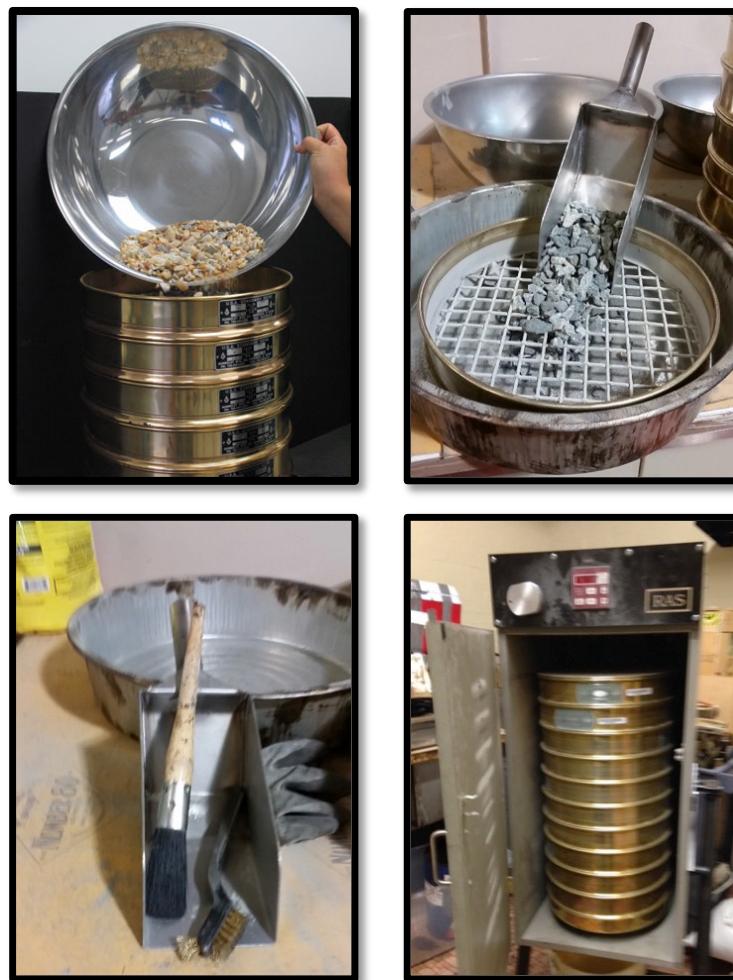


FIGURE 7. Sieves and the shaker used to separate aggregates

The specific gravity of aggregates was used in the volumetric calculations of the mixture. In this project, the ASTM C127 and C128 methods were implemented to measure bulk specific gravities of coarse and fine aggregates, respectively.

This procedure covers the determination of the average density of the particles (not including the volume of the voids between the particles), the relative density (specific gravity), and the absorption of the coarse aggregates. The test is used to determine the density of the essentially solid portion of a large number of aggregate particles and provides an average value representing the sample. Relative density is important for the calculation of the volume that will be occupied by the aggregates in the asphalt mixture. The aggregates usually have some porosity, which leads to absorption of asphalt into their pore structure, affecting the air void calculation that is a control parameter in asphalt mix design. Aggregate properties were tested separately and used as input for the mix design.

Table 10 shows the ASTM D3515 specification for the aggregate gradations for a 9.5 NMAS (nominal maximum aggregate size). This table shows the allowable range for percent passing for each control sieve.

Table 10. ASTM D3515 gradation specification for 9.5 NMAS

Sieve Number	Mesh Size (mm)	Min. Passing Percent	Max. Passing Percent
1"	12.5	100	n/a
3/8"	9.5	90	100
#4	4.75	55	85
#8	2.36	32	67
#50	0.3	7	23
#200	0.075	2	10

Table 11 shows the NCHRP 673 guidelines for the aggregate gradations for a 9.5 NMAS (nominal maximum aggregate size). This table shows the desirable range for percent passing for each control sieve.

Table 11 NCHRP 673 gradation guideline for 9.5 NMAS

Sieve Number	Mesh Size (mm)	Min. Passing Percent	Max. Passing Percent
1"	12.5	100	N/A
3/8"	9.5	90	100
#4	4.75		90
#8	2.36	32	67
#200	0.075	2	10

The final mixture aggregate gradation for this study is presented in Table 12. After selection of gradation (Figure 8), specimens were batched and mixed. Once the temperatures for both the BMR and CRM samples were determined, the samples were compacted using a Gyratory Compactor manufactured by Troxler Inc. The number of gyrations was selected based on the desired mixture properties. As the samples were being compacted, the sample heights were tracked and related to the amount of energy required for compaction as well as workability of the mixtures. Data for both CRM and BMR mixtures were recorded and analyzed.

Table 12. Final mixture gradation for 9.5 NMAS

Sieve Size	Mesh Size (mm)	Percent Passing
1"	12.5	100
3/8"	9.5	98.8
1/4"	6.35	88
#4	4.75	78
#8	2.36	57
#16	1.18	35
#30	0.6	22
#50	0.3	12.5
#100	0.15	9
#200	0.075	8

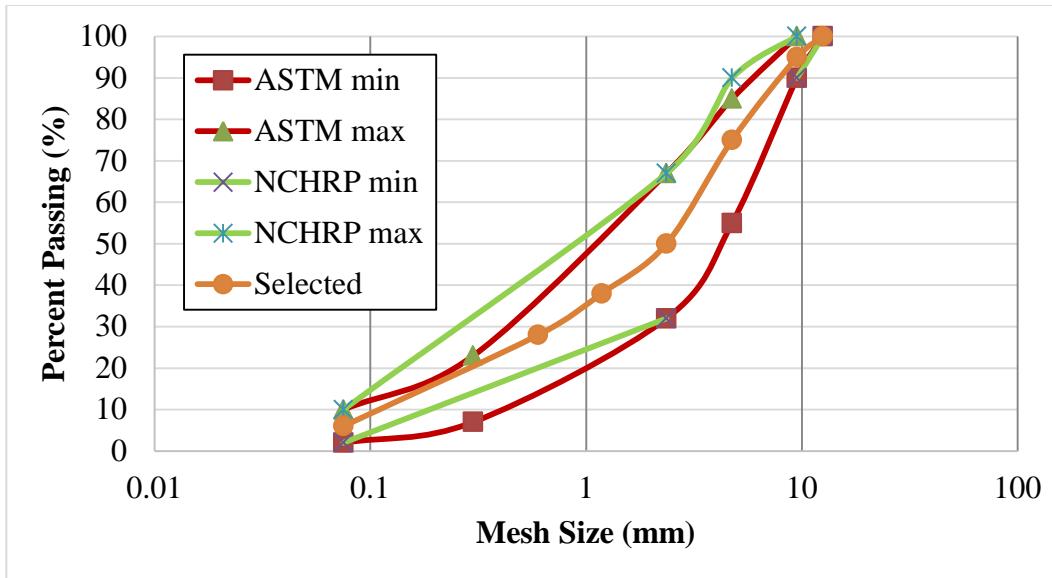


FIGURE 8. The specifications and final selected gradation.

Volumetrics

The compaction of each sample was performed using a gyratory compactor at selected temperature to nearly achieve 4% air void for all specimens. The compaction effort curves were gathered from the gyratory compactor, which shows the sample height vs. number of gyrations for both BMR and the CRM.

The weighted values as well as the calculated volumetric data of the test samples are given in Tables 13–15. To find the optimum asphalt content, four samples were prepared with the final gradations and 6.03%, 6.10%, 6.12%, and 6.20% asphalt content. The maximum theoretical specific gravities of samples were measured and calculated in compliance with ASTM D2041, and the acceptable range of VMA and VFA was checked for those trial samples.

Table 13. G_{MM} for trial asphalt mixtures

Sample No.	Asphalt Content (%)	Weight of Bowl—B (g)	Weight of Sample in Air—A (g)	Weight of Submerged Sample—C (g)	G_{MM}
1	6.03%	149	1851.3	1276.4	2.557
2	6.10%	149	1854.6	1275.9	2.549
3	6.12%	149	1858.9	1277.4	2.545
4	6.20%	149	1859.9	1273	2.527

Table 14. G_{MB} for trial asphalt mixtures

Sample No.	Asphalt Content (%)	Weight in Air (g)	Weight Submerged (g)	Weight SSD (g)	G_{MB}
1	6.03%	5060.8	2995.9	5069.2	2.441
2	6.10%	5069.1	3006.8	5078.1	2.430
3	6.12%	5060.5	2992.2	5068.1	2.429
4	6.20%	5078	2984.4	5084.2	2.444

Table 15. Volumetric properties of trial asphalt mixture samples (Continued)

Sample No.	Asphalt Content (%)	V_a (Air Voids)	G_{sb} (bulk SG)	P_s (% by mass in total mixture)	VMA	VFA
1	6.03%	4.55%	2.73	94.0%	16.0%	71.5%
2	6.10%	4.65%	2.73	93.9%	16.4%	71.7%
3	6.12%	4.56%	2.73	93.9%	16.5%	72.3%
4	6.20%	3.28%	2.73	93.9%	16.0%	79.5%

By reviewing the results and calculations, it can be concluded that the desired values could be reached by the same aggregate gradations at an asphalt content of approximately 6%. Using linear interpolation of the air voids, VMA, and VFA values, the 6.1% asphalt binder content sample is designated as the optimum asphalt content, as shown in Table 16: the air void is 4.0%, which is in the middle while VMA and VFA are 16.0% and 74.3%, respectively, and are within the allowable range.

Table 16. Volumetric properties of the final mix design

Mix Type	AC Content	Design Air Voids	VMA	VFA
Neat	6.1%	3.94	16.0	74.3
15% CRM	6.7%	4.3	17.7	75.5
15% BMR	6.7%	4.4	17.3	73.5

For the rest of the samples, the aggregate gradations, asphalt content, and gyration numbers (N_{design}) of the mixture design were kept constant. Three replicate samples were prepared for each binder (neat, CRM, and BMR) with the

same conditions, except for mixing and compaction temperatures. Accordingly, gyratory specimens were prepared for several performance tests.

EXTENDED LOW-TEMPERATURE CONDITIONING

To evaluate the performance of CRM and BMR modified samples, which undergo extended low-temperature conditions, CRM and BMR modified asphalt binder and asphalt mixtures were isothermally conditioned at -18°C for 12 hours similarly to the procedure followed by Oldham et al. (2014). After conditioning, a Bending Beam Rheometer Test and a Direct Tension Test were performed on the conditioned modified asphalt binder, while a Disk-Shaped Compact Tension Test and the Superpave Indirect Tensile Test were performed on the modified asphalt mixtures.

BINDER TESTING METHODS

Rotational Viscometer

Measurements were conducted using a Brookfield Viscometer RV-DVIII Ultra, following ASTM D4402. In this test, the viscosity, torque, and shear stress are determined by application of a rotational shear on the specimens. To prepare specimens, 10.5 grams of each sample was poured into an aluminum chamber. The tubes were then placed into a pre-heated thermosel to reach thermal equilibrium. The thermosel was preheated for at least 30 minutes. Samples and the spindle were preheated in an oven for 30 minutes before being placed into the thermosel. After loading the sample, an additional 20 minutes was taken to ensure thermal equilibrium. Three replicate readings were taken at 3-minute intervals for each test, and the average of the three values was recorded as the viscosity at the specified temperature and speed.

Dynamic Shear Rheometer

Mixing and Compaction Temperature

To determine the appropriate mixing and compaction temperatures for the 15% CRM and 15% BMR, two mixing and compaction temperature determination methods were used with the DSR. The first method, which is known as the Phase Angle Method, was performed in accordance with NCHRP Report 648. The second method is known as the Shear Flow Method, which was performed in accordance with NCHRP Report 459 from Project NCHRP 9-39.

Fatigue and Rutting Susceptibility

In order to evaluate the fatigue and rutting susceptibility of the binder, the complex modulus (G^*) and phase angle (δ) were measured using the Malvern Kinexus DSR at 0.001 to 1E9 rad/s from 76°C to 10°C , using both the 20 mm and 8 mm spindles. The DSR is used to evaluate the elastic and viscous behavior through monitoring the evolvement of shear stress and shear strain due to the application of a specified oscillation rate. Typically, 10 rad/s is used to represent the shearing action caused by a traffic speed of 90 km/h (AASHTO T 315). The measured data is then used to calculate the complex shear modulus (G^*) and phase angle (δ) (Table 17). The complex shear modulus (G^*) is a measure of material resistance to deformation when repeatedly sheared. G^* and δ values of asphalt binder are typically used to evaluate pavement rutting and fatigue cracking resistance (AASHTO T 315). The limits for unaged, RTFO, and PAV according to Superpave specifications are given in Table 17. Figure 9 shows the Dynamic Shear Rheometer.

Table 17. Intermediate temperature DSR specifications

Material	Value	Specification	HMA Distress of Concern
Unaged binder	$G^*/\sin\delta$	$\geq 1.0 \text{ kPa (0.145 psi)}$	Rutting
RTFO residue	$G^*/\sin\delta$	$\geq 2.2 \text{ kPa (0.319 psi)}$	Rutting
PAV residue	$G^*\sin\delta$	$\leq 5000 \text{ kPa (725 psi)}$	Fatigue cracking

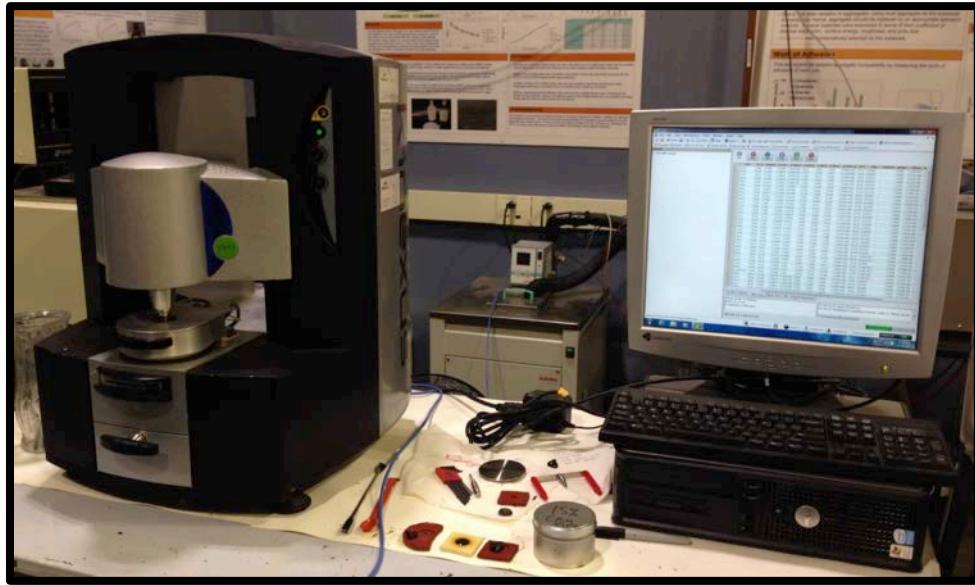


FIGURE 9. Dynamic shear rheometer.

Multiple Stress Creep Recovery

In addition to binder level analysis, the rutting performance of the mixture was evaluated using the method of multiple stress creep recovery (MSCR) as outlined in AASHTO T350. In this test, the binder is subjected to 10 cycles per stress level at 1 second loading at a specified shear stress of 0.1 kPa, then 3.2 kPa. The sample is allowed a 9-second rest period. MSCR is also a good evaluator for determining how the modifier is working in the binder.

Crossover Frequency

The crossover frequency is defined as the frequency at which the elastic modulus (G') and the viscous modulus (G'') are equal, or where the phase angle (δ) is equal to one. This parameter is related to the binder's hardness and is an indicator of the overall consistency of the asphalt (Huang et al. 2015). By generating master curves, the frequencies were converted to crossover temperatures.

Fourier Transform Infrared Spectroscopy

Characterization of molecular structure was done using Fourier Transform Infrared Attenuated Total Reflectance Spectroscopy (FTIR-ATR). Spectra are obtained by dropping thin layers of water samples on top of a zinc selenide (ZnSe) ATR prism. In this method, the total internal reflectance of infrared light in a non-absorbing prism is used. The contact of absorbing substances with the prism surface will attenuate the internally reflected light and provide

an infrared absorption spectra, corresponding to a spectra recorded if the light passed through the surface layer of the material studied (Karlsson et al. 2006).

Bending Beam Rheometer

Accordingly, the binder stiffness was evaluated using the bending beam rheometer (BBR), following AASHTO T 313. In this test, deflection at the mid-span of a binder beam was measured while a constant load of 980 mN was applied to the asphalt binder. The recorded data was then used to calculate binder stiffness after 60 seconds of loading; this value has been found to correlate with the thermal cracking of pavements. In addition to the BBR, the direct tension tester (DTT) test was also used to evaluate the binder's stiffness by determining the energy required to create two surfaces on the material, which is defined as the fracture energy. Samples were prepared and tested following ASTM D6723 at -12°C.

Direct Tension Tester

The direct tension test is another instrument used to determine the low-temperature performance of asphalt binder. The apparatus, developed by Interlaken Inc., applies direct tension to dog-bone-shaped asphalt binder specimens. To prepare samples, ASTM D6723-12 was followed. The binder is heated to a fluid-like state, poured into specific DTT molds, and allowed to sit at room temperature for one hour. Afterwards, the samples are trimmed and allowed to cool for three minutes. Samples are then placed into a freezer for seven minutes before demolding. After demolding, the samples are allowed to sit out for four minutes before being placed into the DTT methanol bath at -12°C for exactly one hour before testing until failure. Using loading and displacement data collected by the test, the fracture energy was calculated using Equation 1. Ductility was also calculated and simply defined as change in length divided by original length.

$$\text{Fracture Energy} = \frac{\sum \text{Area}_{L-D.\text{Curve}}}{\text{Area}_{C.S.A}} \quad \text{Equation 1}$$

Where:

$\text{Area}_{L-D.\text{Curve}}$: The area under the load-displacement curve (N^*m) and

$\text{Area}_{C.S.A}$: The cross-section area at fracture (m^2).

Mixture Testing Methods

Water Stripping FTIR Analysis

For this project the team followed the procedures for a batch leaching test in order to determine the adhesion performance of the modified asphalt binder with fine mineral aggregates. This test was done using deionized (DI) water as the leaching fluid, and a liquid/solid (L/S) ratio of 20.

A 200-gram specimen of uncompacted, loose material was prepared and placed in a 10-liter plastic container, along with the DI water. The sample was then subjected to vibration of approximately 50 Hz for intervals of 3 days and 7 days, while stored at room temperature (22.2°C). The testing apparatus used to collect the fine aggregate from the two modified asphalt mixtures is shown in Figure 10. The final samples from the two mixtures after conditioning are shown in Figure 11. In order to determine if the presence of the modified binder can be found on the fine aggregates after conditioning, the FTIR was utilized.



FIGURE 10. Moisture conditioning testing apparatus for CRM and BMR asphalt specimens.



FIGURE 11. Final BMR and CRM asphalt specimens for adhesion determination

Leaching Test

Leachates of CRM and BMR were tested using the Gas Chromatography–mass spectrometry (GC-MS). The samples were extracted as follows. About 10 g of sample were added to glass tubes with Teflon lids, and the tubes were filled with water, leaving about 2 ml headspace. Then the tubes were left in a water bath at 100°C for one hour. After cooling, the samples were filtered through Whatman filters with approximate particle retention size of 1.2 µm. The filtrate was mixed with hexane with ½ ratio of volume concentration. The samples were then delivered to a glass separatory funnel. The extract water was mixed with methylene chloride, and the organic part was dissolved in methylene chloride and separated in a separatory funnel. The extracted samples were injected into GC vials for determining organic compounds. A Varian 210-Ms Ion Trap Mass Spectrometer with 431-GC Gas Chromatograph was used to detect organic extracted compounds.

Moisture Test

For this study, it was important to measure the effect of water on the tensile strength of the Neat, CRM, and BMR. The test method that was followed was ASTM D4867/D4867M-09; the evaluation of the effect of moisture was done without the use of antistripping additives. The samples were all cut to a height of 50 mm \pm 5 mm with a diameter of 150 mm. There were a total of six samples for each type of asphalt. Three of the six were to be tested dry, while the other three samples were to be conditioned in the water bath as the standard stated. The standard required the samples to have an air void of 7 \pm 1%, a level expected in the field at the time of construction. It should be noted that the samples that were used were all compacted to an air void level of 4 \pm 1%. The samples were also tested using the DC(T) method following ASTM D-7313 at room temperature. The tensile ratio (TSR) values were reported as the ratio of the average of the two wet samples to the average of the two dry samples.

Hamburg Wheel Rutting Test

To determine the rutting and stripping performance of the prepared asphalt mixtures at intermediate temperatures, the Hamburg wheel tracking test (HWTT) was performed following AASHTO T 324 (Figure 12). To prepare samples, mixtures were compacted to 7% \pm 0.5% air voids. A masonry saw was used to cut along the edge of the cylindrical specimens. The samples were then placed in high-density polyethylene molds, which were placed in a stainless steel tray mounting system. The test is performed by rolling a steel wheel across the surface of an asphalt specimen that is submerged in a temperature-controlled water bath. Since the binder is a PG 64-22, the selected temperature was 50°C. Before the test started, the samples were submerged in the water for 30 minutes. The number of wheel passes and the rut depth of the sample is recorded. AASHTO T 324 does not standardize the analysis nor the reporting of the results; therefore, the state DOTs select their allowable rut depths. Based on the Texas DOT, the allowable rut depth is 12.5 mm at 20,000 wheel passes.

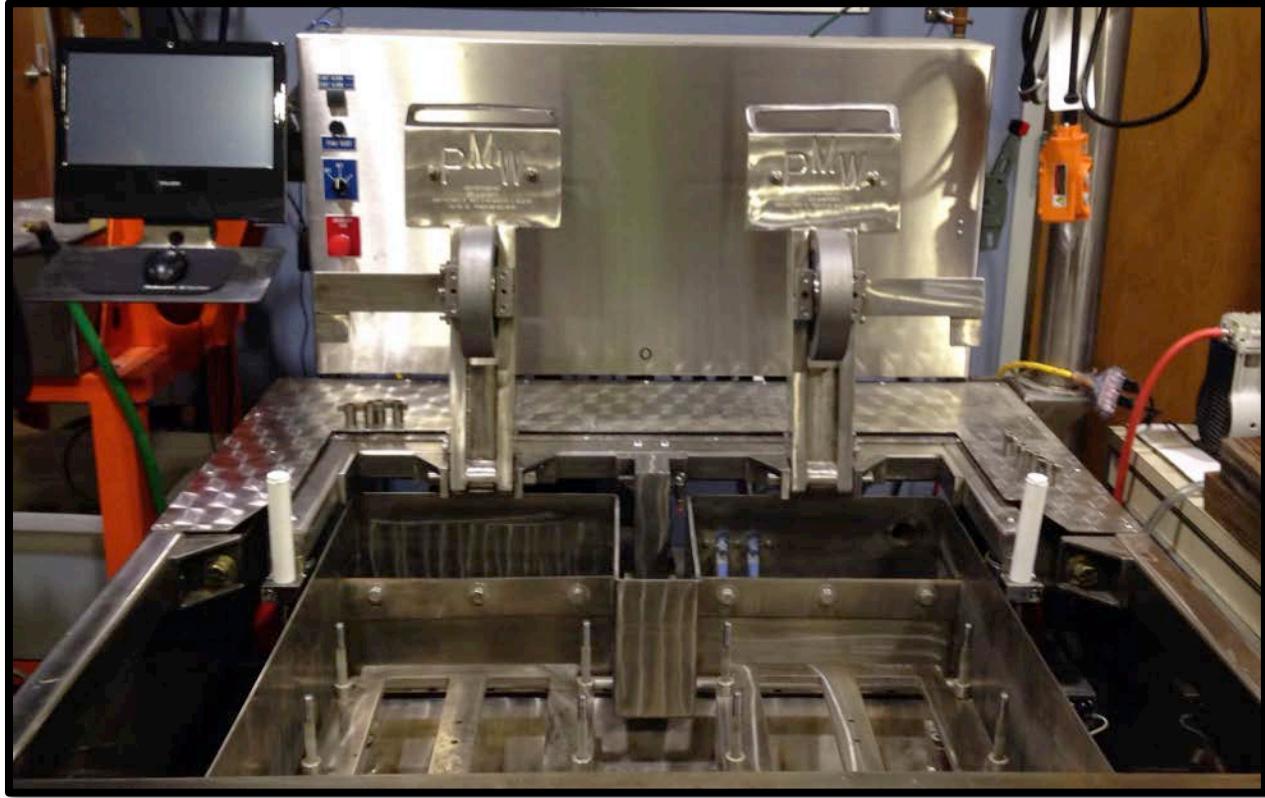


FIGURE 12. Hamburg Wheel Tracking Test (HWTT).

Disk-Shaped Compact Tension Test

In addition to binder testing, the fracture energy of asphalt mixtures at low temperature was evaluated using the disk-shaped compact tension [DC(T)] test. Using Mode I fracture, the DC (T) samples were subjected to an applied load related to a displacement rate of 1 mm/min, which is regulated using a crack-mouth opening displacement (CMOD) gauge. Sample preparation and testing procedures were performed in accordance with ASTM D-7313. It should be noted that nearly all samples were compacted to an air void of 4%. The samples were tested using an IPC Universal Testing Machine (UTM) at -12°C (Figure 13).



FIGURE 13. Disk-shaped compaction tension (DCT) test.

Superpave Indirect Tensile Creep Test

Additionally, in order to determine the creep compliance and relaxation properties of the CRM asphalt and BMR asphalt mixtures, the indirect tensile test (IDT) was utilized, following AASHTO T-322 (Figure 14). In this test, the cylindrical sample was subjected to horizontal and vertical deformation by a constant creep load for 1,000 seconds. The deformation under the load was recorded using four linear variable differential transformers (LVDT). Following the standard, the samples were tested at 0°C, -12°C, and -24°C, and master curves were generated using the principle of time-temperature superposition. The data was then fitted using the power-law model with Equation 2. A least squares fitting method was used to determine D_0 , D_1 , and m for each case.

$$D(T) = D_0 + D_1 t^m \quad \text{Eq. 2}$$

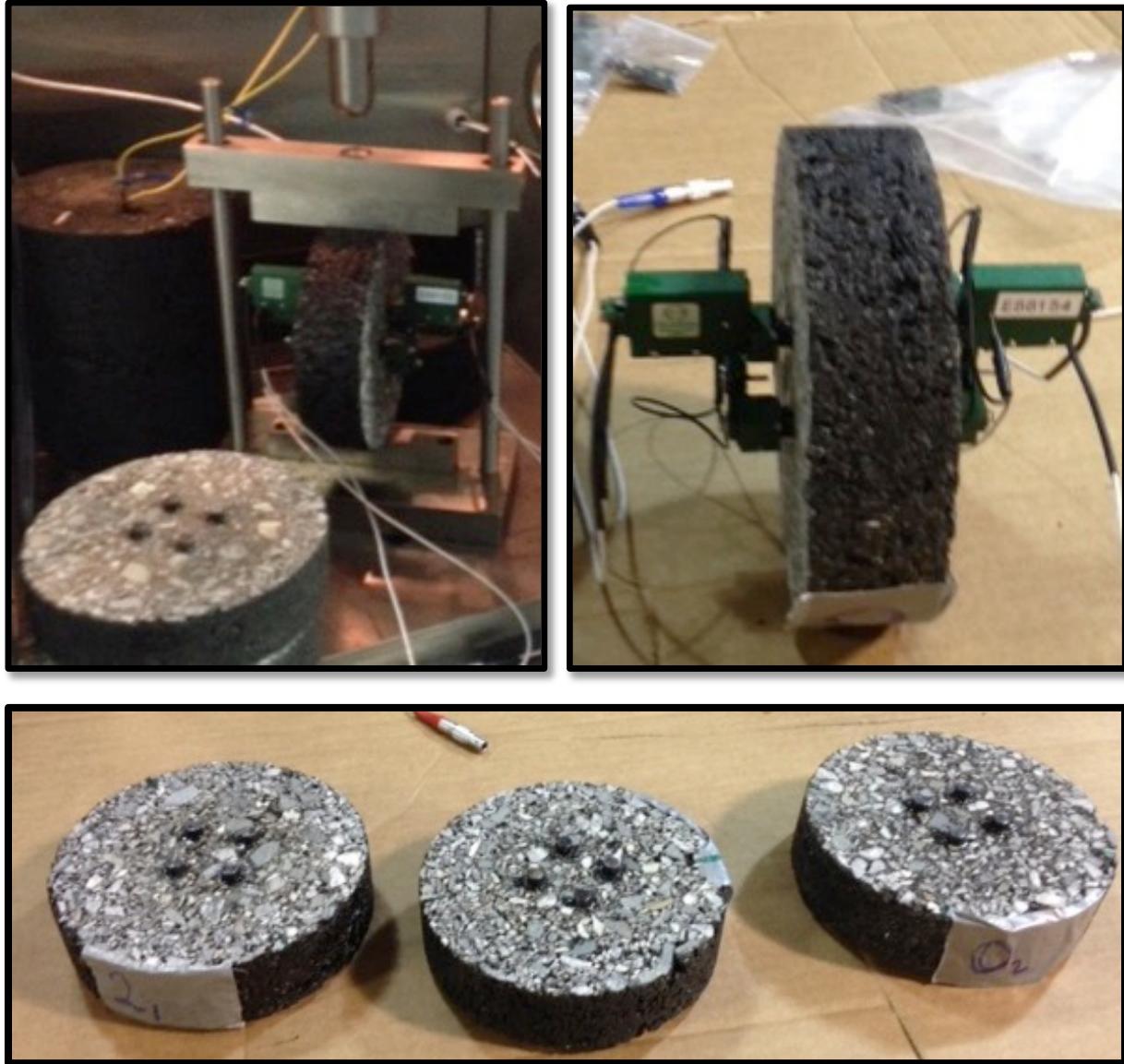


FIGURE 14. Indirect tensile test (IDT) samples.

PRELIMINARY BINDER STUDY

To determine the optimal crumb rubber and bio-binder percentage combination, preliminary specimens were tested using the rotational viscometer, the dynamic shear rheometer, and Fourier Transform infrared spectroscopy.

Rotational Viscometer

Figures 15 and 16 show the viscosity results of 15% and 20% CRM asphalt binder at four different testing temperatures and various percentages of BB. The figures show that the viscosity of each sample decreases as the temperature increases, and the addition of BB results in a reduction of rubberized asphalt's viscosity for both percentages of crumb rubber content. Accordingly, the introduction of up to 20% BB to rubberized asphalt with

concentrations of 15% and 20% of CRM decreased the overall viscosity by 71% and 38%, respectively. For both 15% and 20% CRM, the viscosity reduction was found to be nearly the same for BB percentages of 10%, 15%, and 20%.

To further study the effect of increasing BB content on the rubberized asphalt binder viscosity, Figure 15 shows the results for the asphalt binder modified with 20% of CR and 30% and 50% BB. Similarly to the 15% CRM samples shown in Figure 16, the viscosity remained nearly the same up to 20% BB; however, when 30% BB was added, a significant reduction in viscosity was observed as well as at 50% BB. The fact that the viscosity showed an initial reduction at 5% BB could be attributed to the lubricating effect of BB. However, when the BB content is increased from 10% to 20%, the viscosity remained nearly constant. This could be attributed to the adsorption of BB to the surface of rubber particles, leading to progressive surface treatment of rubber particles with BB and the consequent interaction being with free radicals and the formation of bonds between disulfide and styrene butadiene polymers.

The BB level of 30% and 50% showed a continuous reduction in viscosity, indicating that BB can saturate the rubber surface at a certain percentage above which BB would work as a dilutant. Thus, it was concluded that the addition of BB to rubberized asphalt could be divided into three phases: lubrication, surface treatment, and dilution. However, this depends on the ratio between BB content and rubber effective surface area. Therefore, the BB content should be designed to maximize rubber surface treatment while avoiding dilution.

Figure 15 shows the viscosity vs. temperature for bio-modified rubber asphalts at 15% rubber (15% CRM) concentration with 5%, 10%, 15%, and 20% concentration of BB, abbreviated as BMR15-5, BMR15-10, BMR15-15 and BMR15-20, respectively. Figure 16 shows 20% CRM with two additional percentages of BB: 30% and 50%.

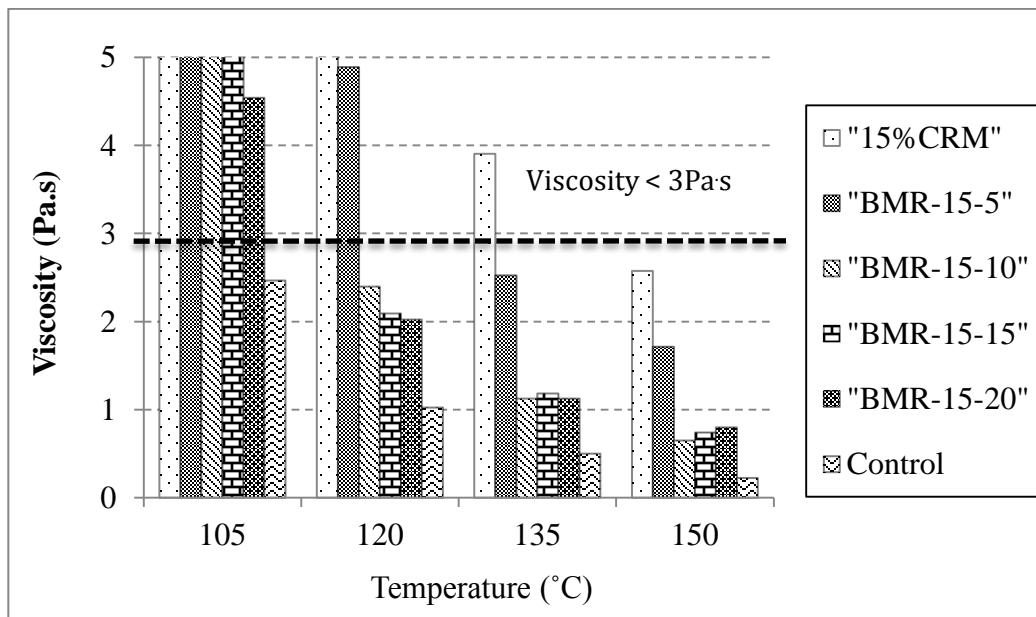


FIGURE 15. Viscosity for BMR asphalt binder at 15% CRM with 0 to 20% BB at 105°C, 120°C, 135°C, and 150°C.

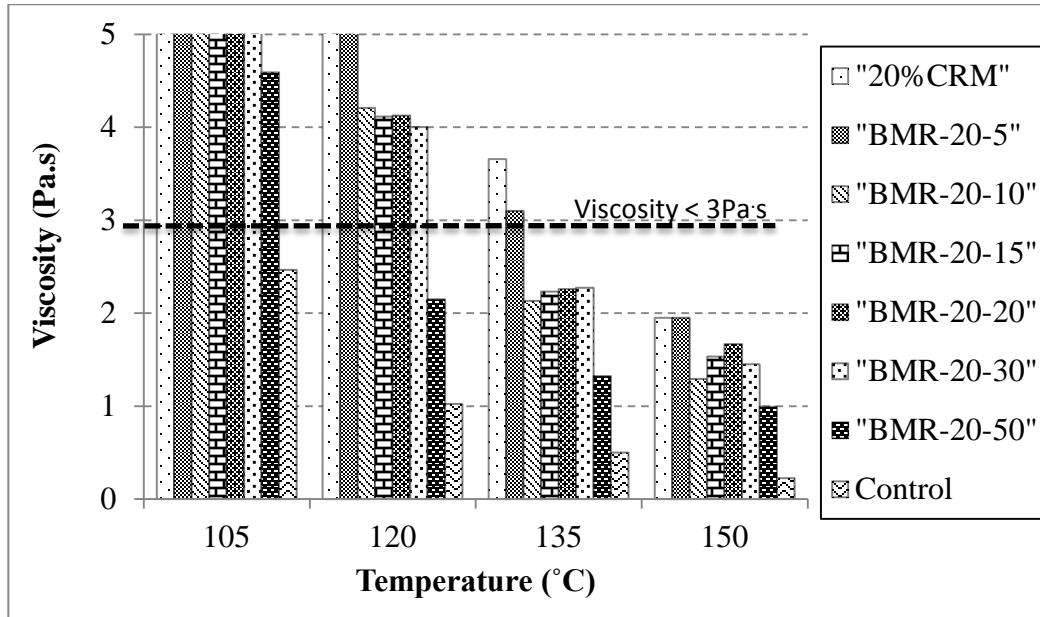


FIGURE 16. Viscosity for BMR asphalt binder at 20% CRM with 0 to 20% BB at 105, 120, 135, and 150°C.

Figure 17 shows the viscosity of all specimens at a standard test temperature of 135°C and 20 RPM, following the Superpave binder specifications (ASTM D4402). It can be seen that for asphalt with 15% or 20% CRM, the viscosity is above the maximum Superpave specification of 3 Pa·s. However, the introduction of 5% bio-binder to both 15% CRM and 20% CRM was shown to reduce the CRM viscosity to a level below the maximum allowed value. Accordingly, all of the BMR samples had a viscosity value below 3 Pa·s, meeting the Superpave specification limit.

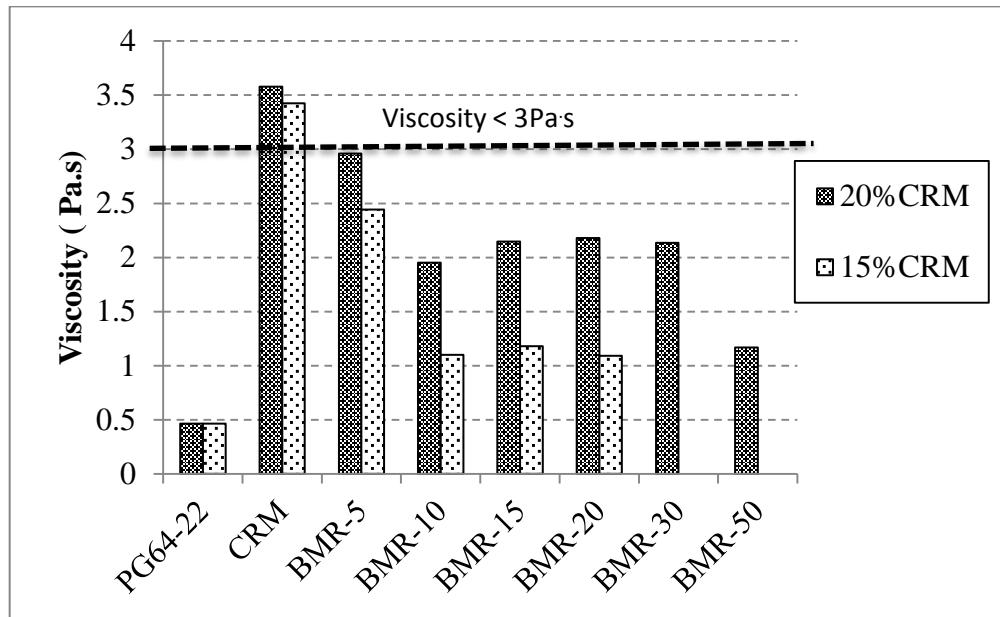


FIGURE 17. Viscosity Results of 15% and 20% CRM with 0 to 50% BB at 135°C.

Dynamic Shear Rheometer

Complex Modulus

The DSR tests were conducted following ASTM D7175 to characterize the viscoelastic behavior of CRM asphalt before and after the addition of BB. Complex modulus master curves for RTFO aged samples are presented in Figure 18 and Figure 19. The figures show that as the CR content increases, so does the complex modulus, regardless of the reduced frequency. In addition, it was found that G^* of the aforementioned specimens decrease as the amount of BB increases. Similar trends were observed before and after samples were exposed to oxidative aging.

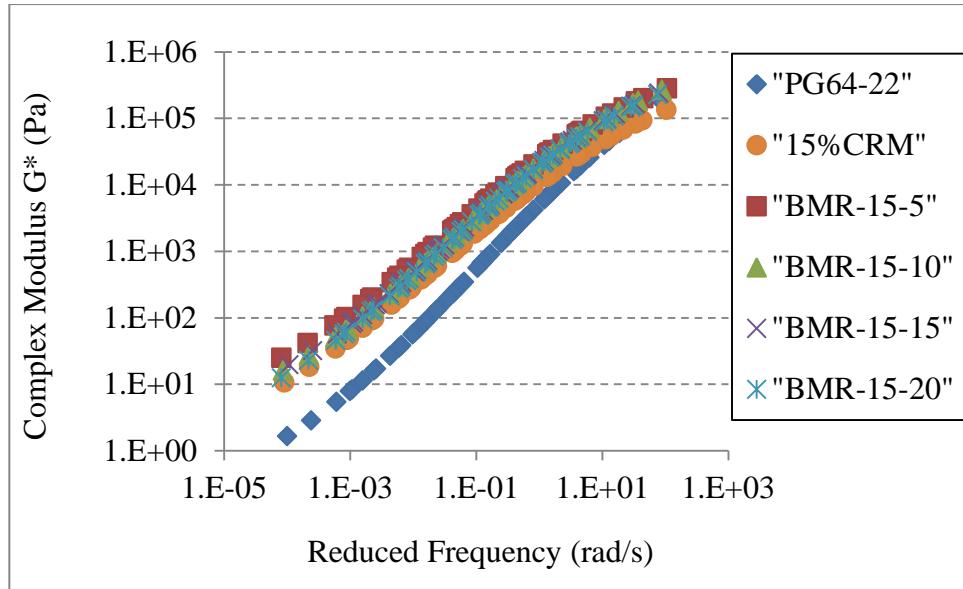


FIGURE 18. Complex modulus master curves for RTFO aged specimens at 15% CRM with 0 to 20% BB.

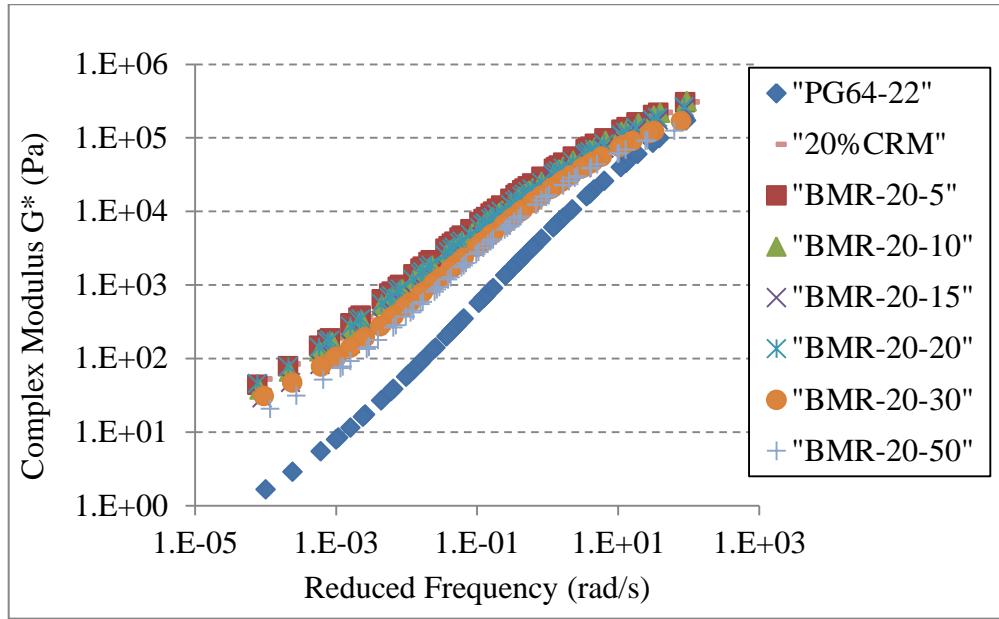


FIGURE 19. Complex modulus master curves for RTFO aged specimens at 20% CRM with 0 to 50% BB.

Temperature Effect on $G^/\sin(\delta)$*

The effect of temperature on $G^*/\sin(\delta)$ for unaged and aged samples is shown in Figures 20 and 21, which are plotted based on the measurement at 64°C at 10 rad/s. The Superpave specification classifies the parameter $G^*/\sin(\delta)$ as an indicator for evaluating the rutting resistance of binders. It was found that for both aged and unaged samples, the rutting parameters for all BMRs are considerably higher than that of control asphalt, and this parameter was increased by increasing the CR content. It was also observed that after adding BB to CRM asphalt, $G^*/\sin(\delta)$ was decreased to some extent; however, all values were still significantly higher than that of control asphalt both before and after aging. According to the Superpave specification, the high-temperature grade of asphalt is defined as the temperature at which $G^*/\sin(\delta)$ of the asphalt binder has a minimum value of 1 kPa before aging and 2.2 kPa after aging. Accordingly, all samples satisfied the rutting criteria at 64°C.

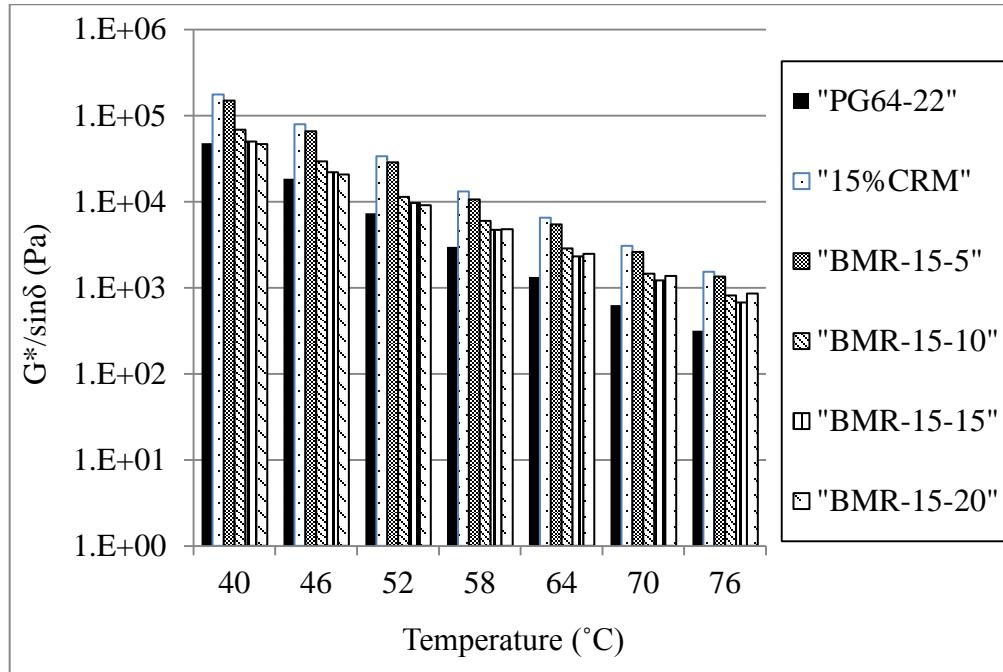


FIGURE 20. $G^*/\sin\delta$ results for Unaged 15% CRM with 0 to 20% BB at 40 to 76°C.

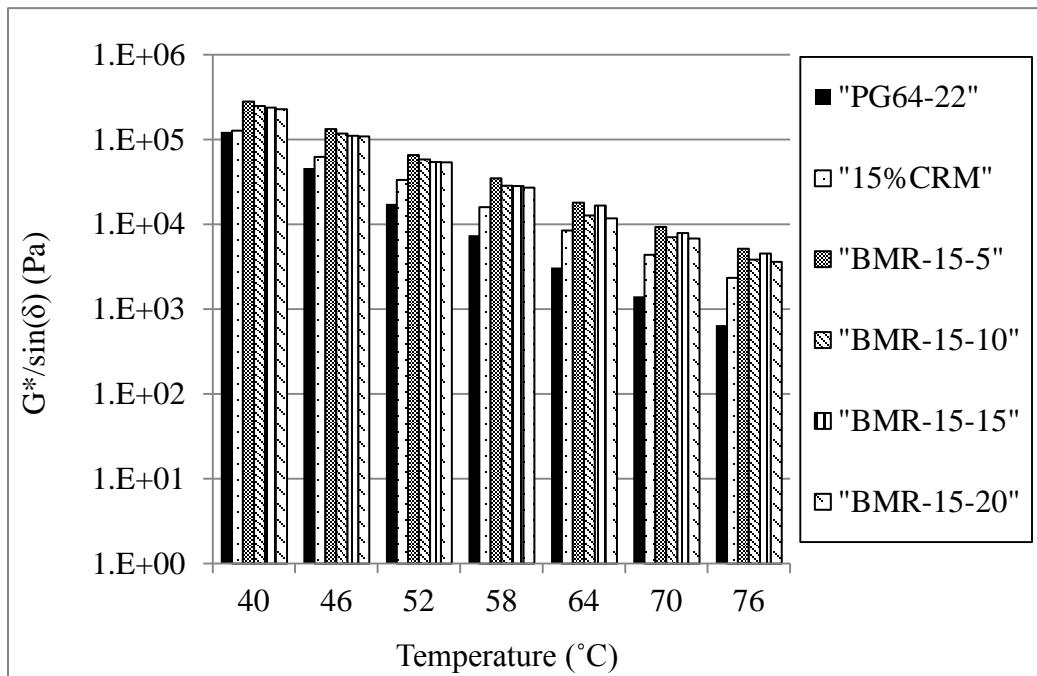


FIGURE 21. $G^*/\sin\delta$ results for RTFO Aged 15% CRM with 0 to 20% BB at 40 to 76°C.

Temperature Effect on Phase Angle (δ) of Unaged and RTFO Specimens

To study viscoelastic properties of bio-modified rubber asphalt (BMR), the phase angle at a frequency of 10 rad/sec was measured at different temperatures for all specimens using a dynamic shear rheometer. The phase angle (δ) is a measure of the lag time between the applied shear stress and the resulting shear strain. Materials with lower phase angle indicate more elastic behavior, while those with higher phase angle show more viscous behavior. As shown in Figures 22 and 23, the phase angle (δ) for the control binder (PG 64-22) has the highest value for both aged and un-aged specimens. It can be further observed that the introduction of 15% or 20% CRM into the control binder decreased the phase angle values significantly for both aged and un-aged specimens; this reduction was found to be proportional to the CRM content. However, when 5% bio-binder was introduced to each CRM, its phase angle increased; to some extent, this was when bio-modification beyond 10% showed a significant shift in the phase angle as well as its temperature dependence. This could be attributed to the interaction of CRM and BB, which was even more evident at high percentages of bio-modification; the BB promoted the surface treatment of rubber particles while interacting with free radicals and the disulfide bonds between the styrene butadiene polymers of rubber particles. While Figures 8 and 9 show the result for 15% CRM, the trend was found to be the same for 20% CRM. In addition, the phase angle continued to decrease when bio-binder content increased from 10% to 15% and 20%.

Binders with a high complex modulus (G^*) and low phase angle (δ) value are more resistant to permanent deformation (rutting). A decrease in phase angle (δ) values could be indicative of improvement in the elastic property of a material. For specimens with 15% CRM concentration, the higher the bio-binder content, the lower the phase angle. This means a 15% CRM specimen with higher bio-binder content (e.g., 20% bio-binder) could be less prone to rutting. For specimens with 20% CRM, a similar trend was observed.

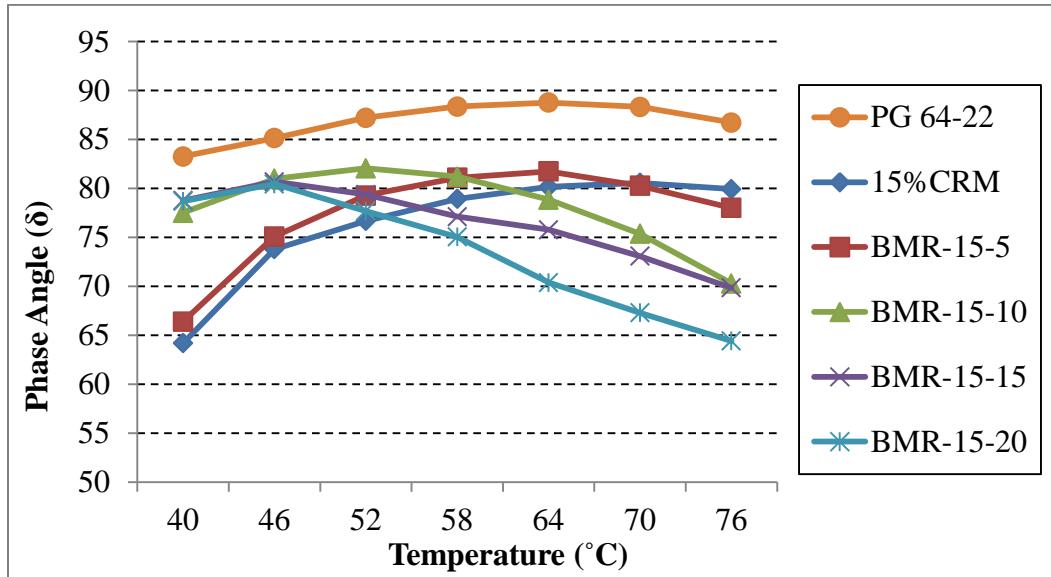


FIGURE 22. Effect of temperature on phase angle (δ), Un-aged 15%CRM.

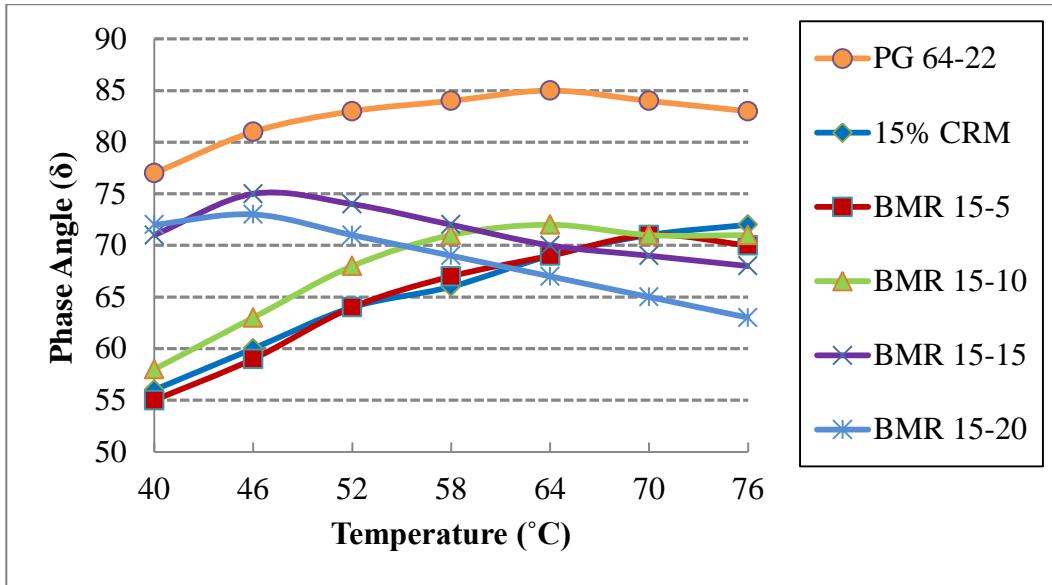


FIGURE 23. Effect of temperature on phase angle (δ), Aged 15%CRM.

FTIR Analysis

The main polymer components of crumb rubber are natural rubber, Butadiene styrene rubber, and cis-butadiene rubber. Each of these polymers has special related peaks; these spectra help to better understand the release of the polymer into the asphalt matrix. Figure 24 shows the spectra for BMR15-10 (15% CRM and 10% bio-binder), for which a significant shift in viscoelastic properties was observed. The presence of the peak around 968 cm^{-1} is attributed to the trans and vinyl double bond out-of-plane C-H bending (Masson et al. 2011). One of the dominant peaks is due to the out-of-plane aromatic C-H deformation at 700 cm^{-1} , which can indicate the dissolution and release of some components of rubber following rubber swelling and absorption of light components of asphalt binder. The peak at 1260 cm^{-1} is attributed to the esters, which may relate to curing part of the rubber. Another peak at 757 cm^{-1} was attributed to the CH out-of-plane deformation (Baeta et al. 2009). The appearance of these new peaks confirms the release of some hydrocarbons from the rubber to the asphalt matrix.

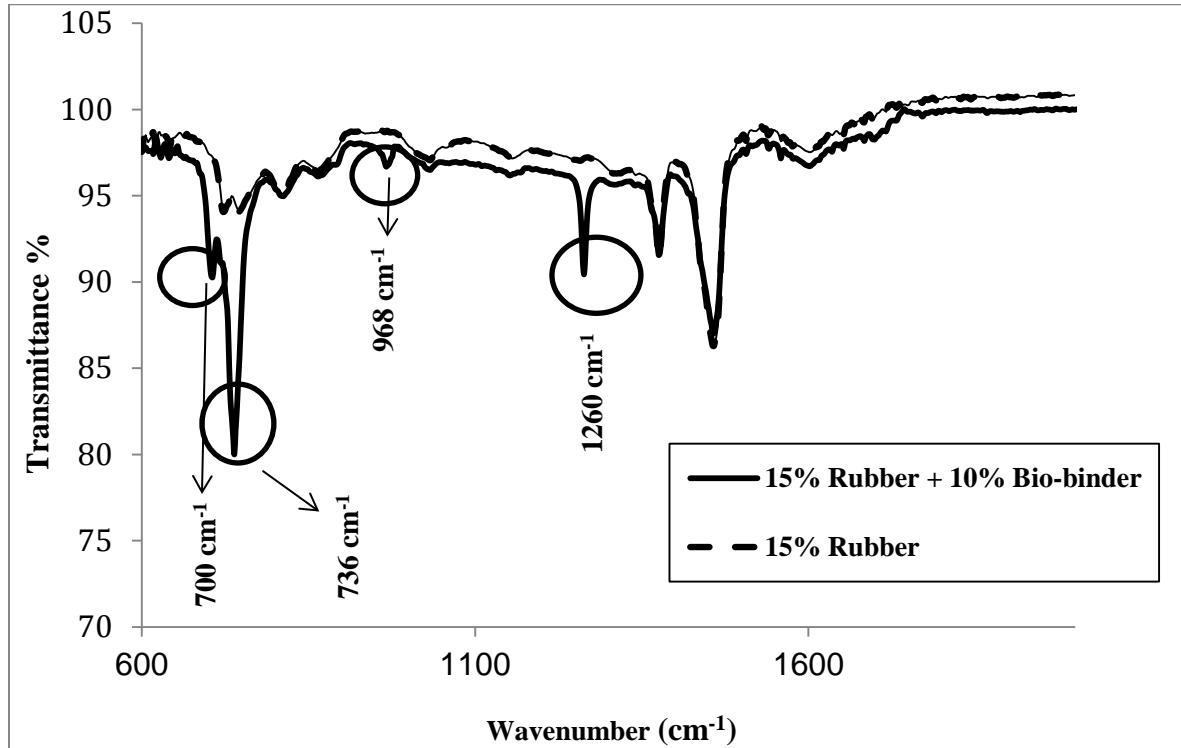


FIGURE 24. FTIR spectra for 15% CRM with and without 10% Bio-binder.

Preliminary Results Conclusion

From the RV, DSR, and FTIR results, the recommended crumb rubber and bio-binder percentages were 15%. Therefore, the following results were based on 15% CRM and 15% BMR modified asphalt.

RESULTS AND DISCUSSION

Binder Section

Rotational Viscometer

To study the effects of rubber and bio-binder on the workability and pumpability of CRM and BMR asphalt binder, the viscosities are given in Figure 25. For selecting the appropriate mixing and compacting temperatures, the proposed viscosity range for the laboratory mixtures is given by ASTM D6925. Based on the aforementioned guideline, the mixing temperature is defined as the temperature at which the viscosity is 170 ± 20 cP, and the compaction temperature is the one at which the viscosity is 280 ± 30 cP. Based on the measured viscosities for each scenario, the mixing temperature range for the neat, CRM, and BMR asphalts were determined to be $154.5^\circ\text{C} - 159.9^\circ\text{C}$, $215^\circ\text{C} - 221.5^\circ\text{C}$, and $207^\circ\text{C} - 215.4^\circ\text{C}$, respectively. In addition, the compaction temperature range for the neat, CRM, and BMR asphalts were selected to be $144.5^\circ\text{C} - 149^\circ\text{C}$, $201.5^\circ\text{C} - 207.5^\circ\text{C}$, and $190^\circ\text{C} - 197.5^\circ\text{C}$, respectively. It has been reported that the aforementioned method may overestimate the mixing temperature for modified binder (REF); therefore, additional mixing and compaction testing was performed using the Dynamic Shear Rheometer.

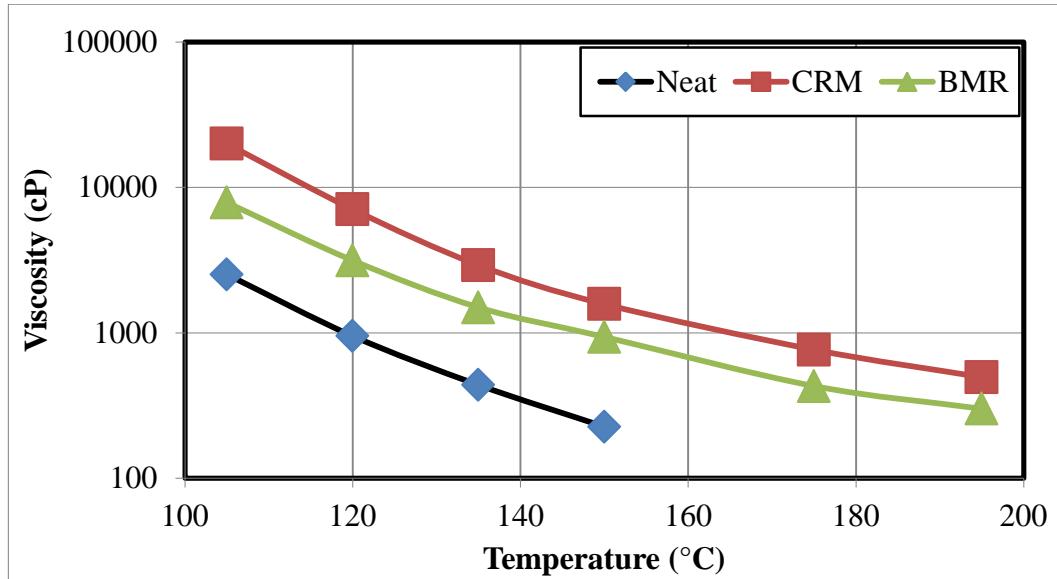


FIGURE 25. Viscosity results for neat, 15% CRM, and 15% BMR at 20 rpm.

Dynamic Shear Rheometer

Mixing and Compaction Temperature

In Table 18, the mixing and compaction temperatures for 15% CRM and 15% BMR are given using both the Phase Angle and the Shear Flow Method. For 15% CRM, the phase angle never went below 86°, so a mixing and compaction temperature could not be determined for 15% CRM using that method; however, the phase angle method showed that 15% BMR could have a significant reduction in mixing and compaction temperatures compared to 15% CRM. Using the Shear Flow method, 15% CRM and 15% BMR appear to show very little difference.

Table 18. Mixing and compaction temperatures from DSR

	Phase Angle Method		Shear Flow Method	
	Mixing	Compaction	Mixing	Compaction
15% CRM	N/A	N/A	363.9°F/184.4°C	363.9°F/184.4°C
15% BMR	316.1°F/157.8°C	292.7°F/144.8°C	366.3°F/185.7°C	366.3°F/185.7°C

Unaged G/sinδ Rutting Performance*

Based on the DSR test results shown in Figure 26 and Table 19, the intermediate temperature grade performance grade of the unaged Neat, CRM, and BMR for rutting was evaluated. The samples were found to have maximum

passing temperatures of 70°C, 82°C, and 76°C for neat, CRM, and BMR respectively. This in turn indicates that the addition of crumb rubber significantly improves the high temperature performance.

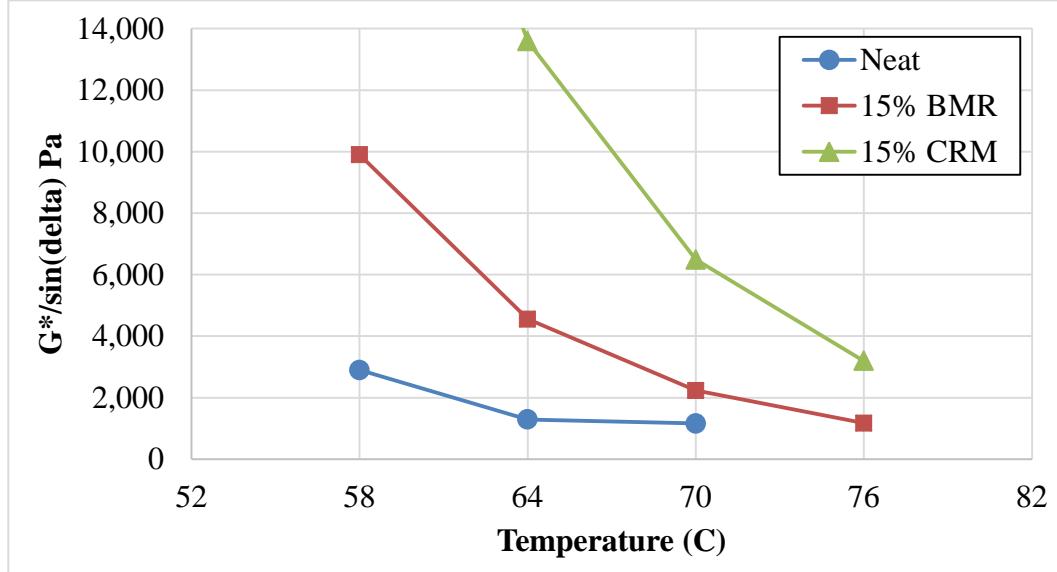


FIGURE 26. DSR results, $G^*/\sin \delta$, of unaged samples at HT range.

Table 19. $G^*/\sin \delta$ DSR results for unaged binder

Unaged Rutting Parameter			Pass / Fail		
Temperature	Parameter	Criteria	Neat	CRM	BMR
52	$G^*/\sin \delta$	≥ 1.0 kPa			Pass
58	$G^*/\sin \delta$	≥ 1.0 kPa	Pass		Pass
64	$G^*/\sin \delta$	≥ 1.0 kPa	Pass	Pass	Pass
70	$G^*/\sin \delta$	≥ 1.0 kPa	Pass	Pass	Pass
76	$G^*/\sin \delta$	≥ 1.0 kPa	Fail	Pass	Pass
82	$G^*/\sin \delta$	≥ 1.0 kPa		Pass	Fail
88	$G^*/\sin \delta$	≥ 1.0 kPa			Fail

Multiple Stress Creep Recovery

Table 20 shows the MSCR results for unaged, RTFO, and PAV CRM and BMR samples. The results show that BMR had consistently higher non-recoverable creep compliance (Jnr) and lower percent recovery, compared to CRM for unaged, RTFO, and PAV aged samples. However, both were higher than measured values for control asphalt, showing the rubber modification had significant effect on improving percent recovery.

Table 20. MSCR results for unaged, RTFO, and PAV aged BMR at CRM at 64°C

	Unaged			Test Result
	$J_{nr} \text{ } 3.2\text{kPa}^{-1}$	$J_{nr} \text{ diff}$	% Recovery 3.2 kPa^{-1}	
15% BMR	2.251	66.4	2.1	Pass [S]
15% CRM	0.5813	58.8	11.2	
	RTFO			Test Result
	$J_{nr} \text{ } 3.2\text{kPa}^{-1}$	$J_{nr} \text{ diff}$	% Recovery 3.2 kPa^{-1}	
15% BMR	0.1043	42.9	50.8	Fail
15% CRM	0.0078	202.8	89.7	
	PAV			Test Result
	$J_{nr} \text{ } 3.2\text{kPa}^{-1}$	$J_{nr} \text{ diff}$	% Recovery 3.2 kPa^{-1}	
15% BMR	0.01249	25.4	82.3	Pass [E]
15% CRM	0.00282	25.9	92.1	

*PAV G*sind Fatigue Performance*

Based on DSR test results shown in Figure 27 and Table 21, the low intermediate temperature grade performance grade of neat, CRM, and BMR for rutting was evaluated. The lowest temperatures of which the samples passed the low temperature fatigue cracking parameter were found to be 22°C, 16°C, and 22°C, for neat, CRM, and BMR. This in turn indicates that addition of crumb rubber decreased the low temperature performance in terms of fatigue cracking; however, the addition of BB helped restore the fatigue performance to that of the neat binder.

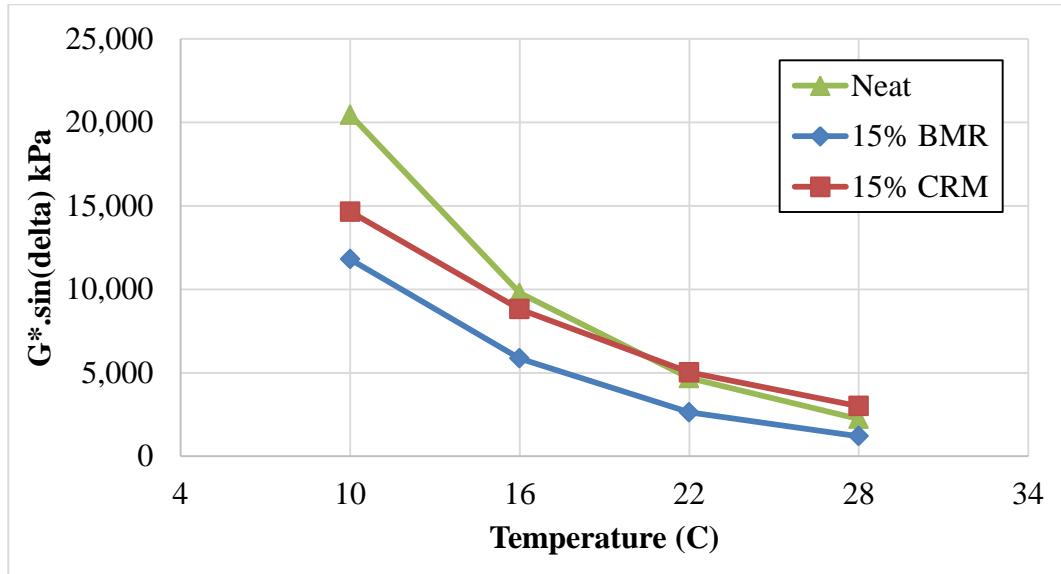


FIGURE 27. DSR results, $G^* \sin \delta$, of PAV aged samples at IT range.

Table 21. $G^* \sin \delta$ DSR results for PAV binder

PAV Fatigue Cracking Parameter			Pass/Fail		
Temperature	Parameter	Criteria	Neat	CRM	BMR
28	$G^* \sin \delta$	≤ 5.0 kPa	Pass	Pass	Pass
22	$G^* \sin \delta$	≤ 5.0 kPa	Pass	Pass	Pass
16	$G^* \sin \delta$	≤ 5.0 kPa	Fail	Fail	Pass
10	$G^* \sin \delta$	≤ 5.0 kPa			Fail

Crossover Frequency

From Figure 28, the crossover temperatures are given for the unaged, RTFO, and PAV CRM and BMR modified binders. The crossover temperatures of the BMR are shown to be consistently lower compared to the CRM specimens, even after short- and long-term aging.

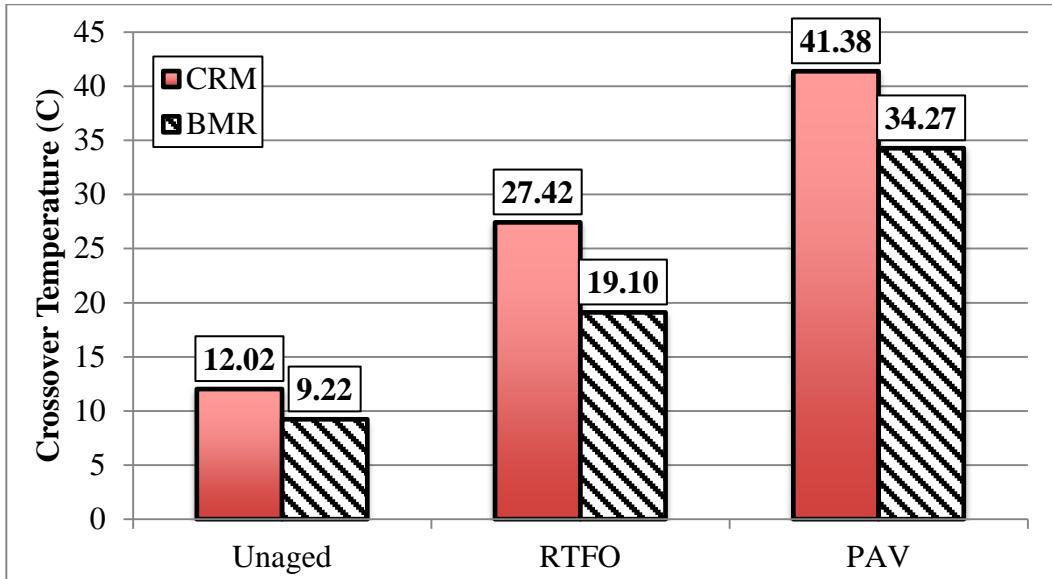


FIGURE 28. Crossover temperatures from BMR and CRM crossover frequencies for unaged, RTFO, and PAV specimens.

Bending Beam Rheometer

To study the effects of conditioning time, the BBR results for both unaged and PAV aged CRM and BMR with 0 and 12 hours isothermal conditioning at -18°C were studied, as seen in Figures 29 and 30. When comparing the unaged material, both the CRM and BMR had stiffness values 34% and 40% lower than that of the neat binder. BMR was shown to have a 15% lower stiffness value compared to the CRM at 0 hour conditioning. However, after isothermal low temperature conditioning, the CRM stiffness was shown to increase 20%, while the BMR increased 10%. When comparing the PAV aged modified binders, the BMR had a 15% lower stiffness value compared to the CRM. After 12-hour conditioning of the PAV aged samples, the CRM was only 4% higher than the BMR, which shows that by this time, both samples are already very stiff from both aging and conditioning. However, the inclusion of 15% bio-binder appears to show improved results compared to the CRM for unaged with and without conditioning, as well as in 0-hour PAV aged binders.

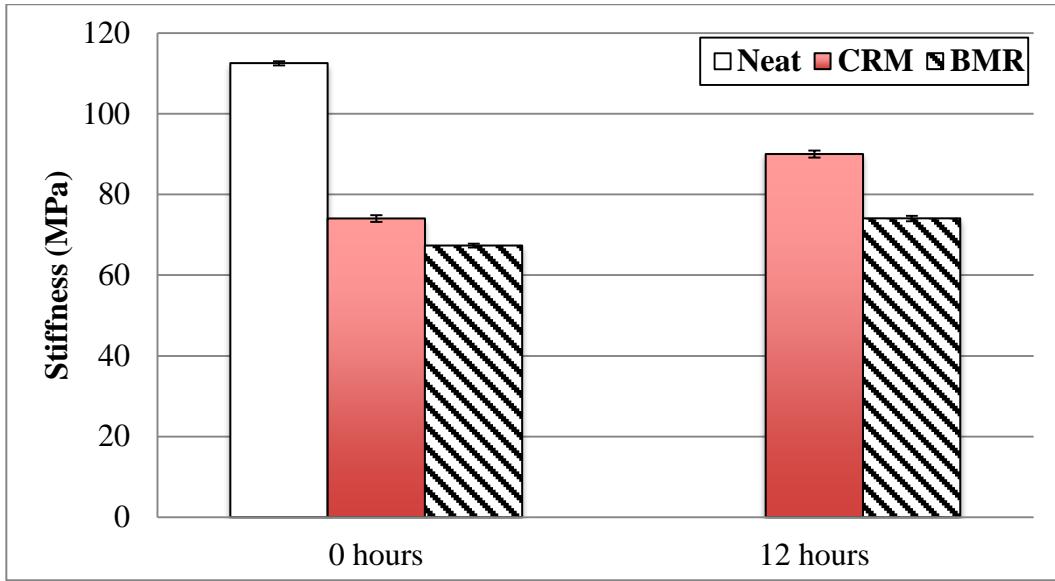


FIGURE 29. BBR stiffness results for unaged neat, CRM, and BMR at 0 and 12 hours conditioning.

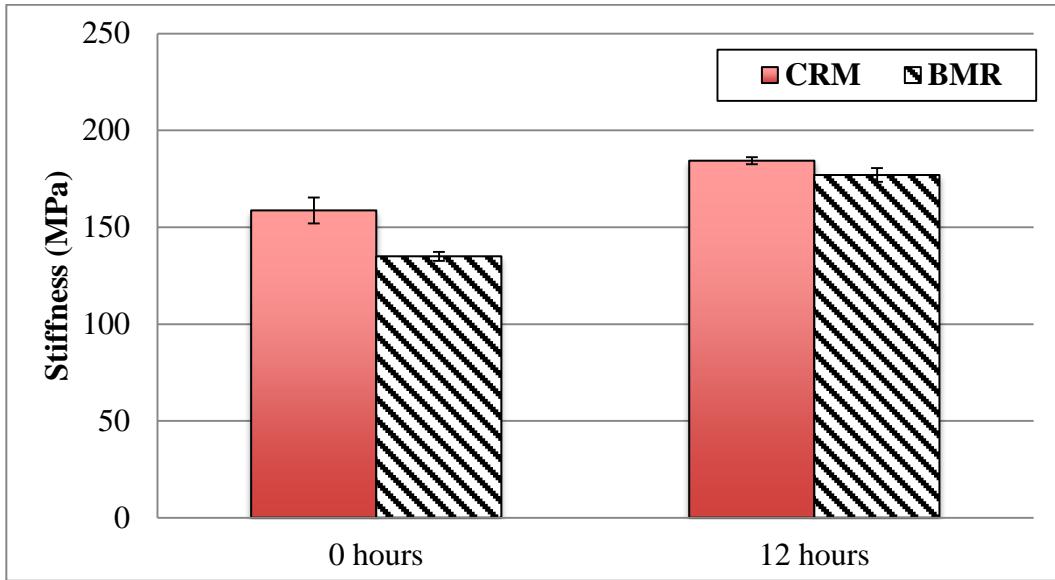


FIGURE 30. BBR stiffness results for PAV aged neat, CRM, and BMR at 0 and 12 hours conditioning.

In Figures 31 and 32, the m-value results from the BBR binder testing are given for both unaged and PAV aged CRM and BMR modified binder after 0- and 12-hour conditioning. For the unaged samples, the results show that both CRM and BMR have respective m-values 8% and 4% lower than the neat binder. After 12-hour isothermal conditioning, the CRM m-value was shown to decrease 7%, while the BMR m-value improved 4% compared to CRM. After PAV aging, the CRM was shown to be 17% lower compared to the neat, while the BMR m-value improved 7%. In comparing the 0- and 12-hour PAV aged samples, the CRM sample was observed to be nearly identical, while the BMR may have decreased slightly. The results indicate that BMR has an improved ability to relax stress compared to the CRM sample, which is closer to the neat binder.

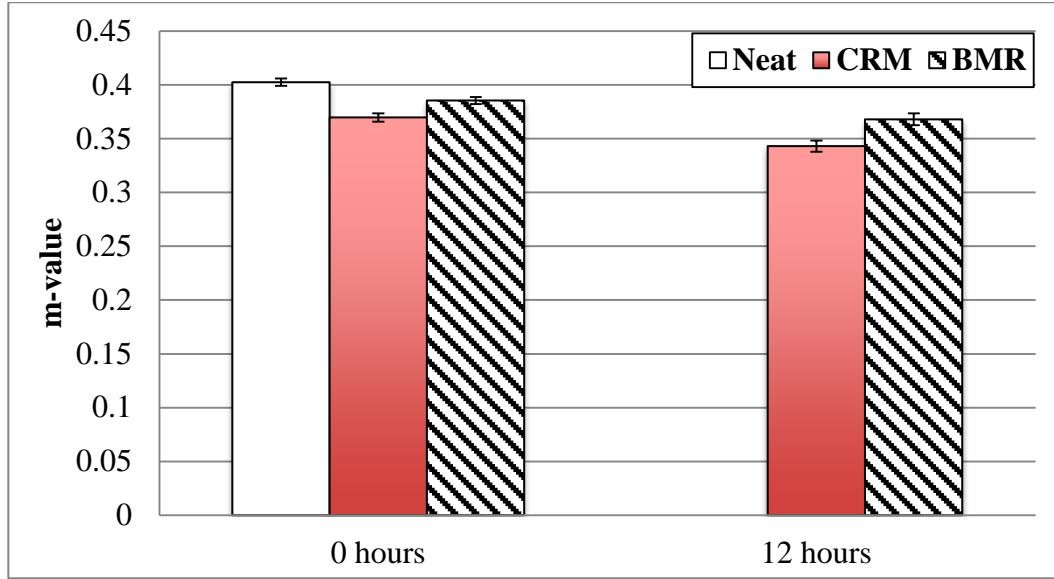


FIGURE 31. BBR m-value results for unaged neat, CRM, and BMR at 0 and CRM and BMR at 12 hours conditioning.

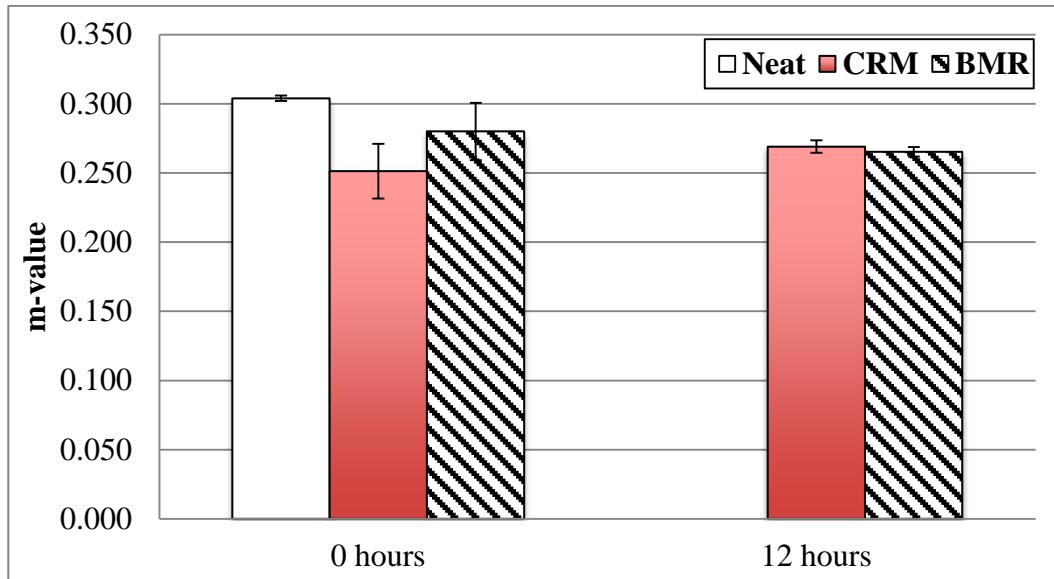


FIGURE 32. BBR m-value results for aged neat, CRM, and BMR at 0 and CRM and BMR at 12 hours conditioning.

Direct Tension Tester

In Figures 33 and 34, the results from the Direct Tension Tester (DTT) are given for BMR and CRM asphalt binder. The corresponding load and displacement for each sample of CRM and BMR are given in Figures 33 and 34. This shows that as samples go from unaged to PAV aged, as well as from 0 to 12 hour isothermal conditioning, the

samples go from ductile to brittle failure. For the CRM PAV aged samples, the results from 0- and 12-hour conditioning appear to be identical, which indicates that the CRM samples have already reached fully brittle failure before isothermal conditioning. However, for the BMR samples, it is shown that even after PAV aging, the samples still become increasingly brittle from 0- to 12-hour conditioning, which indicates that the samples had not reached fully brittle behavior even after PAV aging. The load-displacement data was then used to calculate peak load and fracture energy.

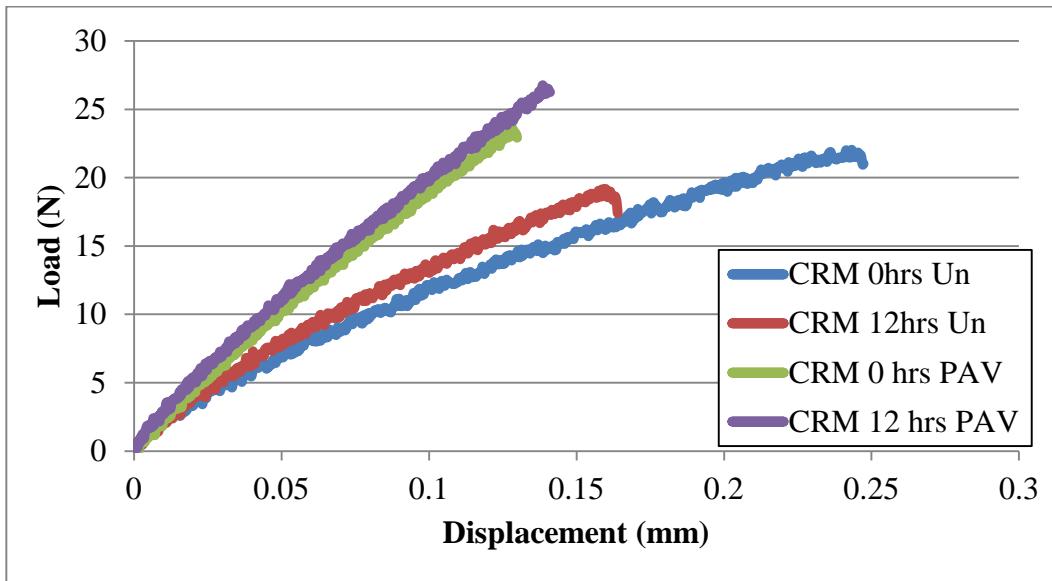


FIGURE 33. Load vs. displacement curves for unaged and PAV aged CRM at 0 and 12 hours conditioning.

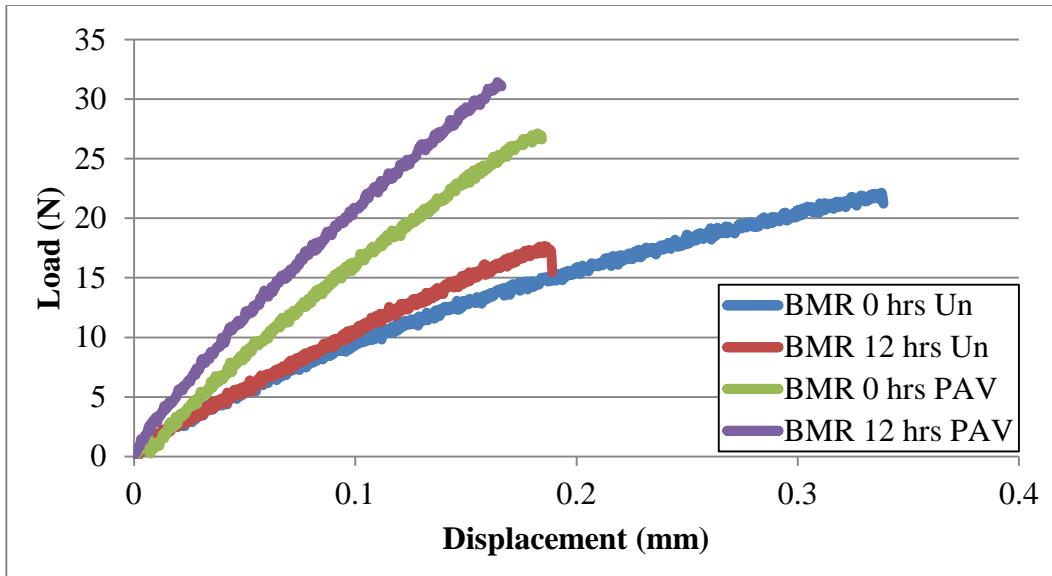


FIGURE 34. Load vs. displacement curves for unaged and PAV aged BMR at 0 and 12 hours conditioning.

Figure 35 shows the fracture energy results for the unaged and PAV aged CRM and BMR samples at 0- and 12-hour conditioning. For the unaged samples, the CRM was shown to have a higher fracture energy at both 0- and 12-hour conditioning, compared to the corresponding BMR samples. However, after PAV aging, the BMR was shown to have significantly higher fracture energy results at both 0- and 12-hour conditioning. In order to better interpret these results, the peak loads were graphed in Figure 36. The trend reflects that of the fracture energy results.

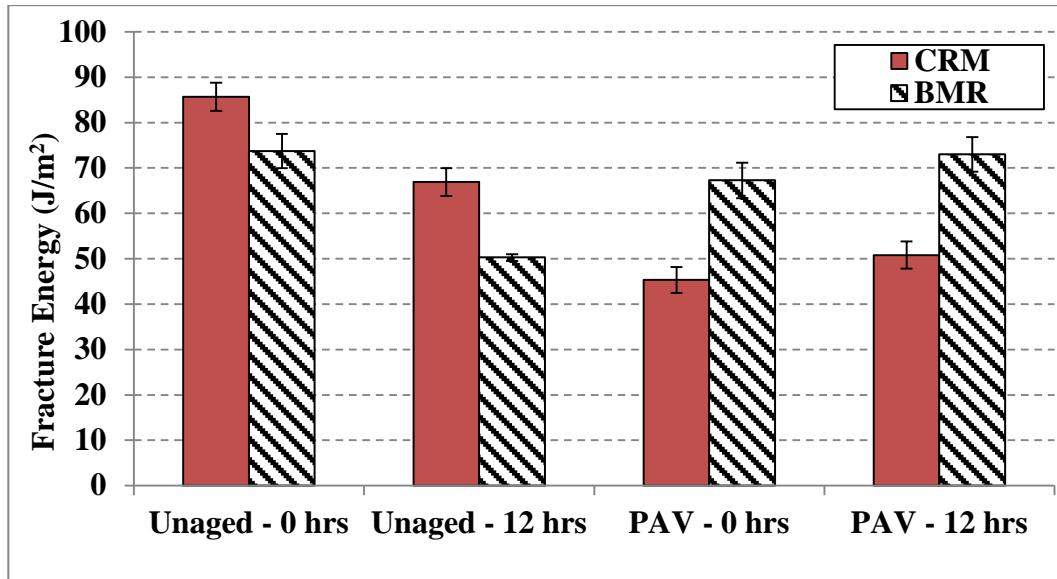


FIGURE 35. Fracture energy for unaged and PAV aged CRM and BMR at 0 and 12 hours conditioning.

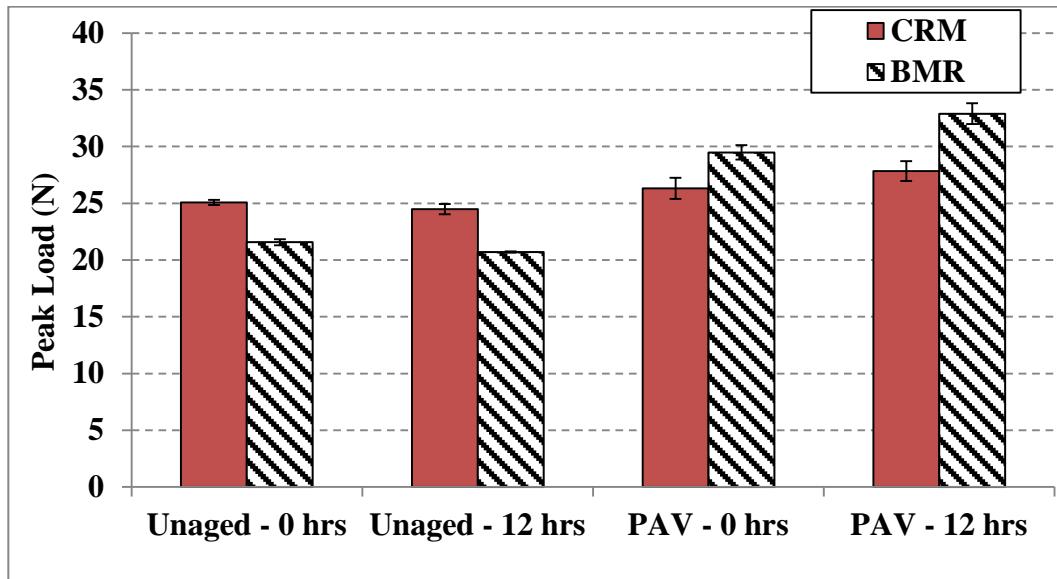


FIGURE 36. Peak load results for unaged and PAV aged CRM and BMR at 0 and 12 hours conditioning.

Mixture Section

Workability and Compaction Comparison

Figure 37 shows the compaction curves of the different mixtures; they have almost the same initial height and the same weight. The compaction curve of the 15% CRM asphalt mixture is located above that of the 15% BMR curve; both the mixtures were compacted at 150 °C and compacted to a total gyration of 199. The graph shows that, of the two mixtures, the BMR asphalt is more workable. The compaction curve of the CRM samples at 150°C placed above the BMR sample shows that it has high viscosity and needs more energy to be compacted.

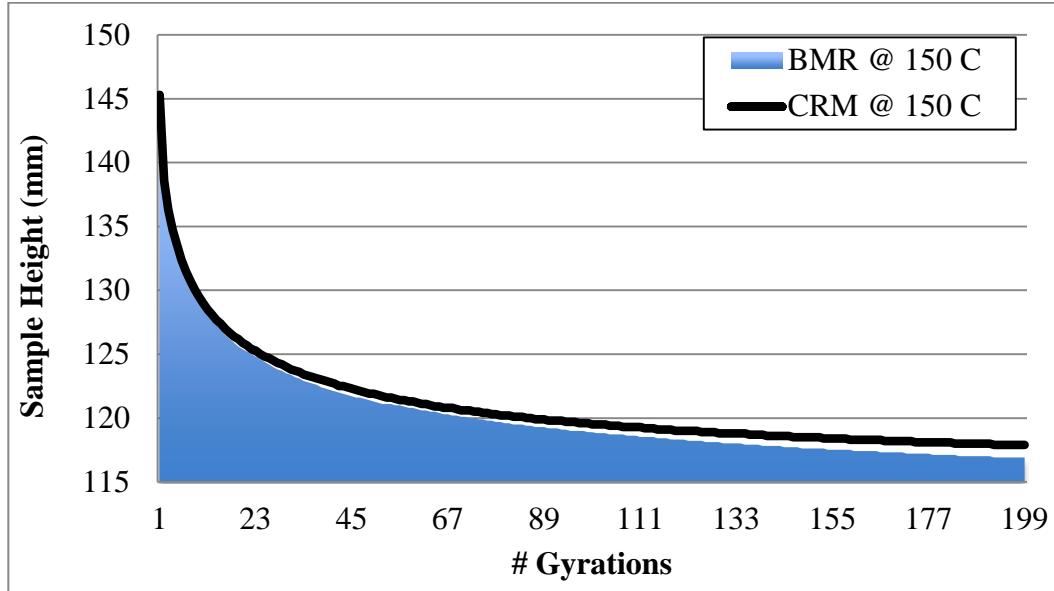


FIGURE 37. Compaction curve for 15% CRM and 15% BMR asphalt shows the effect of CRM and bio-binder on the compaction of the asphalt.

To quantify the compaction effort and compare properties of samples, the compaction resistance ratio is defined as the ratio of the height of the sample at 199 gyrations to the height of the sample at 5 gyrations. A higher compaction resistance ratio means less compaction efficiency. The maximum value of the compaction resistance ratio is 1.00, which means that the sample was not compacted between 5 to 199 gyrations.

In this research, the calculated compaction resistance ratios were 0.88 for CRM and 0.87 for BMR. The compaction resistance ratios for CRM and BMR are shown in Table 22. As can be seen, when CRM was compacted at 150°, the workability is quite low, as evidenced by a higher compaction resistance ratio value of 0.88. To enhance workability, 15% of bio-binder was added to the CRM. When BMR was compacted at 150°, the workability was reduced compared to that of the CRM at the same temperature. Therefore, the introduction of bio-modified binder into crumb rubber in this study led to increased workability.

Table 22. Resistance ratio values for CRM and BMR specimens

Sample Type	Compaction Resistance Ratio
CRM at 150°	0.88
BMR at 150°	0.87

Figure 38 shows the compaction curves of the different mixtures; they have almost the same initial height and same weight. The compaction curve of the neat asphalt mixture located below other curves shows that the neat asphalt mixture at 149°C was the most workable mixture and compacted more than other samples at different temperatures at 75 gyrations. Adding 20% crumb rubber made samples less workable. The compaction curve of the CRM samples at 149°C is higher than the other samples, showing it needs more energy to be compacted.

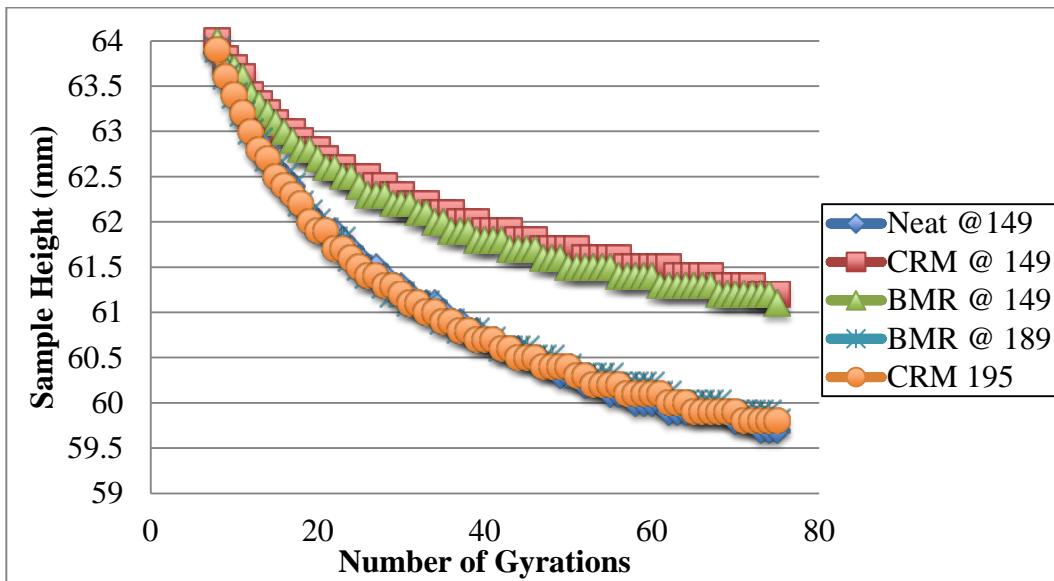


FIGURE 38. Compaction curve for 20% CRM, 20% BMR, and asphalt shows the effect of CRM and bio-binder on the compaction of the asphalt.

In this research, the calculated compaction resistance ratios lay between 0.9182 and 0.9533, corresponding to the neat at 149°C and CRM at 149°C, respectively. The compaction resistance ratios for all samples are shown in Table 23. As can be seen when CRM was compacted at 149°C, the workability is quite low as evidenced by a high compaction resistance ratio value of 0.9533. To enhance workability, the temperature was increased to 195°C, which resulted in 0.9315 resistance ratio. It was shown that 30% increase in temperature could only lead to 3% improvement in workability. Even when compacted at 195°C, the compaction was significantly different from that of neat asphalt.

When BMR was compacted at 149°C, the workability increased by 1% compared to that of the CRM at the same temperature. However, it was still different from that of neat asphalt. In order to improve the workability, the temperature was increased to 189°C; this improved workability by 6% and led to a compaction profile very similar to the neat asphalt. With this increase in energy, there was less than 1% difference from neat asphalt's workability at a compaction temperature of 149°C. Therefore, the introduction of bio-binder in conjunction with crumb rubber in this study led to increased workability but a decrease in compaction energy.

Table 23. Resistance ratio values for CRM and BMR specimens

Sample Type	Compaction Resistance Ratio
Neat 149	0.9282
#1 CRM 149	0.9533
#2 CRM 195	0.9315
#1 BMR 149	0.9443
#2 BMR 189	0.9200

Figure 39 shows the compacted sample height of BMR (the introduction of bio-binder into CRM) at the 75th gyration compared to that of the Neat compacted at a lower temperature but also at the 75th gyration. When BMR and CRM were compacted at a temperature of 150°C, they yielded values that are only 0.5% different. Yet when comparing the Neat to CRM, there is a difference of almost 3%, so adding 15% bio-binder reduces that difference to a 2% difference. These results show that the introduction of bio-binder mixed with crumb rubber increases the workability of asphalt.

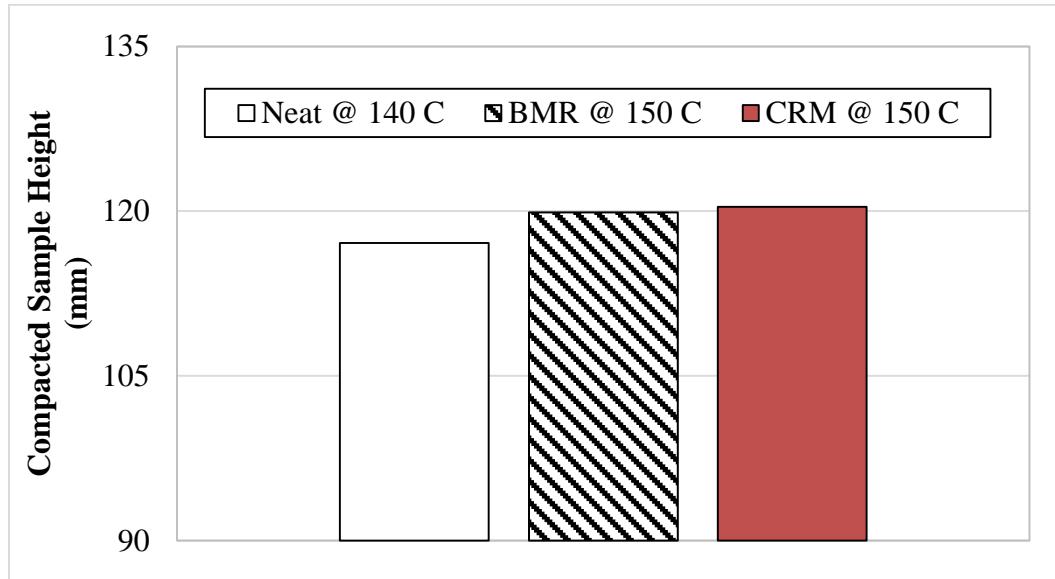


FIGURE 39. Compacted sample height of Neat, CRM, and BMR at the 75th gyration at 150°C.

Moisture Susceptibility

Adhesion

After filtration and separation of particulates from leachates, the samples were analyzed by FTIR method. A Thermo Scientific Nicolet iS10 FT-IR Spectrometer was used in Absorbance mode to acquire the spectra of each sample. Wave numbers ranging from 4000cm⁻¹ to 600cm⁻¹ were covered. The background spectrum was taken after cleaning the prism with methylene chloride.

In Figure 40, the FTIR results are given for CRM and BMR samples. The FTIR spectra seen at 2920 cm^{-1} and 2850 cm^{-1} for BMR and control asphalt are due to asymmetric and symmetric C—H stretching of aliphatic groups; these peaks were in higher intensity for the control binder. The intensity of the peak at 2920 cm^{-1} is greater than the peak at 2850 cm^{-1} for the samples, which indicates the presence of long aliphatic chains. The small peaks within the 650 – 910 cm^{-1} region are typical C—H vibration of benzene (Fang et al.). FTIR results indicated the presence of adhered binders to mineral particulate of aggregates. This adhesion was not seen for CRM; the only peak observed for CRM was in the 2350 – 2370 cm^{-1} region, which can be attributed to the absorbed CO_2 on the surface of mineral particles. These peaks were also observed for BMR (Rege et al. 2005). These results showed better adhesion of BMR to aggregates in comparison to CRM, which can be related to the presence of bio-binder and consequently interaction of bio-binder with base binder and aggregates.

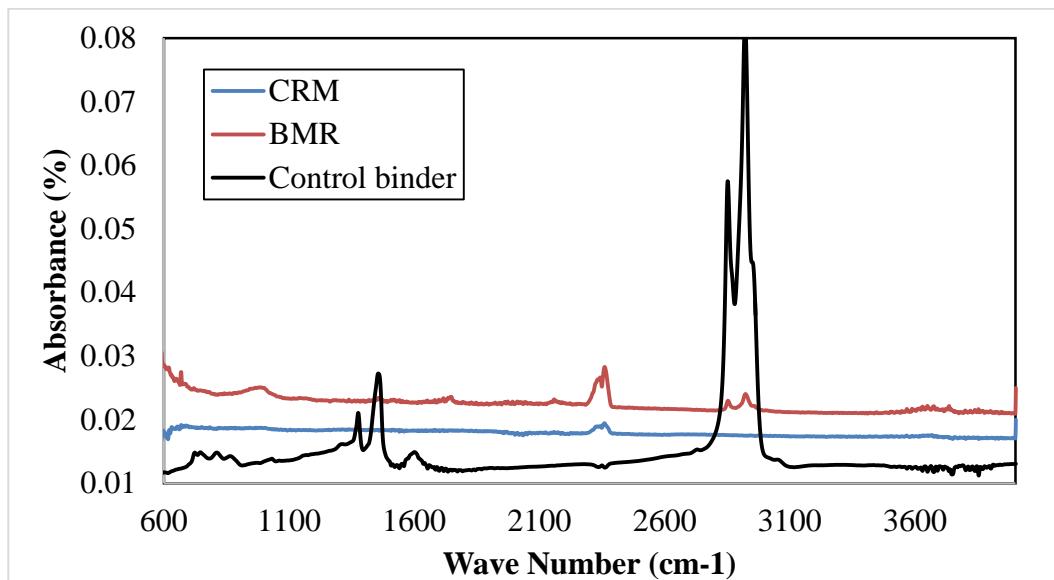


FIGURE 40. FTIR results for CRM and BMR after modified stripping test.

Leachate

GC/MS analysis was used to determine the organic compounds in the leachates; the organic compounds and their chemical formulas are listed in Table 24. Table 24 shows compounds that were seen in CRM and BMR leachates. Some of the compounds were in common for both of the samples but some of them were seen in just one of them. This can be related to the presence of bio-modifier in BMR and consequently the release of different components into the water. None of these compounds were listed under the EPA and NCBI archives as Polycyclic Aromatic Hydrocarbons (PAHs) (EPA et al. 2016; NCBI et al. 2016). Since previous studies showed the presence of PAHs from asphalt and CRM, it appears that this method of study for obtaining leachate was not successful in releasing those compounds. Because of the slow kinetics of leaching when mimicking the natural process, an accelerated laboratory method is necessary to predict long-term leaching behavior.

Table 24. Organic compounds in the leachates from BMR and CRM

Organic Compound	Chemical Formula	BMR	CRM
Cyclohexanamine, N-cyclohexyl	C ₁₂ H ₂₃ N	X	X
1-[3-(2-Hydroxy-ethyl)-octahydro-isoquinolin-2-yl]-ethanone	C ₁₃ H ₂₃ NO ₂	X	X
2H-Pyrimido[1,2-a]pyrimidine, 1,3,4,6,7,8-hexahydro-	C ₇ H ₁₃ N ₃	X	X
1,3-Diazadamantane-6-one, 5,7-dimethyl-	C ₁₀ H ₁₆ N ₂ O	X	X
Chol-11-en-24-oic acid, 3-(acetyloxy)-, methyl ester, (3 α ,5 β)	C ₂₇ H ₄₂ O ₄	X	X
2-Amino-5-chlorobenzophenone, amidinohydrazone	C ₁₄ H ₁₄ ClN ₅		X
Thiocolchicine	C ₂₂ H ₂₅ NO ₅ S		X

Moisture Conditioning Ratio

To determine the moisture conditioning ratio values of CRM and BMR, the DC(T) was utilized to provide not only the peak loads but also the displacement. The recorded peak loads, displacement, and respective degree of saturation of dry and wet Neat, CRM, and BMR samples are given in Table 25. The ratios of the average wet peak loads and the average dry peak loads are given in Figure 41. The results show that CRM had the lowest ratio, followed by the neat, and the BMR had the highest ratio.

Table 25. Neat, CRM, and BMR dry and wet peak loads and degree of saturation

Neat				
	Dry		Wet	
	Peak Load (kN)	Degree of Saturation	Peak Load (kN)	Degree of Saturation (%)
1	0.4288	N/A	0.3386	0.51
2	0.4198	N/A	0.3229	0.53

CRM				
	Dry		Wet	
	Peak Load (kN)	Degree of Saturation	Peak Load (kN)	Degree of Saturation (%)
1	1.0939	N/A	0.6773	0.55
2	1.0725	N/A	0.6311	0.46

BMR				
	Dry		Wet	
	Peak Load (kN)	Degree of Saturation	Peak Load (kN)	Degree of Saturation (%)
1	0.8295	N/A	0.8302	0.40
2	0.8724	N/A	0.7267	0.51

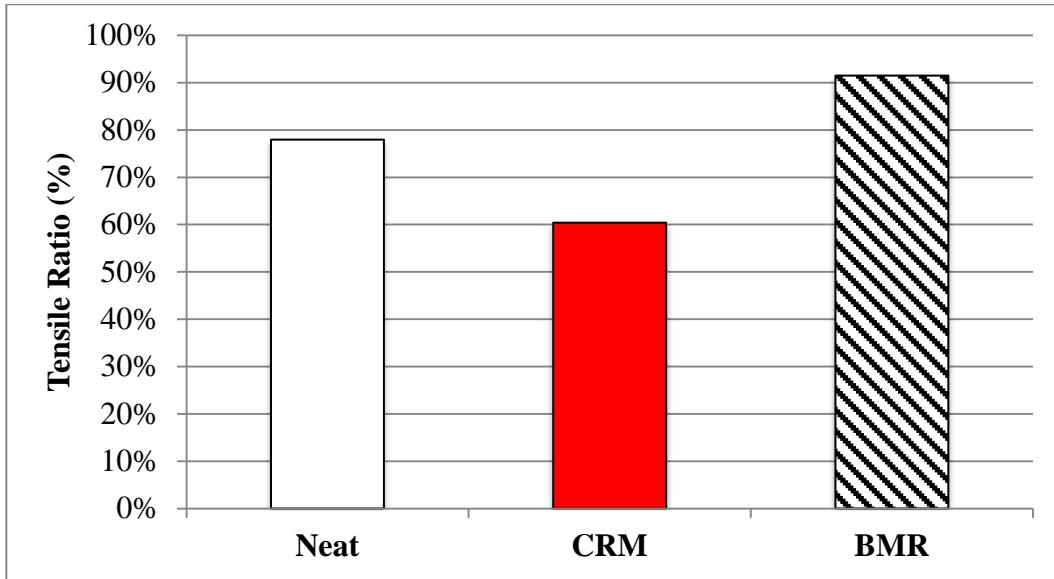


FIGURE 41. Modified tensile ratio results for Neat, CRM, and BMR.

In Figures 42 and 43, the load versus CMOD displacement curves are given for the Wet and Dry DC(T) samples of 15% CRM and 15% BMR. From Figure 42, the water conditioning is shown to have significantly decreased the corresponding peak load, compared to the dry sample. However, at high displacement, the two curves appear to lie on top of each other. In Figure 43, the 15% BMR samples appear to show a different trend. A less drastic drop in peak load is observed between the dry and wet samples. In observing the load at high displacement, it appears that the sample is displaying a more ductile failure compared to the dry sample. While the CRM samples appear to show a significant decrease in fracture energy between dry and wet conditioned samples, BMR appears to show a less significant decrease.

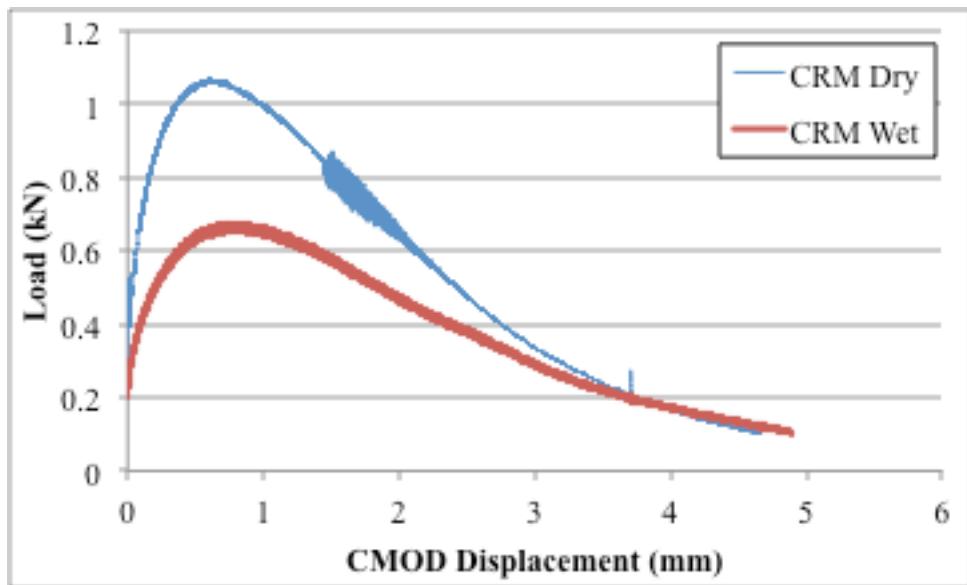


FIGURE 42. Load versus CMOD displacement for wet and dry 15% CRM.

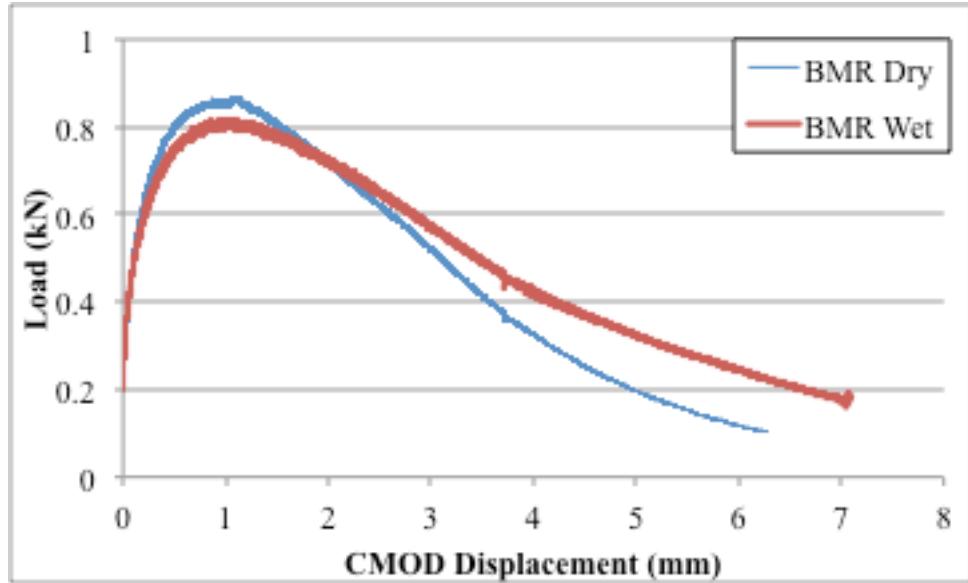


FIGURE 43. Load versus CMOD displacement for wet and dry 15% BMR.

Hamburg Wheel Rutting Test

In order to determine the modified asphalt mixtures' rutting susceptibility, the Hamburg wheel tracking test was performed at a temperature of 50°C in accordance with AASHTO T 324. A graph of the results is given in Figure 44, with a summary of the results given in Table 26. The results show that both BMR and CRM perform well above the 12.5 mm rut depth specification minimum. With respect to stripping, a stripping inflection point (SIP) was not observed in either of the samples. The final HWTT samples are shown in Figure 45; both the CRM (top) and BRM (bottom) samples showed almost no stripping.

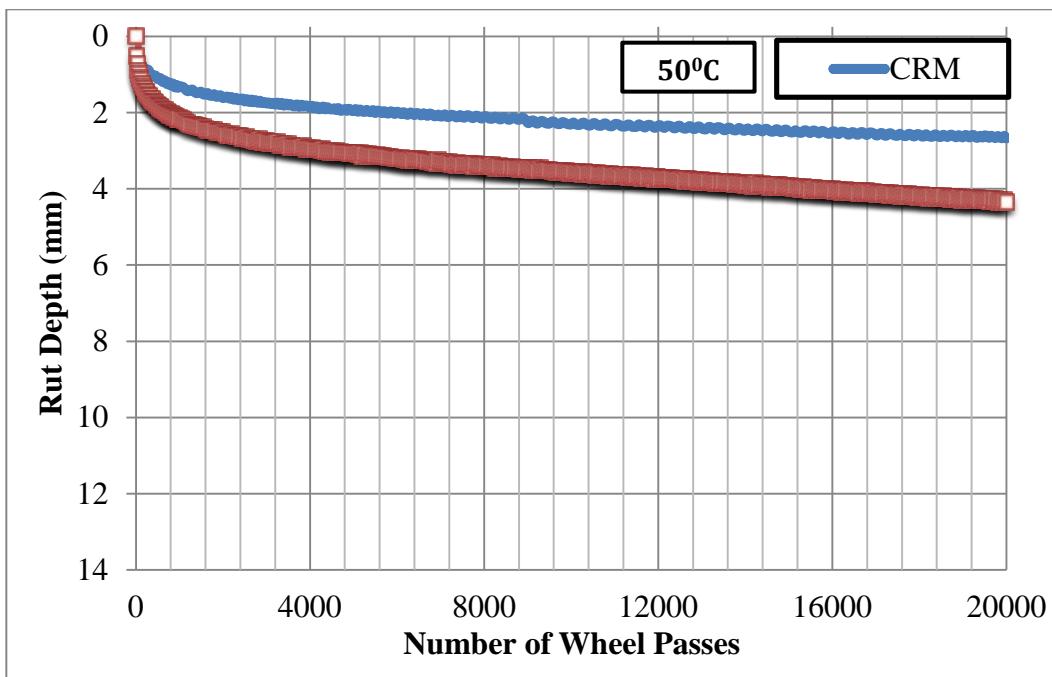


FIGURE 44. Hamburg Wheel Tracking Test (HWTT) results for CRM and BMR.

Table 26. Hamburg Wheel Tracking Test (HWTT) for CRM and BMR

Pass #	Wheel Rutting Depth (mm)	
	CRM	BMR
5,000	1.96	3.08
10,000	2.31	3.55
15,000	2.50	3.95
20,000	2.68	4.36

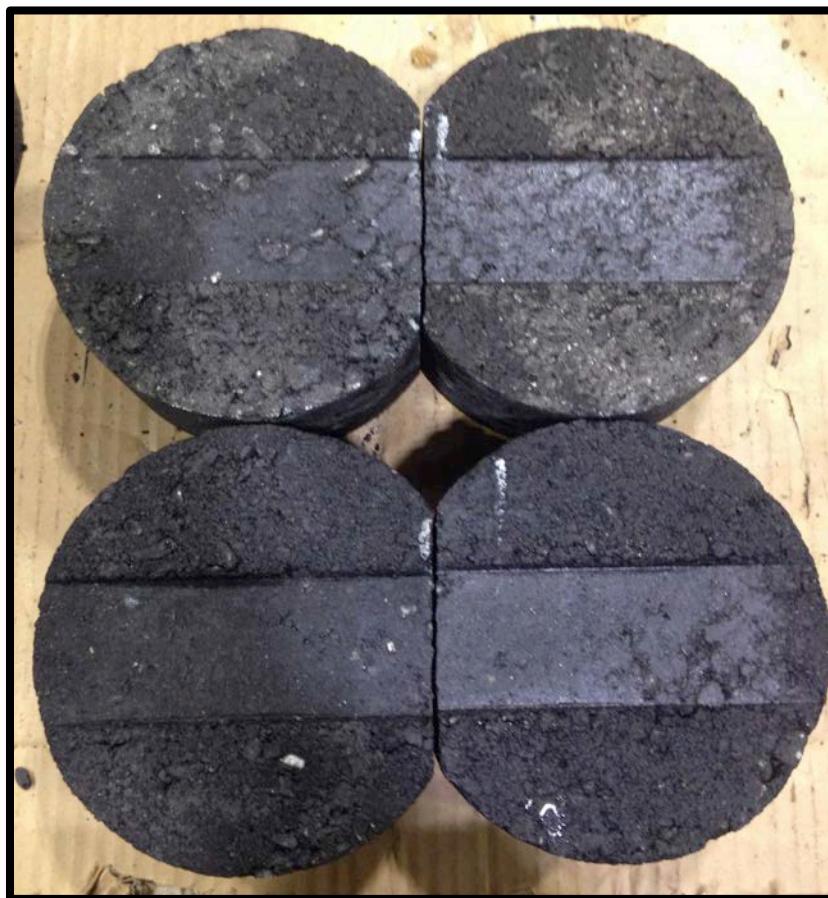


FIGURE 45. HWTT samples after testing: CRM (Top) and BMR (Bottom).

Low-Temperature Performance

Disk-Shaped Compaction Tension Test

The DC(T) results are shown in Figure 46 and Table 27. Prior to testing, the samples were isothermally conditioned at -18°C for 12 hours to determine the effects of the conditioning on the samples. The results show that CRM and BMR had higher fracture energy results compared to that of the neat binder. However, the CRM and BMR appear to have similar fracture energy results. Therefore, to investigate the results more closely, the peak loads and the exact fracture energy results are given in Table 27. The results show that CRM had a slightly higher average peak load compared to the BMR sample; however, the average fracture energy is slightly higher. This indicates that the BMR displayed more ductile behavior than the CRM: the area under the BMR curve is larger than the area under the CRM curve, despite the BMR curve having a slightly lower peak load. This trend is consistent with that seen with the DTT binder results.

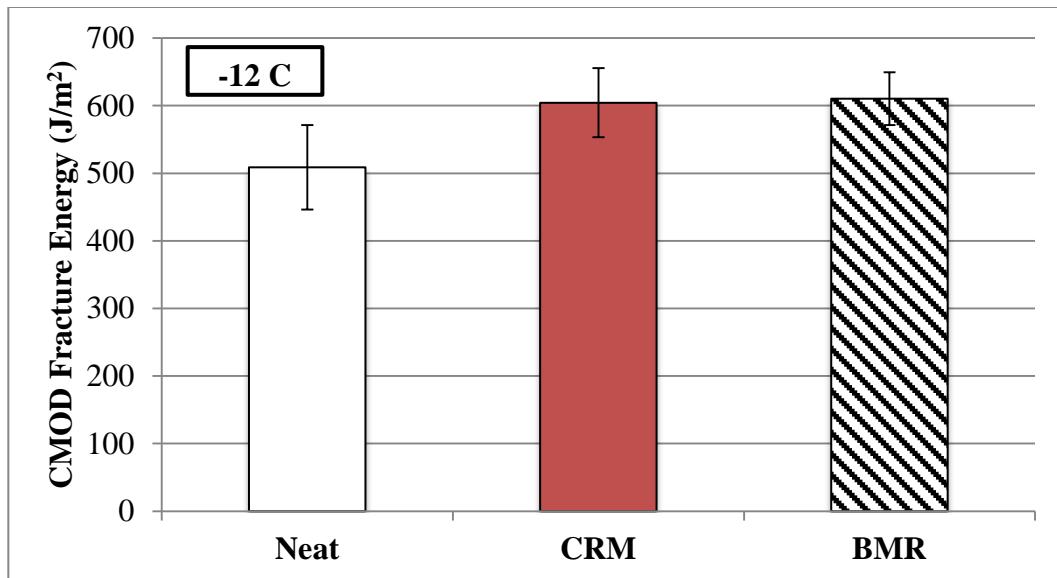


FIGURE 46. The DC(T) CMOD fracture energy results for Neat, CRM, and BMR at -12 °C after isothermal conditioning for 12 hours at -18°C.

Table 27. Neat, CRM, and BMR (DCT) peak load and fracture energy results

Specimen	Avg. Peak Load (kN)	Avg. Fracture Energy (J/m ²)	Fracture Energy COV
Neat	4.170	508.74	21.2%
CRM	4.345	604.19	14.7%
BMR	4.259	610.28	11.1%

Superpave Indirect Tensile Creep Test

The IDT Creep Compliance fitted curves are given in Figure 47 for Neat, CRM, and BMR samples. Initially, the BMR results are shown to be slightly less compliant compared to CRM and Neat; however, afterwards, the CRM samples become the least compliant compared to the Neat. The addition of bio-binder, in the BMR samples, shows similar creep compliance results to that of the Neat samples and even, in some cases, having a slightly higher creep compliance than the Neat. In comparing the m-value results, the same trend is observed as in the binder results from the BBR. The inclusion of CRM was shown to decrease the m-value 23% compared to the Neat. The inclusion of bio-binder was observed to improve the m-value 17% compared to the CRM samples. This indicates that the BMR samples have an increased ability for relaxation, compared to the CRM samples.

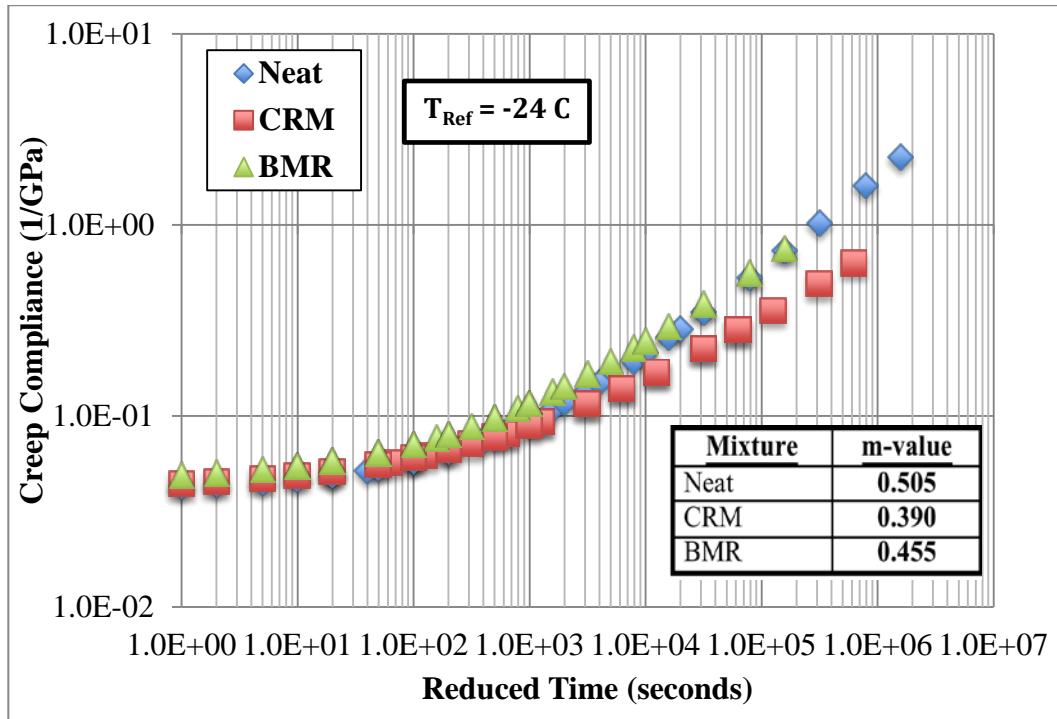


FIGURE 47. Fitted creep compliance results for Neat, CRM, and BMR Samples at 0°C, -12 °C, and -24 °C.

CONCLUSION

NCHRP Project #171 focused on producing a sustainable bio-based alternative to petroleum-based asphalt binder. To do so, crumb rubber particles were treated by an amide enriched bio-modifier made from swine manure to enhance performance of rubber modified asphalt. Traditionally, the use of high rubber percentages in crumb rubber modified (CRM) asphalt has been problematic due to issues with workability, rubber-asphalt separation as well as some concerns with respect to CRM's low temperature performance. Therefore, in this project, the effect of introducing bio-binder (BB) to the CRM asphalt was examined in terms of workability and low temperature performance at both the mixture and binder levels. In addition, moisture susceptibility and leachates evaluation from each specimens as well as chemical characterization was performed.

Workability was investigated on the binder side using the rotational viscometer and the dynamic shear rheometer. The presence of BB showed to decrease the viscosity of CRM asphalt binder. It was found that when 15% BB was introduced to CRM asphalt, it led to a viscosity reduction of 40% and 60% measured at 105 and 195°C, respectively. Based on the phase angle measurements using a dynamic shear rheometer (DSR), mixing and compaction temperature of 15% BMR (containing 15% of bio-modified rubber asphalt) was determined to be nearly similar to that of neat asphalt without rubber; in this study compaction temperature was defined at the temperature at which phase angle was 80degree. Examining mixture workability results, BMR showed some reduction in compaction resistance ratio when compared with CRM compacted at the same temperature. To allow lower mixing and compaction temperature the samples required an increase in number of gyrations to maintain similar volumetrics. It was concluded that a higher dosage of BB (in excess of 20%BB) is needed to maintain similar number of gyrations at a reduced mixing and compaction temperature. Further examination will be performed to determine the optimum dosage of BB at specified compaction temperature.

Furthermore, the addition of BB to CRM showed to decrease the Superpave rutting resistance parameter ($G^*/\sin \delta$) by one temperature grade from 82°C to 76°C. This was further reflected in higher Jnr values in BMR for unaged, RTFO, and PAV aged samples. When observing the Superpave fatigue parameter ($G^*\sin\delta$), the presence of CRM did not show any effect on the binder; however, introduction of BB led to reduction of the low temperature grade. The low temperature improvement was also verified when determining the crossover temperature of the elastic and viscous modulus master curves. The crossover temperature of BMR was 2.8, 8.1, and 7.1 degrees lower than the CRM for unaged, RTFO, and PAV respectively. This, in turn, shows that the improvement in low temperature performance due to presence of BB can be observed even after short and long-term aging.

To further examine the low temperature properties, CRM and BMR samples were tested using standard procedure as well as after 12 hours of isothermal low temperature conditioning at -18°C. At the unaged status, BMR specimens showed to have 9.5% and 18% lower stiffness than CRM after 0 and 12 hours of conditioning, respectively. The BMR's m-value was also observed to be 4% and 7% higher than that of CRM samples after 0 and 12 hours of conditioning, respectively. After PAV aging, the same trend was observed and BMR had 15% lower stiffness and 12% higher m-value than CRM.

When analyzing the DTT results, at unaged status, CRM showed to have 14% and 25% higher fracture energy compared to BMR at 0 and 12 hour conditioning, respectively. However, after PAV aging, the BMR samples had 48% and 44% higher fracture energy compared to the CRM at 0 and 12 hour conditioning, respectively. This, in turn, indicates that the BMR samples maintain a ductile behavior after they are exposed to long-term aging and even after 12hrs conditioning at -18°C. Accordingly, CRM specimens showed a more brittle failure behavior than BMR as evidenced by lower fracture energy in CRM samples compared to BMR.

To further examine the effectiveness of BMR, mixture level analysis was performed. The DC(T) fracture energy of 12 hour conditioned CRM and BMR samples showed no significant difference; however, when examining the peak loads values, the BMR had a slightly lower average peak load which, in light of the similar fracture energy results, indicates the BMR sample could be showing more ductile behavior compared to the CRM sample. Superpave IDT samples were also tested. The addition of CRM showed a decrease in creep compliance compared to the neat asphalt; however, with the addition of BB, the BMR increased creep compliance similar to that of the neat asphalt. The m-value of BMR was higher by 17% compared to that of CRM.

In addition, the study of moisture susceptibility for both CRM and BMR mixtures was performed. After undergoing extensive conditioning for seven days, the fine particles of BMR and CRM were analyzed using the FTIR. The res FTIR spectra showed that the CRM binder was debonded from the fine mineral aggregate after extensive conditioning. However, the fine aggregates from the BMR samples still had BMR absorption peaks; this could be attributed to better adhesion between BMR and fine aggregates compared to that of CRM.

The leachate testing performed on the conditioning water to determine molecular species leached out of each specimen into the water was performed using GPC and FTIR analysis. The identified leachate elements were not significantly different between the two samples. However, the leachate analysis was deemed inconclusive because the detected elements in the leachate were not listed under the EPA archives as recognized polycyclic aromatic hydrocarbons (PAHs). An extended and more severe conditioning method needs to be utilized for future leachate analysis to distinguish between BMR and CRM leachates with respect to the EPA's ground water limits. The Hamburg wheel track (rutting) test results were also obtained for the CRM and BMR samples. The CRM showed superior rutting performance in that after 20,000 passes, the CRM rut depth was only 2.6 mm. The BMR sample had a slightly higher rut depth of 4.3 mm after 20,000 passes; however, both values are well above the maximum rut depth value of 12 mm. In addition, no stripping indication was observed for either of the samples.

The Tensile Strength Ratio (TSR) test was also performed for BMR and CRM samples. The CRM sample was shown to have a 23% lower tensile strength ratio when compared to the neat; however, the BMR sample showed to improve the tensile strength ratio by 52% compared to the CRM and 17% compared to the neat asphalt. The analysis of DC(T) results showed minimal loss of energy after extensive water conditioning in BMR samples, while CRM showed significant reduction in fracture energy after water exposure. This can be attributed to enhanced adhesion of BMR and mineral aggregate promoting water resistance of BMR mixture.

Overall, the study results showed that application of BMR could be a promising approach to facilitate and promote the use of crumb rubber in asphalt while enhancing rubber modified asphalts' low temperature properties and workability. In addition, surface activation of the rubber particles using an amide-enriched bio-modifier could enhance rubber-asphalt interactions while increasing resistance of the resulting rubber modified asphalt to moisture.

FUTURE RESEARCH

The follow-up study will focus on evaluation of technical and scale-up feasibility and market viability of bio-modified rubber asphalt as a sustainable bio-based alternative to petroleum-based asphalt. The outline of this is given in Figure 48. The project team will specifically focus on developing the rubber-activator prototype as well as treating rubber particles to create bio-modified rubber (BMR); the team will further perform molecular dynamics simulations to unveil rubber-asphalt interaction mechanisms. The specific research hypothesis is that during the rubber treatment process, molecules of bio-modifier cleave polysulfidic crosslinks of swollen vulcanized crumb rubber in the absence of oxygen creating free radicals on the surface of rubber particles, which can promote their interactions with asphalt molecules. Accordingly, underlying causes of some of the observed improvements in BMR compared to non-modified scenarios as elaborated in this report could be better understood in an attempt to exploit them for industrial production and prototyping. It should be noted that such surface activation is expected to reduce the propensity of rubber segregation within the asphalt blend while enhancing asphalt rheological properties. To examine the latter hypothesis, the project team plans to build a prototype unit and produce adequate BMR for both laboratory and field evaluation. The project team will select one of the BMR with proved laboratory performance to place a trial field section. The trial section will be placed for two rubber-modified asphalt mixtures after completion of a satisfactory laboratory evaluation (Figure 48), one with BMR (surface treated rubber) and one with un-treated rubber, in collaboration with the project team's industry partner and NC DOT. The mixing temperature and level of agitation for both rubber-modified asphalts as well as the number of roller compaction passes will be monitored to compare the ease of application of the two mixtures. In addition, field samples will be collected before and after compaction to conduct performance testing following NC DOT specification.

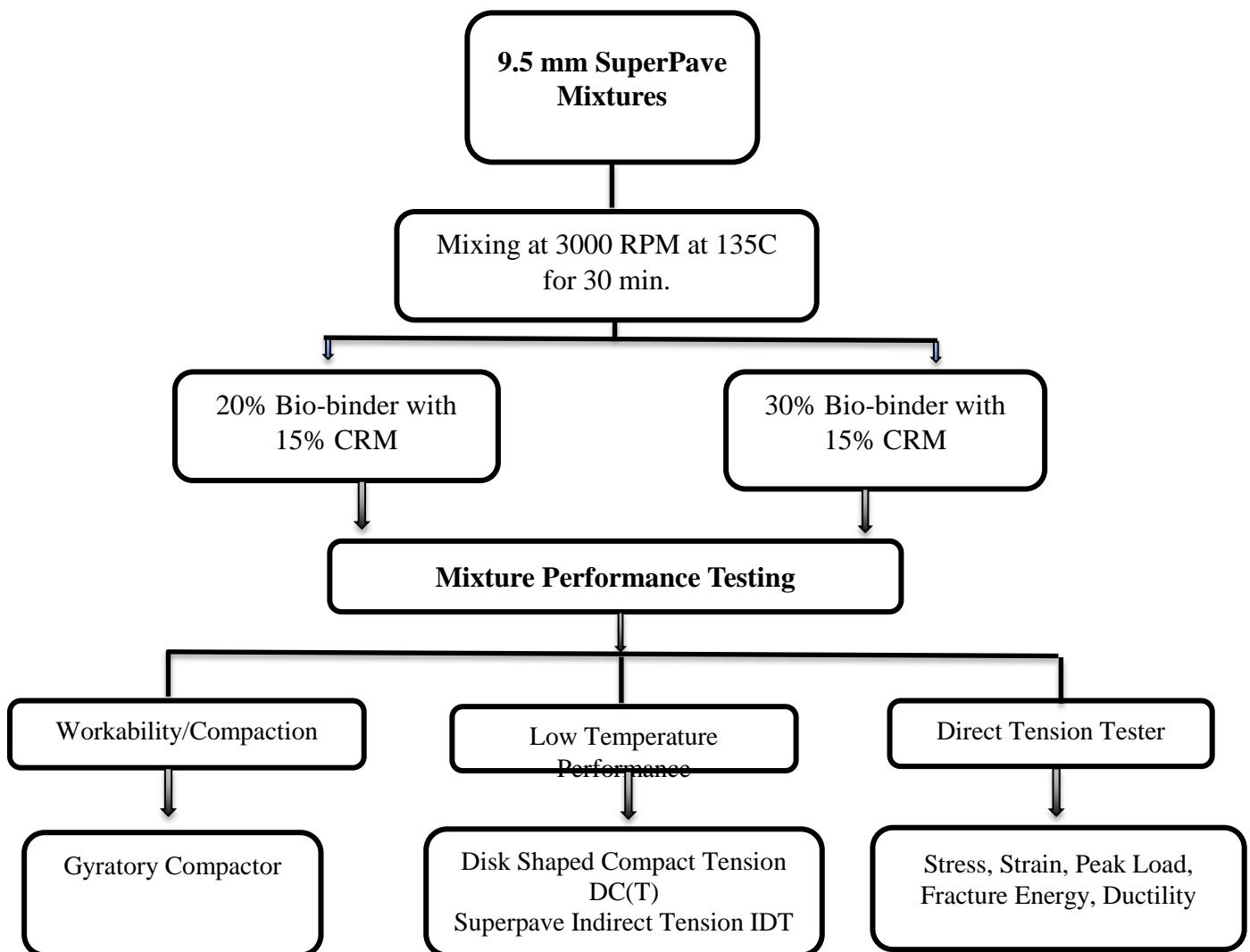


FIGURE 48. Future experimental plan.

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