Louisiana Transportation Research Center

Final Report 594

Sustainable Materials for Pavement Infrastructure: Design and Performance of Asphalt Mixtures Containing Recycled Asphalt Shingles

by

Louay Mohammad, Ph.D., P.E. (WY)
Samuel B. Cooper, Jr., Ph.D., P.E.
Minkyum Kim, Ph.D.

LTRC/LSU

4101 Gourrier Avenue | Baton Rouge, Louisiana 70808
(225) 767-9131 | (225) 767-9108 fax | www.ltrc.lsu.edu

TECHNICAL REPORT STANDARD PAGE

1. Report No. **FHWA/LA.17/594**	**2. Government Accession No.**	**3. Recipient's Catalog No.**
4. Title and Subtitle **Sustainable Materials for Pavement Infrastructure: Design and Performance of Asphalt Mixtures Containing Recycled Asphalt Shingles**	**5. Report Date** July 2019 **6. Performing Organization Code** LTRC Project Number: 12-1B State Project Number: DOTLT1000007	
7. Author(s) Louay N. Mohamad, Ph.D., P.E. (WY), Sam Cooper, Jr., Ph.D., P.E., Minkyum Kim, Ph.D.	**8. Performing Organization Report No.**	
9. Performing Organization Name and Address Louisiana Transportation Research Center Baton Rouge, LA 70808	**10. Work Unit No.** **11. Contract or Grant No.**	
12. Sponsoring Agency Name and Address Louisiana Department of Transportation and Development P.O. Box 94245 Baton Rouge, LA 70804-9245	**13. Type of Report and Period Covered** **Final Report** **April 2014 – March 2017** **14. Sponsoring Agency Code**	
15. Supplementary Notes **Conducted in Cooperation with the U.S. Department of Transportation, Federal Highway Administration**		

16. Abstract

As the price of liquid asphalt continuously climbs, methods are being sought to decrease material costs, which will also have less impact on ecological systems without compromising material or pavement performance. The use of recycled materials is one method that can replace a percentage of their virgin counterparts, thus reducing the negative impacts on the environment. Asphalt mixture performance is affected by the level of blending that occurs between aged and virgin asphalt binders. The interaction and compatibility of recycling agents (RAs) with recycled asphalt shingles (RAS) and reclaimed asphalt pavements (RAP) have not been thoroughly evaluated since the 1970s. To characterize laboratory mixture performance through their mechanistic and volumetric properties, a suite of tests was conducted to evaluate the low, intermediate, and high temperature performance of conventional asphalt mixtures and mixtures containing RAS and/or RAP with and without RAs. Also, the asphalt binders' molecular structure was correlated with their cracking potential through binder fractionation. With respect to the mixes without RAs, results indicate that RAS binder does not fully blend with the virgin binder. The actual availability factor was found to range from 35 to 46%. Also shown was an improvement in rutting performance, with no adverse effects to intermediate temperature or low temperature performance because the mixtures are comprised of approximately 90% virgin asphalt. For mixtures containing RAs, a modified mixture design change was developed to improve blending between the aged and virgin asphalt binders. The actual availability factor was found to range from 50 to 100%. It was determined that RAs adversely affected the intermediate and low temperature properties of the mixtures studied due to the increase in the recycled binder content utilized within the mixture. In terms of low-temperature properties, the use of soft binder performed similar to mixtures containing no RAs. The concentration of the high molecular RAS species exceeds 40% in which 25% of these are highly aggregated with apparent molecular weights approaching 100K. The use of RAs did not significantly dissociate the very high molecular weight species, and thus failed to improve mixture cracking resistance. In addition, Fourier-Transform Infrared Spectroscopy (FTIR) results were inconclusive.

17. Key Words		**18. Distribution Statement** **Unrestricted. This document is available through the National Technical Information Service, Springfield, VA 21161.**	
19. Security Classif. (of this report)	**20. Security Classif. (of this page)**	21. No. of Pages	22. Price

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Louay Mohammad, Ph.D., P.E. (WY)
Samuel B. Cooper, Jr., Ph.D., P.E.
Minkyum Kim, Ph.D.
Louisiana Transportation Research Center
Louisiana State University

Louisiana Transportation Research Center

LTRC Project No. 12-1B
State Project No. DOTLT1000007

conducted for

Louisiana Department of Transportation and Development
Louisiana Transportation Research Center

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July 2019

ABSTRACT

As the price of liquid asphalt continuously climbs, methods are being sought to decrease material costs, which will also have less impact on ecological systems without compromising material or pavement performance. The use of recycled materials is one method that can replace a percentage of their virgin counterparts, thus reducing the negative impacts on the environment.

Asphalt mixture performance is affected by the level of blending that occurs between aged and virgin asphalt binders. The interaction and compatibility of recycling agents (RAs) with recycled asphalt shingles (RAS) and reclaimed asphalt pavements (RAP) have not been thoroughly evaluated since the 1970s.

To characterize laboratory mixture performance through their mechanistic and volumetric properties, a suite of tests was conducted to evaluate the low, intermediate, and high temperature performance of conventional asphalt mixtures and mixtures containing RAS and/or RAP with and without RAs. Also, the asphalt binders' molecular structures were correlated with their cracking potential through binder fractionations.

With respect to the mixes without RAs, results indicate that the RAS binder does not fully blend with the virgin binder. The actual availability factor was found to range from 35 to 46%. Also shown was an improvement in rutting performance, with no adverse effects to intermediate temperature or low temperature performance because the mixtures are comprised of approximately 90% virgin asphalt.

For mixtures containing RAs, a modified mixture design change was developed to improve blending between the aged and virgin asphalt binders. The actual availability factor was found to range from 50 to 100%. It was determined that RAs adversely affected the intermediate and low temperature properties of the mixtures studied due to the increase in the recycled binder content utilized within the mixture. In terms of low-temperature properties, the use of soft binder performed similar to mixtures containing no RAs.

The concentration of the high molecular RAS species exceeds 40% in which 25% of these are highly aggregated with apparent molecular weights approaching 100K. The use of RAs did not significantly dissociate the very high molecular weight species, and thus failed to improve mixture cracking resistance. In addition, Fourier-Transform Infrared Spectroscopy (FTIR) results were inconclusive.

ACKNOWLEDGMENTS

The research work documented in this report was sponsored by the Louisiana Department of Transportation and Development (DOTD) through the Louisiana Transportation Research Center (LTRC). The authors would like to express thanks to all whom have provided valuable help in this study, especially the Asphalt Research Laboratory and the Engineering Materials Characterization Research Facility (EMCRF). The authors also would like to show their gratitude to Dr. William H. Daly and Dr. Ioan Negulescu, the chemistry research group of Louisiana State University (LSU), for providing valuable assistance in understanding the essential knowledge of asphalt chemistry.

IMPLEMENTATION STATEMENT

Based on the results, it is recommended that specifications for inclusion of RAS into mixtures be developed and experimental field projects be constructed. In doing so, the developed laboratory mixture design blending procedure can be validated. In addition, asphalt mixtures from these field projects can be characterized to determine the effects of RAS on the high-, intermediate-, and low-temperature mixture properties.

Also, it is recommended that an ALF (Accelerated Loading Facility) project be constructed. This will enable the evaluation of actual cracking and rutting under accelerated loading of mixtures containing RAS.

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INTRODUCTION

Background

One of the issues concerning environmental sustainability is determining how to make production, distribution, and consumption of goods and services last longer and have less impact on ecological systems consisting of all plants, animals, and microorganisms in an area functioning together with all of the non-living physical factors of the environment. One such method of sustainability in the asphalt mixture industry is using recycled materials to replace a percentage of virgin materials used in the manufacturing process, such as aggregates and asphalt binder, which has a direct impact on cost and the environment.

Agencies and owners must continually find methods to decrease material costs and maximize their benefits as the price of asphalt mixtures continually increases because of the increase in material costs such as aggregates and petroleum products. One such method is to increase and/or begin using readily available recycled materials like RAS. The use of RAS in hot mix asphalt mixtures reduces the negative impact on the environment associated with the extraction, transportation, and processing of virgin materials while also conserving valuable landfill space.

An issue that affects the performance of asphalt mixtures that incorporates sustainable materials (RAP and RAS) with and without recycling agents is the level of blending that occurs between the aged and virgin asphalt binders. The level of binder not only affects the performance of the asphalt mixture, it also affects the economic competitiveness of the recycling process. If the designer assumes that the asphalt materials (recycled binder and virgin binder) blend totally when the RAP or RAS is actually behaving as a black rock, the resulting binder content will be insufficient and less stiff. Likewise, if it is assumed that the recycled binder does not blend with the virgin asphalt binder when it actually is blending (fully or partially), then the resulting binder content is relatively high and stiffer.

With the increased interest in using high contents of RAP and RAS, the use of recycling agents are being incorporated in order to soften and/or to rejuvenate the aged and stiff binders in RAP and RAS. Since the use of recycling agents in asphalt mixtures were mostly researched in the 1970s, the interaction and compatibility of the recycling agents with RAS and RAP have not been thoroughly evaluated. In addition, the chemistry of RAP binders is different than RAS binders as the rheological and physical properties of air-blown asphalt in RAS are not the same as the paving grade asphalt utilized in RAP.

Research has been conducted on mixtures containing RAP, RAS, and the combination of RAP/RAS with and without recycling agents. However, this research has been conducted on either the mixture properties or on the extracted binders from mixtures to determine the effects of these sustainable type products on performance. Herein lies a problem. When an asphalt binder is extracted, 100% of the binder (recycled and virgin binder) is removed from the mixture. During the production of an asphalt mixture, 100% of the RAS recycled binder is not activated and available in the mixture. A portion of the RAS binder blends with the virgin binder and the remaining RAS binder acts as a "black rock" *[1]*. Research has not been conducted on mixtures that have had 100% of the available RAS binder utilized in the asphalt mixtures and related to the rheological and binder fractionation by molecular weight of the extracted binders. A blending procedure is needed to assure that 100% of the available RAS binder is activated and utilized in the asphalt mixture. This is necessary so that research can adequately determine the effects of recycled materials through the evaluation of the rheological and binder fractionation of the extracted binders to the asphalt mixture characterization properties.

Problem Statement

Asphalt binder prices are at an all-time high with no relief in sight. With the asphalt mixtures prices continuously climbing, highway agencies and owners are continually searching for methods to decrease material costs and maximize their benefits without compromising performance. One such method is to develop innovative technologies to incorporate and increase the percentages of waste and recycled materials, such as RAS and RAP in asphalt mixtures. The usage of RAP has increased in recent years. However, despite the potential benefits of increased RAP contents, state agencies have not proceeded in utilizing high percentages of RAP in asphalt mixtures. This is due to their concerns of non-uniformity of RAP materials and the lack of confidence in the long term field performance of mixtures containing RAP. This is further complicated when RAS is used in conjunction with RAP. Some of the main concerns with the utilization of RAS in asphalt mixtures are the consistency, availability, and quality of the RAS asphalt binder. In addition, there are concerns with satisfactory high, intermediate, and low-temperature pavement performance with the usage of RAS.

Literature Review

State of the Practice in Construction Specifications

A challenge of environmental sustainability utilizing different types of construction specifications is determining how to make production, distribution, and consumption of goods and services last longer and have less impact on our ecological systems consisting of all plants, animals and micro-organisms in an area functioning together with all of the non-living physical factors of the environment. The use of available recycled materials in the asphalt industry is an excellent method of sustainability. The addition of recycled materials replaces a percentage of virgin materials used in the manufacturing process such as aggregates and asphalt binder, which has a direct impact on cost and the environment.

Asphalt Shingles are utilized in roughly 67% of the United States residential roofing market. It is documented that there are 11 asphalt roofing manufacturers servicing the United States markets, as shown below in Table 1 *[2]*.

Table 1
List of U.S. asphalt roofing manufacturers *[2]*

Asphalt Roofing Manufacturer	Headquarters Location
Atlas Roofing Corporation	Atlanta, Georgia
Building Product of Canada	LaSalle, Quebec
Certain Teed Corporation	Valley Forge, Pennsylvania
EMCO Building Products Corp	Quebec, Canada
GAF/ELK Materials Corp.	Wayne, New Jersey
IKO Production Inc.	Toronto, Canada
Malarkey Roofing Products	Portland, Oregon
Owens Corning	Toledo, Ohio
Pacific Coast Building Products	Rancho Cordova, California
W. R. Grace and Co.	Cambridge, Massachusetts
TAMKO Building Products, Inc.	Joplin, Missouri

RAS is comprised of the same components as asphalt mixtures: asphalt binder (19-22% on fiberglass matt base, 30-36% on cellulose felt-base made with paper); fiberglass or cellulose backing (2-15%); sand sized aggregate, ceramic-coated natural rock, (20-38%); and mineral filler or stabilizer that includes dolomite, limestone, and silica aggregates (8-40%).

There are approximately 11 million tons of asphalt shingles manufactured and disposed of in the United States. Each year, ten million tons of installation scraps (tear-offs/post-consumer) from re-roofing and one million tons of manufactured shingle waste are disposed of in landfills. Manufactured asphalt shingle wastes are post-industrial wastes which are rejected asphalt shingles due to manufacturing flaws, such as color or shingle tabs, that are discarded in the manufacturing process of new shingles *[2]*.

The use and evaluation of RAS in asphalt mixtures has become a major initiative in the United States. Williams et al. reported on the performance of recycled asphalt shingles in asphalt mixtures as documented from National Pool Study TPF-5(213) *[3]*. The primary goal of this study was to address research needs of state Departments of Transportation (DOT) and environmental officials to determine the best practices for use of RAS in asphalt mixture applications. The objectives of the aforementioned national study were as follows:

- Address concerns of quality assurance/quality control (QA/QC) in the sourcing, processing, and incorporation of RAS to achieve a final product that would meet requirements for use in state asphalt mixture applications.
- Conduct demonstration projects to provide laboratory testing and field surveys to determine the behavior and performance of RAS and asphalt mixtures at varying percentages, climates, and traffic levels.
- To create a comprehensive database on the performance of RAS in asphalt mixture applications.

There have also been numerous studies evaluating RAS in asphalt mixtures and how usage of RAS in various percentages affects mixture characterization.

Kandhal provided a general overview of waste material including the research work conducted and their potential use in asphalt mixture pavements *[4]*. Kandhal stated that roofing shingles are considered a municipal/domestic waste and can be categorized as follows:

- Industrial Wastes:
    - Cellulose Wastes Wood Lignins
    - Bottom Ash
    - Fly Ash
- Municipal/Domestic Wastes:
    - Incinerator Residue
    - Sewage Sludge
    - Scrap Rubber
    - Waste Glass
- Roofing Shingles
- Mining Waste:
    - Coal Mine Refuse

Kandhal stated that shingles need to be shredded to at least 12.5 mm or smaller prior to introduction in the mix to ensure meltdown and uniform dispersion in the asphalt mixture *[4]*. In addition, asphalt mixtures cost can be reduced by $3.08 per megagram (Mg) by introducing only 5% RAS.

Newcomb et al. evaluated the use of manufacturer waste shingles and tear-off waste shingles in dense-graded asphalt mixtures *[5]*. The dense-graded asphalt mixtures evaluated in this study included two grades of asphalt binder, one aggregate gradation, three levels of roofing shingle content, and two roofing waste types. The dense-graded mixtures were designed using the Marshall method to examine the effects of the RAS on the volumetric proportions and compaction behavior. The resilient modulus test was used to characterize the elastic behavior or stiffness of the dense-graded mixtures at various temperatures. The asphalt mixtures sensitivity to moisture susceptibility was evaluated using a modified Lottman conditioning procedure. The indirect tensile test (IDT) was performed at a slow rate of loading in order to simulate volumetric changes induced by daily temperature changes to determine the asphalt mixtures resistance to cold temperature cracking. It was shown that increasing the content of roofing shingles reduced the asphalt mixtures demand for new asphalt binder. The compactability of the asphalt mixture generally increased with RAS content. It was concluded that the mixtures containing roofing waste were easier to compact than the conventional mixtures. As determined from the resilient modulus test, it was reported that the use of manufactured shingle waste resulted in a less temperature susceptible asphalt mixture. The tear-off waste also reduced the mixture temperature susceptibility but to a lesser degree. The asphalt mixtures stiffness was adversely decreased when the RAS content exceeded 5% by weight of the aggregate. It was found that the use of manufactured shingle waste did not significantly change the moisture susceptibility of the mixtures, but that samples containing tear-off waste had increased moisture susceptibility to moisture damage relative to the control mixture. Test results from IDT indicated that tensile strengths at low temperatures were shown to decrease with increasing RAS content. The strain at peak stress increased for the mixture containing felt-backed shingles with the harder asphalt binder. However, the mixtures made with the tear-off waste showed a decrease in strain capacity with increased RAS content, implying that this material was more brittle at cold temperatures than the control mixture.

Ali et al. reported on the mechanistic evaluation of asphalt mixtures containing reclaimed roofing materials *[6]*. The purpose of this study was to determine the feasibility of using reclaimed roofing materials in asphalt mixtures. Mechanistic evaluations were performed on three asphalt mixtures containing 0, 15, and 25% reclaimed roofing materials. To determine the mechanistic properties of the asphalt mixtures, laboratory prepared specimens were evaluated using resilient modulus, creep and permanent deformation, fatigue, and moisture sensitivity tests. In addition, pavement performance was modeled using a VESYS performance prediction model. Performance parameters, such as rut depths, cracking index, and present serviceability index were used to evaluate the possible improvements to asphalt mixtures containing reclaimed roofing materials. The results indicated that the mix containing 25% reclaimed roofing materials exhibited significant improvements in greater resistance to permanent deformation, longer fatigue life, and better overall pavement performance as compared to conventional mixtures containing no reclaimed roofing materials.

Reclaimed roofing materials were added to asphalt mixtures from 0 to 50% by increments of 5% in a preliminary investigation. It was determined that the addition of 5 and 10% of reclaimed roofing materials had little effect in terms of Marshall Stability and flow. When reclaimed roofing materials were added to the asphalt mixtures in excess of 25%, the Marshall briquettes produced were unsatisfactory and crumbled easily. In addition, the Marshall Stability and flow of these asphalt mixtures were not acceptable.

It was concluded that acceptable asphalt mixtures containing up to 25% reclaimed roofing materials by weight result in a cost savings of approximately 3% asphalt binder as compared to conventional mixes. The use of reclaimed roofing materials improved the fatigue life of asphalt mixture pavements, especially at the 25% reclaimed roofing material content. VESYS analysis predicted that mixtures containing 25% reclaimed roofing material content will outperform the other mixtures, resulting in smaller rut depths and less fatigue cracking. These benefits results in an improved serviceability index.

Janish and Turgeon reported on the history of shingle scrap use in Minnesota and presented laboratory and field performance data *[7]*. Minnesota DOT (MDOT) has been experimenting with the use of shingle scrap in asphalt mixtures since 1990 with the source of the shingle scrap being from shingle manufacturers exclusively. It was concluded that there were little difference between laboratory air void results of the shingle and non-shingle mixtures; extracted asphalt binder from the shingle mixtures was harder than the asphalt binder from control mixtures. The slight increase in hardness had not resulted in any additional cracking; each percentage of RAS incorporated into the asphalt mixture contributed between 0.12 and 0.22% AC by weight of mix; and shingle scrap mixtures are expected to be just as resistant to moisture damage as conventional mixtures. Based on this study, MDOT allows the incorporation of manufacturer waste shingles, up to 5% by weight of aggregate, in asphalt mixtures.

Button et al. reported on a limited study for the purpose of providing Texas DOT (TxDOT) with necessary information to specify materials, design, produce, place, and evaluate paving mixtures containing RAS *[8]*. The specific objectives of this study were to: (1) review published information; (2) interview cognizant DOT individuals from various states; (3) develop material specifications for paving mixtures containing RAS; (4) develop or identify suitable mixture design and analysis procedures for paving mixtures containing RAS; (5) develop construction guidelines for applying asphalt mixtures containing RAS; and (6) measure the engineering properties of asphalt mixtures containing RAS. Various laboratory experiments were conducted on asphalt mixtures containing two types of RAS (manufacturer waste shingles and tear-off waste shingles). Two types of asphalt mixtures were modified with RAS and tested in the laboratory. These included a dense-graded, Type D mixture and a coarse matrix-high binder (CMHB) Type C mixture. RAS materials were added to the asphalt mixtures at 5% and 10%, and the engineering properties of the resulting asphalt mixtures were compared to conventional mixtures with no RAS. An asphalt binder, AC-20, was used in all mixtures except one in which AC-10 was used. Laboratory tests measured the effects of RAS on Hveem stability, indirect tension, resilient modulus at several temperatures, moisture susceptibility, TxDOT static creep, air void content, and voids in the mineral aggregate. The following is reported based on results from tests performed on asphalt mixtures:

- All asphalt extracted and tested from tear-off shingles gave penetrations values of 5 dmm or less. Extracted asphalt binder from manufacturer waste shingles proved to be significantly softer.
- Mixing and compaction temperatures were increased by 14°C because initial attempts to incorporate RAS into the dense-graded asphalt mixtures resulted in air void contents greater than 5%. The increase in temperature resulted in much better compliance with air void requirements especially with the manufacturer wasted shingles.
- Optimum asphalt contents based on TxDOT design procedure showed that the tear-off waste shingles contributed little to the optimum binder content of any of the mixtures. However, in utilizing manufacturer waste shingles the optimum virgin asphalt binder content was reduced significantly for the dense-graded mixture.
- Addition of RAS had generally little effect on the resilient modulus of the dense-graded asphalt mixtures mixture. The addition of both RAS types to the CMHB mixture at either quantity exhibited higher resilient moduli at 40°C and lower resilient moduli at 0°C than the control mixture containing no RAS. The use of RAS lowered the temperature susceptibility of the CMHB mixture.

- Addition of either type of RAS reduced the tensile strength as measured by the IDT test. The manufacturer waste shingles had a larger drop in tensile strength than the tear-off waste shingles.
- For dense-graded asphalt mixtures, the tensile strength ratios suggest that RAS (except for 5% manufacturer waste shingles) improved resistance to moisture susceptibility.
- Hveem stability test results showed a consistent decrease upon addition of either type of RAS at the various percentages incorporated into the asphalt mixtures. However, the reduction in stability was not reduced below the acceptable level (35) for any mixtures. It is noted that the fibrous flakes of RAS are not completely disintegrated during the mixing process. The presence of these fibers acts to reduce the stone-on-stone contact which, in turns, reduces the internal angle of friction which manifest as a reduction in Hveem stability.
- Dense-graded asphalt mixture test results from the TxDOT Static Creep test show that the control mixtures met specified strain and creep stiffness criteria but failed the slope criteria. Generally, the incorporation of RAS had negative effects on static creep results. Further increasing the quantity of RAS made the results worse. With the addition of RAS, the creep stiffness dropped significantly for dense-graded mixtures. Only the asphalt mixture containing 5% manufacturer waste shingles satisfied the stiffness criteria.

It was concluded that generally the addition of 5 to 10% RAS into the dense-graded and CMHB mixtures was detrimental to the engineering properties of the asphalt mixtures. However, it was stated that it appeared that quantities of RAS below 5% incorporated into asphalt mixtures would be satisfactory. Standard compaction temperatures may result in higher than desirable air voids. Therefore, the mixing and compaction temperatures of asphalt mixtures containing RAS may need to be increased by approximately 10 to 20°C in the laboratory and in the field to accommodate the relatively stiffer MWS modified mixtures. In regards to asphalt mixture design and construction, RAS can be handled using the techniques already established for RAP.

Watson et al. evaluated the use of waste roofing shingles generated by shingle manufacturers for use in asphalt mixtures for pavement construction in Georgia *[9]*. Two test sections using 5% manufacturer waste shingles (generally consisted of discolored or damaged shingles) by total weight of mix and asphalt binder (AC) 20 and AC-30 were constructed. The manufactured waste shingles were shredded to ½-inch particle size in all dimensions. The RAS contained fiberglass backing and was incorporated to the mixture in the same manner as RAP. Asphalt mixture samples were obtained from the plant facility and tested for gradation, asphalt binder content, maximum specific gravity, bulk specific gravity, stability and flow, rutting susceptibility (loaded wheel test), viscosity, penetration, and moisture susceptibility for both control and modified sections. It was concluded that the mixtures modified with recycled shingles gave similar to slightly improved material properties, as compared to conventional mixtures. The viscosity of recovered asphalt binder from the RAS modified test sections were slightly higher than the virgin AC control sections but a negative effect on performance was not observed. Thermal cracking was not expected to be an issue in Georgia because of the warm climate where the test sections were placed. The additional stiffness from RAS could be beneficial in reducing rutting susceptibility. Both test sections are performing well compared with the unmodified control sections. It was recommended based on the performance of these test sections that up to 5% shingle manufacturing waste is allowed as a recycling material in asphalt mixtures.

Watson et al. evaluated the use of waste roofing shingles generated by shingle manufacturers for use in asphalt mixtures for pavement construction *[9]*. Two sections of roadway were constructed utilizing a control mixture and a mixture containing waste roofing shingles. The asphalt mixtures for the first section were a 19-mm NMAS control containing no shingles and a 19-mm NMAS mixture containing 5% manufactured roofing waste. The second section evaluated contained a 9.5-mm NMAS asphalt mixture control containing no shingles and a 9.5-mm NMAS mixture containing 5% manufactured roofing waste. It was concluded that although the viscosity of the recovered asphalt from modified mixtures was slightly higher than the control mixture, there does not appear to be a negative effect on performance. Thermal cracking is not expected to be a problem because of the warm climate, and the added stiffness should be beneficial in reducing rutting susceptibility. It was recommended that manufacturer roofing shingle waste at dosage rates up to 5% by total weight of the asphalt mixture should be allowed.

Foo et al. evaluated the engineering properties of asphalt mixtures containing RAS as a possible replacement for part of the neat asphalt binder and aggregate *[10]*. Laboratory testing was performed on the extracted and recovered asphalt binder from the asphalt mixtures containing RAS. IDT was performed to evaluate the modified asphalt mixtures susceptibility to cracking. To evaluate permanent deformation, the dynamic creep test and the Asphalt Pavement Analyzer (APA) were performed. It was concluded that:

- Addition of RAS in asphalt mixtures can produce engineering properties comparable to conventional asphalt mixtures.
- Asphalt from shingles causes a significant increase in the stiffness of the recycled asphalt binder, and in order to increase the performance grade of the recycled asphalt by one grade, 5% additional shingle is sufficient.

- Use of shingles in asphalt mixtures improves the rutting resistance of the mix. However, the mix may have a lower fatigue resistance and also lower low temperature cracking resistance. The use of appropriate softer neat asphalt improves the fatigue and low temperature performance of the mix.

Reed evaluated the constructability, asphalt mixture properties, and pavement performance on a test section which utilized RAS in the wearing and binder courses *[11]*. Four types of pavement sections were placed. One section contained wearing and binder control mixtures without shingles. The other three sections contained: (1) wearing/binder courses with shingles; (2) wearing course with shingles and a binder course with no shingles; and (3) a wearing course with no shingles and binder course with shingles. A five year evaluation provided evidence of very good pavement performance from asphalt mixtures containing RAS. The pavement sections with RAS showed minimal transverse cracking and centerline joint cracking similar to the control section. The control section had between ¼ and ½ inch wheel ruts while all three pavement sections containing shingles had no measurable ruts. It was recommended that new manufacturer waste shingles, including tab punch-outs, can be successfully incorporated in asphalt mixture pavements if the RAS are shredded to 100% passing the ½-inch sieve. Shredding RAS to 100% passing the ½-inch sieve would facilitate the replacement of a portion of the required virgin asphalt binder and, therefore, reduce mix costs. It was also recommended to limit the introduction of shingles to 5% by weight of mix.

Mallick and Mogawer evaluated the use of manufacturer waste shingle for reducing the amount of virgin binder in asphalt mixtures and improvement of the performance of asphalt mixtures *[12]*. It was shown that volumetric and low-temperature properties with varying percentages (3, 5, and 7) of waste shingles were not significantly different from the properties of conventional asphalt mixtures. Mixtures containing 5 and 7% RAS had significantly lower rutting potential compared to asphalt mixtures without RAS. It was stated that the standard deviations of test results for mixes with shingles were low, which indicated a consistent quality of the manufacturer waste shingles. It was shown that the shingles contribute a significant amount of asphalt binder to the mix since the asphalt mixture containing shingles were prepared with less asphalt binder than the control mix.

Zickell evaluated 417 samples of tear-off shingles for the presence of asbestos *[13]*. The single biggest obstacle impeding the usage of RAS from re-roofing projects is the concern over potential asbestos content. In the past, asbestos was sometimes used in the manufacturing of asphalt shingles and other shingle installation materials. The asphalt shingle manufacturers generally acknowledge that between approximately 1963 and the mid-1970s, some manufacturers did use asbestos in the fiber mat in some of their shingle products (total asbestos content was always less than 1%). In addition, other materials used in shingling, such as some types of asphalt binder and some tarpapers, also reportedly contained asbestos. It was concluded from this study, in addition to other asbestos testing performed around the country that little to no presence of asbestos in tear-off shingles and related materials collected from the demolition material from asphalt shingle re-roofing projects was observed.

Sengoz and Topal evaluated the utilization of shingle waste addition from the performance of asphalt mixtures in terms of stability and resistance to permanent deformation by varying the percentages of incorporated RAS from 1 to 5% *[14]*. Also, the addition on the reduction of optimum asphalt content was evaluated. Shingle waste was added in the amounts of 1, 2, 3, 4, and 5% to asphalt concrete mixes prepared with the optimum binder content which yielded the best stability value at 5%. After determination of the optimum percentage of shingle to be added, rutting tests were performed. It was determined that waste shingles can be used in asphalt mixtures as an additive to improve Marshall Stability and rutting resistance. Reduction of optimum binder content by 0.5% in the asphalt mixtures mixture containing 1% RAS significantly increases the stability values of the mixture.

Boyle and Bonaquist reported on a laboratory evaluation which compared the expected performance of plant produced asphalt mixtures, control mixture and mixtures containing post-consumer (tear-offs) and post-manufactured (manufacturer waste) recycled asphalt shingles *[15]*. Performance related binder and mixture tests were used to characterize mixture stiffness, rutting stiffness, fatigue cracking resistance, thermal cracking resistance, moisture sensitivity, and aging. Four asphalt mixtures from two experimental projects were evaluated. Each project included a control mixture without RAS and an asphalt mixture containing approximately 5% RAS by weight of total mix. It was concluded that the performance of asphalt mixtures containing RAS depends highly on the degree of mixing of the virgin and RAS asphalt binders that occurs at the hot-mix plant. When complete mixing occurs, RAS has the potential to significantly alter the performance related properties of the mixture. Specifically, the addition of RAS significantly increases the high temperature performance grade of the combined binder while having only a minor effect on the low temperature performance grade. RAS improves the rutting performance of the mixture while having little effect on thermal fracture resistance. Based on a series of performance-related tests, it was determined that limited mixing of the virgin and RAS asphalt binders occurs when mixtures are produced in typical batch plants. Comparisons of back-calculated binder stiffnesses from asphalt mixture dynamic modulus data with recovered binder stiffnesses showed that the effective stiffness of the combined binder in the RAS mixtures is less than the measured recovered binder. The continuum damage fatigue testing showed RAS mixtures have reduced fatigue resistance when compared to control mixtures for plant-aged conditions. This reduction in fatigue resistance is an indication of limited mixing of virgin and RAS asphalt binders. The addition of RAS did not adversely affect the moisture sensitivity of asphalt mixtures. Simulated long-term aging improved the performance related properties of asphalt mixtures containing RAS. The mixtures were long-term aged by exposing them to a temperature of 85°C for 5 days. This conditioning softens the RAS and virgin asphalt binders, allowing them to further co-mingle, and results in increased stiffness of RAS mixtures at high pavement temperatures with little change in the stiffness at low temperatures. Rutting resistance was improved while the resistance to thermal fracture remained unchanged. The simulated long-term aging also improved the resistance of the RAS asphalt mixtures to fatigue damage.

It was recommended that the quantity of RAS incorporated into asphalt mixtures be limited to ensure that the fatigue resistance of asphalt mixtures containing RAS will not be substantially lower than conventional mixtures containing no RAS and mixtures containing RAP. It was further recommended that the limit should be based on the total asphalt binder content of the mixture and that 5% RAS appears to be acceptable for the mechanical properties of laboratory prepared specimens of high asphalt content surface mixtures. A lower limit is needed for lower asphalt content binder and base course asphalt mixtures. In addition, a limit allowing 15% of the effective asphalt binder in the mixture to be replaced with binder from the RAS appears reasonable based on results from this study.

Hughes and Sypolt reported on the evaluation of the performance of the asphalt in tear-off shingles as a substitute for PG 64-22 *[16]*. The asphalt mixture was a Superpave design using 5% by weight of pulverized post-consumer shingle which also replaced 1.3% of the required 5.9% PG 64-22 asphalt binder. The project contained two sections: (1) control section containing no shingles; and (2) an asphalt mixture containing shingles. Results revealed that the recovered asphalt binder from RAS is very stiff and demonstrated an elevated "melting" point. The asphalt binder had to be heated to 180 – 190°C (355 – 375°F) in order to mold the test specimens. This raised the concern that the asphalt binder will not effectively coat aggregate during asphalt mixture production and may not blend significantly with the virgin asphalt binder. It was stated that the tear-off shingles would act more like asphalt coated sand than an actual cement binder in asphalt mixtures. It was also stated that since the amount of PG 64-22 had been reduced in mix design because of the supposed replacement of asphalt binder from the tear-off shingles, that the mix design will actually result in less asphalt binder coating the aggregate. The reduction in asphalt film thickness coating the aggregate will allow the asphalt mixture to deteriorate faster, thus, resulting in poor pavement performance over time. This means the design life of the pavement may be compromised due to tear-off shingle additives. It was concluded that further research would be necessary to determine if tear-off shingles will perform more successfully as coated sand rather than as an asphalt substitute.

Abdulshafi et al. evaluated the benefits of adding manufacturer waste fiberglass asphalt roofing shingles in asphalt mixtures *[17]*. This project addressed asphalt surface mixtures that were produced with the addition of manufacturer waste shingles. A total of twenty-six asphalt mixtures were studied. The variables included aggregate type, shingle producers, level of shingle addition (0, 5, 10, and 15%), and the type of shingle size reduction. Properties of the produced asphalt mixtures were evaluated based on the results from IDT, resilient modulus, Indirect Tensile Creep Modulus, and AASHTO T283. The following was concluded from this study:

- RAS source and reduction method affects the gradation and asphalt binder content of the produced material.
- Air void contents and voids in the mineral aggregate (VMA) were acceptable and easily maintained in the asphalt trial mixtures containing RAS.
- Addition of RAS improved the Marshall stability.
- Addition of RAS improved the indirect tensile strength of asphalt mixtures tested.
- Addition of RAS increased stiffness, as measured by modulus of resilience, of asphalt mixtures tested at 40°C.
- Addition of RAS reduced stiffness, as measured by modulus of resilience, of asphalt mixtures tested at 0°C. This indicates that these asphalt mixtures will perform better in low temperature environment.
- Indirect tensile creep modulus was influenced by the percentage of RAS incorporated into the asphalt mixture. The indirect tensile creep increased with an increase in RAS addition. Generally, the deformation at the end of the creep test decreased as the level of RAS addition increased. This suggests that the addition of RAS in asphalt mixtures can reduce rutting susceptibility.

McGraw et al. investigated the use of both tear-off shingle and manufacturer shingles combined with traditional reclaimed asphalt materials *[18]*. Two studies were conducted to evaluate the influence of RAS addition to the low temperature properties of asphalt mixtures prepared with RAP. The Minnesota study utilized PG 58-28 binder in three mixtures: 20% RAP; 15% RAP + 5% tear-off shingles; and 15% RAP + 5% manufactured waste shingles.

In the Missouri study, two binders (PG 58-28 and PG 64-22) were utilized with a single source of RAP and tear-off shingles, McGraw et al. The Minnesota results indicated that the two types of shingles performed differently. The manufactured shingles seems to be beneficial as it slightly increases the stiffness and did not affect the tensile strength of both mixtures and extracted binders. The asphalt binder critical temperature increased very little. The addition of tear-off shingles appeared to affect properties in a more negative way, although it also slightly increased the stiffness of binders. However, it lowered the strength of the binder significantly at the higher test temperature and increased the binder critical temperatures. This was not confirmed by strength tests, which indicated no significant reduction with the addition of tear-off shingles. The extracted binder rheology showed that the addition of shingles increases only slightly the stiffness but lowers the m-values significantly. This indicates that the addition of RAS lowers the temperature susceptibility of the binders making them stiffer than conventional and RAP modified binders at intermediate temperatures more characteristic of fatigue cracking distress.

The Missouri test results indicate that for the PG 64-22 asphalt mixture, at temperatures below-10°C, the addition of RAS increased the mixture stiffness considerably, McGraw et al. This increase would likely result in large thermal stresses developing in the pavements. This effect was less significant in PG 58-28 mixtures. It remains unclear if using a softer grade was a reasonable solution to meeting the grade for the final product as the use of a softer grade may increase the price of the mixture and make the addition of shingle less cost effective.

McGraw et al. reported that a new provisional AASHTO specification (PP 53) allowing the use of either manufacturers (post-industrial) or tear-off (post-consumer) shingle scrap as an additive to asphalt mixtures *[18], [19]*. There are a three important details within the AASHTO specification: (1) The final RAS product must be sized and screened such that 100% passes a ½-inch sieve screen. This is important because it was found that the size of RAS can be expected to affect the fraction of shingle asphalt that contributes to the final blended binder. RAS ground to a finer size passing a No. 4 sieve can be expected to effectively utilize as much as 95% of the total available binder. (2) The actual addition rate of RAS is left up to the contractor. (3) The new specification states that if the quantity of RAS asphalt binder exceeds 0.75% by weight of the new asphalt mixture, the RAS binder and the virgin binder shall be further evaluated to ensure the performance grade of the final blended asphalt mixture complies with the originally specified performance grade requirements.

Baumgardner and Rowe introduced rheological high and low temperature parameters and the use of rheological properties to replace softening point parameters currently being used in the evaluation of saturants and coating asphalts for asphalt roofing shingles *[20]*. ASTM D 312, “Standard Specification for Asphalt used in Roofing,” is used to characterize asphalt used in roofing as an interply adhesive or flood coats for built-up roof (BUR) membranes *[21]*. It is stated that ASTM standard specifications do not exist for saturants and coating asphalts for roofing shingles. Industry practice is to use softening point as mentioned in ASTM D 312, "Standard Specification for Asphalt Used in Roofing" to specify and evaluate asphalt used as saturants and coating grade asphalt *[21]*.

Tighe et al. measured the performance of five asphalt mixtures with and without RAS and varying percentages of RAP *[22]*. The asphalt mixtures evaluated were: Mix 1 – Virgin material (control); Mix 2 – 20% RAP material; Mix 3 – 20% RAP material, 1.4% shingles; Mix 4 – 20% RAP material, 3.0% shingles; and Mix 5 – 3.0% shingles. Asphalt mixtures were evaluated and analyzed to measure the elastic properties of the mixtures, fatigue and thermal cracking susceptibility, and permanent deformation using the dynamic modulus test, resilient modulus test, Thermal Stress Restrained Specimen Tensile Strength Test (TSRST), and the French wheel rutting test. Test results indicate the following:

- Dynamic Modulus – Mix 1 (control) and Mix 2 (20% RAP) had the highest dynamic modulus at low temperatures which is indicative of lower fatigue susceptibility. At high temperatures, Mix 3 (20% RAP and 1.4% shingles), Mix 4 (20% RAP and 3.0% shingles), and Mix 5 (3.0% shingles) had the lowest dynamic modulus with Mix 4 being the most prominent mix. The introduction of shingles into asphalt mixtures lowered the dynamic modulus which is indicative of lower rutting susceptibility.
- Resilient Modulus – Mix 1 (control) had the highest resilient modulus while Mix 5 (3.0% Shingles) had the lowest. The resilient modulus indicates the fatigue and thermal cracking susceptibility of a pavement and the quality of materials in the asphalt mix.
- Indirect tensile strength test was performed to determine the tensile strength of the specimens. Mix 3 (20% RAP and 1.4% shingles) was shown to have the highest tensile strength, followed by Mix 1 (control) and Mix 2 (20% RAP) respectively. Mix 4 (20% RAP and 3.0% shingles) and Mix 5 (3.0% shingles) were found to have the lowest tensile strength, with Mix 5 being the lowest.
- Rutting – It was determined that Mix 2 (20% RAP) performed the worst having the greatest rut depth for all cycle variations. Mix 4 (20% RAP and 3.0% shingles) had the best overall performance, having the lowest rut depth followed by Mix 5 (3.0% shingles). It was stated that the percentage rut depth for all mixes were very small and was expected to perform well in the field.
- TSRST – was performed to determine the low temperature cracking susceptibility. Mix 3 (20% RAP and 1.4% shingles) reached the highest temperature prior to failure. Mix 1 (control) withstood the highest stress prior to failure. The temperature and stress reached by Mix 4 (20% RAP and 3.0% shingles) and Mix 5 (3.0% shingles) prior to failure was

significantly lower than the temperature and stress reached by Mix 1 (control), Mix 2 (20% RAP), and Mix 3. It was stated that inclusion of large quantities of shingles into a mix, such as 3.0 percent, encourages thermal cracking.

It was concluded that based on the laboratory analysis, Mix 3 (20% RAP and 1.4% shingles) is the better overall asphalt mixture as compared to Mix 4 (20% RAP and 3.0% shingles) and Mix 5 (3.0% shingles).

Maupin evaluated the placement and early performance of a test section of asphalt mixture containing manufacturer waste shingles in Virginia *[23]*. In 1999, the Virginia Department of Transportation (VDOT) implemented a special provision to allow the use of either tear-off or manufacturer waste shingles as requested by the contractor. In 2006, a contractor requested to use 5% manufacturer waste shingles in an asphalt mixture surface mixture and a surface mixture containing 10% RAP on a 4.1-mile two-lane section of roadway. Both asphalt mixtures utilized a PG 64-22 asphalt binder. To compare mixture performance, density tests were performed on the pavement and various laboratory tests such as permeability, fatigue (beam fatigue – The American Association of State Highways and Transportation Officials (AASHTO) T 321), tensile strength ratio (AASHTO T283), rut, and Abson binder recoveries were performed on samples of mix collected during the construction of the section. It was concluded that surface mixes with RAP and surface mixes with shingles behaved similar during placement and compaction. Both mixes were similar in gradation and volume, nearly identical permeability, the same endurance limits, and excellent tensile strength ratios. The performance grade of the recovered asphalt binder from both mixtures increased from PG 64-22 to PG 70-22 with the addition of RAP and RAS. It was noted that the shingle binder was slightly stiffer than the RAP mixtures. It was further concluded that both the field and laboratory tests resulted in identifying that the behavior and performance of the two mixes should be similar and are performing well after 18 months of in-place service.

Anurag et al. conducted research to determine whether homogeneously dispersed roofing waste polyester fibers with varying fiber lengths and different fiber contents improved the indirect tensile strength (ITS) and moisture sensitivity percentages of this fiber on ITS *[24]*. In addition, the effects of aggregate sources on the mechanical properties of the asphalt concrete mixtures containing roofing waste polyester fibers (e.g., air voids, ITS, and toughness) was determined. It was concluded that generally the addition of polyester fiber was beneficial in improving the wet tensile strength and tensile strength ratio (TSR) of the modified mixture. The toughness value in both dry and wet conditions was increased. The addition of polyester fibers increased the void content, the asphalt content, the unit weight, and the Marshall Stability.

Schroer documented the usage of RAS in Missouri. It was reported that Missouri added a provision in the standard specifications to allow the addition of RAS in any mixture requiring the use of Performance Grade (PG) 64-22 *[25]*. RAS modified asphalt mixtures requiring polymer modified asphalt binders are not allowed at this time because of lack of information. Missouri allows up to a maximum of 7 percent RAS in asphalt mixtures and the RAS can be from manufacturer waste or tear-off (post-consumer) waste. The MoDOT had concerns in the usage of tear-off shingles in asphalt mixtures on its effect on the resistance to fatigue and cold weather cracking due to the RAS asphalt binder being much stiffer than ACs commonly used in asphalt pavements. The asphalt binders from tear-off shingles, manufacturer waste shingles, and roadway were blended to determine the performance grading (PG). It was determined that when greater than 70% roadway or virgin asphalt binder was added to the blend, the low temperature grading was not greatly affected by the shingle asphalt. The single asphalt affected the blend more rapidly as the percentage of virgin asphalt binder decreased below 70%. Asphalt binder from tear-off shingles was obtained utilizing chemical extraction. After evaluation of test results from binder blending, the low temperature cracking potential became a concern. It was determined that where asphalt mixtures containing between 60 and 80% virgin binder, the tear-off binder began to take control of the low temperature properties.

Maupin evaluated the suitability of using up to 5% tear-off shingles in asphalt concrete *[26]*. This study was designed to see if asphalt mixtures could be produced where excessive aging of RAS was not detrimental to mix durability. Maupin used laboratory tests to evaluate gyratory volumetric properties, gradation, and asphalt content. In addition, rut tests, beam fatigue tests, Indirect Tensile Test, (ITS), and tests to determine recovered asphalt properties were performed. It was concluded that fatigue durability was comparable to conventional mixtures; permanent deformation was within VDOT's specification; recovered asphalt binders indicated improvement in high-temperature grading, and low-temperature grading was within specification. Maupin stated that Bonaquist in 2009 reported at the 4th Asphalt Shingle Recycling Forum in Chicago Illinois that the addition of 25% RAS binder improved the high temperature grade two levels and reduced the low-temperature grade by one grade.

Robinette and Epps analyzed recycled materials in asphalt mixtures (reclaimed asphalt pavement, post-industrial and post-consumer asphalt shingles), stabilization/treatment of pavement layers, and rehabilitation methods (cold and hot-in-place recycling) for flexible pavements against standard construction materials and methods *[27]*. Life cycle assessment, which includes energy consumption, emissions generation and natural resource consumption, in addition to the price of construction, was used to evaluate various roadway construction activities. Materials evaluated specific to hot mix asphalt mixtures included RAP, RAS, and warm mix asphalt which were compared to conventional materials and construction. It was concluded that in most instances these activities can reduce energy consumption, emissions generation, and conserve natural resources (aggregate and asphalt binder) while reducing the price of construction. It was further concluded that the use of recycled materials in asphalt mixtures reduces the overall environmental impact and produces a price savings.

Scholz investigated the use of RAP and RAS in asphalt mixtures in Oregon *[28]*. This study investigated how various proportions of RAP and ½ inch minus RAS from tear-offs added to hot mixed asphalt mixtures affect the Superpave performance grade of the blended binder. Since this was a limited study, only one virgin asphalt binder (PG 70-28) and one aggregate source commonly used in Oregon was utilized in the asphalt mixtures. Virgin asphalt binder, asphalt binders recovered from the RAP and RAS, and blended binders from each mixture were extracted and tested. The critical temperatures of the blended asphalt binders from mixtures with 5% RAS and 0 to 50% RAP were compared with the critical temperatures of the virgin asphalt binder. In addition, the gradations and asphalt binder contents of the RAP and RAS were determined. It was reported that test results indicated that mixtures with 5% RAS and no RAP resulted in an increase of the performance grade of the blended asphalt binder. Asphalt binders recovered from mixtures containing both RAP and RAS indicated an increase in both high temperature and low temperature performance grades of the blended binder as RAP increased up to about 30%. It was concluded that incorporation of 5% RAS (by total weight of mixture) and no RAP in dense-graded asphalt mixtures results in an increase in both high temperature and low temperature performance grades of the blended asphalt binder as compared to the virgin asphalt binder.

The AASHTO Subcommittee on Materials conducted a recycle survey in 2010 *[29]*. Data from the Excel spreadsheet indicated that there were 34 states reporting, which included Ontario, Puerto Rico, and the Western Federal Lands. About 38% of those entities reported they allow the use of RAS in specifications. The typical range of RAS allowed is 3-5%. Indiana allows less than 25% by weight of total binder whereas New Hampshire allows 0.6% of the total mix replacement binder. It was reported that Oregon and Iowa are developing specifications to allow up to 5% RAS. It was indicated that 21 of the reported states (including Puerto Rico and Western Federal Lands) do not allow the use of RAS. Some reported obstacles: were supply of RAS; durability, fatigue and low temperature cracking of asphalt mixtures; effect of composite binder on thermal, fatigue, and top-down cracking; and the effect of RAS on the high/low asphalt binder properties. Research needs identified during this survey included the addition of rejuvenators to lower viscosity and allow the use of higher RAS percentages, RAS binder contribution, asphalt mixture performance testing, asphalt binder grade bumping, effect of RAS on the mitigation of fatigue and low temperature cracking, durability, effects of increased percentages on RAS on asphalt binder properties, use of RAS in warm mix asphalt (WMA) asphalt mixtures, and the ability of aged, oxidized binders used in shingles to meet paving grade asphalt binder specifications.

Pappas reported on the Recycle Materials Survey to the Recycling Asphalt Pavement Expert Task Group (RAP ETG) in Irvine, California *[30]*. It was shown that in the United States: 15 states allow 5% RAS, Missouri allows 7% RAS, Arkansas allows 3% RAS, 8 states are looking into the use of RAS in asphalt mixtures, 19 states allow no RAS, and 6 states did not report to the survey. Pappas reported on research needs that included the percent binder contribution from RAS, asphalt mixture performance testing, asphalt binder bumping and blending, effects on fatigue and low temperature cracking, use of RAS in WMA, impact of RAS on the asphalt binder, and the use of rejuvenators.

Recycling Agent (RA) Classifications

Figure 1 presents the main classes of recycling agents that have been used in asphalt paving applications *[31]*. RAs are classified as either a rejuvenating agent or a softening agent. There are fundamental differences between softening agents and rejuvenating agents. Both are called recycling agents, but each group acts in a different manner when added into an asphalt mix. Softening agents can lower the viscosity of the aged binder, helping to achieve the proper workability of the RAP and/or RAS mixtures. A similar effect is achieved by the use of warm mixture additives and anti-stripping agents. Nonetheless, their contribution is limited to changing the physical properties of an aged binder. Roberts stated that rejuvenating agents are added for the purpose of restoring physical and chemical properties of the old binder *[32]*. Rejuvenators are, for the most part, organic oils that are rich in maltenes constituents necessary to keep the asphaltenes dispersed *[33]*.

They usually consist of lubricating oil extracts and extender oils with a low content of saturates that do not react with the asphaltenes *[32]*, *[34]*. Shen et al. reported that rejuvenators are used to recover the properties of aged by binders by changing the chemical composition of the aged binder *[35]*. As an asphalt binder ages through oxidation; the aged binder has lower concentrations of the more reactive components (Nitrogen base, N, and A1, the first acidaffins) and higher concentrations of the less reactive components (Paraffines, P, and A2, the second acidaffins). A rejuvenator used for restoring the aged asphalt binders usually has a minimum N/P ratio of 0.5 to ensure the compatibility of the rejuvenator and the aged binder and to prevent syneresis (exudation of Paraffins from asphalts).

Table 2 presents a list of recycling agent types and their classifications. As shown in this table, there are a wide range of rejuvenators and softening agents. Coal-tar based oil and slurry oil have been used as rejuvenators and softening agents but are discouraged due to health concerns.

Rejuvenating Agents	Softening Agents
Lube Extracts	Lube Stock
Extender oils (aromatic oils)	Lubricating or Crankcase Oil
Anti-Stripping Agent	Slurry Oil
Waste Vegetable Grease	Asphalt Flux Oils

Figure 1
Recycling agent classifications *[31]*

Table 2
Recycling agents' descriptions *[31]*

ID	Recycling Agent Type	Classification
1	Aromatic Extract	Rejuvenator
2	Naphthenic Oil	Rejuvenator
3	Petroleum blends (e.g., naphthenic base oil, petroleum asphalt, maltenes, and polymer)	Rejuvenator
4	Vegetable Derived Oils (e.g., soy beans, tall oil)	Rejuvenator
5	Reclaimed Lube Oil Bottom	Rejuvenator
6	Waste Vegetable Grease	Rejuvenator
7	Emulsion-asphalt, rejuvenator oil, polychloroprene polymer	Rejuvenator
8	Industrial Process Oil	Rejuvenator
9	Liquid Anti-Strip Agent (ASA)	Rejuvenator/Softening Agent/ Surfactant
10	Asphalt flux Oil	Softening Agent
11	Plant-Derived Oils/Esters	Softening Agent
12	Lube Stock / Lubricating Oil	Softening Agent
13	Soft Asphalt Binder	Softening Agent
14	Bunker Fuel (#4 and/or #5 Fuel Oil)	Softening Agent

State of the Practice: Use of Recycling Agents in High RAP/RAS Mixtures

Recycling agents can be incorporated into the asphalt mixture by directly mixing them with asphalt binder either at the batching plant or in the field in the case of hot-in-place recycling *[35]*, *[36]*. AASHTO R–14 and ASTM D4552/D4552M–10, both designated "Standard

Practice for Classifying Hot Mix Recycling Agents," are the references whereby additives can be identified for use in recycling of asphalt mixtures *[37, 38]*. These two standards establish their requirements based on viscosity, flash point temperature, weight percent of saturates, specific gravity and selected properties of the RTFO and TFO residues. Recycling agents are classified in six groups; RA 1, RA 5, RA 25, RA 75, RA 250 or RA 500. Slight discrepancies exist between the two standards for the specified thresholds.

Processing RAS

Prior to use in asphalt mixtures, RAS must first be processed. The processes involved are shredding, screening, blending, and watering *[14]*. The technology and RAS grinding process was adapted from the wood grinding process where material is fed into a cylinder hammer mill with teeth on it *[13]*. RAS used in asphalt mixtures are typically shredded into pieces approximately ½ inch in size and smaller using a shingle shredding machine that consists of a rotary shredder or a high speed hammer mill with teeth on it. Often RAS are passed through the processing equipment twice to obtain the appropriate size reduction. The speed of the shredding process is dependent on the type of teeth used and the rotation of the hammer mill. After this operation, shredded shingles are screened to the desired gradation and stockpiled. Experience has shown that the size of the processed RAS particles should be no larger than ½ inch to ensure complete digestion of the roofing shingle scrap and uniform incorporation into the asphalt mixture. It is reported that shredded scrap shingles greater than ½ inch (12.5 mm) in size does not readily disperse and functions much like an aggregate *[14]*. It is stated that tear-off shingles are easier to shred than manufacturer waste shingles because tear-offs have hardened with age. Manufacturer waste shingles tend to become plastic due to heat and the mechanical action of the shredding process which presents a problem with RAS agglomerating during processing. Tear-offs, which have hardened during its service life, are less likely to agglomerate. In addition, it is reported that tear-offs are much more variable in composition than manufacturer waste shingles (Virginia Agency of Natural Resources,). To reduce the effect of the RAS agglomerating, a water process is used during the shredding operation. The application of water, however, is not desirable because the processed RAS becomes very wet and must be dried prior to incorporation into the asphalt mixture. Sometimes it becomes necessary to reprocess and rescreen the RAS prior to introduction at a hot mix plant facility because the RAS can harden during and after stockpiling. To mitigate this, the processed RAS can be blended with sand or RAP to prevent the particles from sticking together *[14]*.

Current Asphalt Mixture Design Utilizing RAS

AASHTO has recently developed a revised standard practice for design considerations when using RAS in asphalt mixtures, AASHTO Designation: PP 78-14: "Standard Practice for

Design Considerations When Using Reclaimed Asphalt Shingles (RAS) in Asphalt Mixtures," 2014 *[39]*. This standard practice provides guidance for designing new asphalt mixtures that contains RAS. The standard provides specific guidance on how to determine the shingle aggregate gradation, determination of the performance grade (PG) and the percentage of the virgin asphalt binder, and how to estimate the RAS asphalt binder contribution to the final blended binder. The RAS asphalt binder availability factor is assumed to range from 0.70 to 0.85. It is stated that the introduction of RAS into an asphalt mixture will affect the gradation properties and that the designer must determine the particle size and percentage of shingle aggregate present and adjust the virgin aggregate composition to ensure that the blended gradation meets requirements. Also, the introduction of RAS affects the virgin asphalt binder content requirements, and the designer must determine the virgin asphalt binder content of the new asphalt mixture as part of the volumetric mix design process. During production, the available RAS asphalt binder will mix with the virgin asphalt binder to produce a final blended binder. The designer must be prepared to adjust the PG of the virgin asphalt binder to compensate for this effect. Binder grade adjustment guidelines from AASHTO M 323 – "Standard Specification for Superpave Volumetric Mix Design" have been adapted based on RAS and/or RAP asphalt binder percentages *[40]*.

It is expected that the particle size of the RAS can affect the percentage of shingle asphalt binder that contributes to the final blended asphalt binder. RAS material that has been ground to a size passing the 12.5-mm (1/2-inch) sieve can be expected to release lower levels of available shingle asphalt binder (20 to 40%). Whereas, RAS material ground to 4.75 mm (No. 4) sieve can expect as much as 95% release of available shingle asphalt binder. Release of shingle asphalt binder into the asphalt mixture can result in reduced virgin asphalt binder requirements. However, it is unlikely that all the shingle asphalt binder will dissolve and blend with the virgin asphalt binders. These undissolved particles of shingle asphalt binders may act like aggregate particles and require more virgin asphalt binder to coat the particles. In addition, the particles may absorb bituminous oils from the virgin asphalt binder. It is stated that since the major portion of the shingle fiber was retained on the 4.75 mm (No. 4) sieve, the fiber fabric can be removed by tweezers or other appropriate methods (AASHTO PP53 – Standard Practice for Design Considerations When Using Reclaimed Asphalt Shingles (RAS) in New Hot Mix Asphalt (HMA)") *[19]*.

In addition, AASHTO has a standard specification for use of reclaimed asphalt shingles in asphalt mixtures, AASHTO Designation: MP 23-14: "Standard Specification for Use of Reclaimed Asphalt Shingles for Use in Asphalt Mixtures," *[41]*. The reclaimed asphalt shingles may be either manufactured shingle waste of post-consumer asphalt shingles. It is stated that neither the manufactured nor the post-consumer asphalt shingles should be blended together for production in asphalt mixtures. It is required that reclaimed asphalt shingles be processed so that 100% passes the 9.5-mm (3/8-inch) sieve. The addition rate of RAS shall be such that the gradation and the volumetric mix design requirements comply with AASHTO M 323 *[40]*. In regards to deleterious materials, RAS shall not contain extraneous materials (glass, metals, rubber, brick, paper, wood, and plastic) more than 1.5% by total mass as determined by material retained on and above the 4.75-mm (No. 4) sieve. In addition, nonmetallic deleterious materials shall not exceed 0.5% by total mass as determined by the amount of material retained on and above the 4.75-mm (No. 4) sieve. Asbestos fibers shall be less than the maximum percentage based on testing procedures and frequencies established by the specifying agencies.

OBJECTIVE

The objective of this study was to characterize the laboratory performance and performance of conventional asphalt mixtures (mixtures containing RAS and/or RAP) with and without recycling agents, RAs. In addition, the molecular structure of asphalt binders of conventional asphalt mixtures, as well as mixtures containing RAS and/or RAP, with and without RAs were correlated with their cracking potential utilizing Gel Permeation Chromatography (GPC) and Fourier Transform Infrared Spectroscopy (FTIR). Also, asphalt mixture blending procedures were developed to ensure that 100% of the available recycle binders were utilized within the asphalt mixture.

SCOPE

This report documents the methodology and findings of the research conducted to characterize asphalt mixtures utilizing high recycled asphalt shingles (RAS), recycled asphalt pavement (RAP) contents, and recycling agents. Details of the material variables are listed below:

- A total of 11 Superpave l2.5-mm nominal maximum aggregate size (NMAS) Level 2 asphalt mixtures meeting DOTD specification were evaluated in this study. These asphalt mixtures were produced using a styrene-butadiene-styrene (SBS) modified PG 70-22M asphalt meeting Louisiana specifications or PG 52-28 with or without recycling agents.
- Gravel aggregates and natural sand (coarse and fine) that are commonly used in Louisiana were included in this study.
- Manufacturer waste shingles (MWS) and post-consumer (tear-off) waste shingles (PCWS) from a Louisiana source were used in this study.
- Four different types of recycling agents (i.e., Hydrogreen, Cyclogen-L; Asphalt Flux (PG 32.3-46.6), and PG52-28 soft asphalt binder) were used with varying contents for asphalt mixtures that contain 5% PCWS except control mixtures.

METHODOLOGY

Research Approach

To completely characterize the asphalt mixtures' fundamental engineering properties under a full range of climatic conditions, a suite of laboratory mechanistic tests were conducted at low, intermediate, and high temperature regimes. These mechanistic tests include the dynamic modulus test for viscoelastic characterization, semi-circular bend (SCB) test for intermediate temperature fracture performance, and the TSRST for low temperature performance. In addition, a Hamburg type loaded wheel tracking (LWT) test was performed to evaluate the mixtures' resistance to permanent deformation and moisture susceptibility. Triplicate samples were used for each test, except the LWT test, in which two replicates were used. The air void content for each specimen was 7.0 ± 0.5%.

A total of 11 Superpave l2.5-mm nominal maximum aggregate size (NMAS) Level 2 asphalt mixtures meeting DOTD specification (i.e., $N_{initial} = 8$, $N_{design} = 100$, $N_{final} = 160$ gyrations) were designed and examined. Gravel aggregates and natural sand (coarse and fine) that are commonly used in Louisiana were included in this study. Manufacturer waste shingles (MWS) and post-consumer (tear-off) waste shingles (PCWS) from a Louisiana source were used in this study.

Comparative evaluations on the 11 asphalt mixtures were conducted, which include:

- a conventional Superpave asphalt mixture that contain PG 70-22M a, styrene-butadiene-styrene (SBS) modified asphalt binder, without RAS, RAP, and RAs as a control mixture;
- mixtures with PG70-22M binder vs. an unmodified PG 52-28 binder;
- mixtures with 5% MWS vs. with 5% PCWS;
- mixtures with 15% RAP vs. without RAP; and
- mixtures with RAs vs. without RAs.

RAS was incorporated into the asphalt mixtures at 5% by total weight of mix. RAP was incorporated into the asphalt mixtures at 15% by total weight of mix. The rate of recycling agents added was based on volumetric analysis through various iterations to assure that the maximum benefit of available recycle binder content.

To evaluate performance, physical and rheological tests were evaluated on asphalt binders and correlated with their cracking potential utilizing Gel Permeation Chromatography (GPC) and Fourier Transform Infrared Spectroscopy (FTIR).

Experimental Design Factorial

To characterize laboratory mixture performance through their mechanistic and volumetric properties, a suite of tests was conducted to evaluate the high, intermediate, and low temperature performance of conventional asphalt mixtures and mixtures containing RAS and/or RAP with and without RAs. Also, the asphalt binders' molecular structure was correlated with their cracking potential through binder fractionation. Table 3 lists the mixture and binder experiments conducted in this study.

Table 3
List of mixture and binder experiment

Mixture Experiments	Binder Experiment
Dynamic Modulus	Asphalt Binder Performance Grading
High Temperature – Loaded Wheel Tracking (Hamburg Type)	Multiple Stress Creep Recovery
Intermediate Temperature – Semi-Circular Bend (SCB) Test	Linear Amplitude Sweep Test
Low Temperature – Thermal Stress Restrained Specimen Tensile Strength Test	Complex Shear Modulus
	Glover-Rowe Parameters
	ΔT_c - Change in Critical Binder Temperature
	Saturate, aromatic, resin, and asphaltene (SARA)
	GPC - Gel Permeation Chromatography
	FTIR –Fourier Transform Infrared Spectroscopy

Table 4 presents a summary of the experimental test factorial considered. The mixture designations and constituents are defined below:

Table 4
Experimental factorial

Mixture		Mixture Variables							High Temp		Inter. Temp	Low Temp
									E*	LWT	SCB	TSRST
12.5 mm NMAS – Superpave Level 2	Mix Designation	Shingle Type	%RAS	%RAP	%RA-1	%RA-2	%RA-3	%RA-4	Unaged		Aged	Aged
	70CO	N/A							3	2	3	3
	70PG5M	MWS	5						3	2	3	3
	70PG5P	PCWS	5						3	2	3	3
	70PG5P5HG	PCWS	5		5				3	2	3	3
	70PG5P12CYCL	PCWS	5			12			3	2	3	3
	70PG5P20FLUX	PCWS	5				20		3	2	3	3
	52PG5P	PCWS	5					4.6%AC	3	2	3	3
	70PG15RAP	N/A		15					3	2	3	3
	70PG5P15RAP	PCWS	5	15					3	2	3	3
	70PG5PHG15RAP	PCWS	5	15	5^{+}				3	2	3	3
	52PG5P15RAP	PCWS	5	15				3.5%AC	3	2	3	3

MWS: Manufacturer waste shingle;
PCWS: Post-consumer waste shingle;
RAS: Recycled asphalt shingle;
RAP: Reclaimed asphalt pavement;
RA-1 = Hydrogreen;
RA-2 = Cyclogen-L;
RA-3 = Asphalt Flux (PG 28-46)
RA-4 = PG52-28 Soft asphalt binder (same mix design as 70PG5P except asphalt binders were different)
5^{+} = 5% RA1 added by total weight of RAS material plus 0.75% RA1 added by total weight of RAP material

- 70CO – Conventional mixture containing PG 70-22 asphalt binder that’s SBS modified, no RAP, no RAS, no recycling agents;
- 70PG5M – Mixture containing PG 70-22 asphalt binder that’s SBS modified, 5% MWS, no recycling agents;
- 70PG5P – Mixture containing PG 70-22 asphalt binder that’s SBS modified, 5% PCWS, no recycling agents;
- 70PG5P5HG – Mixture containing PG 70-22 asphalt binder that’s SBS modified, 5% PCWS, 5% Hydrogreen added by total weight of PCWS;

- 70PG5P12CYCL – Mixture containing PG 70-22 asphalt binder that's SBS modified, 5% PCWS, 12% Cyclogen-L added by total weight of PCWS;
- 70PG5P20FLUX – Mixture containing PG 70-22 asphalt binder that's SBS modified, 5% PCWS, 20% asphalt flux added by total weight of PCWS;
- 52PG5P – Mixture containing PG 52-28 soft asphalt binder, 5% PCWS, no recycling agents;
- 70PG15RAP – Mixture containing PG 70-22 asphalt binder that's SBS modified, 15% RAP, no recycling agents;
- 70PG5P15RAP – Mixture containing PG 70-22 asphalt binder that's SBS modified, 5% PCWS, 15% RAP, no recycling agents;
- 70PG5PHG15RAP – Mixture containing PG 70-22 asphalt binder that's SBS modified, 5% PCWS, 15% RAP, Hydrogreen recycling agent added at 5% by total weight of PCWS plus an additional 0.75% added by total weight of RAP; and
- 52PG5P15RAP – Mixture containing PG 52-28 soft asphalt binder, 5% PCWS, 15% RAP, no recycling agents.

Hot Mix Asphalt Mixture Design Development

Superpave 12.5-mm nominal maximum aggregate size (NMAS) Level 2 asphalt mixtures meeting Louisiana Department of Transportation and Development (DOTD) specification for Roads and Bridges (DOTD, 2006). Specifically, the optimum asphalt binder content was determined based on volumetric properties (VTM = 3.0 – 5.0%, VMA ≥ 13%, VFA = 68% - 78%) and densification requirements (%G_{mm} at $N_{initial} \leq 89$, %G_{mm} at $N_{final} \leq 98$). These asphalt mixtures were designed according to AASHTO R 35-09, "Standard Practice for Superpave Volumetric Design for Hot Mix Asphalt (HMA)" and AASHTO M 323-07, "Standard Specification for Superpave Volumetric Mix Design." The Superpave mixture design procedure requires a specific number of gyrations that are determined by the expected Equivalent Single Axle Load (ESAL) of traffic expected on a given roadway. The desired aggregate structures for each asphalt mixture consisted of coarse and fine aggregate typically used in Louisiana. The aggregate structure for all mixtures considered in this study was similar.

Asphalt binder utilized in this study was a PG 70-22M, which is commonly used in Louisiana, in addition to a PG 52-28 that was considered a RA. Also, rejuvenating and softening type RAs were utilized, Cyclogen-L, Hydrogreen, and an asphalt flux, respectively. In addition, RAS and RAP materials were utilized in this study.

Aggregate Tests

Aggregates from each source were tested to determine aggregate properties. The test items include: coarse aggregate angularity, fine aggregate angularity, flat and elongated particles, gradation analysis, and sand equivalency. Table 5 indicates the aggregate test methods utilized.

Table 5
Aggregate test methods

Test	Test Method
Coarse aggregate angularity	AASHTO TP 61 - Standard Method of Test for Determining the Percentage of Fracture in Coarse Aggregate *[42]*
Fine aggregate angularity	AASHTO T 304 - Standard Method of Test for Uncompacted Void Content of Fine Aggregate *[43]*
Flat and Elongated	ASTM D4791 - Standard Test Method for Flat Particles, Elongated Particles, or Flat and Elongated Particles in Coarse Aggregate *[44]*
Sand Equivalency	AASHTO T 176 - Standard Method of Test for Plastic Fines in Graded Aggregates and Soils by Use of the Sand Equivalent Test *[45]*
Aggregate Gradation	AASHTO T 27 - Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates *[46]*

To determine the aggregate gradation from each source, a washed sieve analysis was performed on aggregates in accordance with AASHTO T 27 "Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates." Additionally, all coarse aggregates were sieved and materials retained on the ¾-in., 1/2-in., 3/8-in., No. 4 sieves, and passing No. 4 sieves were stored in separate containers *[47]*. Separating the aggregates into various size fractions is needed in order to batch the required aggregate blend gradations directly from individual sized fractions for the desired asphalt mixture design. This method allowed for consistent replication of the asphalt mixtures composite aggregate gradation since each sieve size batch weight is mixed at the exact proportions needed for the hot mix job mix formula.

Asphalt Binder and Binder Fractionation by Molecular Weight Tests

The asphalt binder's rheological properties change during the production of an asphalt mixture as the asphalt cement (AC) ages over time due to oxidation and environmental influences. Premature pavement distresses can occur if these changes are not properly controlled throughout the life of an asphalt mixture. Some specific types of pavement distresses that may be caused by the excessive changes in rheological properties of AC include raveling, cracking, stripping, and rutting. To ensure that these premature distresses are prevented effectively, certain requirements on various asphalt rheological properties have been developed and used in practice.

All 11 asphalt binders (virgin, RAS, RAP, RAS/RAP with RA, and RAS/RAP without RAs) were tested and characterized in accordance with AASHTO PP 6, "Practice for Grading or Verifying the Performance Grade (PG) of an Asphalt Binder," in order to determine the PG of asphalt binders *[48]*. "Performance Graded Asphalt Cement" section (shown in Table 6) of "Louisiana Standard Specifications for Roads and Bridges" were also referenced *[47]*.

Both aged and unaged asphalt binders were tested for the rheological properties. The Rolling Thin Film Oven (RTFO) test was performed in accordance with AASHTO T 240, "Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)," to simulate the binder aging that occurs during asphalt mixture production and construction operations *[49]*. The RTFO simulates short-term aging of asphalt binders during the mixture production in the plant. For simulating the long-term aging of asphalt binders during the service life of asphalt pavements, the Pressure Aging Vessel (PAV) test was conducted in accordance with AASHTO R 28, "Standard Practice for Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV) *[50]."* The Rotational Viscometer (RV) is used to measure the viscosity of liquid asphalt cement at high temperatures to assure pumping and handling during production. This test is conducted in accordance with AASHTO T 316, "Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer," and determines the viscosity of the asphalt cement at 135°C *[51]*.

The dynamic shear rheometer (DSR) is used to measure the asphalt binder properties at high and intermediate service temperatures to determine its resistance to permanent deformation (rutting) and fatigue cracking. The Dynamic Shear Rheometer test was conducted in accordance with AASHTO T 315, "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)," method *[52]*. In addition, the Bending Beam Rheometer (BBR) test is used to measure the asphalt binder properties at low service temperature to determine its resistance to thermal cracking. This test was performed in accordance with AASHTO T 313-06, "Standard Method of Test for Determining the Flexural Creep Stiffness of Asphalt Binder Using the Bending Beam Rheometer (BBR) *[53]."*

Table 6
DOTD performance graded asphalt cement specification

Property	AASHTO Test Method	Specification	
		PG 70-22M	PG52-28
Tests on Original Binder			
Rotational Viscosity @ 135°C, Pa.s	T 316 *[51]*	3.0-	3.0-
Dynamic Shear, 10 rad/s, G*/sinδ, kPa	T 315 *[52]*	1.00+ @ 70°C	1.00+ @ 52°C
Force Ductility, (4°C, 5 cm/min, 30 cm elongation, kg)	T 300 *[54]*	0.23+	N/A
Tests on RTFO Residue			
Dynamic Shear, 10 rad/s, G*/sinδ, kPa	T 315 *[52]*	2.20+ @ 70°C	2.20+ @ 52°C
Elastic Recovery, 25ºC, 10 cm elongation, %	T 301 *[55]*	40+	N/A
% Mass Loss	T 240 *[49]*	1.00-	1.00-
Tests on PAV Residue			
Dynamic Shear, @ 10 rad/s, G*sin δ, kPa	T 315 *[52]*	5000-	5000-
Bending beam Creep Stiffness, S, Mpa	T 313 *[53]*	300-	300-
Bending beam Creep Slope, m value	T 313 *[53]*	0.300+	0.300+

Note: N/A: Not Applicable; "M" designation for elastomer type of polymer modified

In addition, AASHTO TP 70, "Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)" was conducted *[56]*. The MSCR test is typically conducted at the PG high temperature binder temperature. For this study, 67°C was selected as the required test temperature. Also, this test is blind to any modification of the asphalt binder. A benefit of the AASHTO TP 70 is that it eliminates the need to run elastic recovery, toughness and tenacity, and force ductility tests which are procedures designed specifically to indicate polymer modification of asphalt binders. AASHTO TP 101, "Standard Method of Test for Estimating Fatigue Resistance of Asphalt Binders Using the Linear Amplitude Sweep," (LAS) was utilized to determine an asphalt binders' resistance to fatigue damage *[57]*.

The molecular structure of asphalt binders of conventional mixtures, as well as mixtures containing RAS, with and without RAP, and with and without RAs were evaluated with their cracking potential through binder fractionation by molecular weight. The tests utilized were Gel-Permeation Chromatography (GPC) and Fourier Transform Infrared Spectroscopy (FTIR).

Asphalt Binder Extraction from Asphalt Mixtures

Asphalt binders from RAS, RAP, mixtures containing RAS and/or RAP with and without RAs, mixtures containing RAS, RAS and/or RAP, with and without RAs were extracted in accordance AASHTO T 164, "Standard Method of Test for Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt HMA – Method A *[58].*"

Afterwards, the solution of solvent (trichloroethylene) and asphalt binder obtained from AASHTO T 164 – Method A is then distilled to a point where most of the solvent is removed and then carbon dioxide gas is introduced to remove all traces of trichloroethylene *[58]*. This procedure was conducted in accordance with AASHTO R 59, "Standard Practice for Recovery of Asphalt Binder from Solution by Abson Method *[59].*" Figure 2 illustrates the extraction method setup indicating a centrifuge and the Abson Recovery Method.

Figure 2
Centrifuge and Abson test setup

Multiple Stress Creep Recovery (MSCR) Test Procedure

The Multiple Stress Creep Recovery (MSCR) test was conducted in accordance with AASHTO TP 70 to evaluate the effects of extracted asphalt binders from mixtures containing RAP, RAS, RAP and/or RAS, with and without recycling agents as compared to the control mixture containing no RAP, RAS, or recycling agents on rutting resistance *[56]*. The MSCR test method is designed to ascertain the elastic response in an asphalt binder in addition to the change in elastic response under two different stress levels while being subjected to ten cycles of creep stress and recovery. The non-recoverable creep compliance is said to be an indicator of an asphalt binder's resistance to permanent deformation under a repeated load. Virgin asphalt binders utilized in this study were first conditioned in accordance with AASHTO T 240 – "Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test) *[49].*" Recovered asphalt binders were not additionally aged since these materials are considered short-term aged during mixture preparation prior to specimen compaction. The testing temperature for this study was the PG high temperature grade utilized in Louisiana, 67°C. In this test, the DSR is utilized in accordance with AASHTO T 315 – "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)" with a 25-mm parallel plate and a 1-mm gap to employ a haversine load for 1-second followed by a 9-second rest period in each cycle *[52]*. Figure 3 illustrates the MSCR loading and unloading sequence. The asphalt binder specimen is tested in creep at two stress levels. The stress levels utilized in this test are 0.1 kPa and 3.2 kPa respectively. Ten creep and recovery cycles are tested and recorded at each stress level.

Two parameters are utilized to evaluate the asphalt binder performance at high temperatures. The first parameter is the non-recoverable creep compliance (J_{nr}) which normalizes the strain response of the asphalt binder to stress as follows:

$$J_{nr} = \frac{\varepsilon_{nr}}{\sigma} = \frac{non\text{-}recoverable\ strain}{applied\ shear\ stress} \tag{1}$$

where,

J_{nr} = non-recoverable creep compliance (kPa^{-1});

ε_{nr} – non-recoverable strain at the end of the rest period; and

σ = constant stress applied in the creep phase of the test (0.1 kPa and 3.2 kPa).

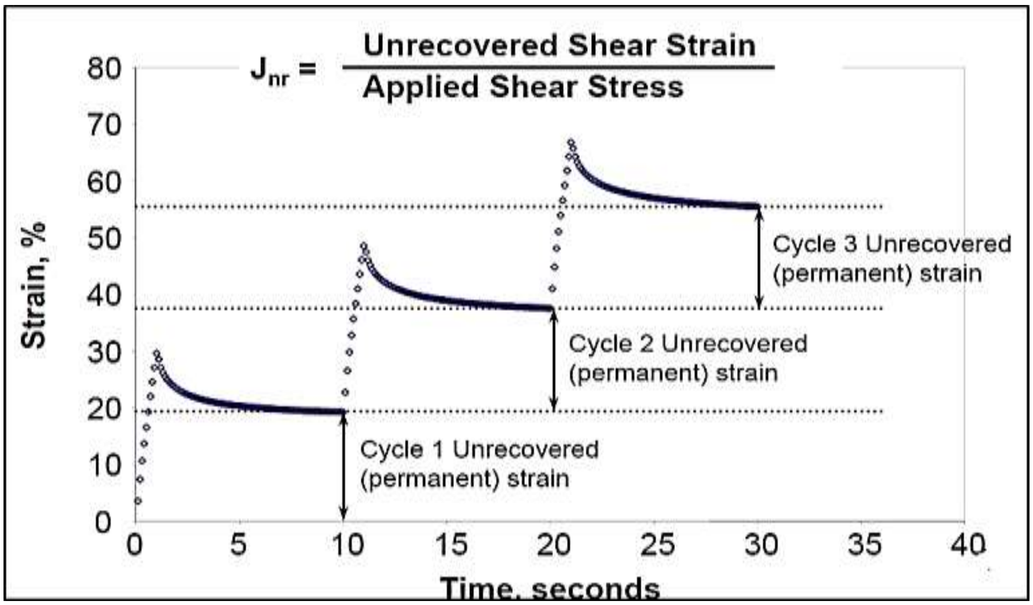


Figure 3
MSCR loading vs. unloading sequence

The second parameter is the percent recovery, which is determined at the end of the recovery period for each applied constant stress. The percent recovery is determined by dividing the difference between the peak strain and the final strain by the peak strain for each individual loading cycle (Figure 4). Mathematically,

$$\text{Percent Recovery} = \frac{\gamma_p - \gamma_u}{\gamma_p} \times 100 = \frac{\gamma_r}{\gamma_p} \times 100 \tag{2}$$

where,

γ_p = peak strain;

γ_r = recovered strain; and

γ_u = un-recovered strain.

To calculate the percent recovery for each cycle and applied stress at the end of the recovery period the following equation is used:

$$\varepsilon_r = \frac{\varepsilon_1 - \varepsilon_{10}}{\varepsilon_1} X100 \tag{3}$$

where,
ε_r = percent recovery;
ε_1 = strain at the end of the creep phase (after 1 second); and
ε_{10} = strain at the end of the recovery phase (after 10 seconds).

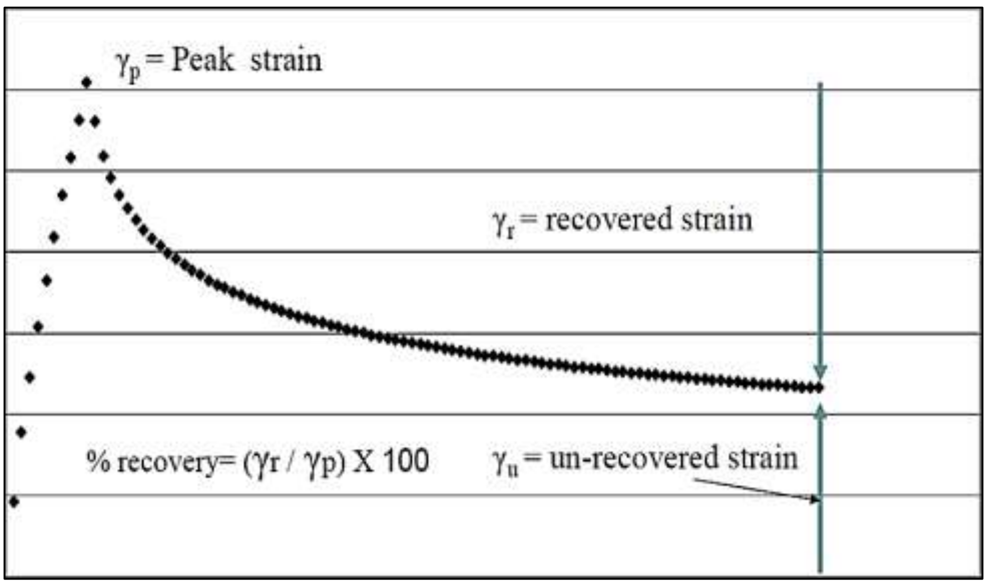


Figure 4
Details of MSCR loading cycle

To determine the percent difference in the non-recoverable creep compliance between 0.1 kPa and 3.2 kPa which represents the stress sensitivity parameter the following calculation is performed:

$$J_{nr-difference} = \frac{J_{nr3.2} - J_{nr0.1}}{J_{nr0.1}} X100 \tag{4}$$

where,
$J_{nr\text{-}difference}$ = percentage difference in non-recoverable creep compliance between 0.1 kPa and 3.2 kPa;

$J_{nr3.2}$ = average non-recoverable creep compliance at 3.2 kPa; and
$J_{nr0.1}$ = average non-recoverable creep compliance at 0.1 kPa.

For acceptable performance, it is desirable to utilize a binder that has a low, non-recoverable creep compliance and a high percentage of recovery. To determine the stress dependency of an asphalt binder, the stress dependency is predicted by calculating the percentage difference in the binder response at the two-applied stress levels (0.1 kPa and 3.2 kPa, respectively) as follows:

$$\varepsilon_{r-difference} = \frac{\varepsilon_{r0.1} - \varepsilon_{r3.2}}{\epsilon_{r0.1}} X100 \tag{5}$$

where,
$\varepsilon_{r\text{-difference}}$ = percentage difference in recovery between 0.1 kPa and 3.2 kPa;
$\varepsilon_{r0.1}$ = percent recovery at 0.1 kPa; and
$\varepsilon_{r3.2}$ = percent recovery at 3.2 kPa.

AASHTO TP 70 introduced the graphical presentation presented in Figure 5 to evaluate the delayed elastic response of the binder at high temperature and it was suggested using the boundary line, defined by the equation $y = 29.37(x)^{-0.263}$ as an indicator of the presence of elastomeric modification *[56]*. The equation was used in this study to evaluate the effects of RAS on the binder rutting performance and on its elastomeric modification.

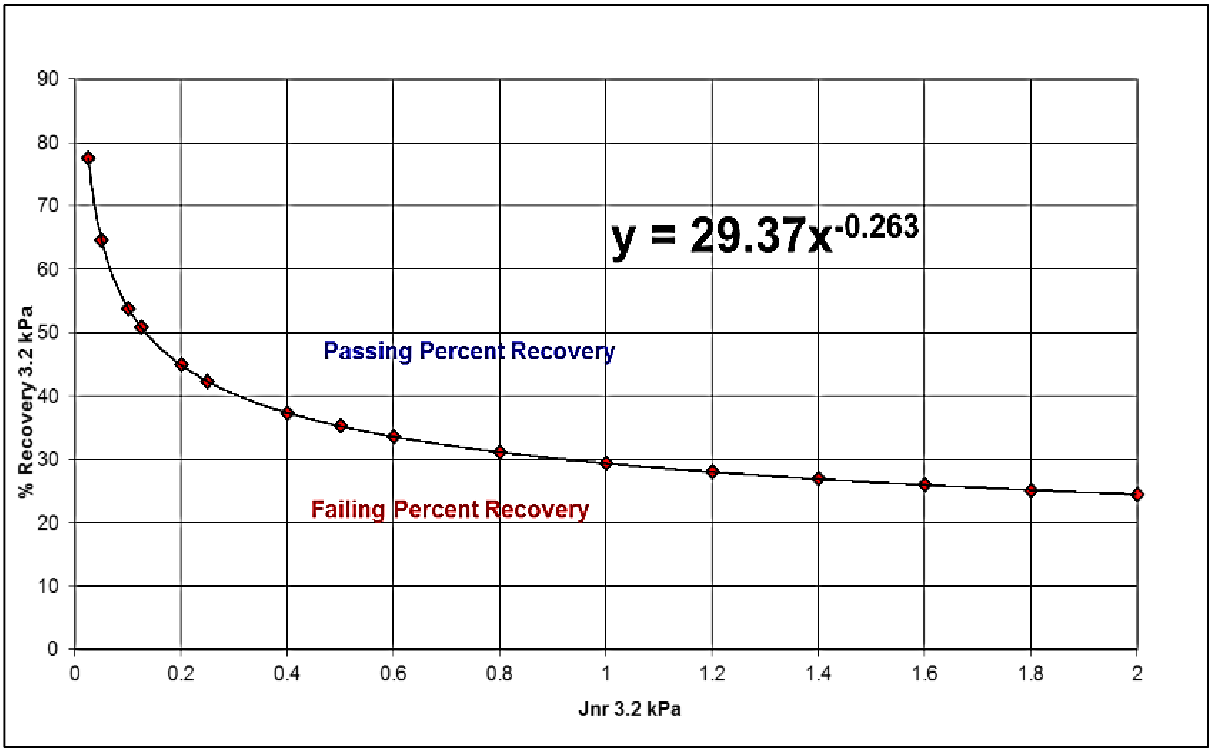


Figure 5
Elastic response curve (AASHTO TP 70)

Linear Amplitude Sweep (LAS)

Currently, asphalt binder fatigue performance is measured by the Superpave Performance Grade System which measures |G*|sin δ at intermediate temperature. The Superpave specification requires that the |G*|sin δ parameter be less than 5000 kPa in order for the binder to show reasonable resistance against fatigue cracking. Deacon et al. reported that the binder loss stiffness, |G*|sin δ, which is utilized to control fatigue cracking may not be adequate especially if the asphalt concrete layer is more than 2 in. in thickness and suggests that an alternate approach be utilized to assure adequate fatigue resistance *[60]*. Bahia et al. reports that the current Superpave specification do not adequately characterize the performance of modified asphalt binders *[61]*. In the National Cooperative Highway Research Program (NCHRP) Report 459 on the characterization of modified asphalt binders in Superpave mix design, Bahia et al. discussed the need for new testing protocols and parameters for predicting the fatigue damage behavior of an asphalt binder *[61]*. Concepts of nonlinear viscoelasticity and energy dissipation were evaluated to develop better asphalt binder parameters that more effectively relate to binder and mixture behavior. Bahia et al. recommended that the current binder specification |G*|sin δ be replaced with the parameter N_f, which is defined as the number of cycles to crack propagation to improve the role of asphalt binders in mixture fatigue. The number of cycles to crack propagation is based on the concept of damage accumulation. An accelerated test method (LAS) based on viscoelastic continuum damage mechanics (VECD) was developed to estimate an asphalt binders resistance to fatigue *[62]*. The LAS test consists of a series of cyclic loads at linearly increasing strain amplitudes at a constant frequency of 10 Hertz. Upon completion of the test, the number of cycles to failure is determined at the peak stress. Hintz et al. stated that the current Superpave fatigue parameter, $|G^*|\sin\delta$, lacks the ability to adequately characterize actual damage because the parameter does not take into account pavement structure or traffic loading since the asphalt binder is subjected to very few loading cycles and the measurement is made at a specific strain level *[63]*. It was also shown that LAS results correlated well with field measurements when comparing the measured fatigue life (N_f) to measured cracking in test pavement sections constructed as part of the LTPP program.

Linear Amplitude Sweep (LAS) Test Procedure

The LAS test uses cyclic loading at linearly increasing load amplitudes to determine an asphalt binder's resistance to fatigue damage. The LAS test was performed in accordance to AASHTO TP 101 *[57]*. Asphalt binders are aged in accordance with AASHTO T 240 – "Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)" for short-term aging and may be further long-term aged in accordance with AAHSTO R 28 *[49]*, *[50]*. An Anton Paar MCR 302 rheometer was used to perform the test at an intermediate temperature of 25°C. Three replicate specimens were tested. Specimens were prepared in accordance with AASHTO T 315 – "Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)" using the 8-mm parallel plate geometry with a 2-mm gap setting *[52]*. Specimens were first tested using a shear frequency sweep to determine its rheological properties. This frequency sweep data was analyzed to determine the damage analysis "alpha" parameter (undamaged material property). The frequency sweep test was performed at a testing temperature of 25°C and an oscillatory shear loading at constant amplitude over a range of frequencies was used. The frequency test applied a load of 0.1±0.01 % strain over a range of frequencies (12 frequencies) from 0.2-30 Hz. Figure 6 indicates a typical frequency sweep test output from test data.

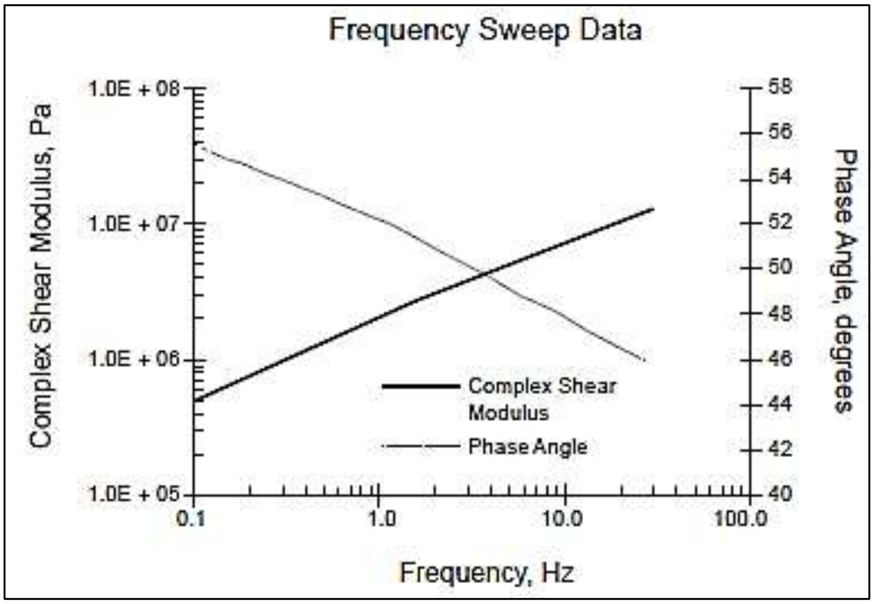


Figure 6
Example output from frequency sweep test (AASHTO TP 101)

After frequency testing, the specimens were tested with an amplitude sweep test in which the specimens were subjected to a series of oscillatory load cycles in strain-controlled mode at a frequency of 10 Hz. The specimens are loaded for 10-s intervals of constant strain amplitude starting from 0.1% to 1% and then increased by each additional 1% up to 30% over the course of 3,100 cycles of loadings as shown in Figure 7. The peak shear strain, peak shear stress, dynamic shear modulus (|G*|, Pa) and phase angle (δ, degrees) were recorded every 10 load cycles. Analysis of results is based on the viscoelastic continuum damage (VECD) approach. Failure damage is based on the peak shear stress as this relates the failure criteria to material response. The binder fatigue parameter N_f is calculated using the following equation:

$$N_f = A(\gamma_0)^{-B} \tag{6}$$

where,

A and B = material property coefficients

γ_0 = applied strain (2.5% for strong pavements and 5.0% for weak pavements) *[63]*

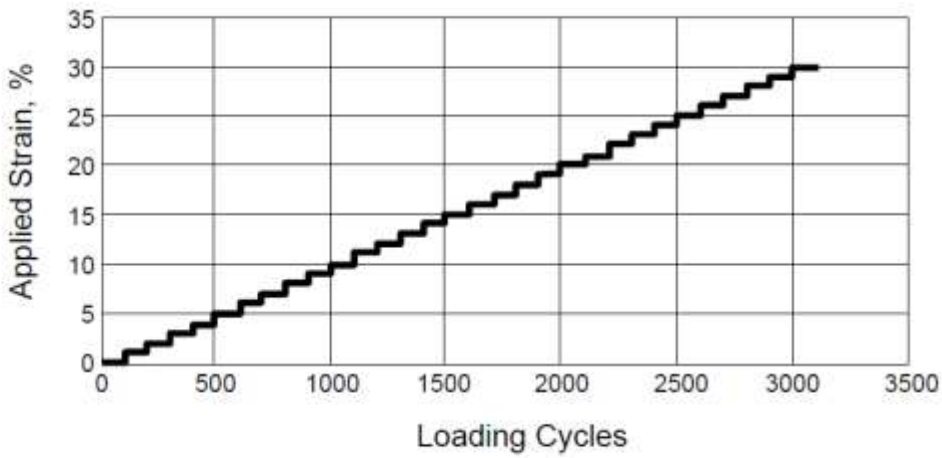


Figure 7
Loading scheme of linear amplitude sweep test (AASHTO TP 101)

Christensen-Anderson (CA) Model

A frequency sweep test using an oscillating parallel plate rheometer, such as the DSR, is commonly conducted as a dynamic mechanical test on asphalt binders. Test temperatures typically range from -35 to 60°C, and a series of loading is applied at varying frequencies ranging from 0.1 to 100 radians/second. A predetermined fixed torque, which induces sinusoidal strain was applied to a specimen and the resulting stress was analyzed as a function of frequency in the dynamic mechanical analysis. This is termed strain controlled testing. Stress controlled testing is where a sinusoidal varying stress is applied and the resulting strain is measured. Christensen and Anderson stated that dynamic mechanical properties are directly related to the creep properties of the asphalt binder. In dynamic testing, the primary response of interest is the complex shear modulus (G*) determined in strain controlled testing. The complex module developed by Christensen and Anderson is commonly referred to as the CA model and was derived on a logistic distribution function to describe the relaxation spectra *[64]*. The CA model describing the complex shear modulus is based on the following formula:

$$G^*(\omega) = G_g[1+(\omega_0 / \omega)^{(\log 2)/R}]^{-R/(\log 2)} \quad (7)$$

where,

$G^*(\omega)$ = complex shear modulus in Pa at a given frequency, ω in radians/second;

G_g = glassy modulus, typically assumed to be 1 GPa;

ω_0 = crossover frequency in radians/second; and

R = rheological index.

Rheological Index (R)

The rheological index (R) as defined by Christensen and Anderson is the difference between the glassy modulus, Gg, and the dynamic complex modulus at the crossover frequency, G(ω0), as seen in Figure 8. Whereas, the cross over frequency, (ω0), is defined as the frequency at a given temperature in which tan δ = 1 and δ is the phase angle. R is a shape factor and is proportional to the width of the relaxation spectrum *[64]*. As the asphalt binder ages, there is an increase in the rheological index. The rheological index was determined using the following formula:

$$R = (\log 2)*\log [G^*(\omega)/Gg]/\log (1-\delta/90) \tag{8}$$

where,

$G^*(\omega)$ = complex dynamic modulus (Pa), at frequency ω (radians/second);

G_g = glassy modulus, 1 GPa; and

δ = phase angle, degrees.

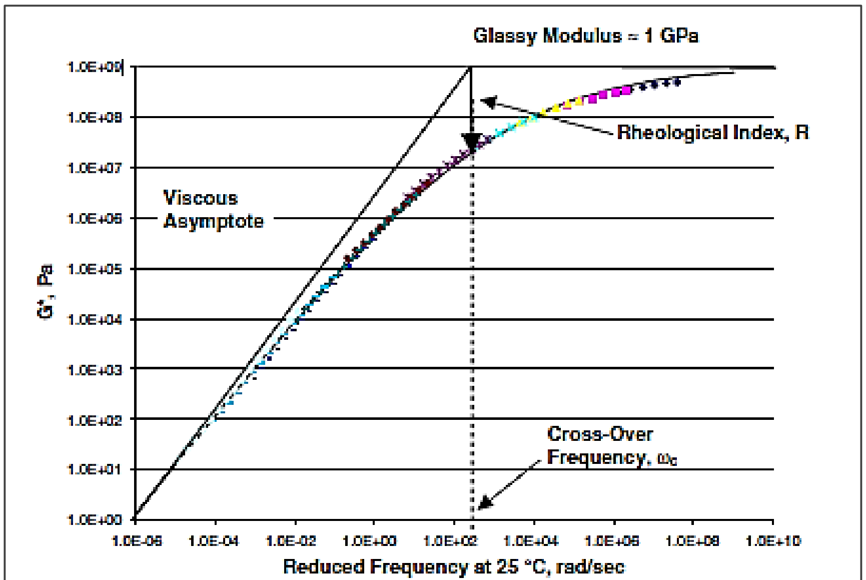


Figure 3-34. Christensen-Anderson Model master curve.

Figure 8
Christensen Anderson model curve (NCHRP Report 709)

Glover-Rowe – Black Space Diagram - ΔTc

Anderson et al. evaluated the use of the Black Space Diagram to evaluate changes in asphalt binder rheology due to aging *[65]*. The Black Space Diagram is a rheological plot of G* and phase angle. The Glover-Rowe fatigue cracking parameter, $G^*(\cos\delta)^2/(\sin\delta)$, was proposed to create a damage curve in black space. Anderson et al. proposed thresholds when non-load associated cracking (block cracking) begins and when there are significant cracking problems. Thresholds have been set at 180 kPa for the onset of cracking, and 450 kPa is significant cracking when tested at 15°C and a loading frequency of 0.005 radians/second. The phenomenon of block cracking is usually associated with cracking that happens as a pavement ages and loses durability. Anderson et al. stated that intermediate temperature research has shown a relationship between ductility and durability. Anderson et al. used ductility as the property that relates to flexibility. Two parameters were chosen that relate well to ductility and the loss of flexibility with aging. The first parameter was the Glover parameter which was modified to the Glover-Rowe fatigue cracking parameter previously mentioned. The second parameter, ΔTc, represents the change in critical temperature and quantifies the difference between the continuous grade temperature for both stiffness and relaxation temperatures as measured by BBR. ΔTc is determined by the following formula:

$$\Delta T_c = T_{c(S)} - T_{c(m)} \tag{9}$$

where,

$T_{c(S)}$ = the temperature where the BBR stiffness at 60 seconds of loading is 300 MPa; and
$T_{c(m)}$ = the temperature where the BBR m-value at 60 seconds of loading is 0.3.

During aging, both the critical temperatures for stiffness and m-value increase; however, the m-value increases at a much greater rate of time. This indicates an asphalt binder's loss of relaxation properties as aging increases. The difference between Tc(m) and Tc(S) become larger as aging time increases. Therefore, the larger the difference between critical temperatures then the greater the loss of relaxation properties of the asphalt binder. When Tc(m) is larger than Tc(S), the ΔTc will be a negative value. This indicates that the material is "m-controlled," otherwise it is "S-controlled." ΔTc is positive. As an asphalt binder ages, it will transform from S-controlled to m-controlled. As the asphalt binder becomes more m-controlled, the ductility of the asphalt binder decreases which adversely effects an asphalt binder's capability to relax under loading.

Binder Fractionation by Molecular Weight

Saturate, Aromatic, Resin, and Asphaltene (SARA) Analysis. The composition of asphalt differs depending on the crude source and the refining process. The components of asphalt have been characterized as saturate, aromatic, resin, and asphaltene (SARA) by extraction techniques to separate the components. Among them, saturates, aromatics, and resins are typically grouped together and called as the maltenes, which are low molecular weight (MW) mixtures (<3000 Daltons) and act as dispersing agents for the higher molecular weight components. The maltenes can be separated by their differences in polarity. The nature of the asphaltenes, the most aromatic of the heaviest components of asphalt, is the major factor controlling the properties of asphalt binders *[66]*. Asphaltenes, as the higher MW component, by virtue of their molecular size are the bodying agent for the maltenes and have a significant influence on asphalt performance *[67]*. The largest "molecules" are assemblies of smaller molecules held together by one or more intermolecular forces. Through changes in the polarity of the solvent used in the analysis, the ability of the samples to undergo self-assembly by different interactive mechanisms has been probed *[68]*. Therefore, by analyzing the asphaltenes in the asphalt cements and polymer-modified asphalt cements, with or without reclaimed asphalt materials (e.g., RAP and RAS), one might correlate physical performances of mixtures containing these materials with the content and MW magnitude of asphaltenes.

Analysis of the asphalt component is facilitated using an Iatroscan instrument, i.e. thin layer chromatography (TLC) of the maltenes absorbed in silica rods and sequential elution with a series of solvents with increasing polarity. The concentration of the components in the elution bands is accessed by burning them from the rod and passing the gases through a Flame Ionization Detector (FID). The heavy metals (typically vanadium and nickel) in the asphaltenes fraction tend to deactivate the silica coating on the chromatographic rods so it is preferable to remove the metals, before spotting the maltenes on the rods. Thus, the asphaltenes fraction is precipitated with n-heptane and removed by filtration for gravimetric determination in accordance with ASTM D-3279, “Standard Test Method for n-Heptane Insolubles.” The test method defines asphaltenes as the insoluble fraction recovered from n-heptane precipitation. The n-heptane soluble fractions (maltenes) can then be separated into three fractions and identified by the increasing polarity of the eluting solvents as saturates, aromatics, and resins *[69]*, *[70]*.

For SARA analysis, the asphalt binders from mixtures containing RAP and/or RAS, with and without RAs were extracted with trichloroethylene according to AASHTO T 164 *[58]*. Extracted binders from mixtures containing trichloroethylene were distilled to a point where most of it was removed and then carbon dioxide gas was introduced to remove all traces of trichloroethylene following the procedure described in AASHTO R 59 *[59]*. Each binder was deasphaltened according to ASTM D-3279 to yield asphaltenes (As) and maltenes, which are dissolved in the n-heptane soluble portion. The maltenes were further fractionated on an Iatroscan TH-10 Hydrocarbon Analyzer to yield the composition in saturates (S), aromatics (Ar), and resins (R). n-Pentane was used to elute the saturates, and a 90/10 toluene/chloroform mixture was used to elute the aromatics. The resins were not eluted and remained at the origin.10

Gel Permeation Chromatography (GPC). The differences in the molecular weights of maltenes and asphaltenes have prompted efforts to separate these components using Gel Permeation Chromatography (GPC). GPC provides a simple separation of molecules in a sample according to their sizes or, more specifically, their hydrodynamic volumes. This molecular size excluding technique can be likened to a sieving process in which the largest materials elute first, followed by successively smaller molecules. The ability of GPC to separate by molecular size rather than by some complex property, such as solubility or absorptivity, is the most desirable advantage of the technique. This feature made GPC especially suited for fractionating complicated mixtures like crude oil residual, asphalt cement, and asphaltenes for almost 50 years *[71], [72], [73]*. GPC very uniquely mirrors the quantitative distribution of all species present in a binder, such as maltenes, asphaltenes, and polymers. The instrument signal, viz., the difference between the refractive indices of the eluting solution containing the asphalt and that of the solvent (ΔRI), is plotted vs. the eluting volume (mL), the molecules of larger size are excluded first, allowing the differentiation of asphalt species on the scale of MW = 106-102 Daltons. A correlation of the eluting volume with the molecular weight of the eluting fraction is achieved using narrow molecular weight standards *[74]*.

Efforts to predict the properties of asphalts using GPC have been reported. Rather than estimate the actual molecular weight the eluting fractions, the GPC chromatograms have been divided into three regions: large molecular size (LMS), medium molecular size (MMS), and small molecular size (SMS). Researchers stated that the LMS and SMS regions are significant with respect to predicting pavement performance *[67], [68], [75], [76]*. Although the arbitrary division of the chromatograms into arbitrary regions, preference is given to calibrate the GPC chromatograms and identify the maltenes, asphaltenes and polymer components on the basis of their molecular weight ranges *[77], [78]*. Using molecular weight regions, it is possible to divide the LMS fraction into ranges which change when the asphalt ages or is modified.

The GPC analysis was performed using an EcoSEC high performance GPC system (HLC-8320GPC) of Tosoh Corporation, equipped with a differential refractive index detector (RI) and UV detector. A set of four microstyragel columns of pore sizes 200 Å, 75 Å (2 columns) and 30 Å from Tosoh Bioscience were used for the analysis. Tetrahydrofuran (THF) at a flow rate of 0.35 mL/ min. was used as the solvent. Columns were calibrated using polystyrene standard mixtures PStQuick B (MW= 5480000, 706000, 96400, 10200, 1000), PStQuick E (MW= 355000, 37900, 5970, 1000), and PStQuick F (MW= 190000, 18100, 2500, 500) from Tosoh Bioscience.

Asphalt samples for GPC analysis were processed according to methodology described by Daly et al., 2015. The neat asphalts were dissolved in Tetrahydrofuran (THF) to a concentration of 0.25-1.00%. The asphalts from mixtures were extracted with THF in closed vials at room temperature (by shaking), left overnight for decantation and filtered using 0.45 micron Teflon filters. The concentration of asphalt solutions was 0.5%. All samples were prepared the previous day and filtered in the day of analysis using 0.45 micron Teflon filters.

Fourier Transform Infrared Spectroscopy (FTIR). Characterization of oxidative asphalt aging with FTIR has been studied extensively in the past few years. The formation of carbonyl (C=O) containing molecules¸ which can be identified in the FTIR spectrum, has been correlated with standard asphalt binder aging techniques, RTFO and PAV *[77]*. It is well established that the main process occurring during this period is the oxidation of asphalt molecules, which then leads to aggregation due to the strongly interacting oxygen containing molecule *[78], [79]*. Since the main process is oxidation, the oxidized species can be used to quantify the amount of aging. FTIR spectra of the aged samples show a peak around 1700 cm^{-1}, which is the characteristic of C=O species. A typical spectrum of an aged asphalt sample is shown in Figure 9, showing the key absorption bands.

In previous investigations related to aging of SBS copolymer modified asphalt binders, Negulescu used FTIR in order to gain a relative understanding of oxidation, which is directly related to asphalt binder aging *[80]*. It was observed that the area of the carbonyl absorbance occurring at 1695 cm^{-1} increased as compared to that of the C-C absorbance occurring at 1455 cm^{-1}. The ratio of the C=O and C-C vibrations gave a relative comparison of how much oxidation is occurring, which is called the carbonyl index. As the carbonyl (C=O) index increased, there was a higher level of oxidation in the asphalt binder and a stiffening of the binder has been observed. Since both MWS and PCWS are highly oxidized materials, it is expected that the carbonyl indices of paving asphalts incorporating MWS and PCWS to be large. A correlation of the C=O index and the size and distribution of asphaltenes given by the maltenes/high end asphaltenes (MW>10K Daltons) ratio might be attempted, to a limited extent, to predict the field performance at intermediate temperatures as reflected by the value of J_c integral.

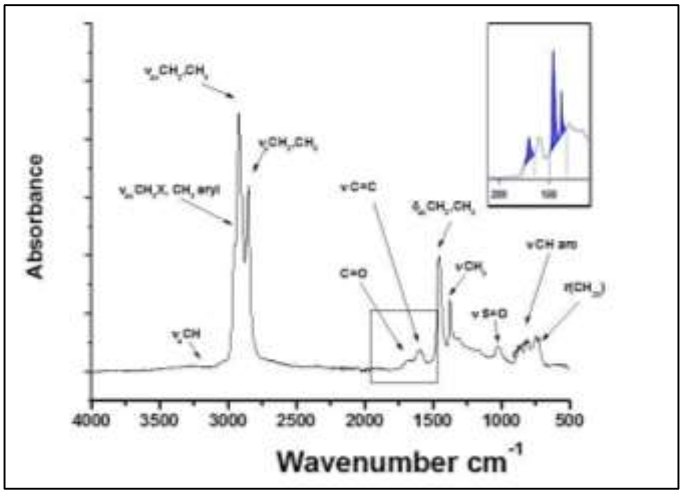


Figure 9
Typical FTIR spectrum of an aged asphalt

The FTIR spectra for the samples were obtained using a Bruker Alpha FT-IR spectrometer (Alpha) using a diamond single reflection attenuated total reflectance (ATR). An OPUS 7.2 data collection program was used for the data analysis. The following settings were used for data collection: 16 scans per sample; spectral resolution 4 cm^{-1}; and wave number range 4000-500 cm^{-1}. Approximately 1% solution of mix samples was made in carbon disulfide (CS2) solvent and filtered using a 0.2 µ filter. A few drops of the solution were kept on the diamond crystal and allowed to evaporate the solvent. Spectrum was collected after the complete evaporation of the solvent. The carbonyl index was calculated from the band areas measured from valley to valley *[81]*. This was done using the OPUS spectroscopy software provided with the Bruker FTIR instrument.

$$Carbonyl\ Index\ (\%) = \frac{Area\ of\ the\ carbonyl\ centered\ around\ 1700\ cm^{-1}}{\sum Area\ of\ the\ spectral\ bands\ between\ 1490\ and\ 1320\ cm^{-1}}\ X100 \qquad (10)$$

Asphalt Mixture Design

Acquisition of Materials

Asphalt binders, RAP, RAS, and RAs were acquired for laboratory evaluation and performance prediction. Sufficient quantities of asphalt cement binder, RAP, and RAS materials from available sources to the State were collected. RAS material was classified as either manufactured waste shingle (MWS) or post-consumer waste shingles (PCWS), and their compositions were characterized by separating individual components using extraction methods. Asphalt binders were acquired from a supplier within the State. RAP materials were obtained from a contractor within the state which had reclaimed the RAP material from an existing State route. Coarse natural sand and gravel aggregates commonly used in the State of Louisiana were also utilized in this study.

RAP, RAS, and Aggregate Characterization

After acquiring the materials necessary for mixture design, the asphalt binder content, specific gravities, and gradation for the collected RAP and RAS materials were determined. In addition, aggregate gradations, specific gravities, and aggregate consensus properties were determined.

Optimum Dosage Rate of Recycling Agents

The optimum dosage rates for the RAs were determined by volumetric and densification criteria aiming 100% utilization of asphalt binder included in the PCWS. It was determined that an average %AC of PCWS was 28.6% (Table 10), which will be discussed in the later section. With 5% PCWS by weight of the asphalt mixture, a 100% contribution of the RAS binder to the mixtures containing RAS is estimated as 1.4% of the total weight of the mix. At 5.3% design AC, it is shown in Table 7 that the virgin AC content of the 70PG5P mixture was reduced to 4.8% as the additional %AC utilized from the recycled RAS without RAs was 0.5%. Thus, aiming 100% utilization of AC from recycled RAS with the three RAs, the virgin AC content was reduced to 3.9% for the subsequent mixtures.

For Cyclogen-L, three dosage rates of 5%, 12%, and 15% were tried, of which 12% was selected as the rate yielded the closest air voids and total %AC (i.e., Virgin plus Recycle) to that of 70PG5P. For Hydrogreen, sole trial dosage of 5% was simply adopted upon the manufacturer's recommendation, and that yielded just perfect air voids and the total %AC. For Asphalt Flux, three dosage rates of 5%, 15%, and 20% were tried, of which 20% was selected as the rate yielded 3.9% of air voids and the exact total %AC of 5.3%.

It is noteworthy that heating the RAS to 163°C prior to incorporating RAs was generally recommended by the manufacturers to achieve 100% contribution of asphalt binder from the recycled RAS. As shown in Table 7, however, Cyclogen-L was not able to yield the contribution of RAS asphalt binders also depends on the dosage rate.

Table 7
Volumetric and densification criteria for selected RA dosage

Volumetrics	70CO	70PG5P	Cyclogen-L			Hydrogreen	Asphalt Flux		
			5%	12%	15%	5%	5%	15%	20%
% G_{mm} at N_{ini}	88.8	88.9	88.2	88.9	88.4	89.4	87.8	88.3	89.5
% G_{mm} at N_{max}	97.0	96.9	95.6	96.5	96.2	96.9	95.4	96.1	97.1

Air Voids %	4.0	4.0	5.3	4.5	4.8	4.0	5.5	4.9	3.9
VMA %	13.3	13.9	14.3	13.6	14.0	13.3	14.6	14.2	13.6
VFA %	70	71	63	67	66	69	62	66	71
Design %AC	5.3	5.3	5.3	5.3	5.3	5.3	5.3	5.3	5.3
%AC (Virgin)	5.3	4.8	3.9	3.9	3.9	3.9	3.9	3.9	3.9
%AC (Recycled)	0.0	0.5	0.9	1.2	1.1	1.4	0.8	1.0	1.4

Shading indicates design level

Asphalt Mixture Preparation

Figure 10 shows the steps followed during the asphalt mixture preparation. For the asphalt mixture preparation, the aggregate blending calculations were first performed to determine the batch weight of each individual aggregate component. According to the determined aggregate batch weights, individual size fractions of aggregates were weighed and placed together in a metal pan, and the pan was placed in a force draft oven at 163°C, i.e., the mixing temperature, until the blended aggregates were thoroughly heated. Approximately one hour prior to mixing of the aggregate with the asphalt binder, the asphalt binder was placed in a force draft oven at 163°C, too. To assure uniform mixing, all mixing equipment were also placed in the force draft oven at 163°C for 30 minutes prior to mixing of aggregate and AC. After all components reached to 163°C, these materials were placed in a mixing bucket. A crater in the center of the blended aggregate was formed for placement of the asphalt binder at the specified batch weight. The mixing operation followed immediately after the asphalt binder component was added to the aggregate to ensure uniform mixing of the materials. After mixing the final asphalt mixture, it was distributed in a flat pan and then placed back in a force draft oven at 163°C for two hours for short term aging before compaction.

Mixing of RAS into the asphalt mixture followed the same protocol as mixing any mixture with reclaimed asphalt pavement (RAP).

Fabrications of Mixture Specimens

Laboratory mix specimens were prepared according to the specific requirements of each individual test. According to the test factorials described, cylindrical samples were fabricated. A typical Superpave gyratory compactor (SGC), as shown in Figure 11, was used to compact all cylindrical specimens as shown in Table 8 with the exception of TSRST specimens.

Prepared RAS and/or RAP

RAS and/or RAP on bottom

Superheated aggregate placed on top of RAS and/or RAP

Aggregate prior to blending

Aggregate and RAS and/or RAP Blended

Steaming occurs during initial mixing. After mixing, back in oven till aggregate blend achieves mixing temperature

Asphalt binder added to prepared aggregate blend

Asphalt binder and aggregate mixed together

Asphalt mixture after mixing

Figure 10
Asphalt mixture preparation procedure

TSRST specimens were compacted with a linear kneading compaction device. After compaction, the final TSRST specimen geometry was achieved by saw cutting to the required test specimen dimension.

Figure 11
Typical Superpave Gyratory Compactor (SGC)

Asphalt Mixture Performance Tests

Laboratory mechanistic performance and material characterization tests were conducted to evaluate the laboratory performance of conventional asphalt mixtures and mixtures containing high RAS content and RAs through their fundamental engineering properties.

Asphalt mixture characterization in terms of low-temperature (thermal cracking), intermediate-temperature (fatigue cracking), and high-temperature (permanent deformation and moisture susceptibility) performance were analyzed and evaluated to determine the effects of RAS and/or RAP, with and without RAs as it related to the conventional asphalt mixture.

Table 8 presents the mixture performance test factorial considered for the asphalt mixtures evaluated in this study. Three fundamental tests, as well as LWT, were conducted to characterize the performance of asphalt mixtures. According to the test factorials described in Table 8, cylindrical samples were fabricated using a Superpave gyratory compactor (SGC) with the exception of the TSRST test. The laboratory prepared TSRST specimens were compacted into a rectangular slab using a linear kneading compactor. After compaction, the required beam specimen geometry for the TSRST were obtained by sawing the rectangular slab to the correct dimensions as detailed in Table 8. The target air void for all specimens prepared in this study was 7 ± ½%. Triplicate samples were used for each test with the exception of the LWT test. A brief description of each test is provided below.

Table 8
Mixture performance tests

Test	Test Protocols	Engineering Properties	Specimen Geometry	Test Temperature
Dynamic Modulus, |E*|	AASHTO T 342 *[82]*	High Temperature: Permanent Deformation and Fatigue Cracking Resistance	150 mm diameter x 100 mm	-10, 4.4, 25, 37.8, and 54.4°C
Semi-circular Bend (SCB)	DOTD TR 330-14 *[83]*	Intermediate Temperature: Fatigue Cracking Resistance	150 mm diameter x 57 mm	25°C
Thermal Stress Restrained Specimen Tensile Strength Test (TSRST)	AASHTO TP 10-93 *[84]*	Low Temperature: Thermal Cracking Resistance	50 ± 5 mm x 50 ± 5 mm x 250 ± 5 mm length	Varies
Loaded Wheel Tracking (LWT)	AASHTO T 324 *[85]*	Rutting Susceptibility and Moisture Resistance	150 mm diameter x 60 mm	50°C, wet condition

Laboratory Performance Tests

Tests were performed to characterize the laboratory performance of mixtures evaluated in this study with respect to resistance to permanent deformation as measured by the Dynamic Modulus. Using the measured Dynamic Modulus and Phase Angles obtained from the laboratory performance test, a rutting factor and a fatigue factor can be developed, which is an indication of an asphalt mixture's ability to resist permanent deformation (i.e., rutting).

Dynamic Modulus, |E*|. The dynamic modulus test is a triaxial compression test, which was conducted in accordance with AASHTO T 342 – "Standard Method of Test for Determining Dynamic Modulus of Hot-Mix Asphalt Concrete Mixtures *[82]*." This test consists of applying a uniaxial sinusoidal (i.e., haversine) compressive stress to an unconfined or confined asphalt mixture cylindrical test specimen, as shown in Figure 12. The stress to strain relationship under a continuous sinusoidal loading for linear viscoelastic materials is defined by a complex number called the "complex modulus" (E*). The absolute value of the complex modulus |E* | is defined as the dynamic modulus. The dynamic modulus is mathematically defined as the maximum (i.e., peak) dynamic stress (σ_o) divided by the peak recoverable strain (ε_o).

$$|E^*| = \frac{\sigma_o}{\epsilon_o} \tag{11}$$

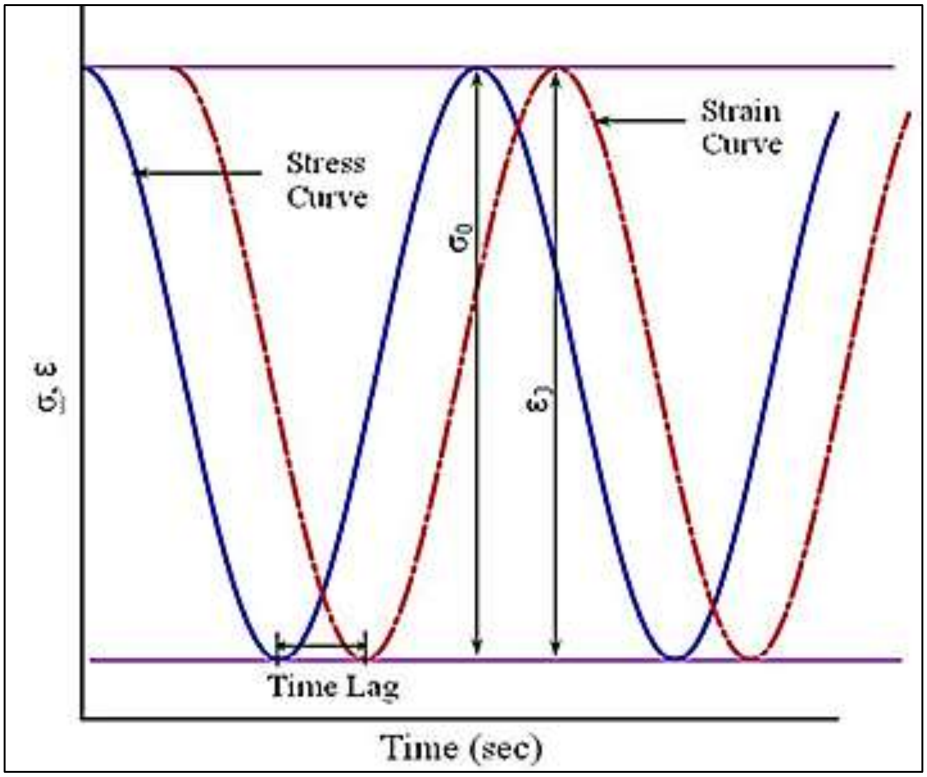


Figure 12
Mixture stress-strain response under sinusoidal load

This test is conducted at five temperatures (i.e., -10, 4.4, 25, 37.8 and 54.4°C) and at six loading frequencies (i.e., 0.1, 0.5, 1.0, 5, 10, 25 Hz) at each temperature *[85]*. This test measures the viscoelastic response of the asphalt mixture. Figure 13 indicates the Dynamic Modulus Test Equipment.

High Temperature Mixture Performance Test

Loaded Wheel Tracking (LWT) (Hamburg Type) Test. This test was conducted in accordance with AASHTO T 324-04, "Standard Method of Test for Hamburg Wheel-Track Testing of Compacted Hot-Mix Asphalt (HMA) *[85]*." In this test, specimens are subjected to a steel wheel weighing 703 N (158 pounds), which repeatedly roll at a speed of 1.1 km/hour and a passing rate of 56 passes/minute across its surface while being submerged in 50°C hot water. The test completion time is predicated upon test specimens being subjected to a maximum of 20,000 cycles or attainment of 20 mm deformation, whichever is reached first. Upon completion of the test, the average rut depth for the samples tested are recorded.

Figure 13
Asphalt mixture performance tester (AMPT) for dynamic modulus

The Hamburg type LWT manufactured by PMW, Inc. of Salina, Kansas was used in this study (Figure 14). The Hamburg LWT can test two specimens simultaneously. The test specimens are subject to two reciprocating solid-steel wheels of 203.5 mm (8 inch) in diameter and 47 mm (1.85 inch) in width while being submerged in hot water at the specified temperature of 50°C, which was utilized in this study. Before testing of the laboratory specimens, they were conditioned by being submerged in hot water for 30 minutes at 50°C. Subsequent to specimen conditioning, a fixed load of 703 N (158 lb.) with a rolling speed of 1.1 km/h (0.68 mi/h) at the rate of 56 passes /min was utilized to induce damage. Each wheel rolls 230 mm (9.1 inch) before reversing direction.

Figure 14
Hamburg loaded wheel tracking device

In order to accurately measure permanent deformation, two Linear Variable Displacement Transducers (LVDT's) were utilized and the subsequent test results (rut depths, number of passes, water bath temperature) were collected and recorded in an automatic data recording system associated with the Hamburg Wheel Tracking Device used in this study. Lower rut depth values are desirable for rut-resistant mixtures. Figure 15 represents a typical Hamburg curve with test parameters.

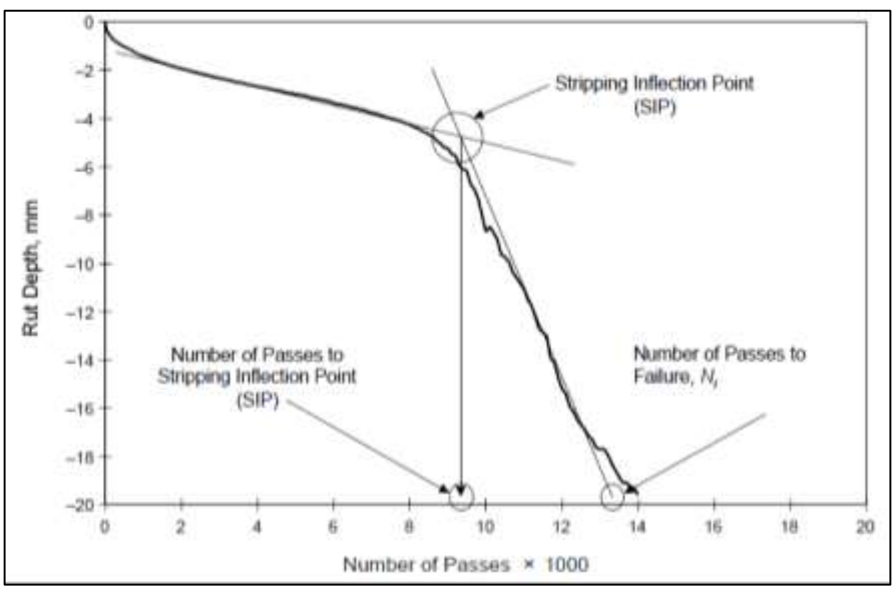


Figure 15
Hamburg curve with test parameters, (AASHTO T 324)

Intermediate Temperature Mixture Performance Test

Semi-circular Bend (SCB) Test. Cracking potential was evaluated using the SCB test procedure (Figure 16), based on fracture mechanics (FM) principles *[83]*, *[86]*. The critical strain energy release rate, also called the critical value of J-integral (J_c), was used to describe the mixture's resistance to fracture.

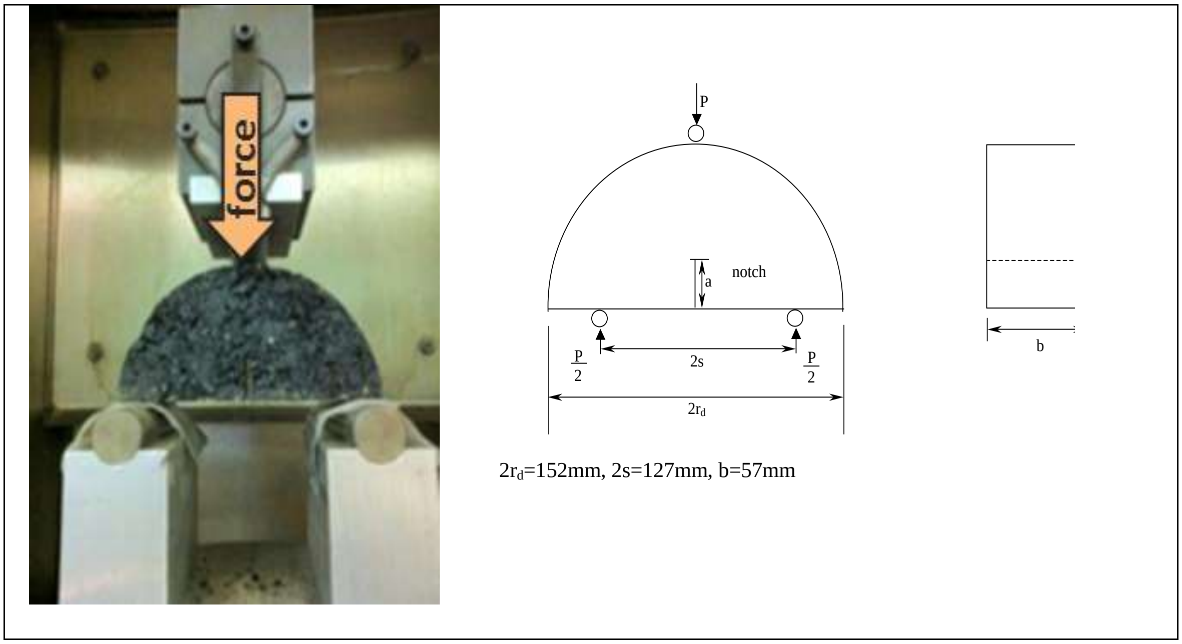


Figure 16
Set-up of semi-circular bending test

$$J_c = -\left(\frac{1}{b}\right)\frac{dU}{da} \tag{12}$$

where,

J_c= critical strain energy release rate (kJ/m^2);

b = sample thickness (m);

a = notch depth (m);

U = strain energy to failure (kJ); and

dU/da = change of strain energy with notch depth (kJ/m).

To determine the critical value of J-integral (J_c) using equation (12), semi-circular specimens with at least two different notch depths should be tested to determine the change of strain energy with notch depth (dU/da). In this study, three notch depths of 25.4 mm, 31.8 mm, and 38 mm were tested to increase the accuracy of slope calculation (dU/da) by fitting a regression line to the change of strain energy with notch depth.

The semi-circular specimen was loaded monotonically until fracture failure under a constant cross-head deformation rate of 0.5 mm/min. in a three-point bending load configuration. The load and deformation were continuously recorded. This test was performed at a temperature of 25°C. The area under the loading portion of the load deflection curves, up to the maximum load, measured for each notch depth, represents the strain energy to failure, U. The average values of U were then plotted vs. the different notch depths to compute a regression line slope, which gives the value of (dU/da). The J_c was computed by dividing dU/da value by the specimen thickness.

Specimens were long-termed aged in accordance with AASHTO R 30 – "Standard Practice for Mixture Conditioning of Hot Mix Asphalt (HMA)" by placing compacted specimens in a forced draft oven for five days at 85°C . Triplicate specimens were utilized for this test. In general, the coefficient of variation was within 15% for the samples tested. High J_c values are desirable for fracture-resistant mixtures.

Low Temperature Mixture Performance Test

Thermal Stress Restrained Specimen Tensile Strength (TSRST) Test. This test was conducted in accordance with AASHTO TP 10 – "Standard Test Method for Thermal Stress Restrained Specimen Tensile Strength *[84]*." The TSRST test can be used in asphalt mixture pavement design and analysis to reduce thermal cracking and to improve life cycle performance. This method determines the tensile strength and temperature at fracture of an asphalt mixture by measuring the tensile load in the asphalt mixture specimen, which is cooled at a constant rate while being constrained from contraction. The data acquired from this test allows the determination of the temperature vs. stress relationship of the asphalt mixture.

Laboratory prepared asphalt mixtures meeting Superpave volumetric and densification criteria were utilized in this test. After blending of the laboratory prepared asphalt mixture, two rectangular asphalt concrete slabs, which are 256.25 mm (10.25 in.) wide by 312.5 mm (12.5 in.) long by 50 mm (2 in.) thick, were compacted using a linear kneading compactor. The rectangular molds were heated to the compaction temperature before charging them with the lab prepared loose asphalt mixture. After compaction, the rectangular slabs were cooled to room temperature and then tested to assure that the percentage of air voids of the compacted asphalt mixture is within 7 ± 0.5%. The required beam specimens for the TSRST were obtained by sawing the rectangular slabs to the correct dimensions of 50 ± 5 mm (2.0 ± 0.2 in.) square and 250 ± 5 mm (10.0 ± 0.2 in.) in length. The prepared beams were then affixed at each ends to platens of the test machine and enclosed in an environmental chamber for conditioning. Afterwards, a tensile load of 50 ± 5 N was applied to the asphalt mixture's beam specimen and the specimen was cooled at a rate of 10.0 ± 1°C per hour until tensile fracture occurs. The thermal contraction along the long axis of the specimen was monitored electronically. The initial length of the asphalt mixture's beam specimen was held constant to the original position. This process was continuous until tensile fracture of the beam specimen occurs. The recorded temperature at tensile fracture was related to the low temperature grade of the Performance Grade asphalt binder. Figure 17 illustrates a typical plot of load vs. temperature for the TSRST results and Figure 18 indicates the TSRST testing setup.

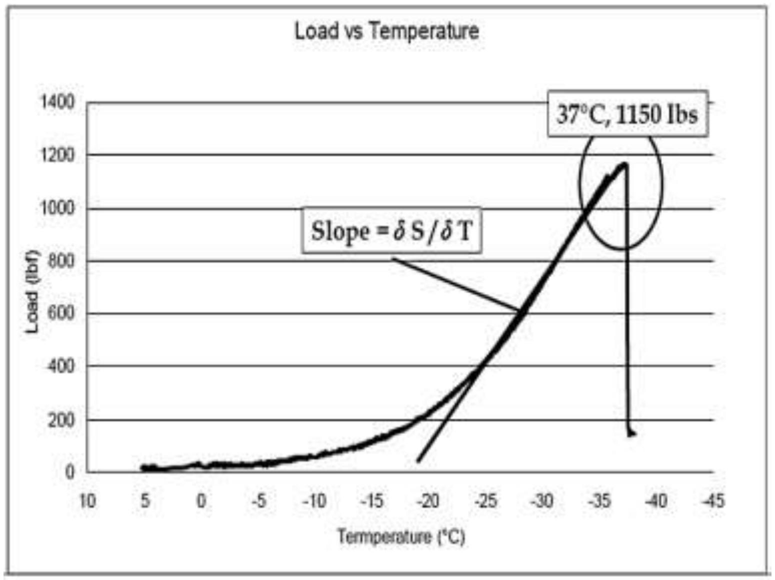


Figure 17
Typical load vs. temperature graph

Figure 18
TSRST test setup and thermally fractured specimen

Statistical Analysis

Statistical analysis of laboratory mechanistic performance and material characterization tests used to evaluate the laboratory performance of conventional asphalt mixtures and mixtures containing high RAP, RAP/RAS content, with and without RAs was conducted through their fundamental engineering properties. A brief description is provided below.

Analysis of Variance (ANOVA)

Laboratory test data was statistically analyzed using the analysis of variance (ANOVA) procedure provided in the Statistical Analysis System (SAS) program from SAS Institute, Inc. A multiple comparison procedure with a risk level of 5% was performed on the means. Tukey's studentized range HSD (honestly significant difference) test was selected as the ANOVA test as it controls the Type I error, and it is the preferred method when making comparisons between large data sets (six or more). The groupings represent the mean for the test results reported by mixture type. The results of the statistical grouping is reported with the letters A, B, C, D, and so forth. The letter A was assigned to the highest mean followed by the other letters in appropriate order. A single letter designation, such as A as compared to a letter B, indicates that there is a significant difference between the means. A double (or more) letter designation, such as A/B (or A/B/C), indicates that in the analysis the difference in the means is not clear-cut, and that the mean is close to either group.

DISCUSSION OF RESULTS

Characterization of Materials

Sufficient quantities of RAP and RAS materials from two sources available to the State were collected. RAS material was classified as either manufactured waste shingle (MWS) or post-consumer waste shingles (PCWS), and their compositions were characterized by separating individual components using extraction methods. Asphalt binder content, effective specific gravities, and gradation for the collected RAS and RAP materials were determined. The percent asphalt binder in PCWS (28.6%) and MWS (25.6%) was obtained by the ignition oven method (AASHTO T308 - Determining the Asphalt Binder Content of Hot Mix Asphalt (HMA) by the Ignition Method) and was verified by extraction method (AASHTO T 164-11 - Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt (HMA)) *[58]*. To determine the aggregate gradation from each source, a washed sieve analysis was performed in accordance with AASHTO T 27, "Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates *[46].*" Table 9 presents the aggregate gradations and specific gravities of the fine and coarse aggregates utilized in this study.

Table 9
Fine and coarse virgin aggregate gradations and specific gravities

Sieve Size Metric (US)	5/8" Gravel	¼ x 0 Gravel	Coarse Sand	Fine Sand
	Average % Passing	Average% Passing	Average % Passing	Average % Passing
19 mm (¾ in)	100	100	100.0	100.0
12.5 (½ in.)	86.7	99.8	100.0	100.0
9.5 mm (⅜ in)	40.2	99.5	97.8	100.0
4.75 mm (No. 4)	11.7	77.2	94.4	99.9
2.36 mm (No. 8)	8.9	48.7	88.4	99.7
1.18 mm (N0. 16)	8.0	30.3	81.5	99.3
0.600 mm (No. 30)	7.4	19.9	71.8	97.2
0.300 mm (No. 50)	6.0	14.9	33.1	80.2
0.150 mm (No. 100)	4.4	8.2	5.4	34.2
0.075 mm (No. 200)	3.5	5.7	2.3	20.5
Specific Gravity				
G_{sb}	2.495	2.492	2.622	2.589
G_{sa}	2.622	2.644	2.656	2.745

Table 10 presents the material properties of RAS (PCWS and MWS), and RAP as it relates to aggregate gradation, asphalt binder content, and specific gravities. It is shown that both the MWS and PCWS had similar gradations. It is also indicated that the ignition oven determined asphalt binder contents were similar to what determined by extractions.

Table 10
Material properties of RAP and RAS

Asphalt Content (%)			
	MWS	PCWS	RAP
Ignition Oven	25.6	28.6	5.5
Extraction (Abson Method)	27.8	28.5	5.3
Fiber Content (%)			
	3.8	2.0	N/A
Aggregate Gradations from Ignition Oven			
Sieve Size Metric (US)	Average %Passing		
	MWS	PCWS	RAP
37.5 mm (1 ½ in,)	100	100	100.0
25 mm (1 in)	100	100	100.0
19 mm (¾ in)	100	100	100.0
12.5 (½ in.)	100	100	98.7
9.5 mm (⅜ in)	100	100	92.1
4.75 mm (No. 4)	99.4	99.6	71.4
2.36 mm (No. 8)	98.7	98.5	55.3
1.18 mm (N0. 16)	82.7	81.6	44.9
0.600 mm (No. 30)	59.7	58.9	37.5
0.300 mm (No. 50)	52.5	52.3	28.2
0.150 mm (No. 100)	44.2	45.4	13.9
0.075 mm (No. 200)	32.7	34.0	10.3
Specific Gravity			
G_{se}	2.553	2.731	2.629

Table 11 presents the aggregate consensus properties: coarse aggregate angularity (CAA), fine aggregate angularity (FAA), flat and elongated (F&E) particle, and sand equivalency (SE) for all virgin aggregates, MWS, and PCWS utilized in this study. Two types of RAs were utilized in this study, rejuvenating agents and softening agents, respectively. The rejuvenators utilized in this study were a naphthenic oil (Cyclogen-L) and a vegetable oil derived from the pyrolysis of the pine tree (Hydrogreen). The softening agents utilized were a PG 52-28 soft asphalt binder and an asphalt flux (PG 28-46). Table 12 indicates the material specifications for the Cyclogen-L and Hydrogreen rejuvenating type recycling agents.

Table 11
Aggregate consensus properties

Property	5/8" Gravel	¼ x 0 Gravel	Coarse Sand	Fine Sand	Manufacturer Waste Shingles	Post-Consumer Waste Shingles
CAA	98	99	—	—	—	—
FAA	—	46	40	44	50	50
F&E	pass	pass	N/A	N/A	N/A	N/A
SE	—	71	93	26	N/A	N/A

Table 12
RA material properties *[87]*

Material Property	Cyclogen-L	Hydrogreen
Viscosity @ 60°C (140°F)	200 – 500 cSt	< 100 cSt
Flash Point (COC), °F	> 400°F	> 425°F
Specific Gravity	0.98 – 1.02	0.92 – 0.95
Appearance	Non-transparent dark green/brown liquid	Clear, dark amber colored liquid

Asphalt Mixture Design

Gravel and coarse natural sand aggregates commonly used in Louisiana were utilized in this study. Post-Consumer Waste Shingles (PCWS) and manufactured waste shingles (MWS) were incorporated into the asphalt mixtures at a dosage rate of 5% by total weight of mix. RAP was added to the asphalt mixtures at a dosage rate of 15% by total weight of mix. The rate of recycling agents added was based on volumetric analysis through various iterations to assure that the maximum benefit of available recycle binder content was achieved. The total amount of recycled binder available for mixtures containing RAS was calculated as the amount of total asphalt recycled binder present in the RAS (PCWS and MWS) multiplied by the amount of RAS added to the mixture by total weight (i.e., 28.6% total PCWS AC* 5% RAS = 1.4% and 25.6% total MWS AC*5% RAS, which results in 1.3%). The total RAP recycle binder available was calculated in the same manner as total RAS recycle binder. The available total RAP recycle binder is (5.5% total RAP AC)*(15% RAP), which results in 0.8%. The asphalt binder available from PCWS, MWS, and RAP was 1.4%, 1.3%, and 0.8%, respectively; see Table 13.

Table 13
Available percent recycled asphalt binder

Recycle Materials	Asphalt Binder Content in Recycle Materials (%)[1]	Dosage of Recycle Material in Mix Design	Recycled Asphalt in the Mixture (%)
PCWS	28.6	5.0	1.4
MWS	25.6	5.0	1.3
RAP	5.5	15.0	0.8

[1] Determined by the ignition oven method, shown in Table 10

The optimum design asphalt content was maintained at 5.3% for all mixtures. Consequently, the recycled binder ratio (RBR) was calculated as the amount of recycled binder available for the mixture divided by the total optimum asphalt binder. During the asphalt mixture design process, the target optimum asphalt binder content was obtained by varying the virgin AC content while utilizing the same composite aggregate blend until volumetric and densification criteria were met. For the Superpave mixtures evaluated in this study, the composite gradation, VMA, and VFA were similar. With the optimum %AC of the mix and the percentage of virgin AC utilized being known, the actual recycle AC contribution in the total binder is calculated for each mixture. In addition, the actual shingle asphalt availability binder factor and the percentage of recycled binder in the total binder content is also determined. Table 14 presents the job mix formulas for asphalt mixture types utilized in this study. It is shown that the Superpave asphalt mixtures evaluated were a 12.5 mm (½ in.) nominal maximum size aggregate. It is noted that the gradation, percent voids filled with asphalt (VFA), and air voids (Va) between mixture types are similar. The asphalt mixtures evaluated are dense-graded and on the fine side of the 0.45-power gradation chart.

Table 15 presents a summary of the various binder contents and ratios used in the research. It is shown in Table 14 that the RBR is 9.4% [(0.5/5.3)*100] for 70PG5P, which contains no recycling agents. For economic benefits, it is important to increase the RBR. However, the increase in RBR could adversely affect the durability performance of asphalt mixtures, intermediate and low-temperature, respectively. Recycling agents were employed to increase the RBR for mixtures containing RAS and/or RAP.

Table 14
Job mix formula

Mixture Designation	70CO	70PG5M	70PG5P	70PG5P5HG	70PG5P12CYCL	52PG5P
Mixture Type	12.5 mm (½”) NMSA Superpave					
% G_{mm} @ N_{ini}	88.8	89.0	88.9	89.4	88.9	88.2
% G_{mm} @ N_{max}	97.0	97.1	96.9	96.9	96.5	96.0
Va (Air Voids), %	4.0	4.0	4.0	4.0	4.5	3.9
VMA %	13.3	13.8	13.9	13.3	13.6	13.3
VFA %	70	71	71	69	67	70
Total %AC	5.3	5.3	5.3	5.3	5.3	5.3
%AC (Virgin)	5.3	4.7	4.8	3.9	3.9	4.6
%AC from recycle	0.0	0.6	0.5	1.4	1.2	0.7
Metric (U.S.) Sieve	Gradation					
19 mm (¾ in)	100	100	100	100	100	100
12.5 mm (½ in)	97	96	97	97	97	97
9.5 mm (⅜ in)	85	84	86	85	86	86
4.75 mm (No.4)	63	62	64	63	63	63
2.36 mm (No.8)	44	44	45	45	45	45
1.18 mm (No.16)	32	31	32	32	32	32
0.600 mm (No.30)	24	23	24	24	24	24
0.300 mm (No.50)	17	16	17	16	16	16
0.150 mm (No.100)	8	9	9	9	9	9
0.075 mm (No.200)	5.3	5.3	5.2	5.2	5.2	5.2
Actual Recycled Binder Availability Factor, %	0.0	46.2	35.7	100.0	85.7	50.0
Recycle Binder Ratio (RBR), %	0.0	11.3	9.4	26.4	22.6	13.2
P_s, %	94.7	94.7	94.7	94.7	94.9	94.7
G_{sb}	2.522	2.506	2.514	2.514	2.514	2.514
G_{se}	2.582	2.561	2.568	2.598	2.585	2.573
G_{mm}	2.391	2.374	2.380	2.400	2.400	2.391
P_{ba}, %	0.9	0.9	0.9	1.3	1.1	0.9
P_{be}, %	4.4	4.5	4.5	4.0	4.0	4.4
D/P_{be}	1.2	1.2	1.1	1.3	1.3	1.2

Mixture Designation	70PG5P20FLUX	70PG15RAP	70PG5P15RAP	70PG5PHG15RAP	52PG5P15RAP
Mixture Type	12.5” NMSA Superpave				
% G_{mm} @ N_{ini}	89.0	89.0	88.9	89.0	88.9
% G_{mm} @ N_{max}	97.3	97.0	96.9	96.8	97.2
Va (Air Voids), %	3.9	4.0	4.1	4.0	3.9
VMA %	13.6	13.2	13.6	13.7	13.8
VFA %	71	70	70	71	71
Total %AC	5.3	5.3	5.3	5.3	5.3
%AC (Virgin)	3.9	4.5	4.1	3.1	3.5
%AC from RAS and/or RAP	1.4	0.8	RAS 0.4 RAP 0.8	RAS 1.4 RAP 0.8	RAS 1.0 RAP 0.8
Metric (U.S.) Sieve	Gradation				
19 mm (¾ in)	100	100	100	100	100
12.5 mm (½ in)	97	96	97	97	97
9.5 mm (⅜ in)	86	83	85	85	85
4.75 mm (No.4)	64	61	63	63	63
2.36 mm (No.8)	45	44	42	42	42
1.18 mm (No.16)	32	32	32	32	32
0.600 mm (No.30)	24	25	24	24	24
0.300 mm (No.50)	17	17	17	17	17
0.150 mm (No.100)	9	8	9	9	9
0.075 mm (No.200)	5.2	5.4	5.4	5.4	5.4
Actual Recycled Binder Availability Factor, %	100.0	100.0	54.6	100.0	81.8
Recycle Binder Ratio (RBR), %	26.4	15.1	22.6	41.5	34.0
P_s, %	94.7	94.7	94.7	94.7	94.7
G_{sb}	2.514	2.525	2.528	2.528	2.528
G_{se}	2.571	2.579	2.562	2.594	2.581
G_{mm}	2.382	2.389	2.375	2.400	2.390
P_{ba}, %	0.9	0.9	0.5	1.0	0.8
P_{be}, %	4.4	4.5	4.8	4.3	4.5
D/P_{be}	1.2	1.2	1.1	1.3	1.2

Table 15 indicates that the RBR generally increased with the use of recycling agents. It is noted that the increase in the RBR was only achieved after a new mixture design method was developed and utilized for the mixtures evaluated, as shown in Figure 19.

Table 15
Percent RAS and/or RAP asphalt binder availability

<table>
<tr><th rowspan="2">Mixture ID</th><th rowspan="2">% Optimum Design AC</th><th colspan="3">Recycled Binder Contribution (%)</th><th rowspan="2">Virgin Binder Content (%)</th><th rowspan="2">Virgin Binder Ratio (%)</th><th rowspan="2" colspan="2">Actual Recycled Binder Availability Factor (%)</th><th rowspan="2" colspan="2">Recycled Binder Ratio, (%)</th></tr>
<tr><th colspan="2">Available</th><th>Actual</th></tr>
<tr><td>70CO</td><td>5.3</td><td colspan="2">0.0</td><td>0.0</td><td>5.3</td><td>100.0</td><td colspan="2">0.0</td><td colspan="2">0.0</td></tr>
<tr><td>70PG5M</td><td>5.3</td><td colspan="2">1.3</td><td>0.6</td><td>4.7</td><td>88.7</td><td colspan="2">46.2</td><td colspan="2">11.3</td></tr>
<tr><td>70PG5P</td><td>5.3</td><td colspan="2">1.4</td><td>0.5</td><td>4.8</td><td>90.6</td><td colspan="2">35.7</td><td colspan="2">9.4</td></tr>
<tr><td>70PG5P5HG</td><td>5.3</td><td colspan="2">1.4</td><td>1.4</td><td>3.9</td><td>73.6</td><td colspan="2">100.0</td><td colspan="2">26.4</td></tr>
<tr><td>70PG5P12CYCL</td><td>5.3</td><td colspan="2">1.4</td><td>1.2</td><td>3.9</td><td>77.4</td><td colspan="2">85.7</td><td colspan="2">22.6</td></tr>
<tr><td>52PG5P</td><td>5.3</td><td colspan="2">1.4</td><td>0.7</td><td>4.6</td><td>86.8</td><td colspan="2">50.0</td><td colspan="2">13.2</td></tr>
<tr><td>70PG5P20FLUX</td><td>5.3</td><td colspan="2">1.4</td><td>1.4</td><td>3.9</td><td>73.6</td><td colspan="2">100</td><td colspan="2">26.4</td></tr>
<tr><td>70PG15RAP</td><td>5.3</td><td colspan="2">0.8</td><td>0.8</td><td>4.5</td><td>84.9</td><td colspan="2">100</td><td colspan="2">15.1</td></tr>
<tr><td>70PG5P15RAP</td><td>5.3</td><td>RAS
1.4</td><td>RAP
0.8</td><td>1.2</td><td>4.1</td><td>77.4</td><td>RAS
18.2</td><td>RAP
36.4</td><td>RAS
7.5</td><td>RAP
15.1</td></tr>
<tr><td>70PG5PHG15RAP</td><td>5.3</td><td>RAS
1.4</td><td>RAP
0.8</td><td>2.2</td><td>3.1</td><td>58.5</td><td>RAS
63.6</td><td>RAP
36.4</td><td>RAS
26.4</td><td>RAP
15.1</td></tr>
<tr><td>52PG5P15RAP</td><td>5.3</td><td>RAS
1.4</td><td>RAP
0.8</td><td>1.8</td><td>3.5</td><td>66.0</td><td>RAS
45.4</td><td>RAP
36.4</td><td>RAS
18.9</td><td>RAP
15.1</td></tr>
</table>

Cooper et al. reported that only a portion of the recycled binder from RAS is activated and the remaining RAS acted as a black rock *[1]*. To address this issue, a modified mixture design change was developed. Figure 19 presents the mixture design flow chart that outlines the procedural steps that were developed to assure that 100% of the available recycle binder was utilized within the asphalt mixture. In order to properly evaluate the asphalt mixture properties with the extracted asphalt binder's rheological properties and binder fractionation, the amount of actual recycle binder activated within the asphalt mixture would need to be 100%. This is necessary because during the asphalt binder extraction procedure, 100% of all binders would be extracted regardless of the actual percentage of the recycled binder activated during the asphalt mixture design. The flow chart has a series of questions that leads the asphalt mixture designer to the appropriated design procedure.

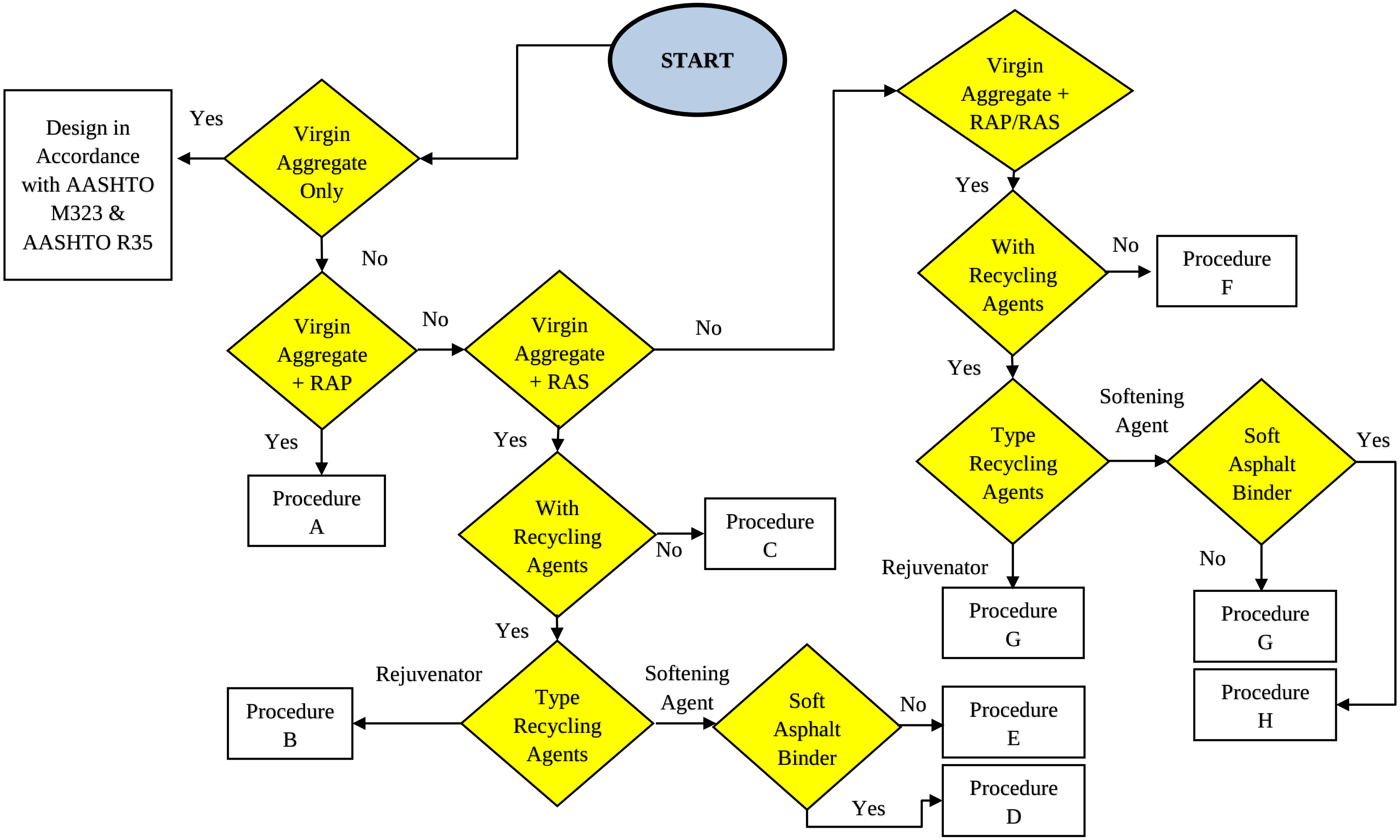


Figure 19
Mix design flow chart

(Figure 19 continued)

Procedure A

- Determine specific gravities, gradations, RAP % asphalt content, consensus properties for virgin aggregate and RAP;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAP;
- Mix superheated virgin aggregate and RAP together resulting in steaming.
- Continue mixing until steam seizes.
- Place blended aggregate and RAP into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Procedure B

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Heat RAS to 163°C, then mix with rejuvenator in heated mixing bowl. After mixing put RAS back in oven at 163°C for 30 minutes;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate and RAS into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

(Figure 19 continued)

Procedure C

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place room temperature RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Procedure D

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place room temperature RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend soft asphalt binder (softening agent) and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

(Figure 19 continued)

Procedure E

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Heat RAS to 163°C, then mix with softening agent in heated mixing bowl. After mixing put RAS back in oven at 163°C for 30 minutes;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate and RAS into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40], [87]*

Procedure F

- Determine specific gravities, gradations, RAP & RAS % asphalt content, consensus properties for virgin aggregate and RAP & RAS;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket, then the RAS (at room temperature) on top of the RAP and then superheated virgin aggregate place on top of the RAP and RAS;
- Mix superheated virgin aggregate and RAP & RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40], [87]*

(Figure 19 continued)

Procedure G

↓

- Determine specific gravities, gradations, RAP & RAS % asphalt content, consensus properties for virgin aggregate and RAP and RAS;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Heat RAS to 163°C, then mix with RAs in heated mixing bowl. After mixing put RAS back in oven at 163°C for 30 minutes;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket, then the RAS on top of the RAP and then superheated virgin aggregate place on top of the RAP and RAS;
- Mix superheated virgin aggregate and RAP & RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Procedure H

↓

- Determine specific gravities, gradations, RAP & RAS % asphalt content, consensus properties for virgin aggregate and RAP & RAS;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket, then the RAS (at room temperature) on top of the RAP and then superheated virgin aggregate place on top of the RAP and RAS;
- Mix superheated virgin aggregate and RAP & RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend soft asphalt binder (softening agent) and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Asphalt Binder Test Results

The asphalt binder's rheological properties have an effect on the performance of the asphalt mixture pavement. Changes in the asphalt binders' rheological properties due to production and aging, which result from oxidation and environmental influences, must be addressed to reduce asphalt binder related pavement distresses such as raveling, cracking, stripping, and rutting. It is essential that the asphalt binders are tested to assure that the binder rheology meets specified criteria necessary to reduce pavement distresses due to changes of its rheological properties as a result of aging. Therefore, asphalt binders and extracted asphalt binder from mixtures were conducted to characterize an asphalt binder's rheology, which is necessary to minimize the ACs contribution to durability, high temperature (permanent deformation), intermediate temperature (fatigue) cracking, and low temperature (thermal) cracking performance.

Asphalt Binder Performance Grading

The binder from compacted mixtures was extracted and recovered according to AASHTO T319, "Standard Method of Test for Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixtures" using trichloroethylene (TCE) as the solvent agent *[89]*. All of the binders presented in this study were graded according to AASHTO R29, "Grading or Verifying the Performance Grade of an Asphalt Binder" and AASHTO M320, "Standard Specification for Performance-Graded Asphalt Binder *[48]*, *[90]*." The actual PG grading is presented in Table 16. As shown in Table 16, the extracted binders were stiffer than their virgin counterparts. Comparison between the extracted control mixture (no RAS and/or RAP) (70CO) and the neat binder (PG70-22) show this trend. This can be explained by the short-term aging effects of mix/compaction heating and extraction heating. Binders extracted from the 70PG5PHG15RAP mixture showed the highest stiffness which corresponds with this mixture having the highest RBR, 41.5%. Comparing mixture 70PG5P with 70PG5P5HG, Table 16 indicates that the use of Hydrogreen adversely affected both the high temperature and low temperature PG properties. The low temperature PG performance was also adversely affected from the use of asphalt flux as shown in the comparison of extracted asphalt binder from mixture 70PG5P20FLUX. Whereas, binders from the 70PG5P and 70PG5P12CYCL had similar results. In comparison with the extracted asphalt cement binders from 52PG5P and 70PG5P, Table 16 indicated an improvement in low temperature properties from the use of soft asphalt binder. From Table 16, it is indicated that the use of 15% RAP with no RAs (70PG15RAP) as compared to the 70CO extracted asphalt binder showed no effect to the high temperature and low temperature grade. However, when 5% PCWS is included in the design (70PG5P15RAP), the extracted binder results as compared to the extracted 70PG15RAP asphalt binder showed a significant increase (4-PG grades) in the high temperature properties and decreases the low temperature property by 2-PG grades. It is shown that the use of Hydrogreen did not improve the high temperature and low temperature properties of the extracted asphalt binders utilizing PCWS and RAP. It is noted that the PCWS continuous PG Grading and designated PG grading is not shown in Table 16. An attempt was made to characterize the extracted binders from the PCWS however due to limitations of the DSR high temperature range (120°C) this was not possible. The extracted PCWS asphalt binder far exceeded the limitations of the DSR. Also the low-temperature capabilities of the bending beam rheometer (BBR) could not properly grade the post-consumer waste shingles.

Table 16
Asphalt binder PG characterization

	PG70-22	PG76-22	Asphalt Flux	RAP	70CO
Continuous PG Grading	73.5 - 22.9	77.7 - 22.7	32.3-46.6	99.3 - 16.2	80.7 - 22.0
PG Grading	70 -22	76 - 22	28 - 46	94 - 16	76 - 22
	70PG5P	70PG5M	70PG5P5HG	70PG5P12CYCL	70PG5P20FLUX
Continuous PG Grading	85.1 - 19.3	90.7 - 16.7	91.4 - 15.0	82.2 - 21.6	86.1 - 11.6
PG Grading	82 - 16	88 - 16	88 - 10	82 - 16	82 - 10
	70PG15RAP	70PG5P15RAP	70PG5PHG15RAP	52PG5P	52PG5P15RAP
Continuous PG Grading	80.7 - 22.0	104.1 -12.9	105.1 - 1.0	68.3 - 24.2	74.1 - 22.0
PG Grading	76 - 22	100 - 10	100 - 0	64 - 22	70 - 22

Rotational Viscosity

This test was conducted in accordance with AASHTO T 316-06, “Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer,” for determining the viscosity of the asphalt binder at 135°C *[51]*. The test purpose of the Rotational Viscometer (RV) was to measure the binder properties at high construction temperatures to assure pumping and mixing during production. In addition, an asphalt binder’s viscosity can also influence workability, which is the ability of the asphalt mixtures being placed and compacted with reasonable effort. Figure 20 indicates that the viscosity of the asphalt binders with the addition of RAP and/or RAS, with and without RAs generally had higher viscosities than the original virgin binder (PG70-22M). The asphalt mixture designated as 52PG5P had a lower viscosity than PG70-22M. When comparing the extracted asphalt binder viscosities of the mixtures evaluated to the conventional mixture, 70CO, extracted asphalt binder it is shown that all mixtures had higher viscosities with the exception of the two asphalt mixtures utilizing the soft asphalt binder, 52PG5P and 52PG5P15RAP respectively. Figure 20 illustrates that the 70PG5PHG15RAP asphalt mixture resulting rotational viscosity was unmeasurable (too stiff at 135°C) because of equipment limitations. A display of "EEEEE" on the digital viscometer appeared when testing this material. This display means the reading is over-range and the percent torque readings exceeded 100%. For the extracted asphalt binder for mixture 70PG5PHG15RAP, this error occurred at an approximate viscosity of 11 Pa·s. It is shown that the extracted binders from 70PG5M and 70PG5P5HG had the highest measured rotational viscosity, 4.04 Pa·s and 3.99 Pa·s, respectively.

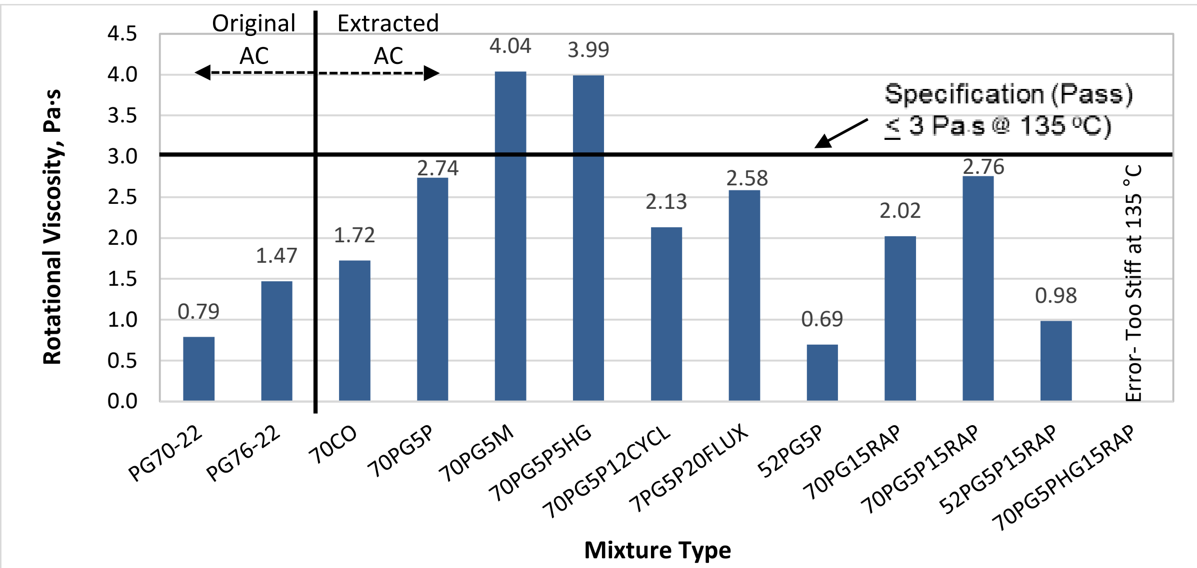


Figure 20
Viscosity of asphalt binders used in various types of mixtures

Linear Amplitude Sweep Test Results

This test was performed in accordance with AASHTO TP 101 at a testing temperature of 25°C *[57]*. The purpose of the LAS test is to evaluate an asphalt binder's ability to resist fatigue damage under cyclic loading by increasing the strain amplitudes to accelerate damage. The rate of damage accumulation is used to indicate fatigue performance. The extracted asphalt binders from the asphalt mixtures were considered as short-term aged since the asphalt mixtures were short-term aged during blending. The extracted binders were then long-term aged in accordance with AASHTO R 28 *[50]*. The extracted asphalt binders were then tested under PAV conditions. In this test, the greater the number of cycles to failure indicates an asphalt binder's resistance to fatigue damage.

Table 17 indicates N_f, the number of cycles to failure, from the LAS test. It is shown that there are two applied strains, 2.5% and 5.0%. These strain levels were chosen because the 2.5% applied strain is for strong pavements and 5.0% applied strain for weak pavements according to Hintz et al. *[63]*. It is shown that the extracted asphalt binder from mixture 70PG5PHG15RAP has the lowest N_f at both strain levels, which corresponds to an observation that the 70PG5PHG15RAP mixture has the highest RBR of 41.5%. Also, it is presented that both extracted asphalt binders from mixtures containing Hydrogreen (70PG5PHG15RAP and 70PG5P5HG) had the lowest N_f at both strain levels. In comparing the extracted asphalt binders from the 70CO mixture to the extracted binders from 70PG5P and 70PG5M, it is shown that both extracted asphalt binders utilizing MWS and PCWS had higher number of cycles to failure than the control at 2.5% strain level. It is indicated in Table 17 that the extracted asphalt binder from 70PG5M had a higher N_f than did the 70PG5P extracted binder. This would be expected since the PCWS are more aged due to oxidation than MWS. Table 17 presents that the use of recycling agents (Cyclogen-L, PG52-28 soft asphalt binder, and asphalt flux) generally increased the number of cycles to failure with the exception of Hydrogreen.

Table 17
LAS number of cycles to failure

	N_f, Number of Cycles to Failure	
Mixture Type	2.5% Applied Strain	5.0% Applied Strain
70PG5PHG15RAP	24733	223
70PG5P5HG	43936	1015
70CO	65481	3435
70PG5P15RAP	67121	1338
70PG15RAP	74975	3427
70PG5P	78762	2695
70PG5M	82790	2763
70PG5P12CYCL	106213	5016
52PG5P	106213	6934
52PG5P15RAP	112113	4924
70PG5P20FLUX	117114	3074

Christensen-Anderson Model, Glover-Rowe Parameter, and ΔTc Test Results

Table 18 presents the critical stiffness and m-value temperatures as measured by the bending beam rheometer, BBR. The critical temperature is the temperature at which the stiffness and the m-value meets specification of 300 MPa and 0.300 respectively. It is noted that these critical temperatures are independent of each other. As the aging takes place, in general, the m-value of asphalt binders changes at a much faster rate than the stiffness does. At a point during the aging, the material will change from S-controlled (stiffness controlled) to m-controlled. Also shown in Table 18 is the ΔTc, which indicates the difference between the critical stiffness temperature and the m-value critical temperature. Asphalt binders that are m-controlled have negative values, while S-controlled have positive values. All of the asphalt binders evaluated in this study clearly show that they are m-controlled, which would show the aging effects they were subjected to during the evaluations. The range of ΔTc was from -3.5 to -26.3°C. The extracted binder from the mixture 70PG5PHG15RAP had the largest difference as shown by ΔTc, followed by the 70PG5P20FLUX. The significance in this test that as the ΔTc increases oxidation increases which results in loss of mixture durability.

Table 18
ΔT_c from bending beam rheometer test results

	BBR				
	Stiffness, S		Relaxation Rate, m		
	Stiffness, MPa	$T_{c(S)}$, (°C)	m-value	$T_{c(m)}$, (°C)	ΔT_c (°C)
70CO	300	-25.5	0.300	-22.0	-3.5
70PG15RAP	300	-30.3	0.300	-22.0	-8.3
70PG5P12CYCL	300	-30.4	0.300	-21.6	-8.8
70PG5P	300	-29.7	0.300	-19.3	-10.4
70PG5P5HG	300	-25.7	0.300	-15.0	-10.7
70PG5M	300	-28.2	0.300	-16.7	-11.5
70PG5P15RAP	300	-28.2	0.300	-12.9	-15.3
52PG5P15RAP	300	-37.5	0.300	-22.0	-15.5
52PG5P	300	-44.0	0.300	-24.2	-19.8
70PG5P20FLUX	300	-35.6	0.300	-11.6	-24.0
70PG5PHG15RAP	300	-27.3	0.300	-1.0	-26.3

Figure 21 presents a comparison chart of the Glover-Rowe intermediate temperature cracking criteria and the conventional Superpave (i.e., $G^*\sin\delta$) criterion at the intermediate temperature of 25°C. This figure is a damage curve in black space where the complex shear modulus, G^*, at 25°C is plotted on the ordinate axis and the corresponding phase angle is plotted on the abscissa axis. Two Glover-Rowe parameter thresholds were proposed by Anderson et al.; one where non-load associated cracking begins and another when the cracking becomes significant *[65]*. These thresholds are respectively set at 180 kPa for the onset of cracking and 450 kPa for significant cracking, respectively. A total of 22 G* and phase angle (δ) data pairs of 11 asphalt binders measured by DSR tests at intermediate temperatures are plotted on this black space diagram. According to the data presented in Figure 21, 8 samples failed the G*sinδ criterion, while 12 samples failed the Glover-Rowe significant cracking criterion and 16 samples failed the cracking on-set criterion, respectively. In other words, 36.4, 54.5, and 72.7% of samples failed G* sinδ, Glover-Rowe significant cracking, and Glover-Rowe cracking on-set criteria, respectively. The observed disagreements between the conventional G*sinδ criterion and the two Glover-Rowe criteria may be in-line with a common belief that the G*sinδ parameter does not adequately capture the asphalt binder's susceptibility to the intermediate temperature cracking *[65]*.

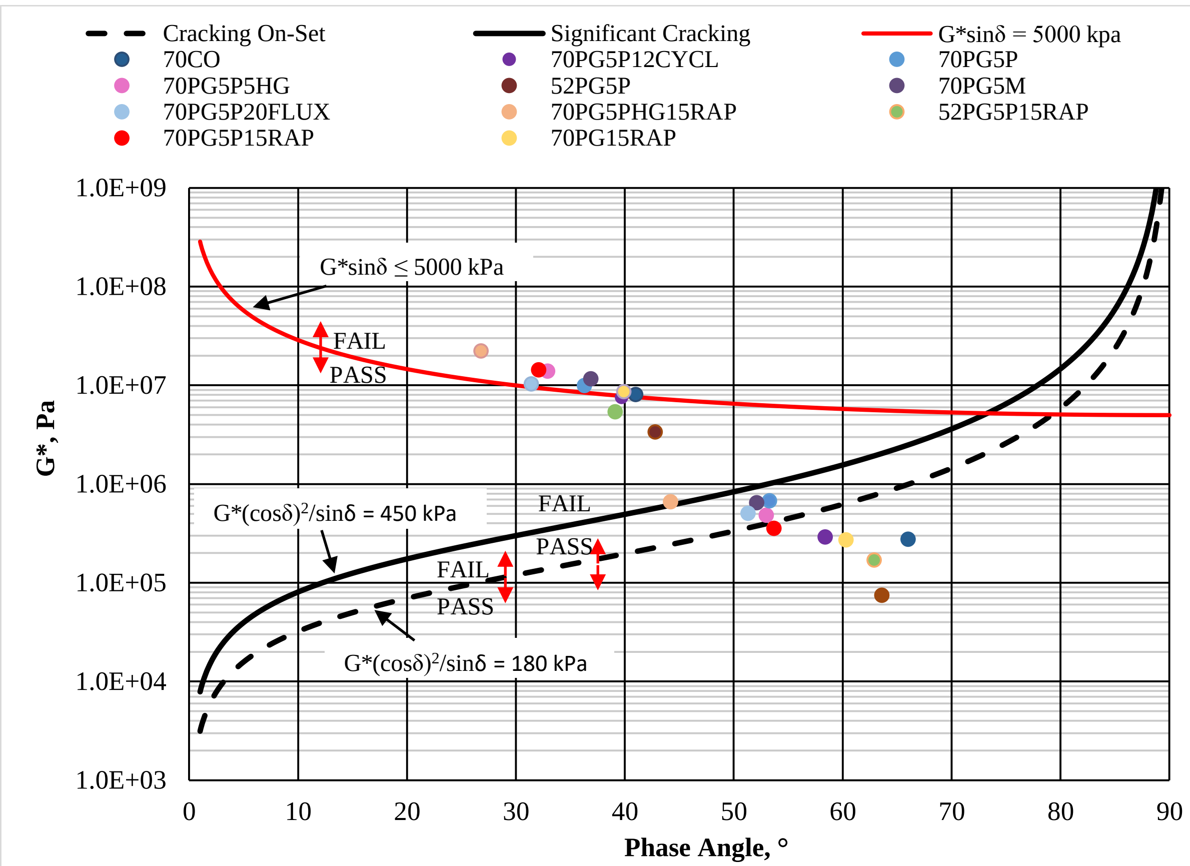


Figure 21
Comparison of conventional superpave and Glover-Rowe failure criteria

Figure 22 indicates the correlation between ΔTc and the rheological Index, R. Anderson et al. chose two parameters that relate ductility and the loss of flexibility with aging, ΔTc and R

[65]. As the asphalt binder becomes more m-controlled, the ductility of the asphalt binder decreases, which adversely affects an asphalt binder's capability to relax under loading and resist fracture. This is indicated by the ΔTc. Likewise, as the asphalt binder ages, there is an increase in the rheological index, R. It is shown in Figure 22 that there is a high correlation between ΔTc and R. As the rheological index increases, the ΔTc also increases.

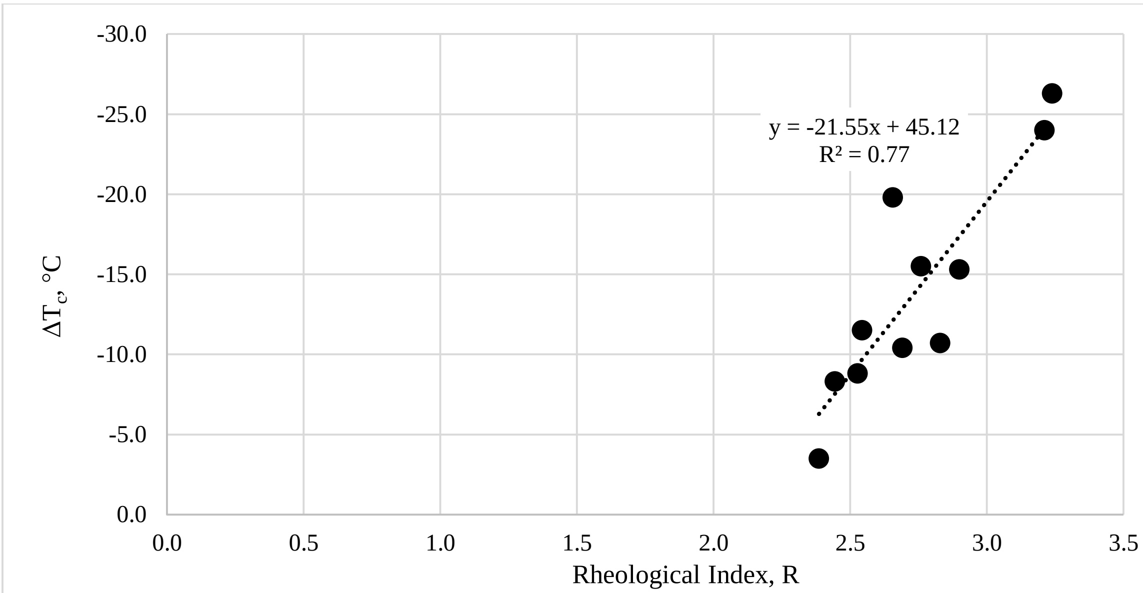


Figure 22
ΔT_c vs. R

Figure 23 illustrates the rheological index as it relates to mixture type. Also indicated is the recycle binder ratio, RBR, for each mixture. It is shown that asphalt mixture 70PG5PHG15RAP has the greatest R value of 3.24. It is also the asphalt mixture having the largest RBR (i.e., aged asphalt from recycled material within the asphalt mixture) of 41.5%. In addition, the conventional asphalt mixture, 70CO, containing no recycled binders had the lowest rheological index of 2.39, which indicates it is less aged than all mixtures evaluated in this study.

Figure 24 shows the correlation between ΔTc and RBR. This figure indicates that there is a trend between these parameters for the mixtures evaluated. It is indicated that as the RBR increases the ΔTc parameter increases. This would seem logical since the change in critical temperatures between stiffness and relaxation is representative of aging. Thus, the more aged binder one has in the asphalt mixture the greater the difference in ΔTc.

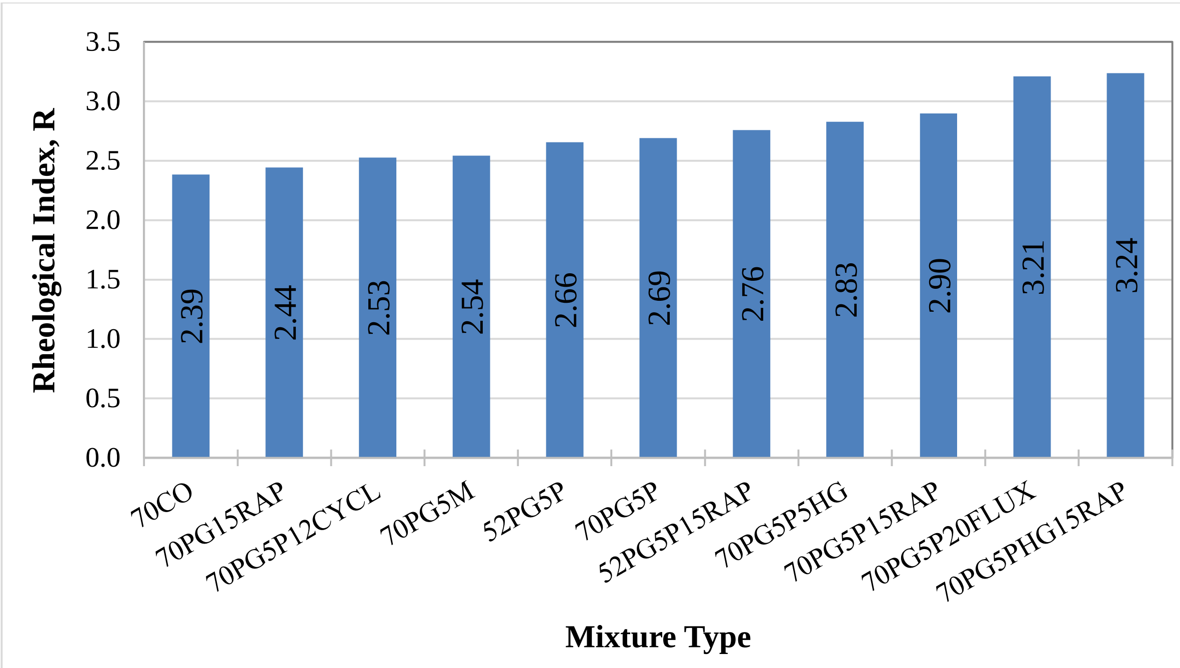


Figure 23
R vs. asphalt mixture type

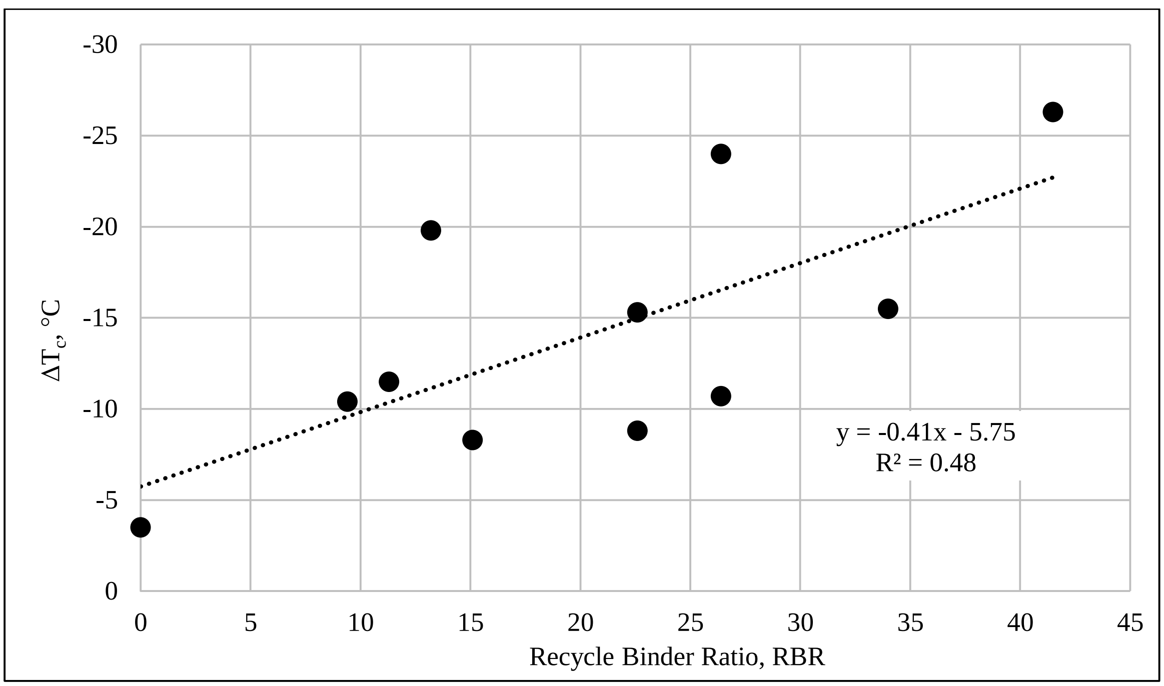


Figure 24
ΔT_c vs. RBR

Complex Shear Modulus, G*, Test Results

Figure 25 presents the complex shear modulus (G*) from the extracted binders at various test temperatures and frequencies for the 11 asphalt mixtures evaluated in this study. It is shown that the 70PG5PHG15RAP mixture had the highest G* values and the 52PG5P mixture had the lowest G* values at the low frequency range, which was expected because the 52PG5P mixture utilized the softest virgin binder. Whereas, the high stiffness results for the 70PG5PHG15RAP are due to 100% of the available aged RAS and RAP binder being utilized in this mixture. Also, there appears to be four groupings. The first grouping is mixture 70PG5PHG15RAP, which has the highest recycle binder ratio. The second grouping is comprised of mixtures 70PG5P5HG, 70PG5P, 70PG5M, 70PG5P20FLUX, and 70PG5P15RAP.

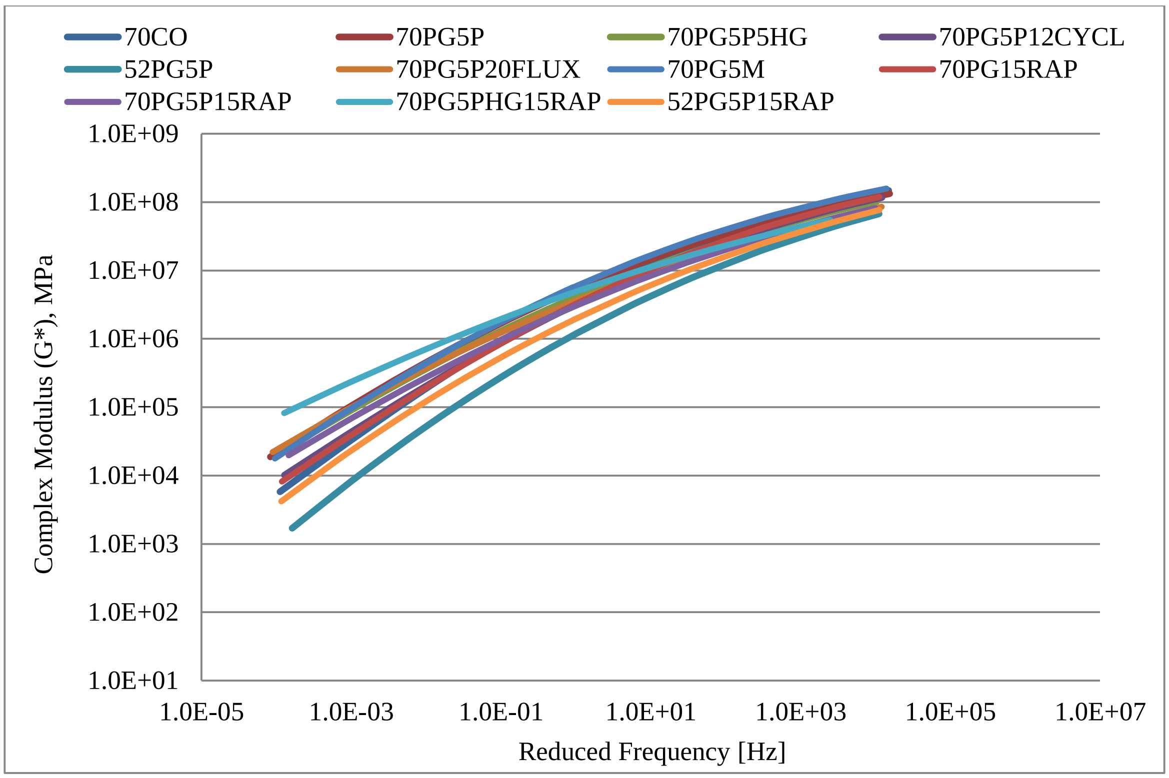


Figure 25
Dynamic shear rheometer test result

The remaining mixtures are grouped together with the exception of mixture 52PG5P. It can also be observed (Figure 26) that the low frequency part of the curves, which correspond to the higher testing temperatures, followed the same trend observed in the E* results obtained during the asphalt mixture testing. It is presented in Figure 25 that the 70PG5PHG15RAP asphalt mixture had the highest stiffness at the lower frequency. This would be indicative of a rut resistant mixture. Likewise, the 52PG5P mixture had the lowest stiffness at the lower frequency which could indicate a mixture’s propensity to rutting. It is shown in Figure 25 that all mixtures converge at the highest frequency which represents the low temperature response of the asphalt cement binders. It is indicated that the 52PG5P had the lowest stiffness at the highest frequency, which is indicative of a mixtures resistance to low temperature cracking.

Multiple Stress Creep Recovery Test Results

The multiple stress creep recovery (MSCR) test was conducted in accordance with AASHTO TP 70-13 to evaluate the effects of mixtures containing RAS and/or RAP, with and without recycling agents as compared to the control mixture containing no RAP, RAS, or recycling agents on rutting resistance *[56]*. This test was introduced to characterize the binder rutting resistance at high temperatures. D'Angelo et al. reported that the MSCR test parameters correlate well with mixture rutting performance as measured by accelerated pavement testing *[91]*.

As shown in Table 19, the increase in RAP and RAS content was associated with an increase in the percentage recovery and a decrease in the non-recoverable creep compliance. These are desirable characteristics as it would decrease the rutting susceptibility of the binders. Asphalt binders (virgin and extracted) were tested at 67°C, which is Louisiana's PG high temperature grade. Triplicate specimens were analyzed.

Table 19
MSCR test results

Extracted Binders from Mixtures	MSCR		
	$J_{nr3.2}$ @ 67°C, kPa^{-1}	%J_{nr} diff	% Recovery
70CO	0.51	16.6	35.07
70PG5P	0.23	13.1	43.25
70PG5M	0.10	7.1	53.85
70PG5P5HG	0.13	13.2	50.26
70PG5P12CYCL	0.33	17.2	39.39
52PG5P	3.37	22.1	21.33
70PG5P20FLUX	0.16	13.2	47.59
70PG15RAP	0.49	25.2	35.44
70PG5P15RAP	0.01	1.1	98.75
70PG5PHG15RAP	0.01	0.9	98.75
52PG5P15RAP	1.49	23.5	26.44

Asphalt Mixture Experiment Results

Several laboratory tests were conducted and evaluated to measure the performance characteristics of the asphalt mixtures considered in this study. The pavement performance characteristics were analyzed for the asphalt mixtures durability as measured by the Loaded Wheel Tracking Test (Hamburg Type) in terms of moisture sensitivity and permanent deformation, rutting. The asphalt mixtures performance in terms of resistance to fatigue cracking was evaluated from results obtained from the Semi-Circular Bend (SCB) and Dynamic Modulus (i.e., fatigue factor, $E^*\sin\delta$) tests. Furthermore, Dynamic Modulus (i.e., rutting factor, $E^*/\sin\delta$) and Loaded Wheel Tracking Test (Hamburg Type) was used to determine the mixtures' resistance to permanent deformation. Triplicate samples were prepared and tested for each laboratory test. To measure the low temperature performance characteristics (thermal cracking), the TSRST was conducted on the asphalt mixtures studied. The detailed analysis for these test results is included in the following sections of this chapter.

Laboratory Performance Tests

Dynamic Modulus (E*) Test Results. The purpose of the Dynamic Modulus test is to evaluate the viscoelastic response characteristics of asphalt mixtures over a given range of temperatures and frequencies. Figure 28 presents the dynamic modulus ($|E^*|$) at 5 test temperatures (-10, 4.4, 25, 37.8, and 54.4°C) and 6 frequencies (25, 10, 5, 1, 0.5, and 0.1 Hertz) for the 11 asphalt mixtures evaluated in this study. It is shown that the 70PG5PHG15RAP mixture had the highest $|E^*|$ values and the 52PG5P mixture had the lowest $|E^*|$ values. It was expected that the 52PG5P mixture would have the lowest stiffness as measured by $|E^*|$ because of the very soft virgin binder utilized in this mixture. Further, the high stiffness results for the mixture 70PG5PHG15RAP are attributable to the 100% of the available aged RAS and RAP binder being included in this mixture. Also there appears to be four mixture groupings. The first mixture grouping is mixture 70PG5PHG15RAP, which has the highest recycle binder ratio. This mixture also had the highest extracted binder stiffness as measured by the dynamic shear rheometer. The extracted asphalt binder from this mixture was approximately 270% higher than the others binders measured. The second mixture grouping is comprised of mixtures 70PG5P5HG and 70PG5P15RAP. The remaining mixtures are ranked together with the exception of mixture 52PG5P. It is worth noting that the low frequency part of the curves, which correspond to the higher testing temperatures, followed the same trend observed in the G* results from the PG grading of extracted binders; see Figure 25.

Figure 27 shows the mean phase angle results vs. the mean Dynamic Modulus values for all 11 asphalt mixtures considered in this study. This figure shows the phase angle for all materials increases with an increase in temperature and a decrease in frequency. Then the phase angle peaks, followed by a decline as the temperature increases further and the frequency continually decreases.

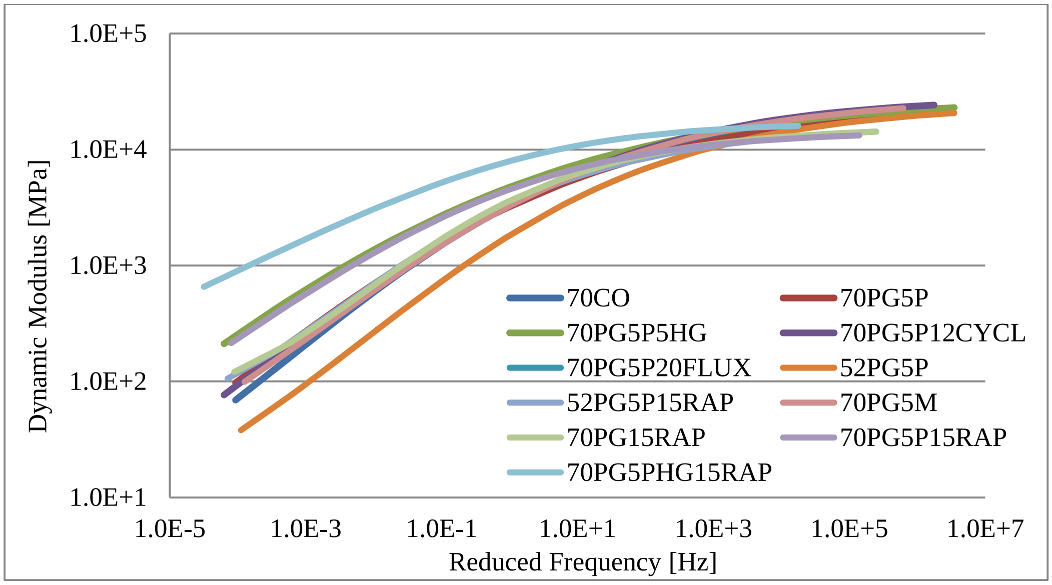


Figure 26
Dynamic modulus (|E*|) vs. reduced frequency

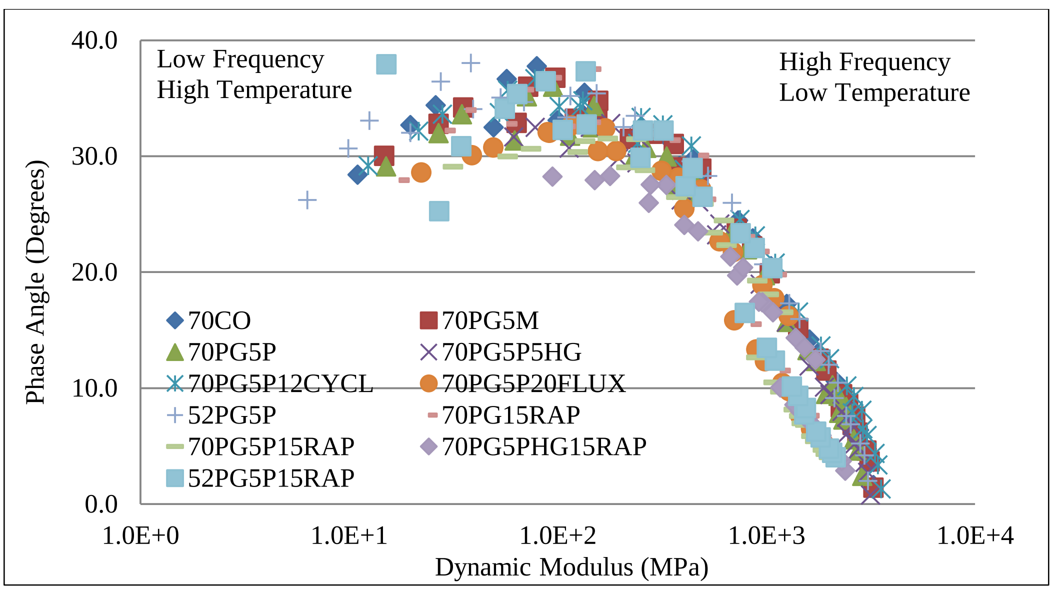


Figure 27
Phase angle vs. dynamic modulus (|E*|) relationship

Figure 28 indicates a normalized comparison to the control mixture (70CO) for all asphalt mixtures evaluated in this study based on the dynamic modulus |E*| at 5 test temperatures (-10, 4.4, 25, 37.8, and 54.4°C) and 6 frequencies (25, 10, 5, 1, 0.5, and 0.1 Hertz). For the purpose of comparison, the E* values calculated at various test temperatures and frequencies for the 70CO asphalt mixture was considered as the unit value (i.e., E* = 1.0).

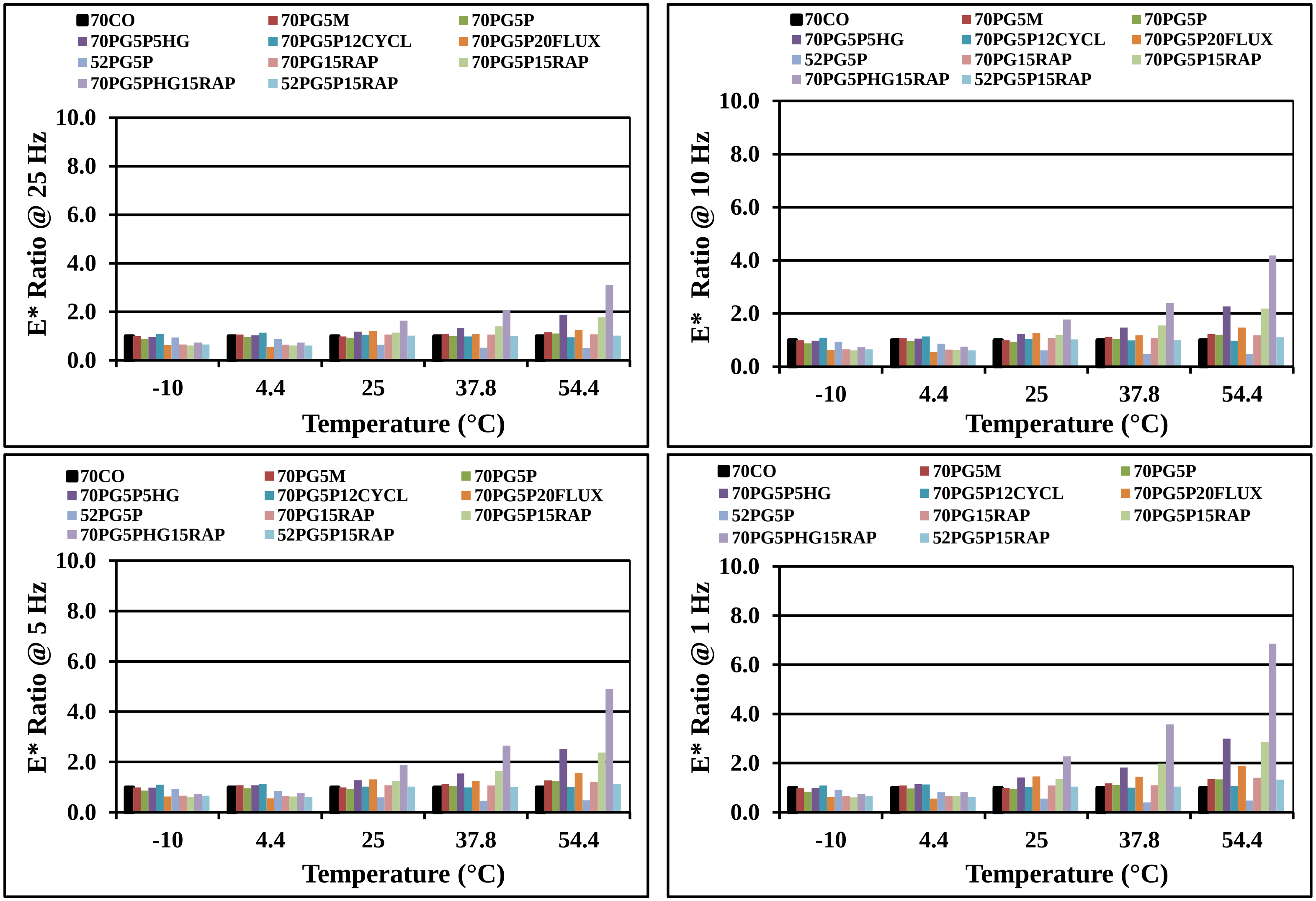
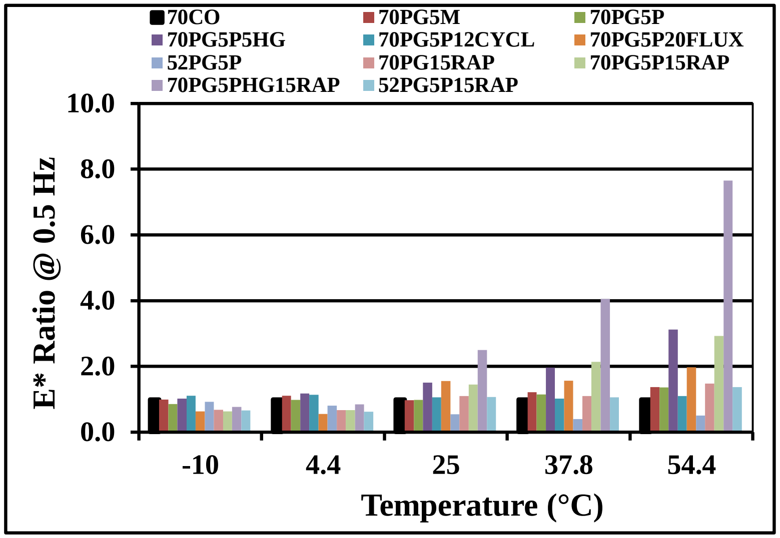


Figure 28
Dynamic modulus ratio comparison (E* Ratio)

(Figure 28 continued)



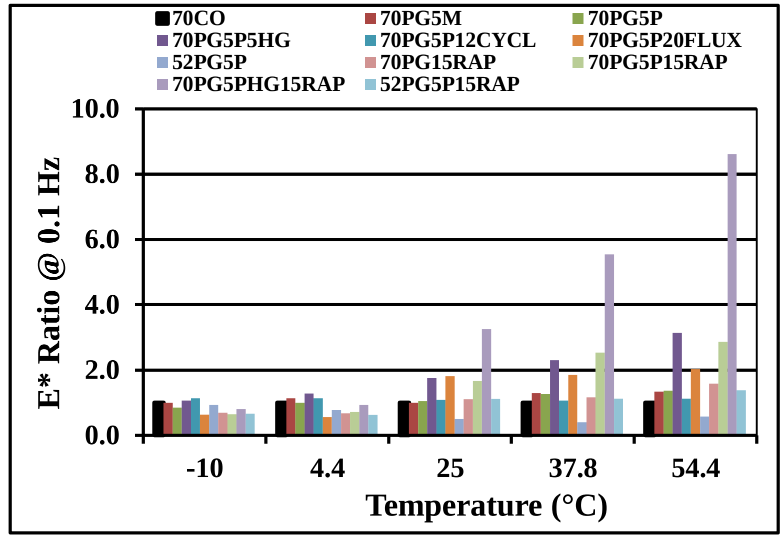


Figure 28
Dynamic modulus ratio comparison (E* Ratio)

To illustrate this concept, the E* values for the 70PG5P and 70CO mixtures at 54.4°C and 5 Hz were 71.2 ksi and 57.4 ksi, respectively. The comparative E* ratio is 71.2/57.4 = 1.2. Any mixtures exhibiting an E* ratio greater than 1.0 has greater stiffness than the 70CO mixture. For high temperature and low frequency, it is important to have an E* ratio greater than 1.0 because this is indicative of a mixtures propensity to resist rutting. Likewise, it is advantageous to have an E* ratio less than 1.0 at the other extreme (low temperature and high frequency). These mixtures have the potential to resist low temperature cracking. Mixture 70PG5PHG15RAP exhibited the highest stiffness at the temperatures of 25°C, 37.8°C, and 54.4°C for all frequencies. Figure 28 illustrates that the mixtures containing RAS and/or RAP, and PG 52-28 asphalt binder generally had lower stiffness than 70CO for temperatures at or below 25°C for all frequencies. In addition, generally the mixtures containing MWS (70PG5M) and PCWS (70PG5P) without recycling agents had comparable stiffness with 70CO for all temperatures and frequencies.

To determine a mixtures resistance to fatigue cracking, a parameter termed fatigue factor is calculated from dynamic modulus test results at a test temperature of 25ºC and a loading frequency of 5 Hz for this study *[92]*. The fatigue factor is computed as E*sinδ, where δ is the phase angle at the selected temperature and frequency *[92]*. For a mixture to resist fatigue cracking, its corresponding E* value should be lower as well as the phase angle at the in-service temperature of 25 ºC. A lower fatigue factor value indicates a better resistance to fatigue cracking.

Figure 29 shows the fatigue factor values for all mixture types and statistical analysis evaluated in this study. There are three distinct groups as shown in Figure 29. Figure 29 indicates that there is a statistical difference between mixtures 52PG5P, 70PG5M, and 70PG5P (Group A) and mixture 70PG5PHG15RAP (Group B). The remaining mixtures (Group A/B) indicate that there is no clear-cut statistical difference between Groups A and B since their mean values are close to both groupings. Statistically, the grouping of mixtures 52PG5P, 70PG5M, and 70PG5P (as shown in Figure 29) are best in fatigue cracking resistance of the 11 mixtures evaluated in this study. Mixture 70PG5PHG15RAP exhibited the highest fatigue factor values; therefore it is the least resistant to fatigue cracking. This can be contributed to this mixture having the highest RBR, the stiffest complex shear modulus (G*), and the highest dynamic modulus (E*). The remaining mixtures (Group A/B) indicate that there is no clear-cut statistical difference between Groups A and B since their mean values is close to both groupings.

An asphalt mixture's propensity to resist permanent deformation (rutting) can be characterized by using the dynamic modulus test results from various temperatures and frequency. The rutting factor is defined as E*/sinδ, where δ is the phase angle, at a particular temperature and frequency. A loading frequency of 5Hz and test temperature of 54.4ºC was used for computation of the rutting factor, E*/sinδ in this study *[92]*. For mixtures to be rut resistant and exhibit higher stiffness necessitates a higher E* value and a lower phase angle. The higher the rut factor value indicates a mixture greater resistance to permanent deformation.

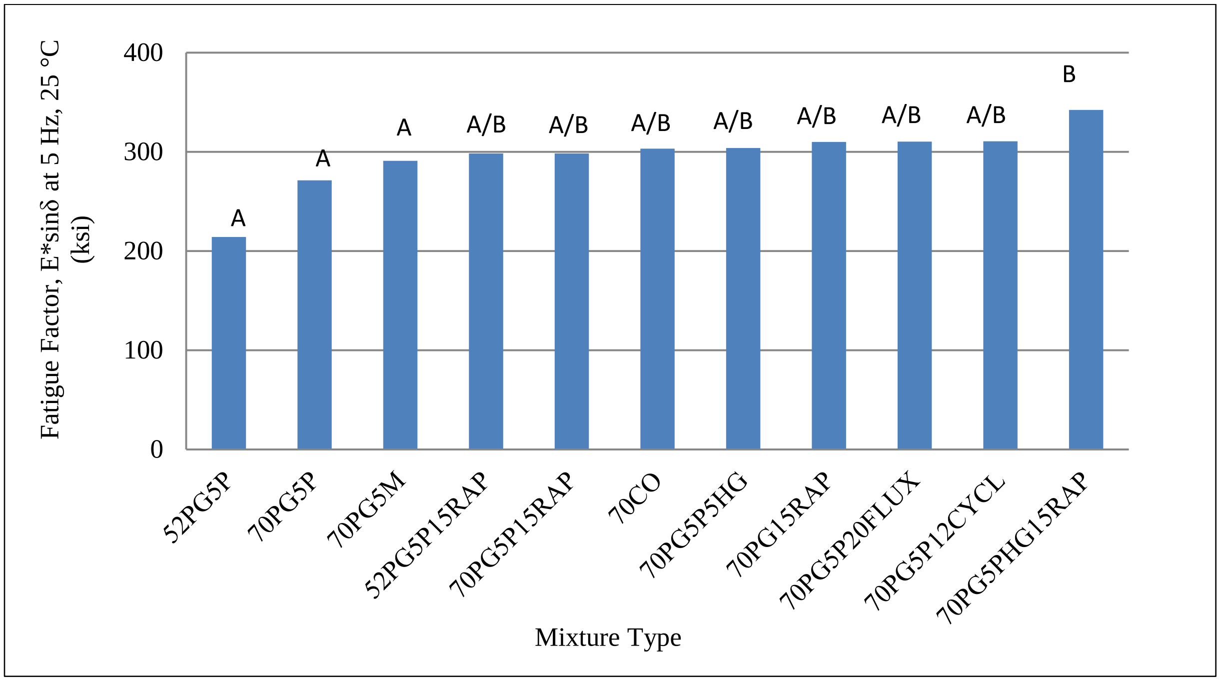


Figure 29
Dynamic modulus test result – fatigue factor

Figure 30 shows the rutting factor values and statistical analysis for all mix types evaluated in this study. Figure 30 indicates that there are five statistical groupings. It clearly shows that the 70PG5PHG15RAP mixture has the greatest resistance to rutting followed by 70PG5P5HG and 70PG5P15RAP. Mixture 70PG5PHG15RAP's resistance to rutting can be contributed to this mixture having the highest RBR, the stiffest binder complex shear modulus (G*), and the stiffest dynamic modulus (E*). Mixtures having the least resistance to permanent deformation are 52PG5P, 70CO, 70PG5P12CYCL, and 52PG5P15RAP. It is noted that there is a grouping of similar results for the 70PG15RAP, 70PG5M, 70PG5P, and 70PG5P20FLUX asphalt mixture types.

Figure 30 indicates that there is a statistical difference between mixture 70PG5PHG15RAP (Group A) and all other mixtures studied. Group A has the highest rut factor and is the most resistant to rutting. Five mixtures are grouped in the "B" and "B/C/D," 70PG5P5HG, 70PG5P15RAP, 70PG5P20FLUX, 70PG5P, and 70PG5M, respectively. There are no discernable statistical difference in this grouping. The remaining mixtures (C/D and D) are the least resistant to rutting because they have the lowest rut factor value. Statistically, the grouping of mixtures 52PG5P, 70PG5M, and 70PG5P (as shown in Figure 29) are best in fatigue cracking resistance of the 11 mixtures evaluated in this study.

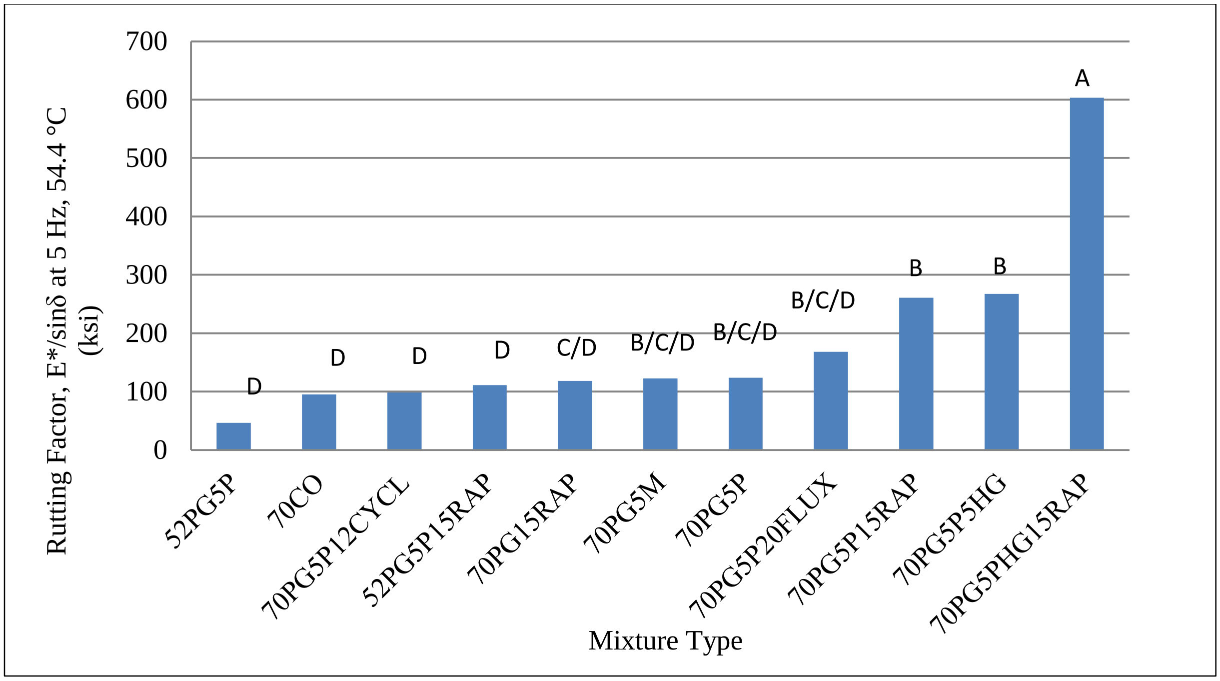


Figure 30
Dynamic modulus test result – rutting factor

High Temperature Mixture Performance

LWT (Hamburg Type) Test Results. Figure 31 illustrates the average permanent deformation depth for the 11 asphalt mixtures evaluated in this study. It is shown that the mixture 70PG5PHG15RAP was the most resistant to permanent deformation, whereas, the mixture 52PG5P containing the PG 52-28 soft asphalt binder and no recycling agent was the least resistant to rutting. However, it is noted that all mixture evaluated performed less than many state specifications for a 12.5-mm NMAS mixture. It is observed that the addition of RAP and RAS reduced the terminal rut depth as compared to the asphalt mixture with no RAS, 70CO. It is also noted that the mixture containing the Cyclogen-L ranked second in least resistance to rutting. These findings are in agreement with the rutting factor results shown in Figure 30. The mixture containing Hydrogreen, RAP and RAS (70PG5PHG15RAP) had the highest stiffness, highest RBR (Table 15), and, therefore, one would expect that it would be the most resistant to permanent deformation. Likewise, it is seen that the mix containing the soft binder (52PG5P) had the lowest dynamic modulus values, and, therefore, should be the most susceptible to rutting. As shown in Figure 32, generally the remaining mixtures were clustered together and are expected to perform similarly against rutting. No tertiary regions were seen in the asphalt mixtures studied (no stripping inflection points); therefore, no susceptibility to moisture damage as measured by the LWT could be observed.

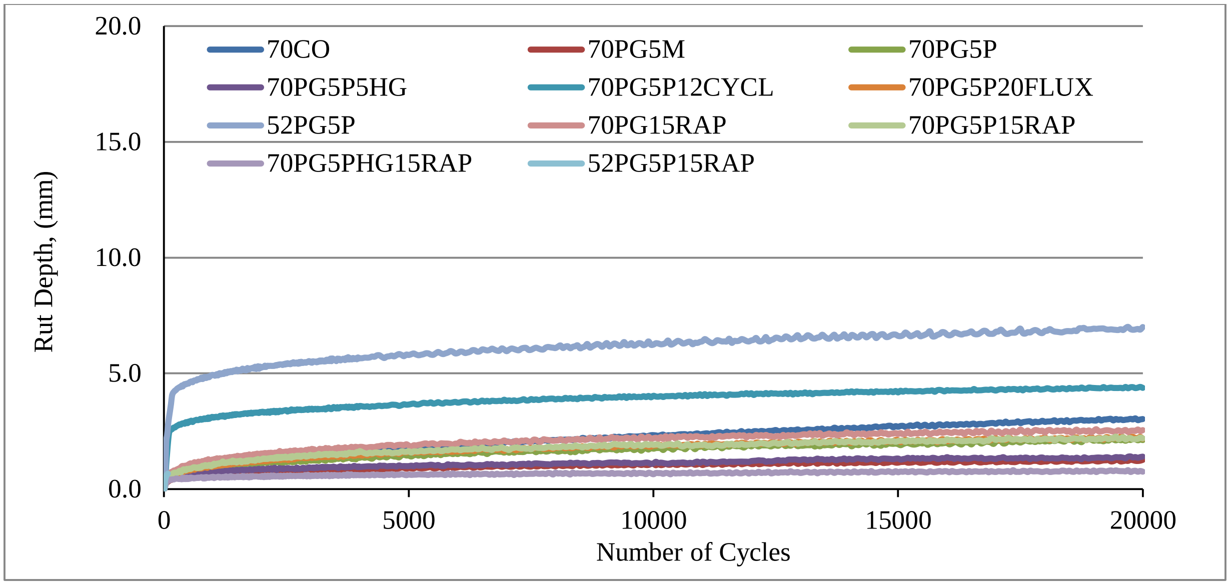


Figure 31
LWT test result, 50°C, wet

Figure 32 indicates the statistical differences between permanent deformation and mixture type. Laboratory test data were statistically analyzed using the analysis of variance (ANOVA) procedure (Tukey's studentized range HSD (honestly significant difference)) provided in the Statistical Analysis System (SAS) program. A multiple comparison procedure with a confidence level of 0.05 was performed on the means. The groupings represent the mean for the test results reported by mixture type. The results of the statistical grouping are reported with letters A, B, C, D, and so forth. Letter A was assigned to the highest mean followed by the other letters in appropriate order. A double (or more) letter designation, such as A/B (or A/B/C) indicates that the difference in the means is not clear-cut, and that the mean is close to either group in the analysis. Figure 32 indicates that there are only two statistical groupings. Statistically there is not much difference in these groupings and the difference is not significant.

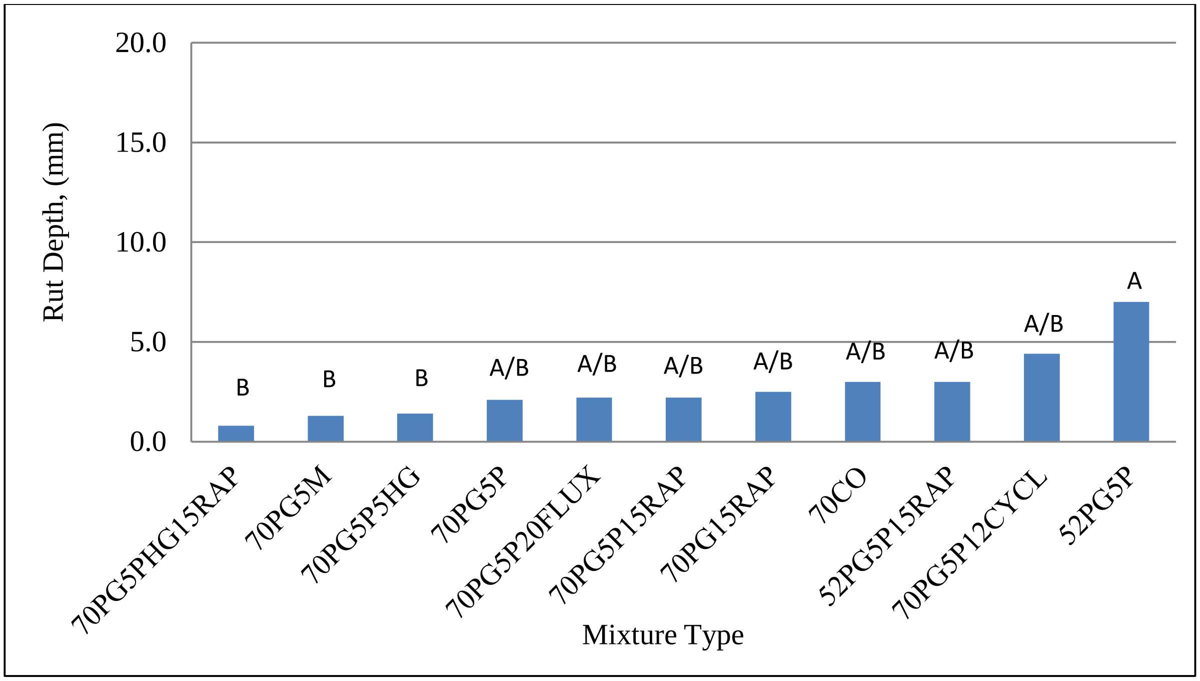


Figure 32
Statistical comparison: LWT test result, 50°C, wet

Figure 33 presents the characterization laboratory test correlation between the binder non-recoverable creep compliance, J_{nr}, (measured at an applied constant stress of 3.2 kPa and at a testing temperature of 67°C), and the LWT rut depth (permanent deformation) measured at 20,000 passes at a testing temperature of 50°C submerged in water for the asphalt mixtures evaluated in this study. A decrease in the non-recoverable creep compliance indicates an improved resistance to rutting damage. This figure shows that as the J_{nr} decreases the rut depth also decreases. It is indicated in Figure 33 that there is a good correlation between the non-recoverable creep compliance, J_{nr}, and LWT test results.

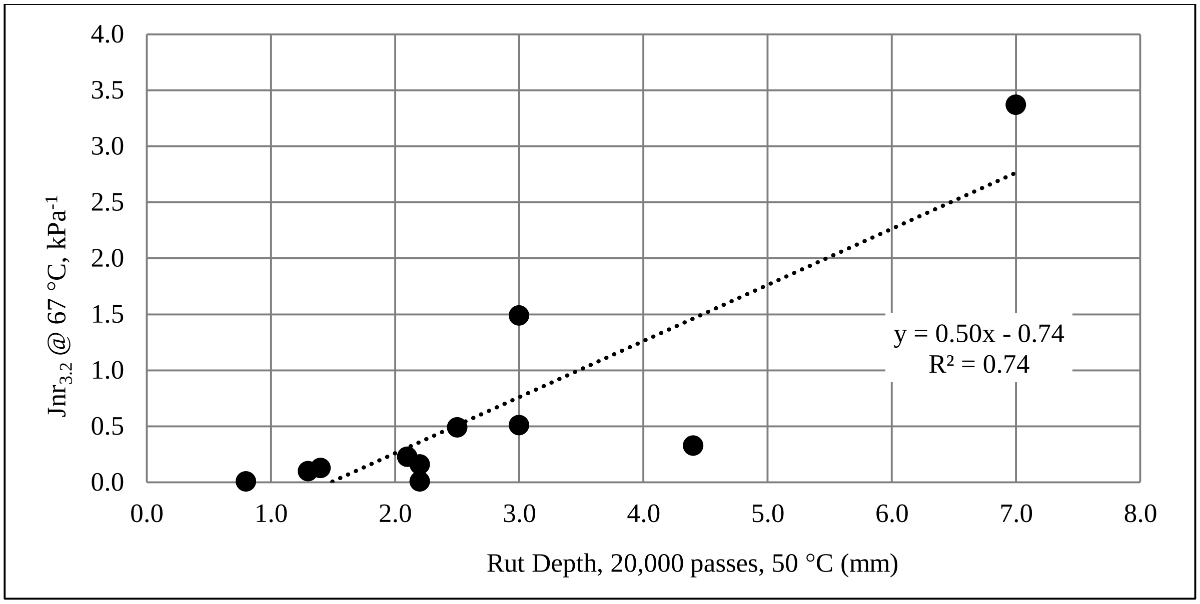


Figure 33
$J_{nr3.2}$ @ 67°C vs LWT rut depth

Figure 34 indicates the characterization laboratory test correlation between the Rutting Factor, E*/sinδ at 5 Hz, 54.4°C, and the LWT rut depth (permanent deformation) measured at 20,000 passes at a testing temperature of 50°C for the asphalt mixtures evaluated in this study. This figure shows that there is a trend between the Rutting Factor and rut depth test results. For mixtures to be rut resistant and exhibit higher stiffness, this necessitates a higher E* value and a lower phase angle. The higher the rutting factor value indicates a mixture greater resistance to permanent deformation. It is illustrated in Figure 34 that as the Rutting Factor increases the rut depth decreases. This is desirable trend since higher rutting factor values indicate an asphalt mixtures stronger propensity for rut resistance.

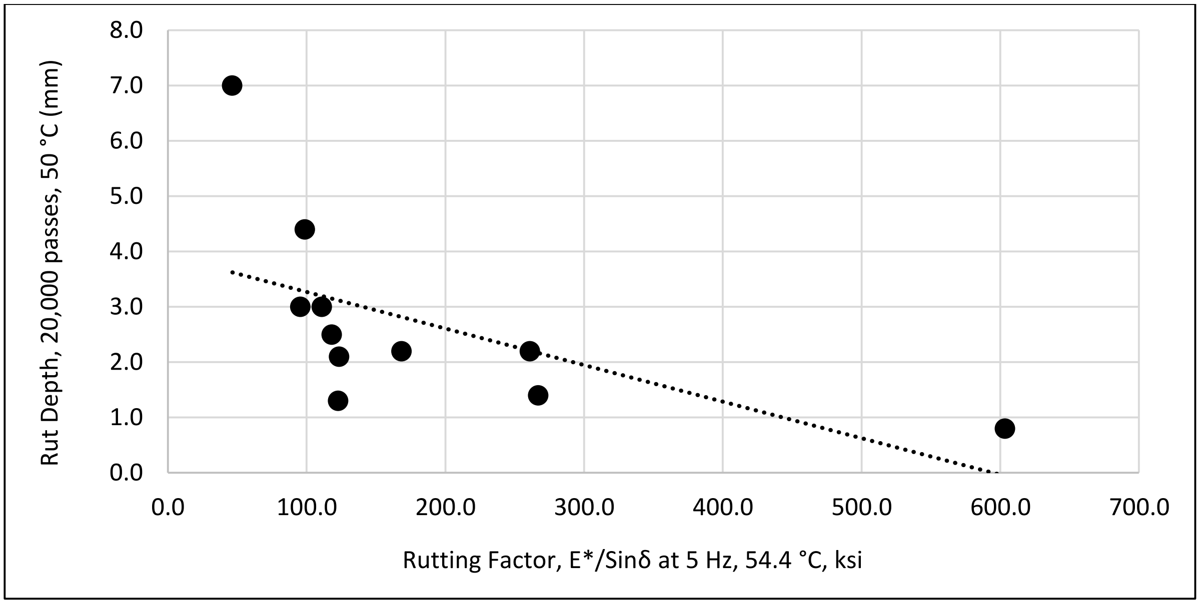


Figure 34
LWT rut depth vs rutting factor, E*/sinδ at 5 Hz, 54.4°C

Intermediate Temperature Mixture Performance

Semi-circular Bend Test Results. Figure 35 presents the calculated critical fracture resistance (J_c) values for the 11 asphalt mixture types evaluated. Mixture aging was performed according to AASHTO R30 by placing compacted specimens in a forced draft oven for five days at 85°C *[93]*. After aging, the specimens were loaded at a monotonic rate of 0.5mm/minute until failure. The higher the J_c value, the greater the fracture resistance the asphalt mixtures possess. It is shown that the 70PG15RAP asphalt mixture had the highest J_c value, and, therefore, has the greatest fracture resistance of all mixtures evaluated in this study. It is suspected that the 70PG15RAP had a the highest J_c value as compared to the control mixture (70CO) because the RAP utilized in this study comprised of a high percentage of polymer modified asphalt as measured by the molecular weight species by gel permeation chromatography.

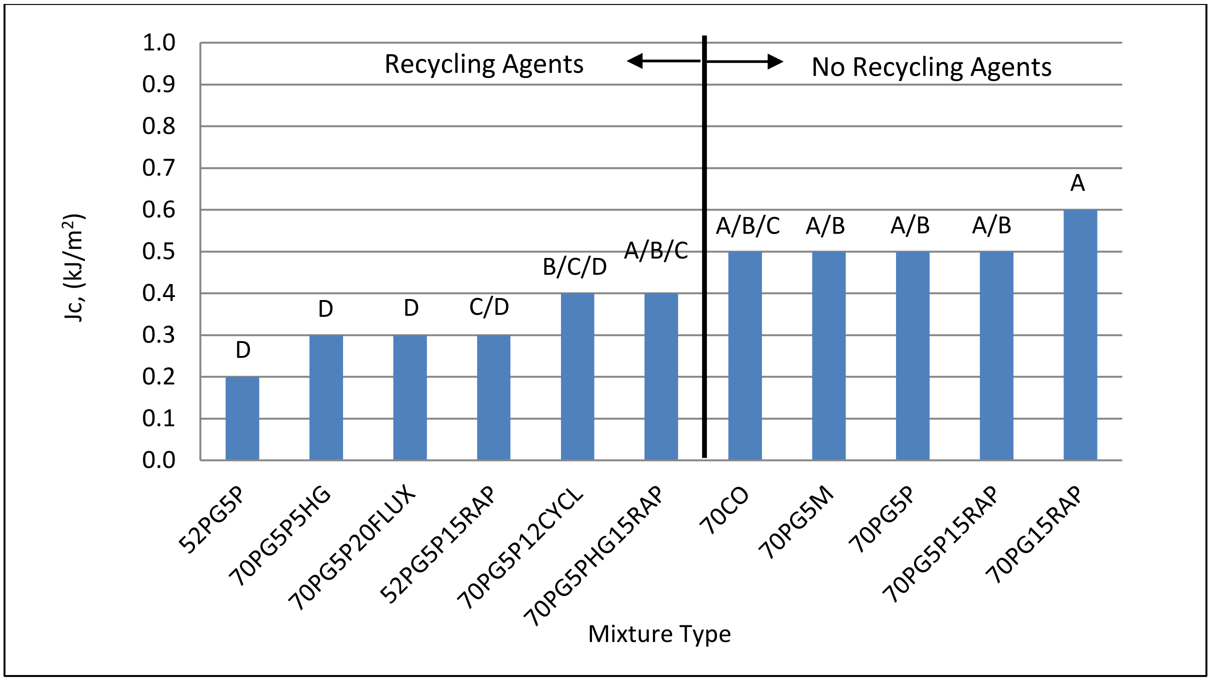


Figure 35
Semi-circular bend test results, 25°C

A minimum threshold J_c value of 0.50 kJ/m^2 is typically used as a failure criterion and is currently being implemented in Louisiana as an acceptance criterion for mixtures containing a PG 70-22 asphalt binder. A J_c of 0.5 kJ/m^2 or above is considered resistant to intermediate temperature fracture *[83]*. It is shown in Figure 35 that asphalt mixtures containing no recycling agents had higher J_c values than the mixtures containing recycling agents, rejuvenators and softening agents. In addition, those mixtures that do not have recycling agents also passed Louisiana's proposed J_c threshold specification of 0.5 kJ/m^2. This can be contributed to these mixtures having more virgin asphalt binder and lower recycle binder ratios than those mixtures containing recycling agents. Although some activation of the RAS binder was achieved without the use of recycling agents, some of the RAS acted as a black rock. This necessitated the use of more virgin asphalt binder to meet volumetric and densification criterion. When the RBR ratio for mixtures increased due to the addition of recycling agents, the resistance to fracture was adversely affected even though the recycling agents are classified as rejuvenators and softening agents. In addition, the use of the soft asphalt binder (PG 52-28) showed very little to no improvement in its resistance to fracture, mixtures 52PG5P and 52PG5P15RAP respectively. In fact, the asphalt mixture designated as 52PG5P is the least resistant to fracture as evaluated in this study.

Figure 35 indicates the statistical differences between J_c and mixture type. It is shown in Figure 35 that there are three statistical groupings. The first statistical grouping is represented by letter designations of A, A/B, A/B/C (mixtures 70PG15RAP, 70PG5P15RAP, 70PG5P, 70PG5M, 70CO and 70PG5pHG15RAP). The second grouping is B/C/D and C/D comprised of mixtures 70PG5P12CYCL and 52PG5P15RAP. The last grouping is "D" which is comprised of mixtures 52PG5P, 70PG5P5HG, and 70PG5P20FLUX and had the lowest SCB test results as measured by J_c. It is indicated in this figure that there is a statistical difference between mixtures containing recycling agents and those mixtures not containing recycling agents. Generally, there is no statistical difference between mixtures that did not utilize recycling agents. It is noted that some of these mixtures have statistical designations of A, A/B, and A/B/C, but this indicates that there is no clear-cut statistical difference between Groups A, B, and C since their mean values are close to both groupings.

Low Temperature Mixture Performance

Thermal Stress Restrained Specimen Tensile Strength Test (TSRST) Results. Figure 36 shows the low temperature fracture for the mixtures studied as measured by TSRST. Mixture aging was performed according to AASHTO R30 by placing compacted specimens in a forced draft oven for five days at 85°C *[93]*. After aging, the specimens were loaded at an applied rate of -10°C/hour.

The test was stopped either at -50°C (coolant limitation) or at fracture, whichever occurred first. The asphalt binder utilized in this study was modified with SBS with a low temperature grade of -22°C with the exception of the PG 52-28 asphalt binder. It is shown that the results indicate seven statistical groupings. However, several of the groupings have double (or more) letter designation, such as A/B (or A/B/C), which indicates there are no clear-cut statistical difference between Groups A, B, and C since their mean values are close to both groupings. Thus there are essentially three groupings. It is shown that mixture 70PG5P had the lowest fracture temperature and that this mixture is statistically different from mixture 70PG5PHG15RAP. It is noted that mixture 70PG5PHG15RAP had the highest fracture temperature (more susceptible to low temperature fracture) of the groupings, and this mixture had the highest recycle binder ratio and utilized a rejuvenating type recycling agent. Figure 36 also shows that generally the mixtures that contained no recycling agents were less susceptible to low temperature fracture, with the exception of mixture 52PG5P which utilized a soft asphalt binder (PG 52-28). Fracture occurs at the low temperature PG grade due to thermal contraction as the specimens are cooled at a rate of 10°C per hour. This may be contributed to only a portion of the recycled binder being activated within the mixture and the remaining recycled materials acting as a black rock.

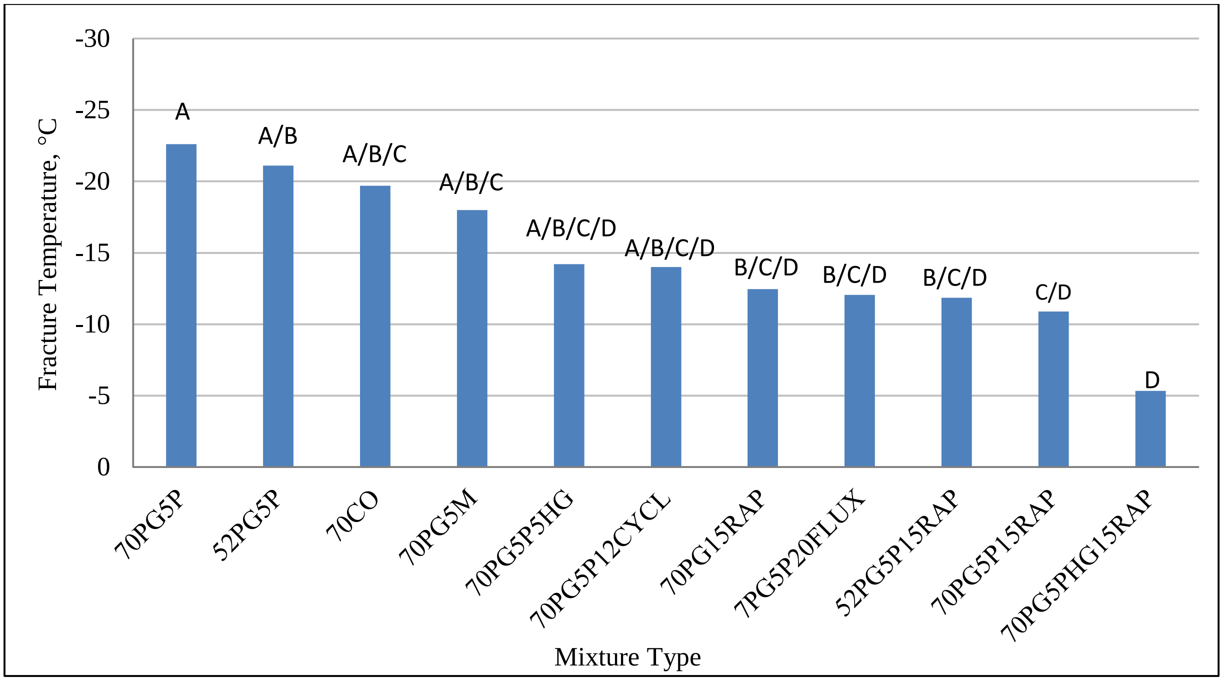


Figure 36
TSRST temperature vs. mixture type

Figure 37 compares the critical low temperatures of asphalt binders determined by the TSRST and by the BBR. It is generally expected that the fracture temperature measured by the TSRST corresponds to the PG low temperature of the asphalt binder utilized in the mixture. However, this assumption would only be valid for mixtures containing virgin asphalt binders. In fact, it has been observed that the correspondence, at some degree, depends on the contents of recycled materials utilized in the asphalt mixtures. As shown in Figure 37, the TSRST fracture temperatures were generally in good agreement with the extracted asphalt binders' PG low temperature values, but for the mixtures 70PG5P12CYCL, 70PG15RAP, and 52PG5P15RAP, the extracted asphalt binders' PG low-temperature were significantly lower than the TSRST fracture temperatures.

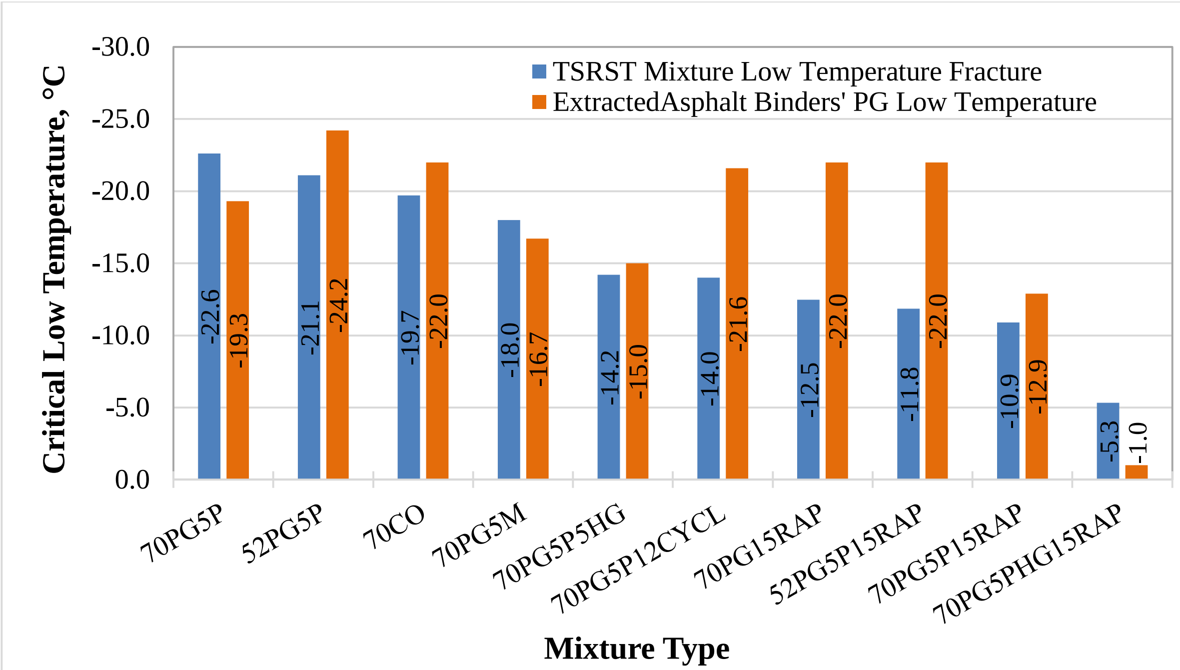


Figure 37
Critical low temperatures: TSRST vs. BBR

Figure 38 shows the correlation between the rheological index, R, and the PG low-temperature of the extracted asphalt binder as determined by BBR. This figure shows that there is a high correlation between these parameters. It is indicated in Figure 38 that as the rheological index becomes greater there is a decrease in the low temperature grade of the asphalt binder. It is expected that since an increase in R represents the increase in aging, which is known to adversely affect the low-temperature performance of the asphalt binder. As the binder ages, the asphalt binder loses the ability to relax under loading, and this is represented in the determination of the low-temperature PG property of the material, m-value. The 70PG5PHG15RAP as shown in Figure 37 has the highest fracture temperature (-5.3°C) and also has the highest rheological index of 3.24 as previously shown in Figure 23.

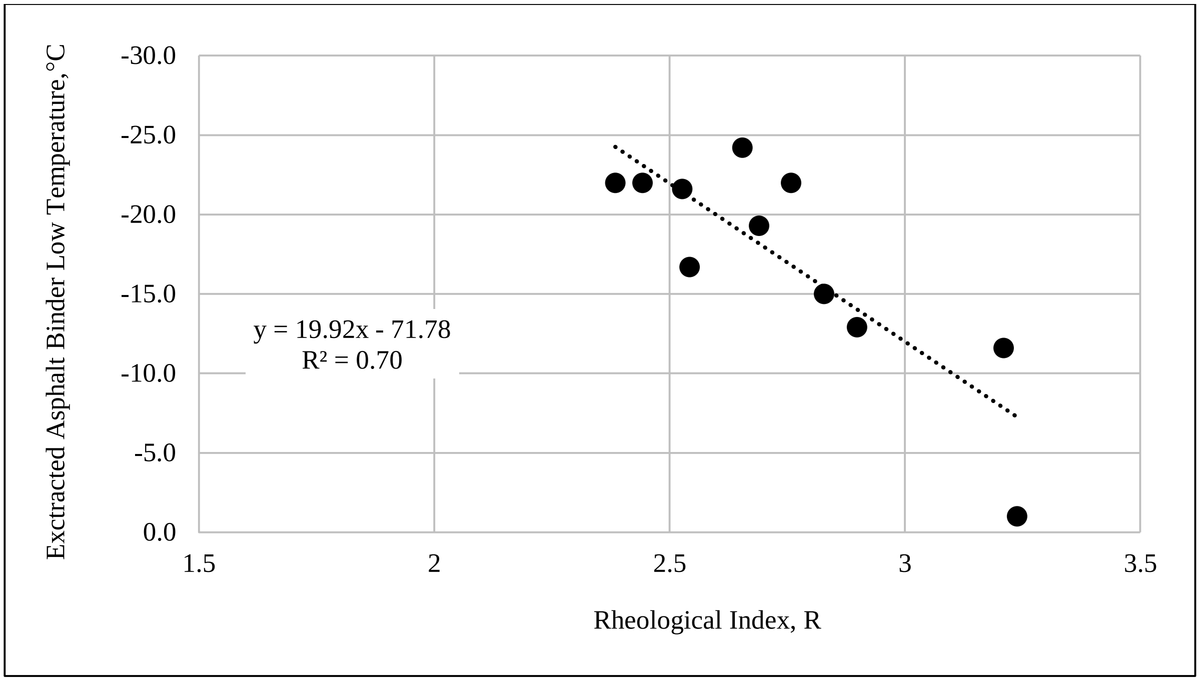


Figure 38
Extracted binder low temperature vs. R

Binder Experiment Results

Performance of RAS in Asphalt Pavements

The industry has been addressing the stiffness and blending concerns by using softer binders when using higher RAP/RAS contents. Since fatigue cracking is influenced more by the intermediate temperature binder properties, using soft (modified) binders is an effective method to improve cracking resistance of RAS mixes. Zhou et al. conducted a comprehensive investigation of asphalt mixtures containing RAS *[94]*. This study characterized the RAS asphalt binder including the evaluation of blending charts for virgin binder blended with RAS binders. In addition, the impact of RAS content on the optimum asphalt binder content and respective engineering properties on mixtures containing RAS was evaluated. It was concluded that the use of RAS had no significant influence on dynamic modulus, though it improved mixture resistance to rutting and moisture damage. However, mixtures containing RAS had very poor cracking resistance as compared to mixtures containing no RAS. Zhou et al. explored two approaches to improve cracking resistance of mixtures containing RAS *[94]*. It was stated that the use of soft binder and increasing the design density can improve cracking resistance. In terms of rutting and moisture damage, the use of soft binders was superior to increasing the design density. When using the softer binder and low air void approaches, one should be aware that, if the RAS is not well blended into the mixture or, if segregation occurs during mixing and/or placement, spots appear on the pavement of "softer" mix, which may fail due to rutting *[94]*.

Asphalt rejuvenating and softening agents are manufactured to restore the rheological properties of the reclaimed asphalt binder by diffusing into it and restoring its colloidal structure and reconstituting its chemical components. Therefore, rejuvenators have been extensively used in pavement preservation to revive the hard and oxidized top layer by penetrating into the pavement and fluxing with the aged binder to balance the maltenes to asphaltenes ratio *[95]*.

Diffusion is the key factor in blending rejuvenators with asphalts. Diffusion of rejuvenators in RAP binders has been of interest since several decades ago. Although there is experimental evidence that the rejuvenators penetrated the RAP binders, corresponding evidence on the impact of these agents on RAS is limited *[96], [97]*. Im et al. studied the impacts of three different rejuvenators on mixtures containing various contents of RAS and RAP, using LWT, the Overlay Test Repeated load test, and the dynamic modulus to characterize the mixtures *[98]*. It was concluded that the rejuvenators improved the cracking resistance, moisture susceptibility, and rutting resistance comparatively to the control mixture. However, the ranking of the three rejuvenators used in the study depended on mixture types and properties evaluated.

With the increased interest in using RAS, the use of recycling agents is considered essential in order to soften and/or to rejuvenate the aged and stiff binders in RAS. Recycling agents are classified as two types: rejuvenating agents and softening agents. Softening agents lower the viscosity of the aged binder while rejuvenating agents are intended to restore the rheological and chemical properties of the aged binder *[98]*. Examples of softening agents include asphalt flux oil, lube stock, and slurry oil. Examples of rejuvenating agents include lubricating and extender oils, which contain a high proportion of maltenes constituents and low saturate contents that do not react with asphaltenes *[95]*. The design and production of asphalt mixtures containing RAS requires provisions to assure that the final product will meet and/or exceed the expected pavement life as required by construction and performance specifications.

Extraction of Binder from RAS Samples

The RAS samples and mixtures containing RAS were extracted with refluxing toluene under nitrogen using a Soxhlet extractor. The solution of asphalt binder in toluene was cooled to room temperature and then filtered to remove most of the fine particles of sand present. The filtered solution was allowed to stand overnight, decanted, and concentrated under vacuum using a rotary evaporator. The concentrated asphalt binder solution in toluene was then dried for 36 to 48 hours in a vacuum oven first at room temperature (ca. 24 hrs), then at 50°C for 12 hrs.

Saturate, Aromatic, Resin, and Asphaltenes (SARA) Analysis Results

The SARA analysis of the binders performed in this study is compiled in Table 20. The asphaltenes are reported as n-heptane insolubles and the maltenes containing resins, aromatics, and saturates were determined by TLC/FID with an Iatroscan instrument. It is noteworthy that the asphaltenes component as determined by precipitation is considerably smaller than the sum of all the components with molecular weights greater than 3,000 Daltons estimated from deconvoluted GPC chromatograms, designated as DEC ASPH in Table 20. The higher percentage of DEC ASPH is composed of associated asphaltenes species with molecular weights as high as 100,000 *[99]*. The high molecular weight component (HMW) of the DEC ASPH (i.e., a sum of the species with molecular weights >19,000 Daltons), is also reported in Table 20. It is noted that the SARA asphaltenes analysis by precipitation does not capture the total amount of associated asphaltenes in the binder as some fractions of associated asphaltenes remain in the resin fraction.

Table 20
Chemical composition of extracted binders

Mix Designation	SARA Analysis, %					DEC ASPH, %	HMW, %	DEC MALT, %	Jc, kJ/m^2
	Asphaltenes	Resins	Aromatics	Saturates	Sum resins, aromatics, saturates				
70CO	23.2	32.7	42.4	1.7	76.8	30.0	1.0	70.0	0.5
70PG5M	23.3	36.0	42.4	1.7	80.1	34.0	3.0	66.0	0.5
70PG5P	23.6	26.7	46.0	3.7	76.4	39.0	5.7	61.0	0.5
70PG5P5HG	27.6	30.0	39.3	3.0	72.3	41.0	8.5	59.0	0.3
70PG5P12CYCL	24.8	26.7	42.1	6.4	75.2	38.6	6.0	61.4	0.4
52PG5P	20.2	29.2	45.6	5.0	79.8	30.0	4.7	70.0	0.2
70PG5P20FLUX	25.6	22.8	44.2	7.5	74.5	35.3	6.0	64.8	0.3
70PG15RAP	22.1	30.7	45.3	1.9	77.9	41.8	8.3	58.2	0.6
70PG5P15RAP	23.5	30.3	43.5	2.7	76.5	46.4	5.5	53.7	0.5
70PG5PHG15RAP	29.8	28.4	36.9	5.0	70.3	44.7	5.1	55.4	0.4
52PG5P15RAP	22.9	29.8	44.1	3.2	77.1	33.2	4.0	66.8	0.3

DEC ASPH = deconvoluted asphaltenes;
HMW = high molecular weight;
DEC MALT = deconvoluted maltenes; and

J_c = critical strain energy release rate.

Figure 39 presents a correlation between the cracking resistance of asphalt mixtures expressed by critical strain energy release rate, J_c, and the SARA analysis derived asphaltenes contents of extracted binders from corresponding mixtures. For the mixtures with PG70-22m binder, a decent linear relationship (R^2 = 0.6) is observed: it is noteworthy that the mixture's cracking resistance is inversely proportional to the asphaltenes contents. On the other hand, mixtures prepared using PG52-28 exhibited no appreciable relationships between the J_c and asphaltenes contents. It can be also noted that the asphaltenes content was increased in 70PG5PHG15RAP compared to 70PG5P5HG, but the J_c was also increased, which is opposite from the general trend. This could be attributed to the fact that the RAP binder used in this mixtures contains approximately 2% polymer, which was beneficial in increasing the intermediate temperature cracking performance.

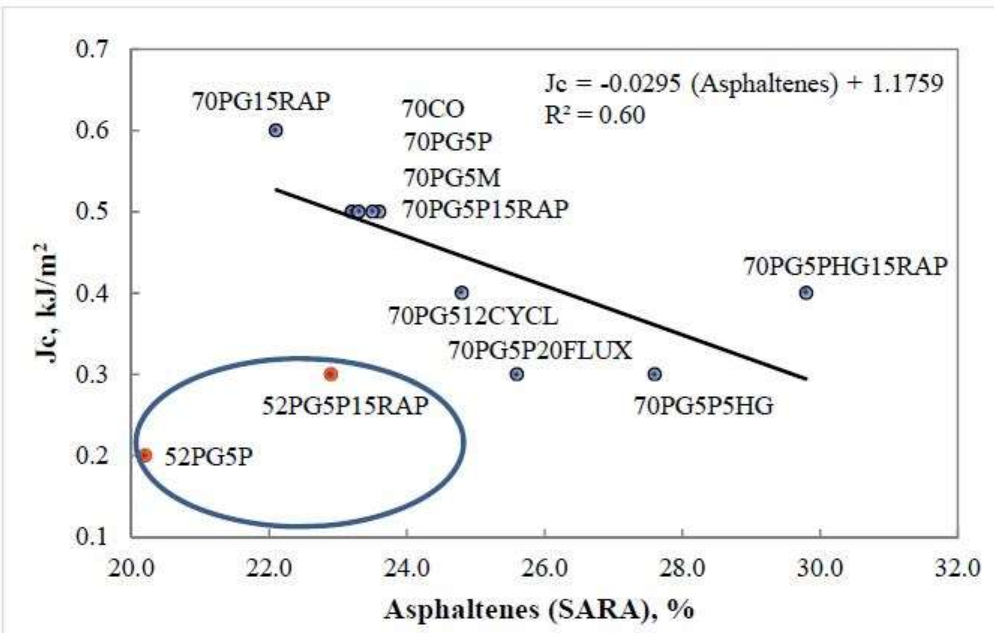


Figure 39
Mixture critical strain energy release rate (J_c) vs. asphaltenes content obtained from SARA fractionation

GPC Analysis Results

As presented in Figure 40 for a PG 64-22 binder, quantification of asphalt components is readily made by determination of area of the respective eluted fraction calculated based on the fact that the area under the curve represents 100% of the sample molecules injected into the GPC system. The total GPC curve can be deconvoluted to show the contributions of the asphalt components using commercially available software Origin 7.

Earlier determinations by osmometry indicated that the average MW of maltenes (as heptane soluble binder fraction) is 700-900 Daltons and that of asphaltenes (as heptane insoluble binder fraction) ranges between 2,000 and 10,000 Daltons *[100]*. These MW data have been confirmed by GPC method, which became a routine technique in Louisiana for analysis of asphalt binders *[101]*.

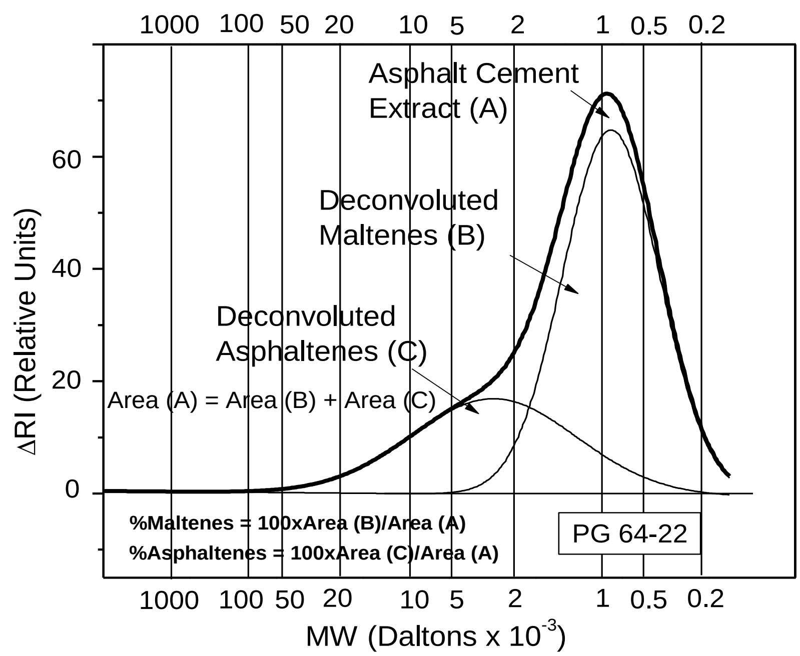


Figure 40
Maltenes and asphaltenes content of PG 64-22 binder by deconvolution of the GPC curve

Since the MW of polymers used in asphalt industry is higher than 10,000 Daltons, the polymer and asphalt components of polymer modified asphalt binders could be separated completely with accurate determination of molecular weight of species achieved by calibration with standard polystyrenes of narrow MW (Figure 41). It is noted that the GPC elution curve was from a mixture that was aged for 5 days at 85°C.

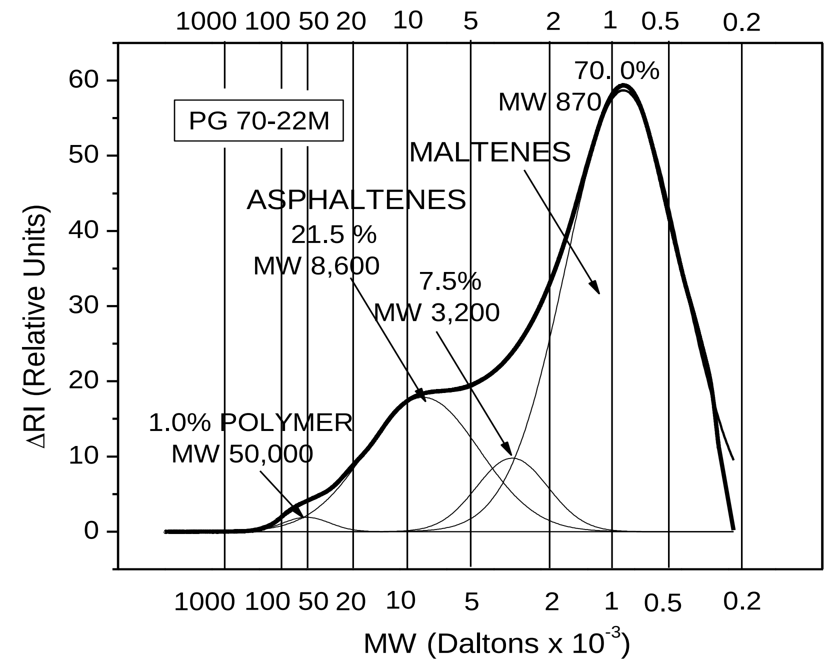


Figure 41
GPC elution curve of PG 70-22M containing 1% polymer extracted from an aged mixture

Asphaltenes, by virtue of their molecular size as the higher MW component, are the bodying agent for the maltenes, having a significant influence on asphalt performance *[101]*. The largest "molecules" are assemblies of smaller molecules held together by one or more intermolecular forces. Through changes in the polarity of the solvent used in the analysis, the ability of the samples to undergo self-assembly by different interactive mechanisms has been probed *[102]*. Therefore, by analyzing the asphaltenes in various asphalt mixture combinations, such as asphalt binders and polymer-modified binders, with and without reclaimed asphalt materials (RAP and RAS), one can correlate the performance of various asphalt mixtures to the content and MW magnitude of asphaltenes species.

The material used to manufacture roofing shingles is a highly oxidized blown asphalt as confirmed by the GPC chromatograms; the assemblies of asphaltenes species from blown asphalt and MWS are practically identical (Figure 42). In addition to oxidation, the blowing process increases the asphalt aromaticity (conjugation) and average molecular size, which improves opportunities for self-assembly. A bi- or tri-modal peak shape showing the presence of two or three distinct populations of molecular sizes is regarded as evidence of intermolecular association in the large molecular size (LMS) region on the left of the chromatogram *[103]*. Over 25% of associated asphaltenes in blown asphalt and MWS have an apparent average MW’s of 10K-50K Daltons. Asphalt binders extracted from MWS and post-consumer waste shingles (PCWS) typically have different properties because of the aging of the later that occurs on roofs. A further major concern with using recycled asphalt shingles relates to the variability in the properties of the RAS materials originating from different sources *[76]*, *[77]*.

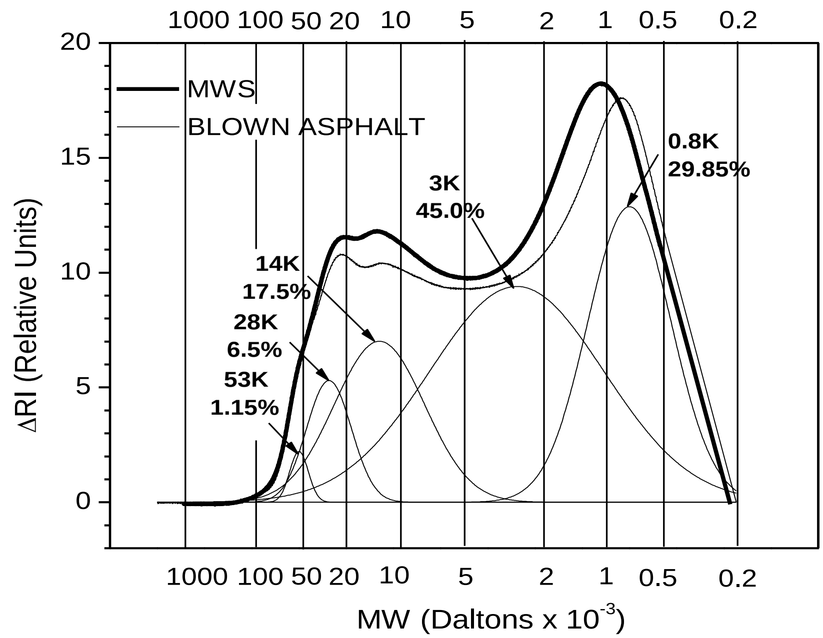


Figure 42
GPC data of blown asphalt and MWS extracted binders

The GPC traces of extracted binders from the Texas RAS used in this study is shown in Figure 43. Discernible differences between RAS sample are evident when one compares the maltenes component to the high end asphaltenes (MW>10K Daltons) component of the extracts. The ratio of the areas shown in the de-convoluted chromatograms identified possible problems with the compatibility of component species when blended with virgin asphalts. For example, the ratio for the binder extracted from RAP originating from the Texan source is ~55/33. In contrast, the corresponding ratio for a Minnesota PCWS extract is quite different, viz., ~80/20. The high molecular weight asphaltene content in the Texas PCWS suggests that this material was less compatible with virgin asphalt binders.

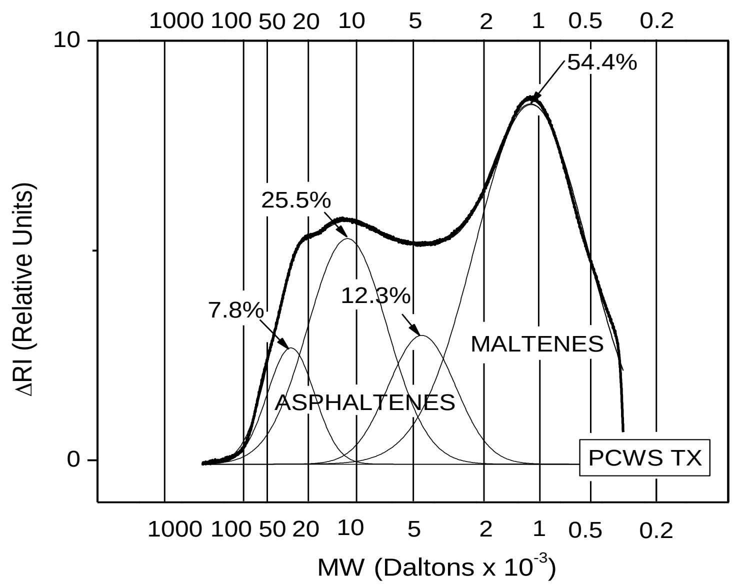


Figure 43
GPC traces of RAS binders extracted from PCWS of Texas origin

In Figure 44, the asphaltene fraction isolated by heptane precipitation of an asphalt binder extracted from Texas MWS was further examined. The MW of the asphaltenes components of MWS greatly surpasses that of a similar precipitation of a PG 64-22 binder in Figure 45. Asphaltenes from PG 64-22 can be separated into two fractions (i.e., molecules with an average MW of 2,000 Daltons (60%) and that with a peak average MW of 6,700 Daltons (22%)). The asphaltenes from MWS could be separated into three fractions with an average MW of 3,000 Daltons (52%), 12,000 Daltons (32%), and 24,500 Daltons (15%). Thus, the asphaltenes content of the MWS sample with molecular weights higher than that in PG 64-22 is approximately 47%.

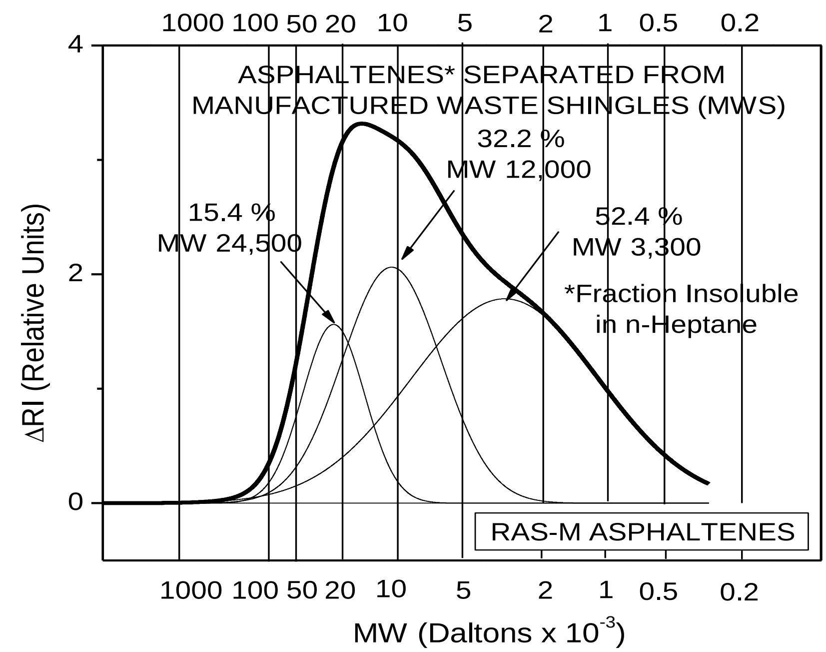


Figure 44
Average MW distributions of n-heptane insoluble asphaltenes species isolated from MWS

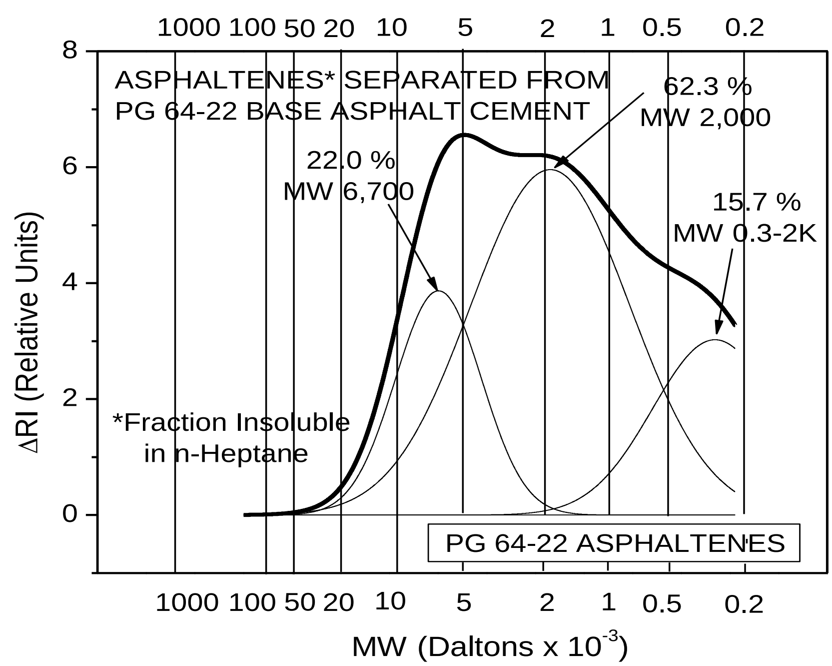


Figure 45
Average MW distributions of n-heptane insoluble asphaltenes species isolated from PG64-22 binder

The GPC chromatograms from an asphalt binder extracted from mixtures 70PG5P and 70PG5M, containing 5% PCWS and 5% MWS, are presented in Figures 46 and 47, respectively. The contribution of extremely oxidized components in PCWS is apparent in Figure 46. The 70PG5P mixture contained over 18% species with average MW>10K Daltons, out of which approximately 6% are of MW averaging 33,000. In contrast, a mixture prepared with 5% MWS, 70PG5M, contained only 3% of species with an average MW of 24,000 Daltons as shown in Figure 47. The maltenes to high end asphaltenes (MW>10K Daltons) ratio of 70PG5M (66/3) is significantly different from that of mixture 70PG5P (61/18), suggesting a higher potential compatibility of mixtures containing MWS.

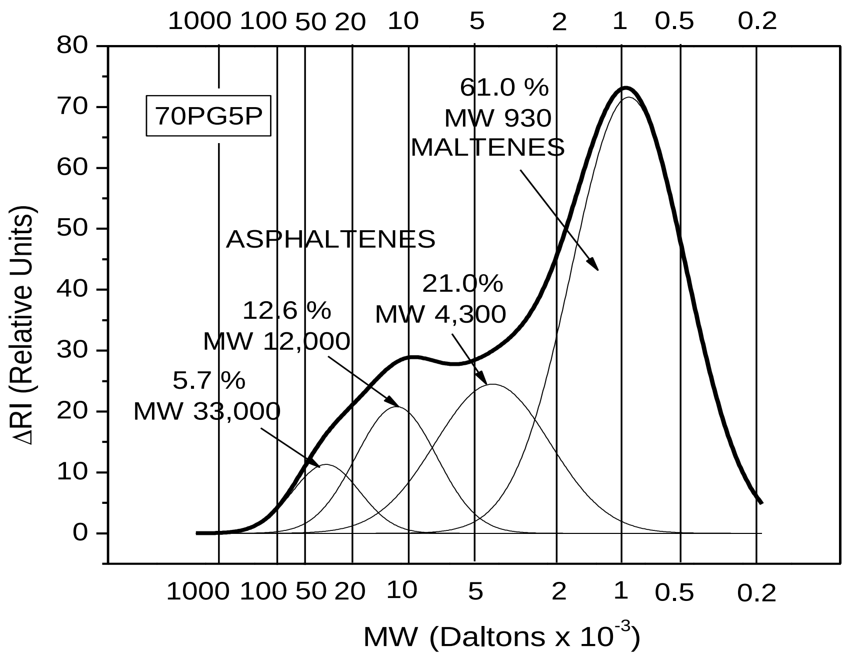


Figure 46
MW distribution of molecular species of extracted 70PG5P binder

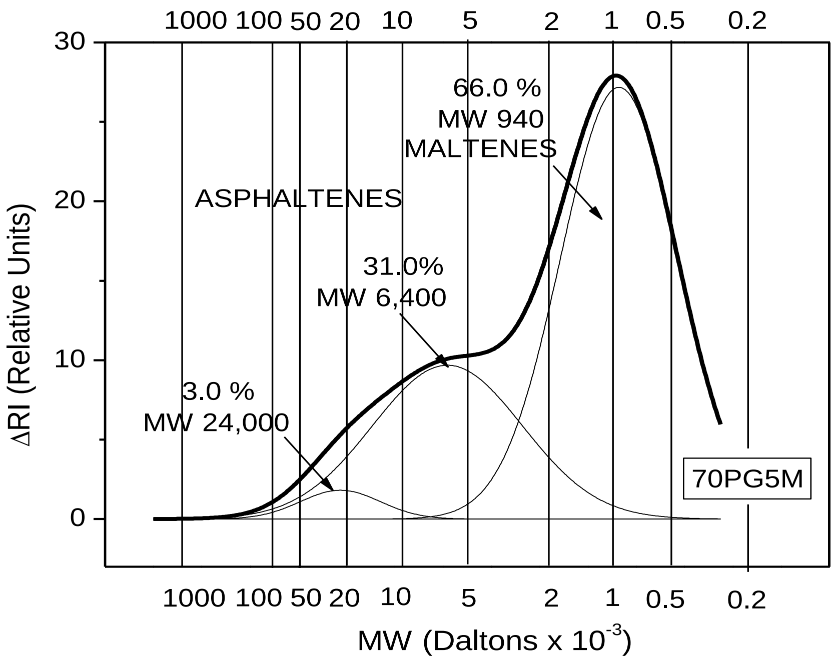


Figure 47
MW distribution of molecular species of extracted 70PG5M binder

According to Cooper, Jr., the high end MW of RAS asphaltenes can exceed even 100,000 Daltons *[87]*. The extremely large difference between MW's of associated asphaltenes from RAS and those from base binders when shingles are incorporated in paving asphalt materials impacts the compatibility of the mixes. It has been reported that some blending occurred between virgin PG 64-22 binder and RAS binder during the asphalt mixture mixing and curing (or short-term aging) processes *[100]*. The blending was not 100% since the high-temperature grades of the MWS and PCWS extracted binders, were 122°C and 166°C. The mixing temperature for a PG 64-22 binder is approximately 143°C (290°F). Zhou et al. stated that "extremely, impractically high temperature is required in order to make the RAS binder flow and comingle with virgin binder" whether using tear-off asphalt shingles or manufactured waste asphalt shingles *[94]*. Therefore, much higher blending temperatures are required in order to make the RAS binder flow and comingle with virgin binder. Moreover, earlier investigations on cross-blending of asphaltenes and maltenes fractions among several asphalts indicated that the asphaltene fractions are not as equally interchangeable as the maltenes components, and the effect of both molecular weight and the chemical nature of the asphaltenes must be taken into account to predict properties of asphalts from chemical composition *[103]*. Two types of segregation causing phase separations can occur when blending of asphalts containing dissimilar asphaltenes and maltenes fractions: one is simply that separation occurs in supersaturated solutions when components are ejected because of insufficient solvent (e.g., flocculation in blends of high asphaltenes content); the other is the rejection of a component when its amount exceeds the mutual compatibility limit, in the form of physical ejection of a liquid from a gel *[67]*. In view of these observations, the large difference between the MW of asphaltenes fractions of the base PG64-22 (ca. 20% of maximum MW ≈ 7-8,000) and the asphaltenes present in Waste Shingles (MWS and PCWS), ~ 40% of MW >12,000, with 15% MW ≈ 25-30,000 Daltons) makes difficult the dispersion of large RAS asphaltenes associations by the maltenes of the base asphalt with which the shingles was blended. To this aim, one must consider also the maltenes/high end asphaltenes (MW>10K Daltons) ratio mentioned above: the higher the ratio, the better. It has been shown earlier that an increase in the binder content of LMW (i.e., MW<3K), or in other words of the content ratio of Maltenes/Asphaltenes, resulted in an increase in its elongation properties at intermediate and low temperatures *[103]*.

Intermediate Temperature Cracking Performance of Asphalt Mixtures Containing RAS

Since the asphaltenes content in asphalts is related to the stiffness, the question arises: When blending virgin asphalt binder with RAS, will the asphaltenes content of the resulting binder follow the additive rule and will the J_c change correspondingly? The answer is no in most cases because the non-polar maltenes of the virgin asphalt are not compatible with the very highly oxidized RAS asphaltenes species. It has been shown that the virgin/RAS binder blending was nonlinear, unlike the well-known virgin–RAP binder linear blending *[94]*. PCWS binders were much stiffer than MWS binders *[100]*, *[98]*. Compared with PCWS, MWS binders had much less impact on properties of blended virgin/RAS binders *[9]*, *[1]*. Cooper et al. reported that the addition of 5% PCWS decreased the intermediate temperature cracking resistance (expressed by critical strain energy J_c) of a PG 70-22M binder when compared to that of a similar mixture in which PCWS has been substituted with MWS *[1]*.

Analyzing the GPC data obtained for the same materials (i.e., 70PG5P and 70PG5M) presented in Figures 46 and 47, respectively, it is considered that the main reason for higher stiffness of the 70PG5P PCWS binder reported by the authors is the degree of association of its large MW end asphaltenes (~ 6% of MW 33,000), which is higher than that found in the 70PG5M binder containing 5% MWS (3% MW 24,000 Daltons) *[1]*. It has been pointed out before that the ability of asphalts to form an intermolecular network by associations could lead to cracking with time and under cold conditions *[84]*.

Table 21 summarizes the data for a series of asphalt binders extracted from RAS containing mixtures. The asphaltene and maltenes fractions of 11 asphalt are the values of carbonyl index and of J_c integral in order to find a correlation between these data to predict, to a limited extent, the field performance of considered mixtures. Data listed in Table 21 show that rather large carbonyl indices and lower than 0.5 kJ/m^2 of J_c values were registered for mixtures containing PG 52-28 and PG 70-22M binders in which 3-6% asphalt species had MW > 19K (i.e., asphaltenes).

Table 21
MW distribution, C=O, and J_c values of materials utilized

Mixture Designation	VHMW (%) (Polymer & highly associated Asphaltene) 1000K - 19K	Asphaltene (%) 19K - 3K	Maltene (%) <3K	Carbonyl Index (C=O)	J_c (kJ/m^2)
Hydrogreen	0.00	0.88	99.12	4.0000	----
Cyclogen-L	0.00	0.00	100.00	0.0606	----
Asphalt Flux	0.00	0.00	100.00	0.0348	----
PG52-28	0.36	17.65	81.99	-0.0058	----
PG70-22M	3.15	22.85	74.00	0.0000	----
PCWS	15.56	32.04	52.40	0.2200	----
MWS	13.18	32.85	53.97	0.2500	----
RAP	2.06	27.83	70.11	0.1691	----
70PG5M	5.29	26.51	68.20	0.1202	0.5
70CO	3.57	25.00	71.43	0.0681	0.5
70PG5P	7.15	28.22	64.63	0.2124	0.5
70PG5P5HG	10.88	29.05	60.07	0.1713	0.3
70PG5PHG15RAP	13.07	29.84	57.09	0.2120	0.4
70PG5P12CYCL	9.08	27.35	63.57	0.1305	0.4
70PG5P20FLUX	8.56	29.23	62.21	0.1667	0.3
70PG15RAP	8.38	28.26	63.36	0.0950	0.6
70PG5P15RAP	10.50	30.23	59.27	0.1406	0.5
52PG5P	4.34	25.10	70.56	0.1174	0.2
52PG5P15RAP	5.81	28.56	65.63	0.1256	0.3

Figure 48 presents a comparison plot of the content of asphalt species with MW larger than 19K Daltons vs. J_c values of the 11 asphalt mixtures. Note that 19K Daltons of MW is the MW threshold of asphaltenes related to the stiffness of asphalt binders *[101]*. Higher J_c values are desirable for fracture-resistant mixtures. A minimum J_c value ranging from 0.50 to

0.65 kJ/m^2 is typically used as a failure criterion *[83]*. In Louisiana, a minimum J_c value of 0.5 kJ/m^2 is being considered as an acceptance criterion for mixture design.

Apparently, more than half of the 11 asphalt mixtures failed to meet the minimum design J_c requirement for Louisiana asphalt pavements. Among the five mixtures that showed greater than or equal to 0.5 kJ/m^2 of J_c value, four were on the borderline and only one asphalt mixture, i.e., 70PG15RAP, passed the requirement. In addition, it can be observed that all mixtures meeting or exceeding the minimum J_c criteria of 0.5 kJ/m^2 were mixtures that were produced with PG70-22M SBS modified asphalt binders and did not contain recycling agents.

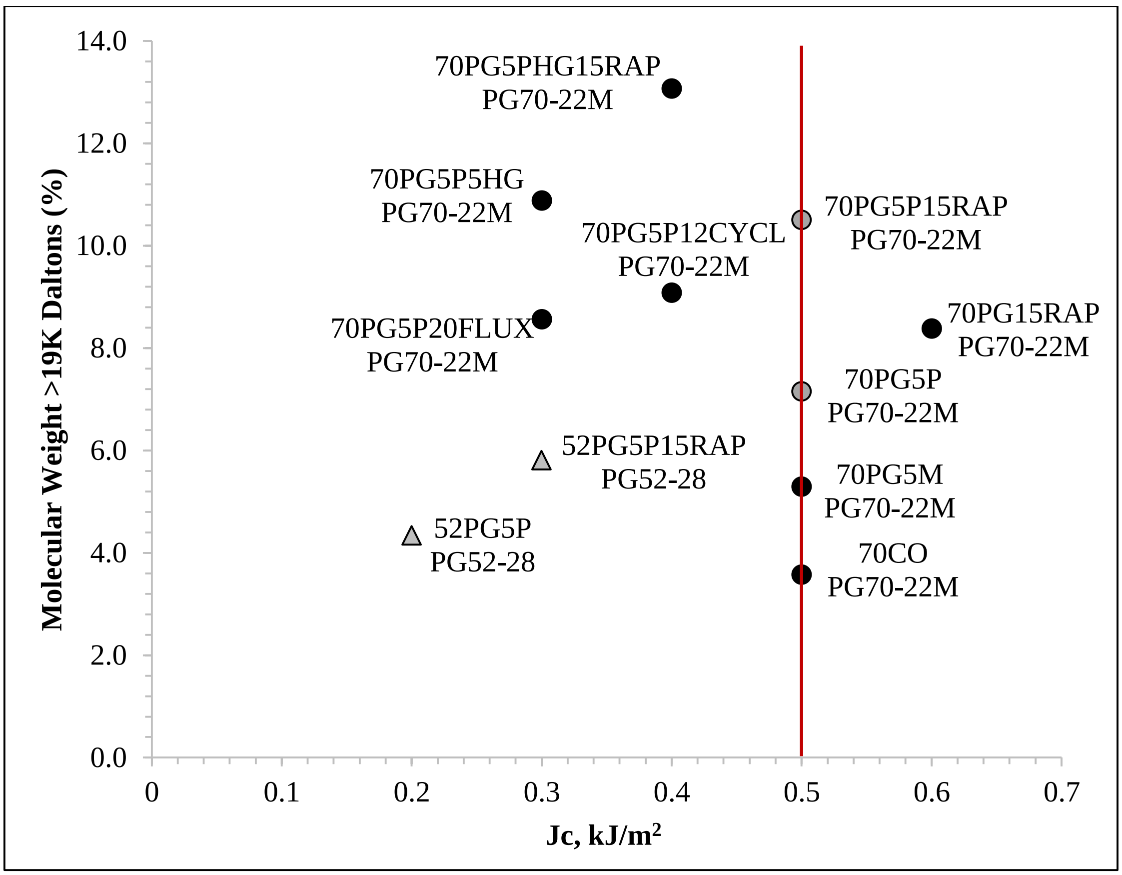


Figure 48
High MW fractions of 11 asphalt binders vs. corresponding J_c values

When comparing 52PG5P and 70PG5P, it looks clear that the high PG binder (i.e., PG70-22M) contains considerably more percentage of high MW fractions (7.15%) than the PG52-28 binder does (4.34%), which may have contributed to the higher fracture resistance of the 70PG5P mixture (0.5 kJ/m^2) than that of the 52PG5P mixture (0.2 kJ/m^2). This observation holds true when comparing 52PG5P15RAP and 70PG5P15RAP mixtures, i.e., 5.81% vs.

10.5% high MW fractions and 0.3 kJ/m^2 vs. 0.5 kJ/m^2 of J_c values for 52PG5P15RAP and 70PG5P15RAP, respectively. Generally speaking, polymers have very high MW and the SBS used in the PG70-22M binder is believed to be responsible for the observed increase in the high MW fractions of 70PG5P and 70PG5P15RAP compared to 52PG5P and 52PG5P15RAP, respectively. Thus, the increase in J_c of the two-70PG mixtures can be attributed to the SBS polymer modification, which is a direct evidence of the improved fracture resistance.

In a similar way, correlations can be found between various binders' high MW fractions and the intermediate temperature cracking resistance.

Intermediate Temperature Cracking Performance of Asphalt Mixtures Containing RAS and RAs

Figure 49 separates four asphalt mixtures (i.e., mixtures containing PG70-22M binder and 5% PCWS) from Figure 48 to see the effects of three RAs (i.e., Cyclogen, Hydrogreen-L, and Asphalt Flux) on the high MW fractions and J_c values clearer.

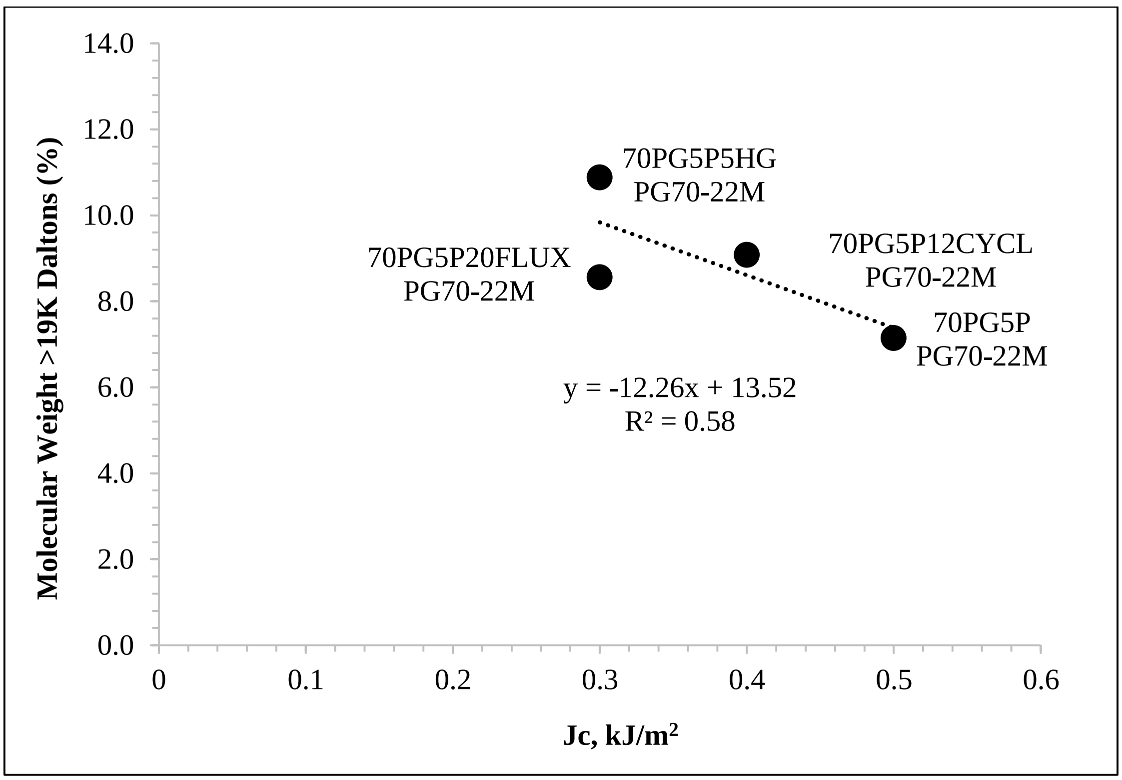


Figure 49
High MW contents for mixtures containing PG70 and 5P vs. J_c

A decent linear correlation can be observed between the high MW fractions and J_c values of these four asphalt mixtures. Interestingly, addition of the three RAs to the 70PG5P mixture increased the high MW fractions and decreased J_c values. Considering the anticipated benefits of these recycling agents, such as rejuvenating or softening of hardened asphalt binders, the trend is in the opposite direction.

Figure 50 illustrates the %MW asphalt fraction species >19K Daltons as it relates to the critical strain energy release rate, J_c, from mixtures containing PG70-22M asphalt binder, 5% PCWS, and 15% RAP. It is shown that there is a good correlation between the MW >19K Daltons and J_c. Figure 50 illustrates that as the MW >19K Daltons decrease the J_c increases.

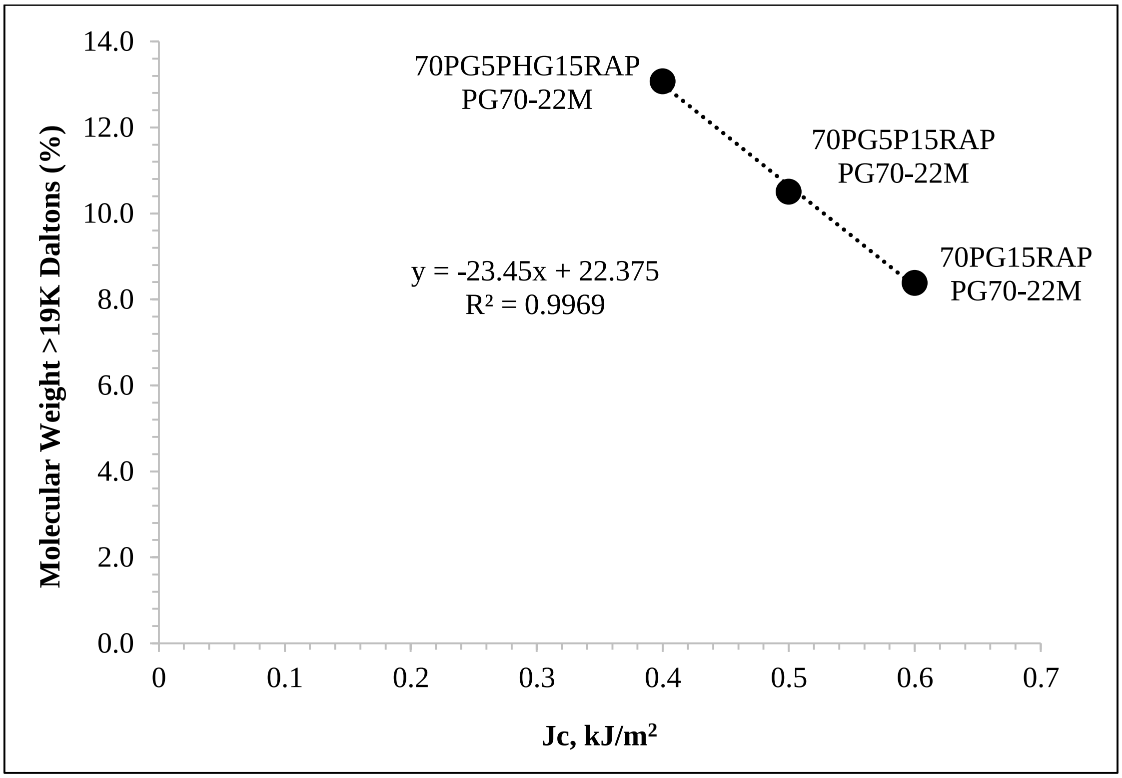


Figure 50
High MW contents for mixtures containing PG70, 5P, and 15RAP vs. J_c

One of the recycling agents chosen for the study was Hydrogreen (HG), which is an esterified derivative obtained from rosin, a by-product of the pulp and paper industry. This environmentally green rejuvenator is a low molecular weight product with the MW distribution shown in Figure 51. Its oxygen content is reflected by a significant carbonyl index (C=O = 0.04). Only 25% of its species matches the molecular weight of maltenes from an asphalt binder (MW ≈ 800-1500). The other recycling agents considered were naphthenic oil (Cyclogen-L), also a rejuvenator, and an asphalt binder meeting a PG 52-28, a softening agent. The anticipated role of the rejuvenators for RAS mixtures is to lower the association of high-end large MW asphaltenes present in RAS binders. However, the addition of 5% HG to a PG70-22M binder containing 5% PCWS (70PG5P5HG) does not seem to affect the distribution of high MW fractions derived from PCWS (Figure 51).

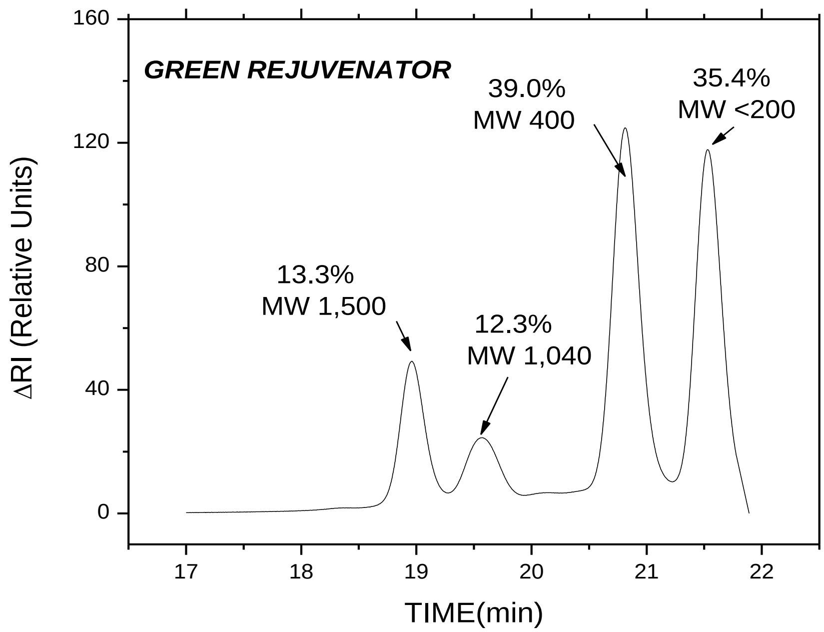


Figure 51
Molecular weight distribution of Hydrogreen green rejuvenator

At the same time, data listed in Table 22 and Figure 50 show that the addition of 5% HG to a PG70-22M binder containing 5% PCWS (70PG5P5HG) did not reduce the high MW asphaltenes and failed to retard the increase of high MW fraction due to the mixture aging for 5 days at 85ºC. This was clearly evident by the increase of asphaltenes from 9.59% to 11.50%, the corresponding reduction of the content of maltenes, and the increase in carbonyl index. These factors may have precluded the decrease of J_c from 0.37 to 0.26 kJ/m^2.

The blending and aging of 70PG/PCWS binders with other rejuvenating agents thought to improve the low temperature performance of the mixtures provided similar results. Adding softening agents instead of the Hydrogreen rejuvenator did not seem to alter the MW distribution of asphalt components (Table 22). GPC traces and MW distribution remained practically the same after SCB aging for both Cyclogen-L containing PG70-22M binder and the PG52-28 binder (Figures 52 and 53, respectively). While C=O index increased accordingly after aging, the cracking resistance expressed by critical strain energy, J_c, remained below the accepted limit (J_c<0.5 kJ/m^2), with decreased values for SCB aged mixtures, save for PG 70-22M containing 5% PCWS with 4.6% PG 52-28 by total weight of mix (Table 21).

Table 22
MW distribution, C=O, and J_c of related RAS binders containing RAs

Sample ID		Total VHMW (%) (Polymer &Highly Associated Asphaltenes) 1000K-19K	Total HMW (%) (Asphaltenes) 19K-3K	Maltene (%) < 3K	C=O $\times 10^2$	J_c (kJ/m^2)
70PG5P5HG	Unaged	9.59	28.06	62.34	13.9	0.37
	Aged 5days @85°C	11.50	29.06	59.43	17.1	0.26
70PG5P12CYCL	Unaged	8.30	25.98	65.38	9.9	0.13
	Aged 5days @85°C	8.77	27.17	64.06	13.0	0.36
52PG5P	Unaged	4.62	24.78	70.59	9.1	0.17
	Aged 5days @85°C	5.68	26.10	68.22	11.7	0.24

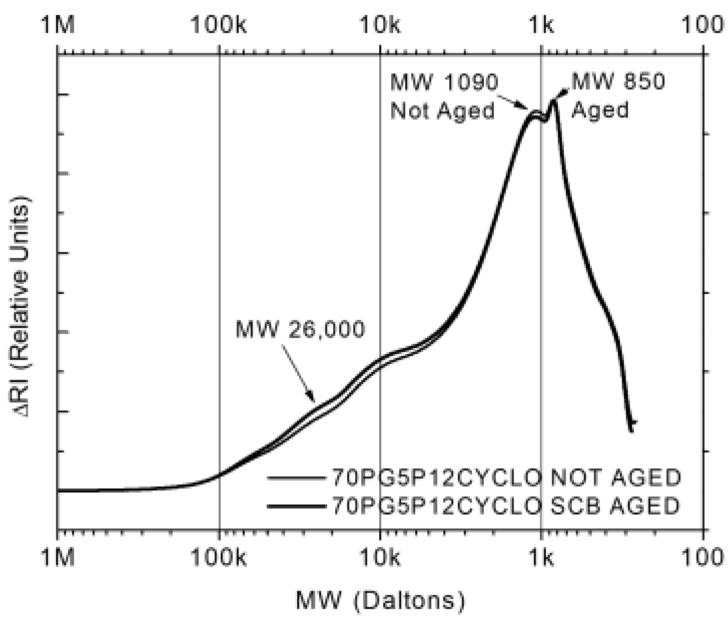


Figure 52
GPC traces of SCB aged and un-aged PG70-22M containing PCWS with Cyclogen-L

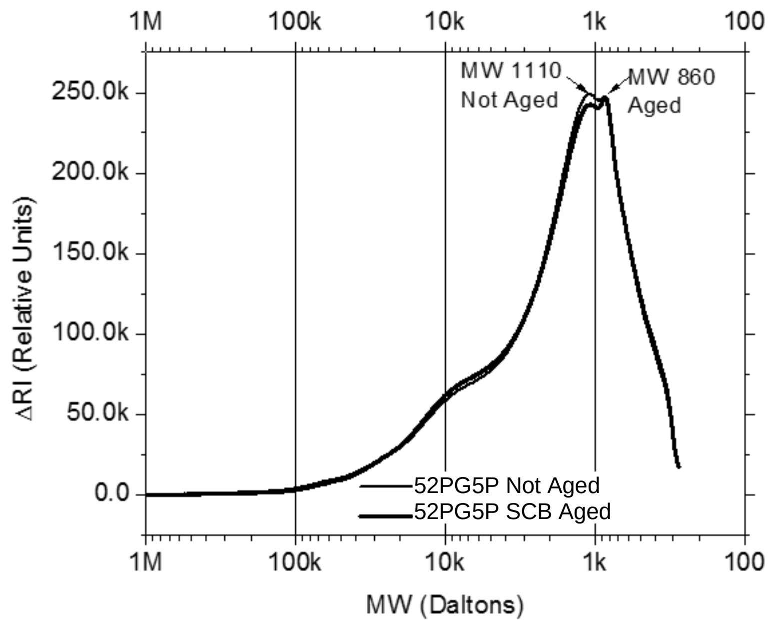


Figure 53
GPC traces of SCB aged and un-aged PG52-28 containing PCWS

Figure 54 presents the percentages of the HMW >3000 Daltons and LMW <3000 Daltons for the asphalt mixtures evaluated in this study. It is shown that the rejuvenating type recycling agents had the lowest percentage of HMW species and the highest percentages of LMW species followed by the neat asphalt binders, PG 52-28 and PG 70-22M. It is indicated that the PCWS followed by the MWS had the highest percentage of HMW species and the lowest percentages of LMW species. Figure 54 shows that as recycled materials (RAP and RAS) are added, regardless if a recycling agent is utilized in the asphalt mixture, there is an increase in the HMW species fraction and a decrease in the LMW species fraction. The increase in the HMW species fraction results in the mixtures being more brittle and more susceptible to fracture.

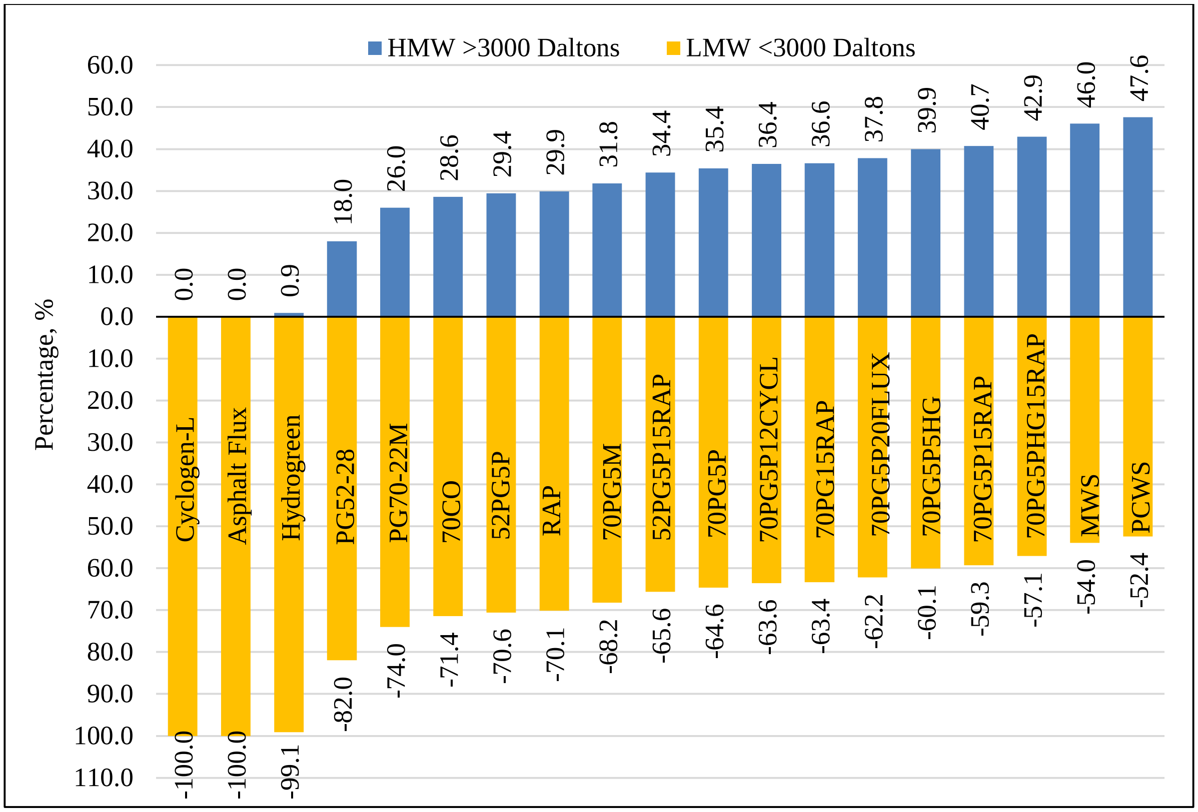


Figure 54
Percentages HMW >3000 daltons and LMW <3000 daltons per mixture type

Figure 55 shows the correlation between the Carbonyl Index, C=O, and the intermediate temperature performance parameter, J_c. The Carbonyl Index increases as an asphalt binder ages and increases in stiffness. As an asphalt binder oxidizes it becomes more brittle and stiffer. The complex shear modulus, G*, increases while the phase angle decreases. This results in a shift toward the loss modulus (plastic) and therefore, the binder is not as elastic before oxidation takes place. This figure indicates there is no correlation between the Carbonyl Index and J_c. However, it is noted that the trend shown is what would be expected. Figure 55 illustrates that as C=O increases the J_c decreases.

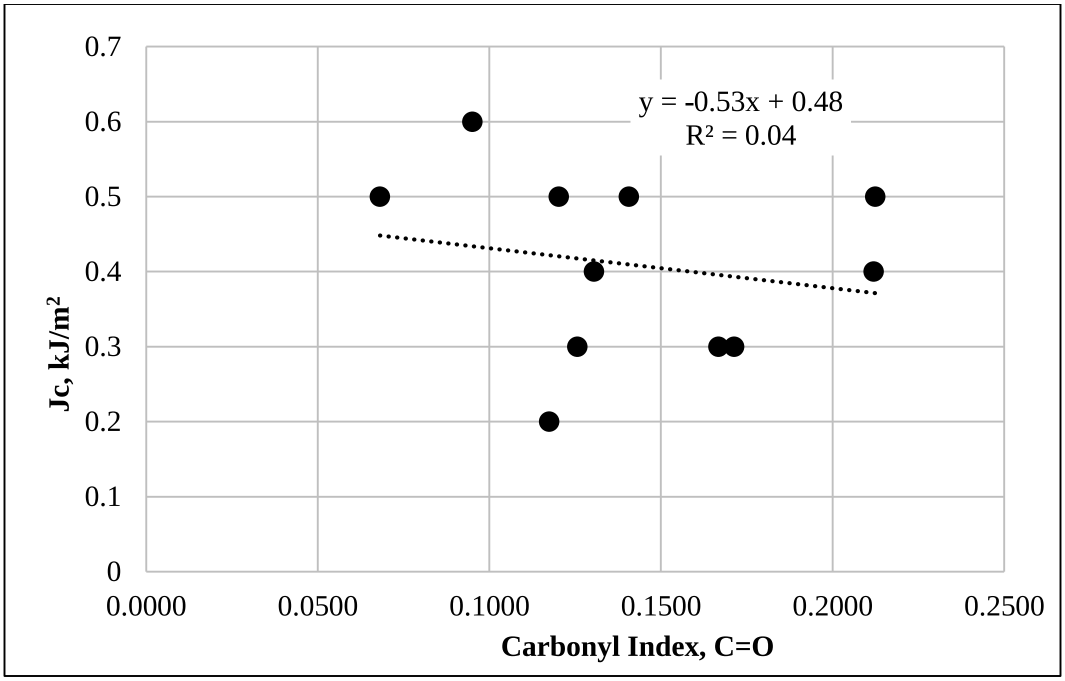


Figure 55
Carbonyl index correlation with J_c

Figure 56 indicates the carbonyl index as it relates to the critical strain energy release rate, J_c, from mixtures containing PG70-22M asphalt binder and 5% PCWS. It is shown that as the carbonyl index increases, the J_c increases. This is contrary to what is expected. It is anticipated that as the carbonyl increases, the J_c decreases. This expectation is because carbonyl index increases as an asphalt binder ages. As an asphalt binder ages the asphalt material becomes stiffer and brittle. This phenomenon results in a mixtures ability to resist intermediate temperature fracture.

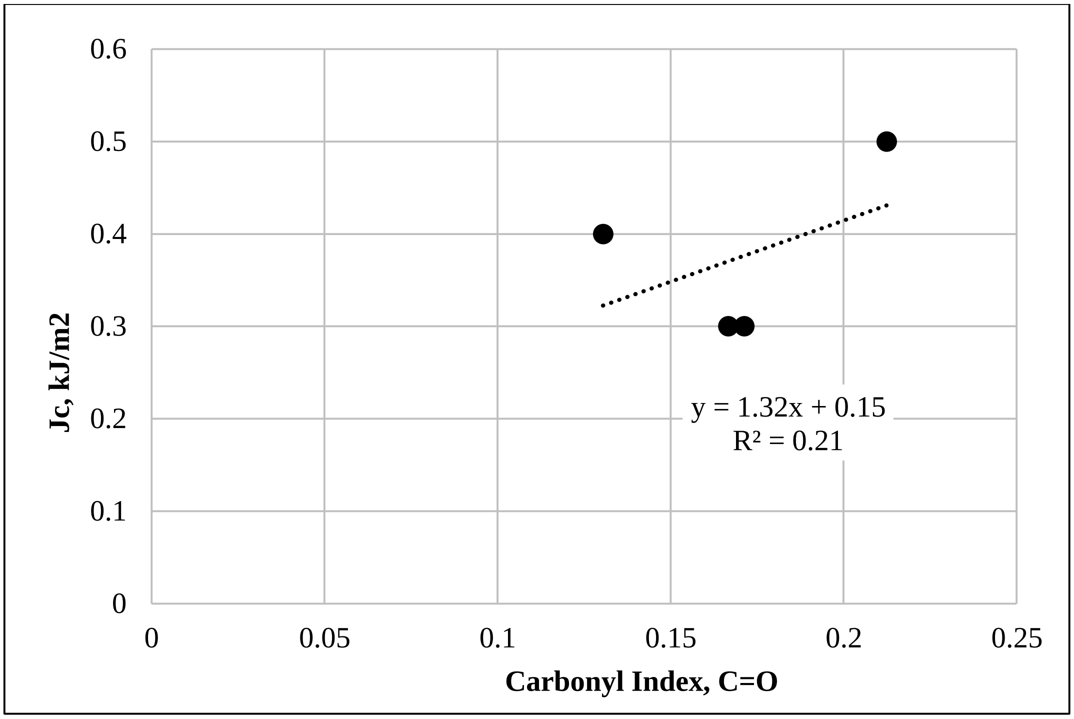


Figure 56
Carbonyl index correlation with J_c (mixtures containing PG70, and 5P)

Figure 57 illustrates the carbonyl index as it relates to the critical strain energy release rate, J_c, from mixtures containing PG70-22M asphalt binder, 5% PCWS, and 15% RAP. It is shown that there is a high correlation between the carbonyl index and the mixture J_c. Figure 57 indicates that as the carbonyl index increases the J_c decreases. This is the expected trend. When comparing Figure 56 and Figure 57, they indicate contrary results. Therefore, the use of carbonyl index as it relates to the critical strain energy release rate, J_c, is inconclusive for the mixtures evaluated. It is anticipated that the evaluation of additional mixtures should provide more conclusive results.

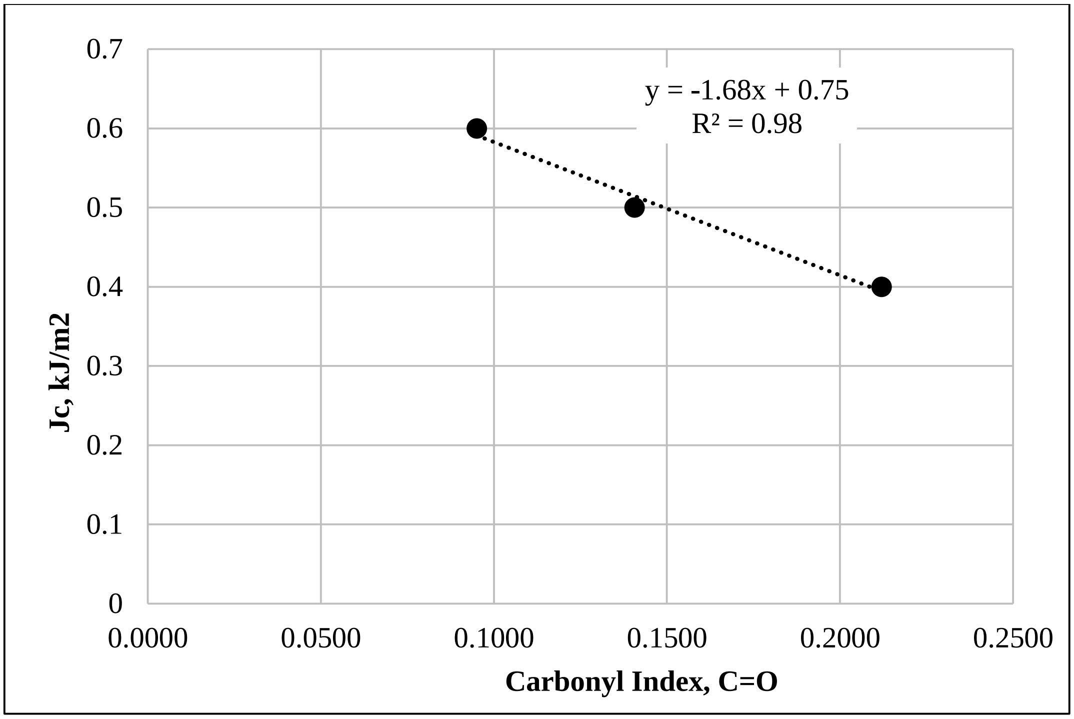


Figure 57
Carbonyl index correlation with J_c (mixtures containing PG70, 5P, and 15RAP)

Figure 58 indicates the GPC traces for mixtures 70PG15RAP, 70PG5P15RAP, and 70PG5PHG15RAP. Daly et al. indicated that the HMW threshold of larger than 19K Daltons relate to the stiffness of asphalt binders *[101]*. In comparing the molecular weights of mixtures 70PG5PHG15RAP and 70PG5P15RAP it is shown that mixture 70PG5PHG15RAP had 5.0% of its molecular structure greater than the 19K Dalton threshold. Whereas mixture 70PG5P15RAP had 1.3% greater than 19K Daltons. The corresponding J_c values for mixtures 70PG5PHG15RAP and 70PG5P15RAP were 0.4 and 0.5 kJ/m^2, respectively (Table 21). This would seem that the use of Hydrogreen did not improve the intermediate temperature performance even though Hydrogreen is considered a rejuvenator. It is however noted that the RBR for mixture 70PG5PHG15RAP is greater than mixture 70PG5P15RAP, 41.5 and 22.6 respectively (Table 15). In review of the MW at approximately 8000 Daltons as shown in Figure 58, it is indicated that mixture 70PG5P15RAP had the highest percentage of HMW species followed by 70PG5PHG15RAP and 70PG15RAP respectively. At the MW greater than 10K Daltons, it is shown that mixtures 70PG5PHG15RAP and 70PG15RAP have similar percentages. This would indicate that Hydrogreen did, in fact, rejuvenate portions of the higher molecular weight of RAS.

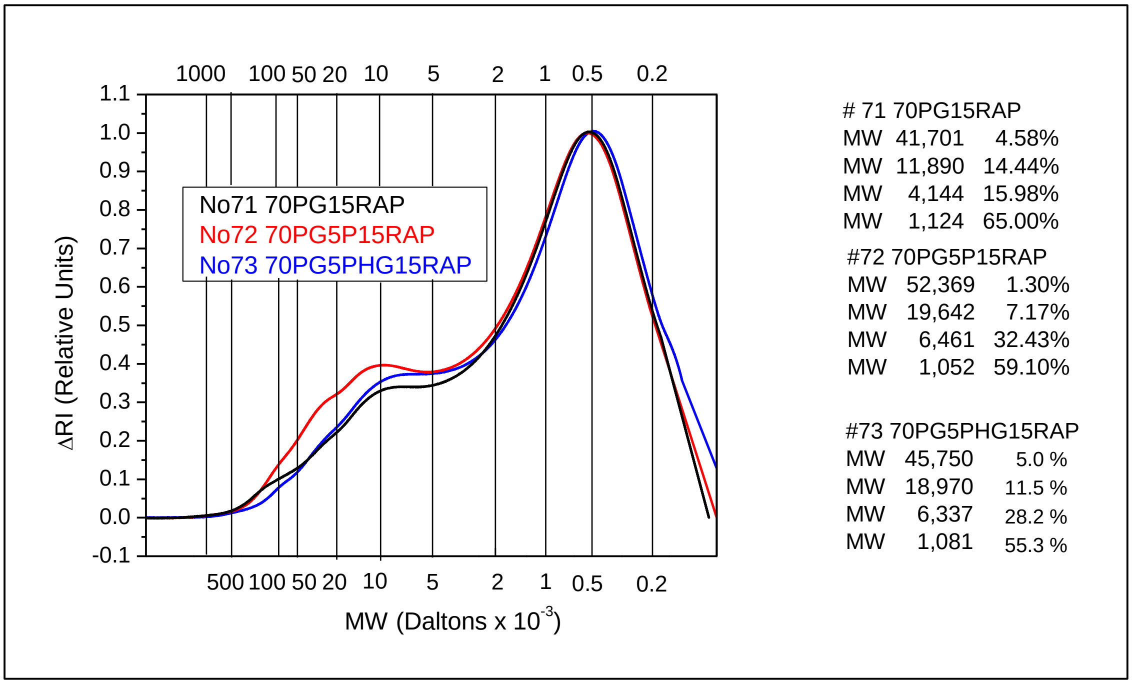
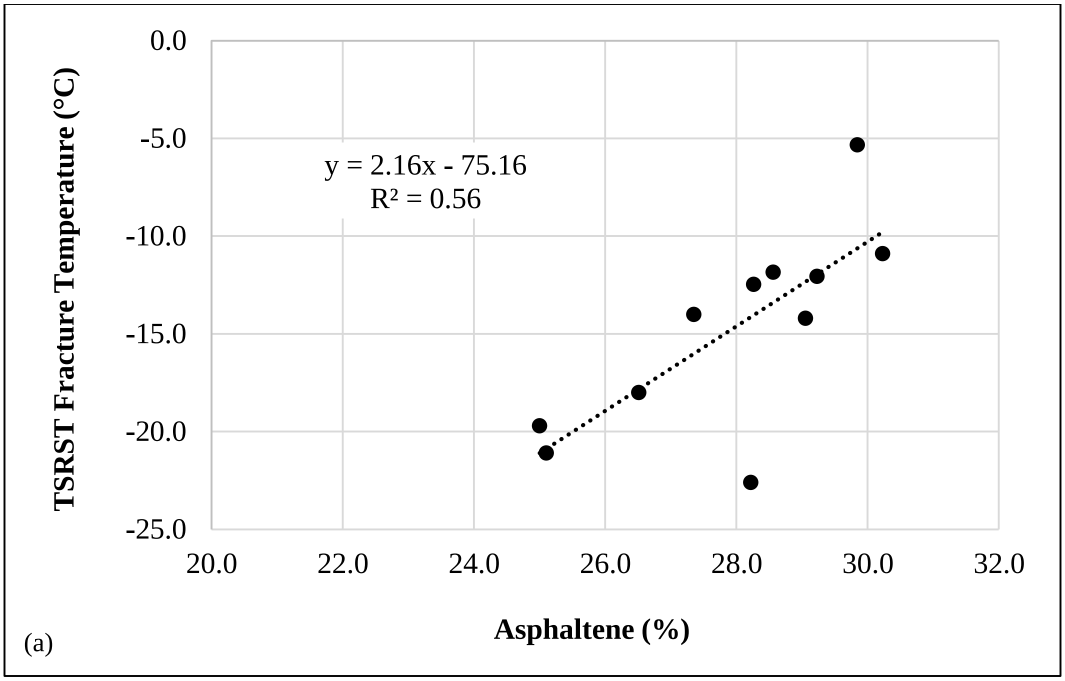


Figure 58
GPC traces of PG 70-22M containing RAP, RAS, RAP/RAS with or without HG

Low-Temperature Mixture Performance vs. Molecular Compositions

Figure 59 presents the relationships between the low-temperature cracking performance parameter and molecular composition of asphalt binders. It is generally indicated that as the asphaltenes content increases the TSRST fracture temperature rises and fracture work decreases. These trends are indicative of a more brittle asphalt mixture that would be more susceptible to low-temperature cracking development, Figures 59 (a) and (b). The superimposed linear trend lines show that the fracture temperature and fracture work done as measured and evaluated by TSRST have solid correlations with the asphaltenes content determined by GPC analysis, showing the R^2 values of 0.56 and 0.84, respectively. Similarly, correlations between each of the two mixture low temperature parameters (i.e., fracture temperature or fracture work) and various molecular compositional groups were determined. Table 23 summarizes the correlation analysis results.



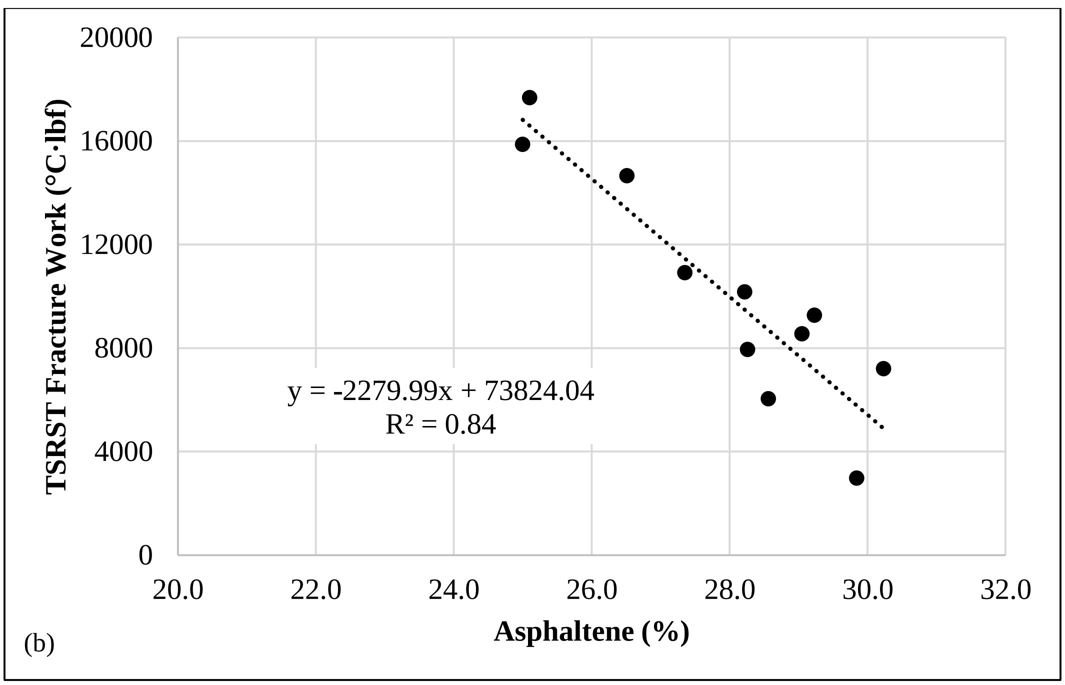


Figure 59
Mixtures' low-temperature cracking performance vs. %ssphaltenes: (a) fracture temperature and (b) fracture work

The total polymer content, asphaltenes, and maltenes determined from GPC analysis show good correlations with both fracture temperature and work done until fracture as compared to the four components (asphaltenes, resins, aromatics, and saturates) derived from SARA analysis. As discussed earlier, asphaltenes is generally regarded as the molecular component responsible for the stiffness and brittleness of asphalt binder and mixtures. Combining this result with an earlier observation from Table 14, it can be said that the addition of RAS and/or RAP into asphalt mixtures with and without RAs increases the formation of larger molecular weight species within the blended asphalt binder, which can considerably reduce the low-temperature cracking resistance of asphalt mixtures.

Table 23
R^2 values between dependent and independent variables

Dependent Variables	Independent Variables							
	Polymer (GPC)	Asphaltenes (GPC)	Maltenes (GPC)	Carbonyl Index	Asphaltenes (SARA)	Resins	Aromatics	Saturates
Fracture T (°C)	0.59	0.56	0.62	0.09	0.40	0.04	0.33	0.05
Work	0.64	0.84	0.77	0.39	0.40	0.11	0.17	0.02

SUMMARY AND CONCLUSIONS

Summary

The objective of this study was to assess the laboratory performance of conventional asphalt mixtures, mixtures containing RAP, RAS, RAP and RAS, with and without recycling agents (RAs), through laboratory measurements of mechanistic properties.

Comparative evaluations on the 11 asphalt mixtures were conducted, which include:

- a conventional Superpave asphalt mixture that contain PG 70-22M a, styrene-butadiene-styrene (SBS) modified asphalt binder, without RAS, RAP, and RAs as a control mixture;
- mixtures with PG70-22M binder vs. an unmodified PG 52-28 binder;
- mixtures with 5% MWS vs. with 5% PCWS;
- mixtures with 15% RAP vs. without RAP; and
- mixtures with RAs vs. without RAs.

A suite of laboratory mechanistic tests was conducted to characterize the high, intermediate, and low temperature properties for asphalt mixtures. Tests conducted include the dynamic modulus test for viscoelastic characterization, semi-circular bend (SCB) test for intermediate temperature fracture performance, and thermal stress restrained specimen tensile strength test (TSRST) for low temperature performance. A Hamburg type loaded wheel tracking (LWT) test was also performed to evaluate the mixtures' resistance to permanent deformation and moisture susceptibility. In addition, the molecular structure of asphalt binders of conventional asphalt mixtures as well as mixtures containing RAP, RAS, and both, with and without RAs were correlated with their intermediate and low temperature cracking potential using the SARA, GPC, and FTIR analyses results.

Conclusions

Based on the results of this study, the following conclusions can be drawn:

- With respect to the mixes without recycling agents, it is concluded that RAS binder does not fully blend with the virgin binder. The availability factor was found to range from 35 to 46%. Based on this fact, it was determined that the inclusion of RAS showed an improvement in rutting performance by resulting in a lower rut depth as compared to the control mixture without RAS. Further, because the RAS binder does not fully blend with the virgin binder, asphalt mixtures containing 5% recycled shingles showed no adverse effects to intermediate temperature properties (fatigue cracking) when compared to control mixture containing no RAS. In addition, the inclusion of 5% RAS did not adversely affect low temperature performance (thermal cracking) as compared to the control mixture. It was also determined that the addition of RAS did not adversely affect moisture susceptibility and no moisture susceptibility was predicted by LWT for the mixtures studied.

- In regards to the asphalt mixtures containing recycling agents, it was shown that RAS binder did blend with virgin binder when the mixtures were blended in accordance with the developed blending procedures. However, the availability factor was found to range from 50 to 100%. It was indicated that the addition of RAS with recycling agents generally showed an improvement in rutting performance by resulting in a lower rut depth as compared to the control mixture without RAS. The RAS mixture containing soft asphalt was the least resistant to permanent deformation. However, the inclusion of recycled shingles with recycling agents adversely affected the resistance to fracture at intermediate temperature even though the recycling agents are classified as rejuvenators. Further, the use of soft asphalt binders generally resulted in the least resistant to fracture at intermediate temperature. Also, RAS mixtures containing recycling agents adversely affected low temperature performance. It was also determined that asphalt mixtures containing RAS and recycling agents did not adversely affect moisture susceptibility and no moisture susceptibility were predicted by the LWT for the mixtures studied.
- In reference to the binder fractionation of the extracted asphalt binders from RAP/RAS mixtures with and without recycling agents, it was concluded that there were higher concentrations of high molecular weight species in the RAS binders as compared to the RAP binders. The concentration of the high molecular RAS species exceeds 40% in which 25% of these are highly aggregated with apparent molecular weights approaching 100K. In addition, the use of rejuvenating agents did not reduce the concentration of the very high molecular weight associated species, and thus they failed to improve the cracking resistance of the asphalt mixtures evaluated in this study. Also, it was shown that RAS is much more highly oxidized than RAP as indicated by FTIR spectroscopy. In addition, a relationship between the carbonyl index and fracture at intermediate temperature is inconclusive for the mixtures studied.
- The percentage of asphaltenes species fractionated from the SARA analysis was slightly less than that determined from the GPC analysis. The SARA asphaltenes analysis by precipitation did not capture the total amount of associated asphaltenes in the binder as measured by GPC. Some associated asphaltenes may remain in the resin fraction which is not captured by SARA analysis. The fracture temperature and fracture work measured by TSRST have good correlations with asphaltenes contents determined by GPC analysis. Similarly, the low-temperature cracking performance parameters have good correlations

with GPC determined polymer and maltenes contents, but showed considerably weaker correlations with SARA fractionated species. The addition of RAS and/or RAP, with and without RAs apparently increase the larger molecular weight species in the blended asphalt binder. In turn, the increased amount of larger molecular weight species adversely impacts the low-temperature fracture properties of asphalt mixtures and increase the likelihood of cracking in the asphalt pavement.

RECOMMENDATIONS

Based on the results, it is recommended that chemical analyses be performed on the asphalt binders containing recycled materials. It is recommended that SARA (Saturate, Aromatic, Resin and Asphaltenes) analysis be conducted to compliment GPC. While GPC has the capabilities to determine the molecular weight of species within a specimen, SARA analysis will divide the asphalt binder components according to polarity and polarizability. This will lend to a better understanding of the effects of RAS, RAP, and recycling agents on asphalt mixtures.

It is recommended that specifications for inclusion of RAS into mixtures be developed and experimental field projects be constructed. In doing so, the developed laboratory mixture design blending procedure can be validated. Also, asphalt mixtures from these field projects can be characterized to determine the effects of RAS on the high, intermediate, and low-temperature mixture properties. Furthermore, binder fractionation by molecular weight can be conducted on the extracted binders to further our understanding of the effects of RAS on mixture performance.

Also, it is recommended that an ALF (Accelerated Loading Facility) project be constructed. This will enable the evaluation of actual cracking and rutting under accelerated loading of mixtures containing RAS.

ACRONYMS, ABBREVIATIONS, AND SYMBOLS

ΔTc	Change in Critical Temperature
AASHTO	American Association of State Highway and Transportation Officials
BBR	Bending Beam Rheometer
C=O	Carbonyl Index
CAA	Coarse Aggregate Angularity
$\|E^*\|$	Dynamic Modulus
DOTD	Louisiana Department of Transportation and Development
DSR	Dynamic Shear Rheometer
FAA	Fine Aggregate Angularity
F&E	Flat and Elongated
FHWA	Federal Highway Administration
FTIR	Fourier Transform Infrared Spectroscopy
G*	Complex Shear Modulus
GPC	Gel Permeation Chromatography
G_{mb}	Bulk Specific Gravity of Mixture
G_{mm}	Maximum Specific Gravity of Mixture
G_{sb}	Bulk Specific Gravity of Aggregate
Gse	Effective Specific Gravity
Hz	Hertz
in.	Inch
ITS	Indirect Tensile Strength
J_c	Critical Strain Energy Release Rate
kJ/m^2	kilo-Joule per meter square
kPa	kilo Pascal
ksi	Kips per Square Inch
LAS	Linear Amplitude Sweep
LWT	Loaded Wheel Tracking
min	Minutes
MWS	Manufactured Waste Shingles
MPa	mega Pascal
mm	Millimeter
MSCR	Multiple Stress Creep Recovery
MW	Molecular Weight
NMAS	Nominal Maximum Aggregate Size
J_{nr}	Non-recoverable Creep Compliance

N_f	Number of Cycles to Failure
Pa·s	Pascal Second
%	Percent
PAV	Pressure Aging Vessel
PCWS	Post-consumer Waste Shingles
PG	Performance Grade
RA	Recycling Agents
RAP	Reclaimed Asphalt Pavement
RAS	Recycled Asphalt Shingles
RTFO	Rolling Thin-Film Oven
SARA	Saturate, Aromatic, Resin, and Asphaltene
SE	Sand Equivalency
sec.	Second
Tc(m)	Critical Low Temperature – m-value
Tc(S)	Critical Stiffness Temperature
TSR	Tensile Strength Ratio
TSRST	Thermal Stress Restrained Specimen Tensile Strength Test
U.S.	United States
Va	Air Voids
VFA	Voids Filled with Asphalt
VMA	Voids in the Mineral Aggregate

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APPENDIX

GPC Curves

COMPARATIVE DATA

71 70PG15RAP

MW	16,961	8.30%
MW	5,066	33.52%
MW	1,069	58.18%

#72 70PG5P15RAP

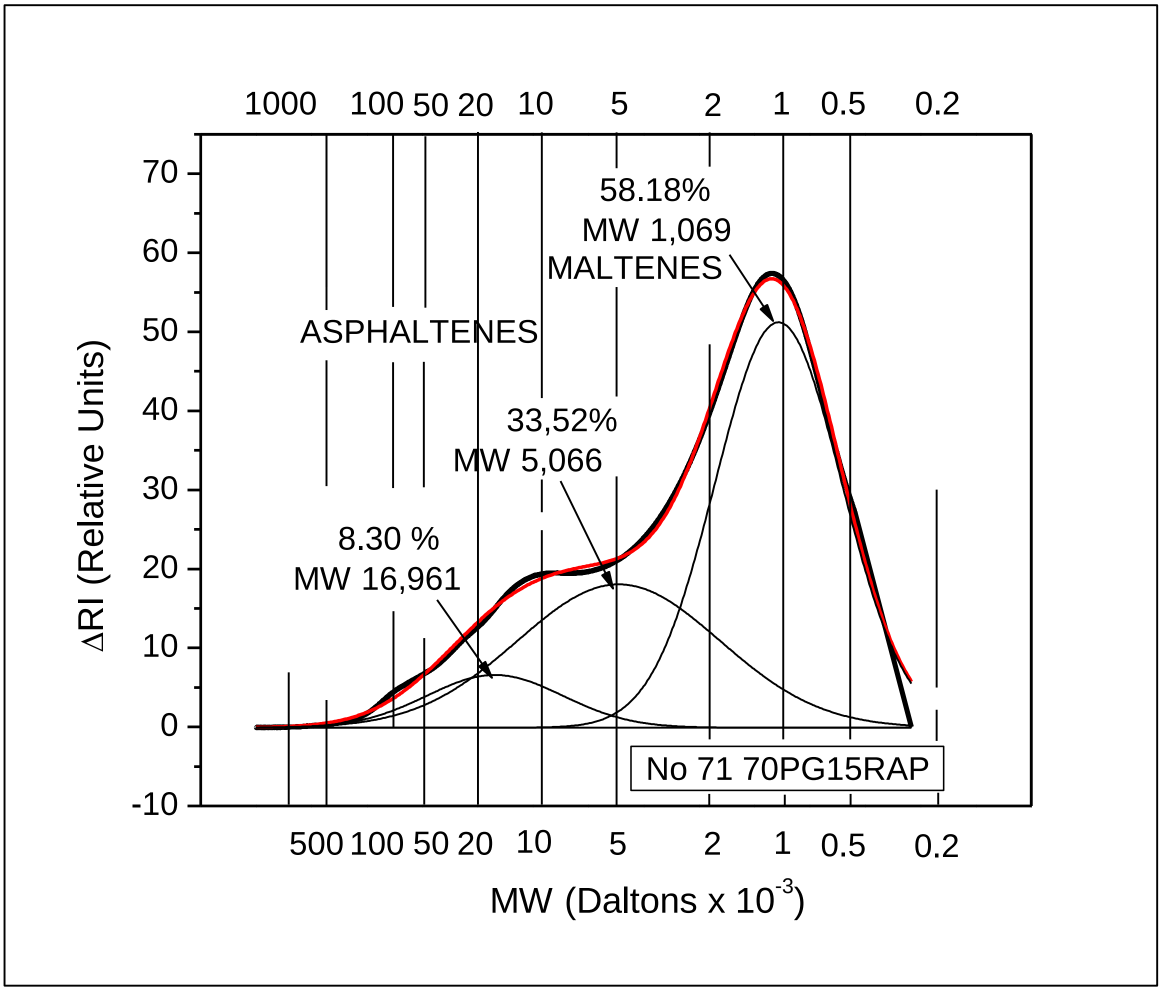
MW	32,571	5.70%
MW	12,945	7.17%
MW	3,691	41.88%
MW	1,052	45.25%

#73 70PG5PHG15RAP

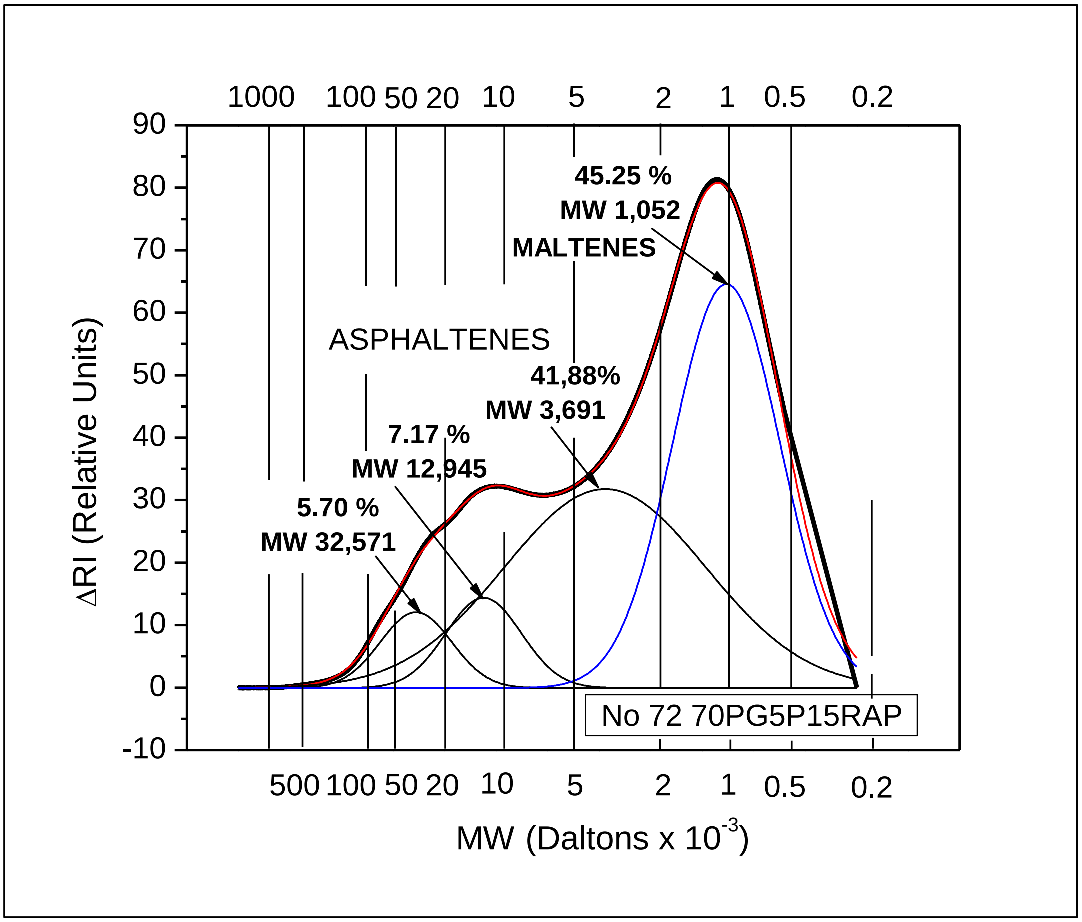
MW	45,750	5.0%
MW	18,970	11.5%
MW	6,337	28.2%
MW	1,081	55.3%

#74 52PG5P15RAP

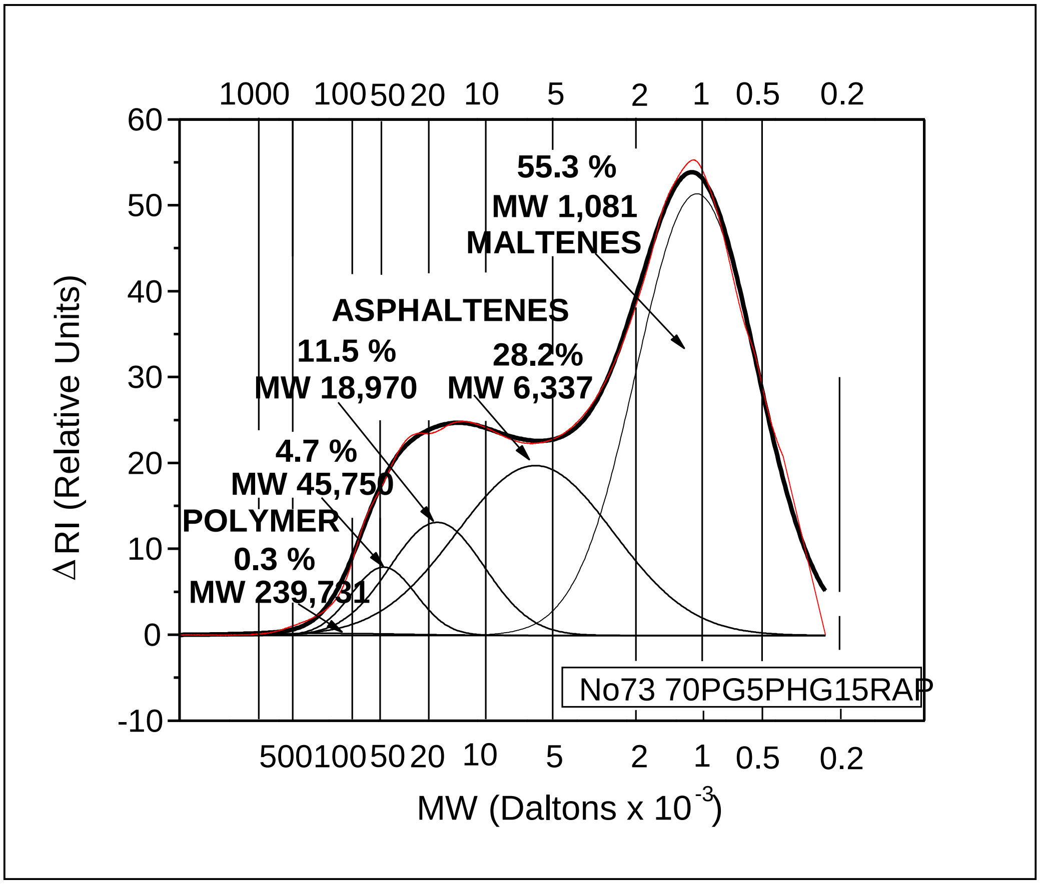
MW	93,814	0.06%
MW	21,137	3.85%
MW	7,815	29.27%
MW	1,069	66.82%



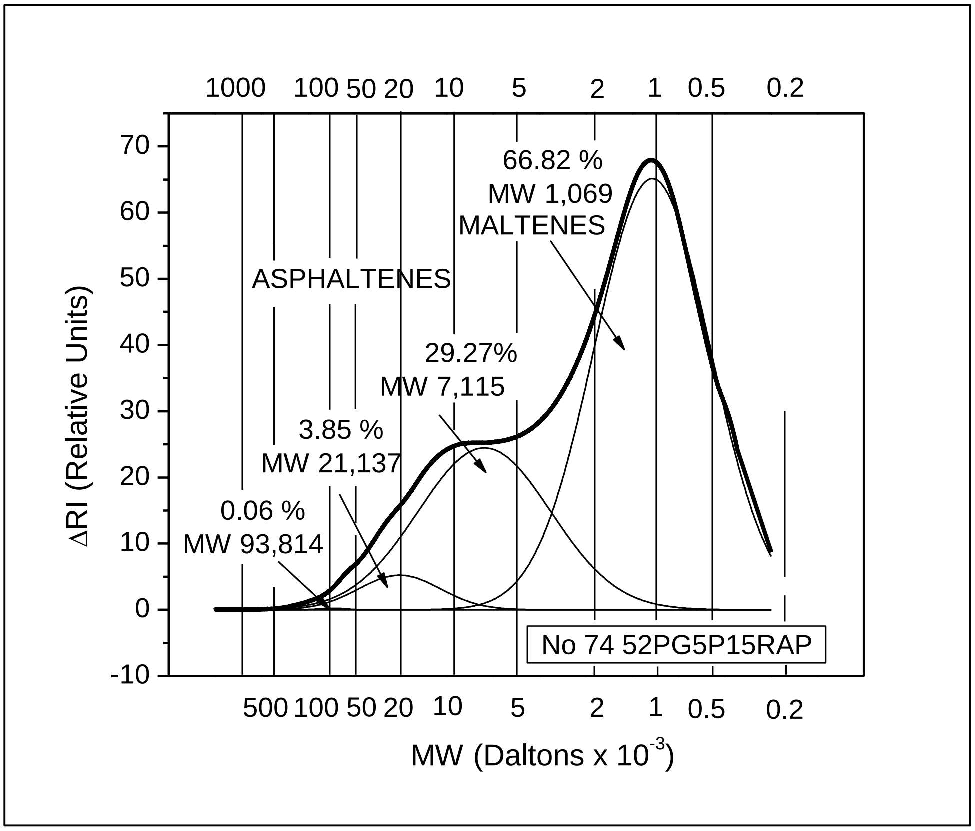
<u># 71 70PG15RAP</u>
MW 16,961 8.30%
MW 5,066 33.52%
MW 1,069 58.18%



#72 70PG5P15RAP		
MW	32,571	5.70%
MW	12,945	7.17%
MW	3,691	41.88%
MW	1,052	45.25%



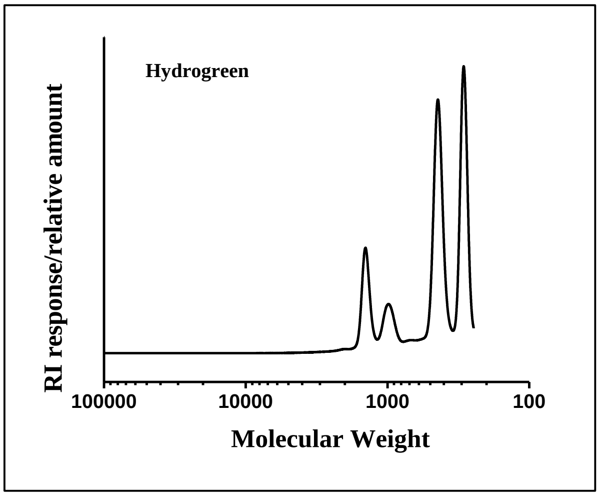
#73 70PG5PHG15RAP		
MW	45,750	5.0%
MW	18,970	11.5%
MW	6,337	28.2%
MW	1,081	55.3%



#74 <u>52PG5P15RAP</u>

MW 93,814 0.06%
MW 21 ,137 3.85%
MW 7,115 29.27%
MW 1,069 66.82%

#74 <u>52PG5P15RAP</u>	#72 <u>70PG5P15RAP</u>
MW 93,814 0.06%	--------
MW 21,137 3.85%	MW 32,571 5.70%
MW 7,815 29.27%	MW 12,945 7.17%
------------	MW 3,691 41.88%
MW 1,069 66.82%	MW 1,052 45.25%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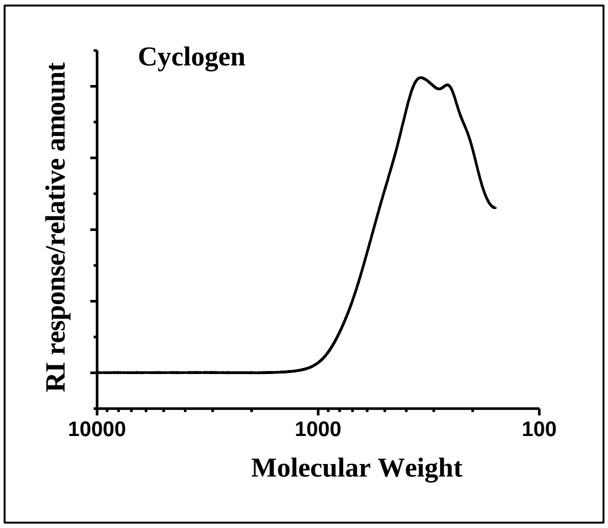
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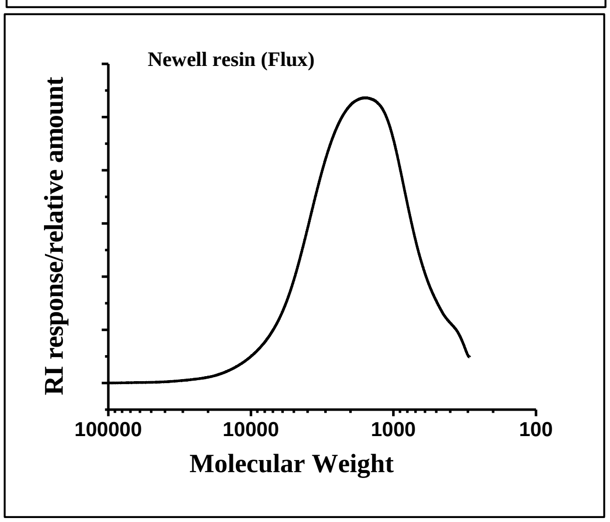
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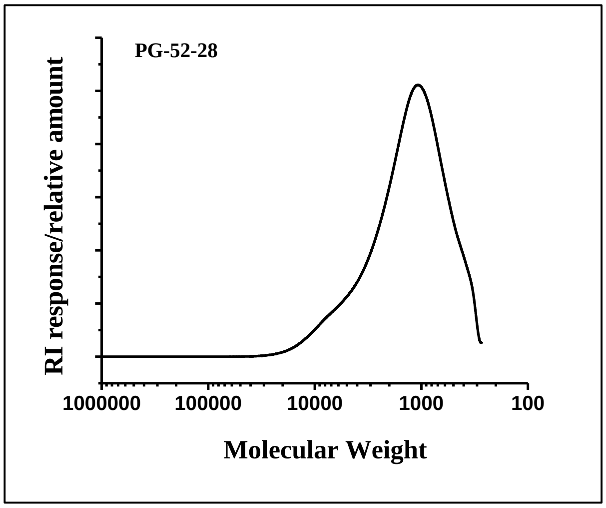
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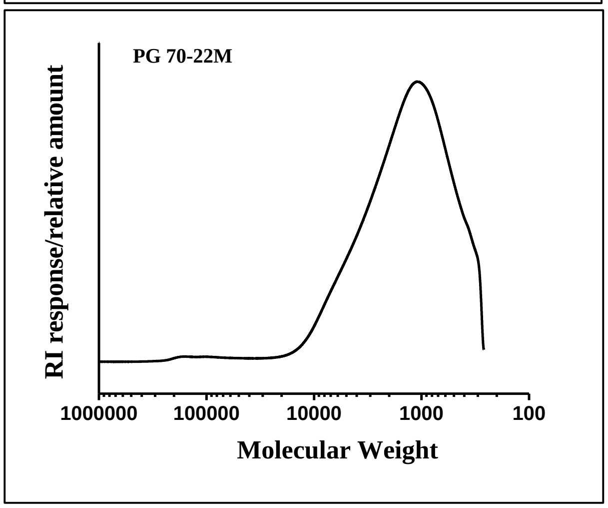
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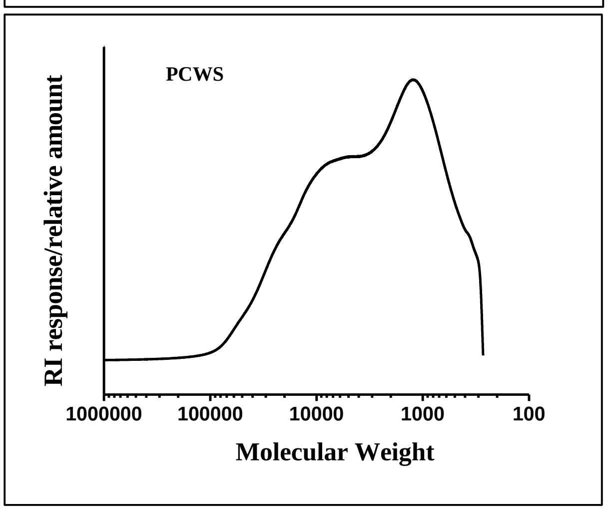
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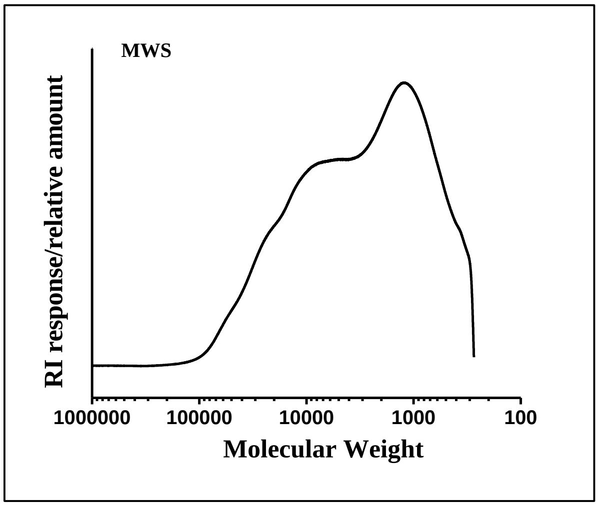
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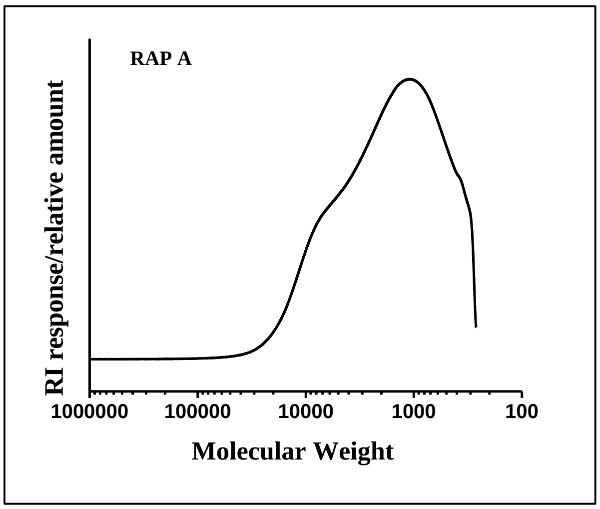
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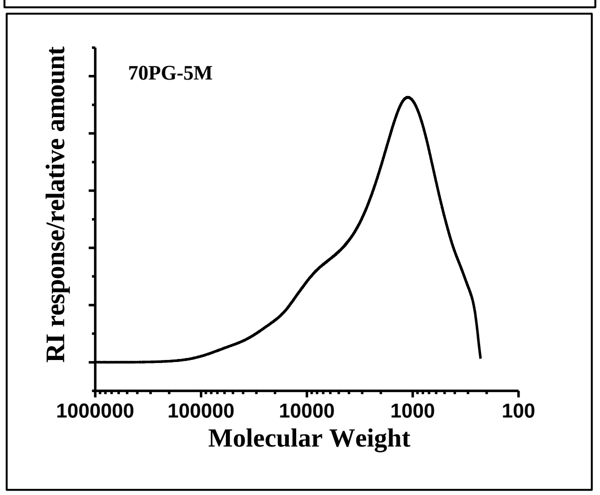
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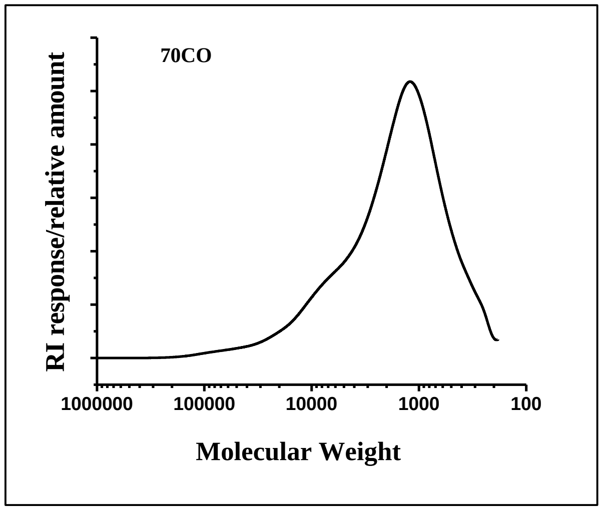
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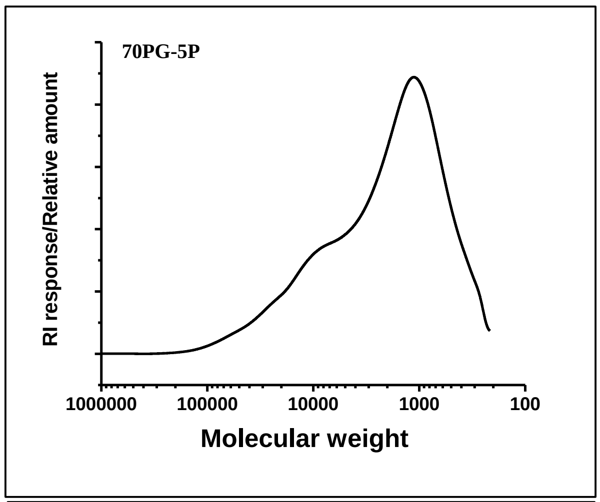
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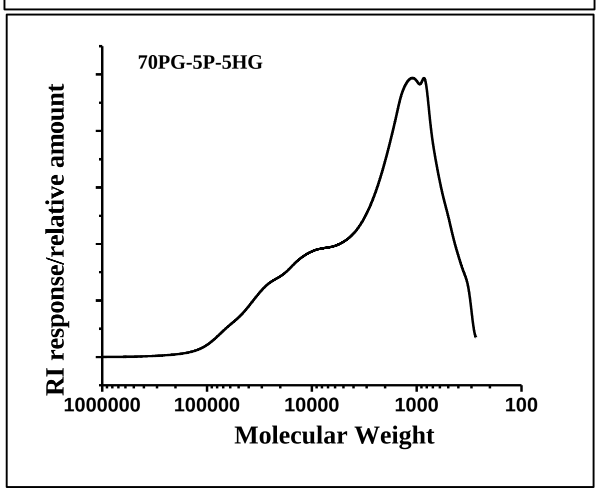
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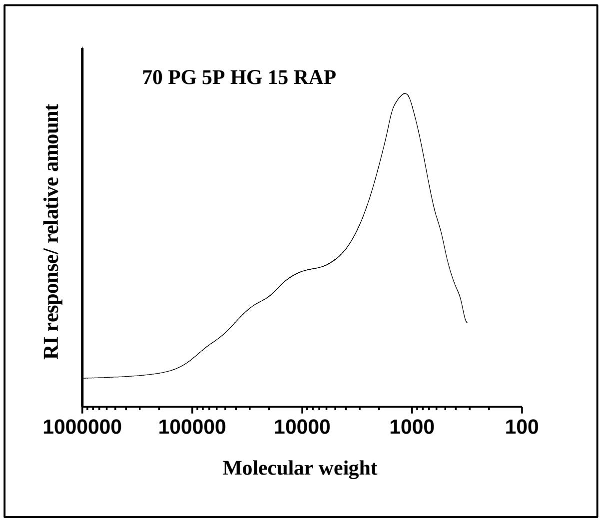
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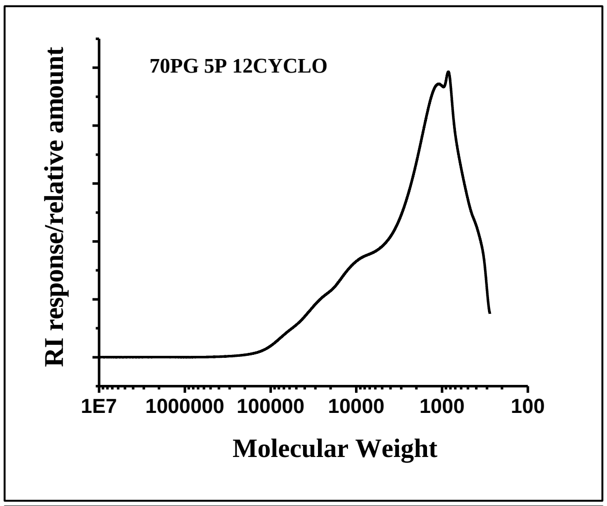
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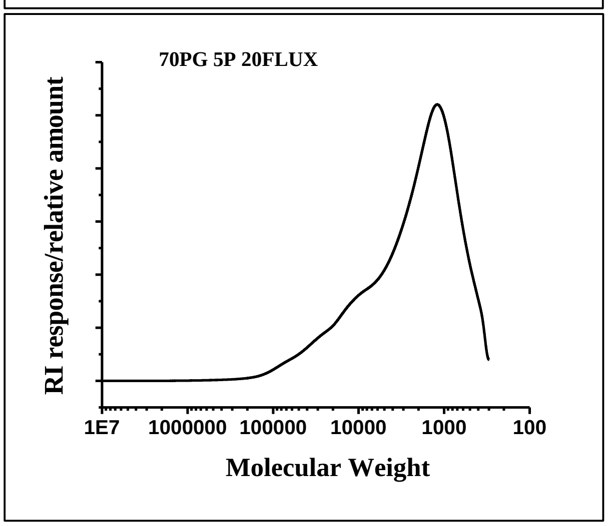
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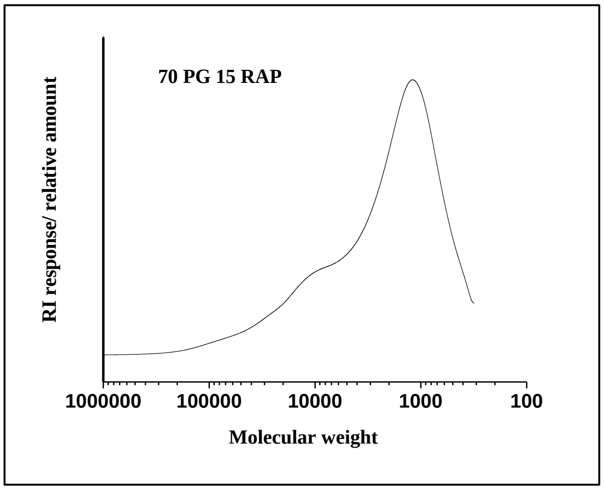
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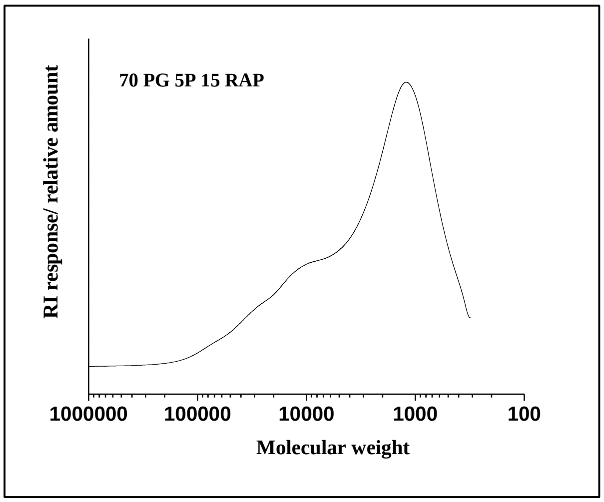
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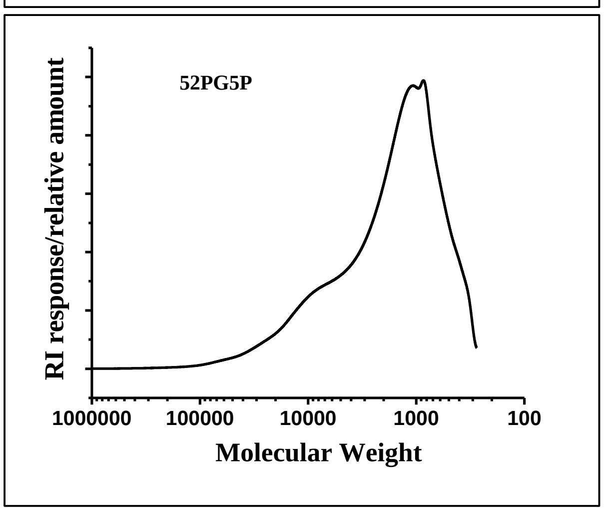
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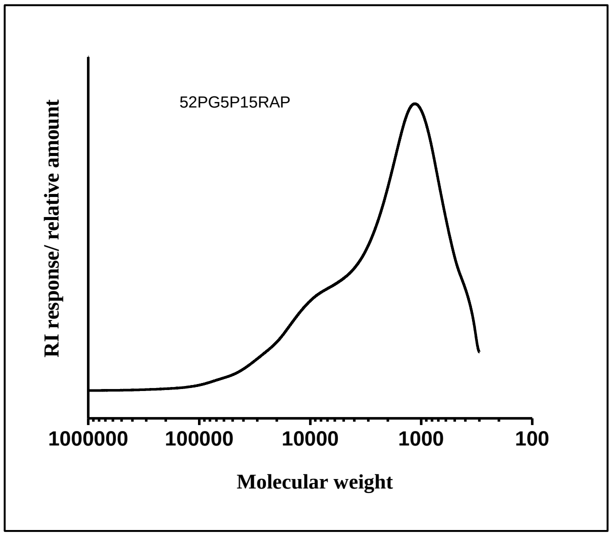
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Molecular weight

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