EFFECT OF REJUVENATORS ON

RHEOLOGICAL PROPERTIES OF ASPHALT BINDERS

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Thesis

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ABSTRACT

Reclaimed Asphalt Pavement is usually used to replace the expensive virgin binder in the asphalt mixture design. Ohio Department of Transportation allows 10% RAP in the mixture design as higher percentage of recycled binder will make mixture aged. In recent years the cost of virgin binder is increasing at extremely rate. This has garnered attention to maximizing the use of RAP to minimize the production and manufacturing cost. This research project is a part of a project of Ohio Department of Transportation. The goal of ODOT's project is to assess the feasibility of RAP in the surface course of municipal and local roadways. The objective is to develop cost effective mix design and quality control recommendations for RAP use on local roadways in Ohio that does not adversely affect the performance or durability of the asphalt mixtures. This study evaluates the effect of three rejuvenators (SylvaroadTM RP 1000, Industrial Soybean Oil and Hydrolene H90T) to restore the viscoelastic properties of PG 64-22 when mixed with recycled asphalt binder. Rejuvenators are known to reversing the aging effect in aged asphalt binder. Therefore, use of rejuvenators will allow higher percentage of RAP being used in asphalt mixture design. In this study PG 64-22 is the virgin binder and the three rejuvenators are SylvaroadTM RP 1000, Industrial Soybean Oil and Hydrolene H90T. The RAP binder percentage was used 40% of total binder's weight. At first recycled binder was extracted from RAP material using centrifuge method and recovered by Abson method. PG 64-22 and RAP binder is mixed with two selected dosage (low and high) for each of the rejuvenators.

In order to evaluate the effect of the rejuvenators at different dosages testing had been done on unaged binders, short term aged binders, and long term aged. Short term aging has been simulated using the Rolling Thin Film Oven Test (RTFO) and the longterm aging has been simulated using the Pressurized Aging Vessel (PAV).

Finally, virgin binder, recycled blend without the rejuvenators and with two different dosage of rejuvenators were tested at different aging conditions using the Dynamic Shear Rheometer (DSR), and the Bending Beam Rheometer (BBR). DSR was used to evaluate viscoelastic properties at high temperatures and Bending Beam Rheometer was used to determine the effect of rejuvenator at low temperature.

These observations had been used to determine an optimum dosage for each of the rejuvenators. The results had suggested that Sylvaroad exhibited the best softening capability at lower dosage. Whereas Hydrolene was unable to leave significant effect even after using higher dosage. Comparison of observations affirmed that at higher dosage Industrial Soybean Oil reverse the aging effect significantly in recycled blend.

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CHAPTER I

INTRODUCTION

1.1. Problem Statement

The insufficiency of availability and increased cost of virgin asphalt along with strict environmental rules have motivated local and state transportation agency to increase the use of recycled asphalt binder in roadway construction. During service years asphalt binder undergoes a change in its viscoelastic behavior from the original binder. Recycled binder in RAP is stiff and brittle compared to original binder. Though this recycled binder gives higher rutting resistance but fails in resistance to fatigue cracking. In an effort to reduce the increased stiffness and brittleness of the recycled mixture, several state highway agencies recommend addition of softer virgin binders with moderately lower viscosity. Even then the resulting mixture may still be very stiff. This problem addresses the importance of controlling the blending process between aged and new binder. At small percentages (up to 20%), an aged binder does not significantly affect the properties of the blend of virgin and RAP binder (Kennedy et al. 1998).

Recycled Asphalt Pavement (RAP) is the milled pavement material obtained from distressed aged pavement. RAP primarily consists of aged asphalt binder and aggregate. Aging affects the binder immensely over the service years by changing the viscoelastic properties. The asphalt binder in RAP goes through two stages of aging, i.e. short-term aging right after manufacture and placement and long-term aging during service years. During the aging process the viscosity is gradually increased and makes the binder stiff. When RAP is added to the softer new binder, the blend behaves differently than the RAP binder and the new binder. The recycled blend contributes to the poor resistance against fatigue and thermal cracking which is undesirable. However, addition of RAP improves the rutting resistance of the asphalt mixtures.

The main limitation of using higher percentage of RAP is the resulting stiff nature of the asphalt mixture. This concern can be resolved by introducing recycling agents or rejuvenators in the mix design for asphalt mixtures. Researchers have been studied the effect of rejuvenator on the blend of new binder and RAP binder for past decades. It is widely acknowledged that rejuvenators can significantly improves the fatigue resistance of the asphalt mixture. Rejuvenating agents act as catalyst which restores the viscoelastic properties of the aged RAP binder. Rejuvenators diffuse through the aged RAP binder and reduces the viscosity. As a result, the recycled blend performs significantly in favor of asphalt mixture by improving the fatigue resistance. Recent research has shown that the use of a high (90% - 100%) RAP content in HMA with rejuvenators is successful (Zaumanis et al.,2014). Fatigue resistance of an asphalt pavement is a concern in the long-term of the service year. Whereas rutting is a problem that needs to be addressed at the very beginning of the service year. If the asphalt mixture is soft then the traffic load can cause depression along the tire path. These depression along the travel path is called rut which is causes major riding discomfort. Rejuvenator is capable of improving the fatigue resistance of asphalt mixture. However rutting problem can be detrimental for asphalt pavement if the dosage of rejuvenator is not selected carefully.

As addition of RAP can reduce the total roadway construction cost up to a great extent based on the percentage of RAP that has been selected to use in the asphalt mixture. However, the effect of increased amount of RAP in mix design is still unclear. Ohio Department of Transportation allows less than 10% RAP in their local roadways. Previous studies on the use of RAP in asphalt mixtures only focuses on the highways and interstate systems. Local roads have different factors affecting the mix design such as traffic volume, type and pattern. Additionally, bus routes, tighter lane width, roadway diet, underground utilities, ADA curb ramps contribute to local road design. Thus, it can be assumed that performance of local roadways may vary than the interstate and highways due to use of RAP.

Three rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene) had been used in this study to evaluate their effect of recycled blend binder. Initially a low and a high dosage have been selected to evaluate the influence that each rejuvenator would leave on the recycled binder blend. Many factors are responsible for the change in rheological properties of the binder after introducing the rejuvenator (i.e.: type, dosage rate, rate of diffusion, mixing rate, temperature, aging etc.). In this study, light had been shed on the following factors: temperature, aging, dosage rate and frequency of the loading. Based on these evaluations an optimum dosage rate had been determined for each of the three rejuvenators.

This study has been initiated to assess the feasibility of use of RAP in the surface course of municipal and local roadways in Ohio. The purpose is to develop cost effective mix design and quality control suggestions for RAP use at higher percentage. As a part of the objective, the optimum dosage of rejuvenators will be determined based on the binder study.

1.2. Research Objectives

The primary objectives for this study are to:

- Evaluate the effect of different rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene) on the viscoelastic properties of blend of PG 64-22 and RAP at high, intermediate and low temperatures.
- Evaluate the effect of different rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene) on the viscoelastic properties of the recycled blend at target temperature and at 10 rad/sec frequency.
- Evaluate the effect of different rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene) on the PG grade of the recycled blend compared to target binder (PG 64-22).

 Determining the optimum dosage for each of the three rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene).

1.3. Thesis Organization

This thesis is organized into 6 chapters. Chapter 1 gives general introduction that includes the problem statement and the research objectives. Chapter 2 is a detailed literature review section on the topics that are relevant to this study. Chapter 3 outlines the research methodology. Chapter 4 discusses the experimental plan. The results obtained from the lab work and a statistical analysis are presented in Chapter 5. Chapter 6 includes the conclusions and recommendations for future study based on the findings from the research.

CHAPTER II

LITERATURE REVIEW

2.1. Introduction

Improvement of sustainability approaches in pavement recycling has gained importance over last couple of decades. The roadway industry is therefore leaning towards alternative materials and construction technology, which are environment friendly as well as energy efficient and cost effective for the construction and maintenance of roads. One of the vital components in road construction, Aggregate, comes from quarries. The extraction of these aggregates from their natural sources results in loss of forest cover and pollution on a large scale leading to environmental degradation. Sequentially this has raised environmental concerns in many parts of the world (Indoria et al, 2011). Moreover, the fluctuating cost of virgin binder has fueled the research for alternative methods of replenishing strength of aged asphalt binder.

Asphalt and concrete pavements are commonly recycled and reused construction materials. Recycled concrete pavements are used as aggregate in new concrete mixture. But reclaimed asphalt pavement can be an addition to both the binder and aggregate. In recent years a remarkable importance has been given in using recycled binder from Reclaimed Asphalt Pavement. Asphalt industries are leaning towards introducing higher percentage of RAP in Hot Mix Asphalt. But there are some concerns associated with it. RAP binder is age-hardened. Introducing RAP binder with virgin binder in higher percentage might have an undesirable effect like low fatigue resistance and low temperature cracking. Current practices allow 20%-30% RAP in mix design. But recent research has shown that use of 100% RAP is possible with appropriate dosage of rejuvenators (Zaumanis et al, 2014). To reverse this effect introduction of rejuvenators with higher percentage of RAP material is getting recognition. Rejuvenators are oil-based product designed to bring back original properties to aged asphalt binders by restoring the original ratio of asphaltenes to maltenes. In this chapter, a detailed comprehensive study was carried out to determine the practice of use of RAP in higher percentage with addition of various rejuvenators in optimum dosage.

2.2. Reclaimed Asphalt Pavement (RAP)

RAP is removed or reprocessed pavement material containing aggregates and asphalt binder. When properly crushed and screened, RAP consists of high-quality, well-graded aggregates coated by asphalt cement. The majority of the RAP that is produced is recycled and used, Recycled RAP is almost always returned back into the roadway structure in some form, usually incorporated into asphalt paving by means of hot or cold recycling, but it is also sometimes used as an aggregate in base or subbase construction. RAP material mainly consists of asphalt binder and aggregates. The asphalt binder generally undergoes various physical and rheological changes during their service life. The binder properties in RAP are significantly affected by the rheological changes. There are two principal factors which contributes to the change in RAP binder properties: chemical structure of the original binder used during its construction and amount of aging undergone during its service. There are two stages of aging that the binder undergoes during the functioning years, i.e. short-term aging (during construction) and long-term aging (during service). During the service years the viscosity of the binder gradually increases due to natural phenomena like oxidation, volatilization, polymerization and syneresis making it a hard and a stiff material (Al-Qadi Imad L. et al, 2007)

2.3. RAP in Hot Mix Asphalt:

The scarcity and increased cost of virgin asphalt along with strict environmental rules have motivated local and state transportation agency to increase the use of recycled asphalt binder in roadway construction. During service years asphalt binder undergoes a change in its rheological behavior from the original binder. Recycled binder in RAP is stiff and brittle compared to original binder. Though this recycled binder gives higher rutting resistance but fails in resistance to fatigue cracking. In an attempt to reduce the increased stiffness and brittleness of the recycled mixture, several state highway agencies recommend addition of softer virgin binders with relatively lower viscosity. Even then the resulting mixture may still be very stiff. This emphasis on the importance of controlling

the blending process between aged and new binder. At small percentages (up to 20%), an aged binder does not significantly affect the properties of the blend of virgin and RAP binder (Kennedy et al. 1998). But a higher percentage of RAP requires the use of a blending chart.

2.4. Rejuvenators

Despite the economic and environmental advantages incorporation of large amount of recycled mixtures into HMA operations is burdened with challenges like poor fatigue resistance, raveling and other durability problems (F. Yin et al., 2017). To mitigate the effect of aged recycled material highway agencies and asphalt paving industries are exploring the possibility of using higher amount of recycled material and getting desirable performance. One of the solutions is incorporation of rejuvenators in the asphalt mixtures. According to the Asphalt Institute (1986), Recycling Agents or Rejuvenators are defined as organic materials with chemical and physical characteristics selected to restore aged binder to desired specifications (F. Yin et al., 2017). Currently, there are several different types of rejuvenators that are commercially available in the United States, and they can be categorized as paraffinic oils, aromatic extracts, naphthenic oils, triglycerides and fatty acids, or tall oils (NCAT, 2014). Waste Engine Oil, Waste Engine Oil Bottoms, Valero VP 165[®], Storbit [®] are paraffinic oil; Hydrolene[®], Reclamite[®], Cyclogen L[®], Valero 130A[®] are aromatic extracts; SonneWarmix RJTM, Ergon HyPrene® are naphthenic oils; Waste Vegetable Oil, Waste Vegetable, Grease, Brown Grease, Delta S* are triglycerides and

fatty acids and SylvaroadTM RP1000, Hydrogreen® are tall oils (Arámbula-Mercado et al., 2018).

2.5. Mechanism of Rejuvenator

A recycling agent or rejuvenator restores the aged recycled binder by decreasing the stiffness for construction purposes and mixture performance in the field and increases fatigue resistance by increasing phase angel (Arámbula-Mercado et al., 2018). A rejuvenator contains high amount of maltenes which is basically condensed aromatic hydrocarbons. According to Tran et al. (2012) rejuvenator rebalances the proportion of maltenes of aged binder which is lost during construction and service years of pavement.

Carpenter and Wolosick (1980) used a stage extraction method to study the movement of rejuvenator into old bitumen. They concluded that when rejuvenator is added to asphalt mixtures it forms a coating around the particle. Upon mixing the rejuvenator penetrates the thick film of asphalt around the aggregate and thereby softening the aged asphalt. Zaumanis et al. (2013) gave a detailed description on the process of diffusion of rejuvenator agents into bitumen. In that description it was stated that the in-plant addition of rejuvenator involves three main interface mechanisms between the aged binder and rejuvenator – dispersion, diffusion and mechanical mixing. Dispersion is the phenomenon of distribution of the rejuvenator on the RAP, diffusion is the penetration of rejuvenator into aged RAP binder, and mechanical mixing is instigated by the friction between aggregate particles. The rejuvenating agents diffuse through the aged RAP binder up to a

certain depth of the aged binder film and reestablish the original maltene to asphaltene ratio in it, making it a softer material.

The process of diffusion is defined by temperature, viscosity, mixing, transportation and storage time (Zaumanis et al., 2013). There have been different studies to analyze the process of diffusion. The method of rejuvenator diffusion into bitumen is a very complex procedure. Karlsson et al. (2003) conducted a research in which he concluded that FTIR-ATR is suitable for monitoring the diffusion process involved in bituminous binder and rejuvenator mixing. Oliver et al. (1975) studied the diffusion of dodecylbenzene and two oil fractions into three different bitumen, using a method of tritium labeling. The conclusions of this study were that the diffusion rate could be increased by adding diluent oil fractions or by raising the temperature and molecule size is important for diffusion rate. Zaumanis et al. (2013) analyzed diffusion rate of rejuvenator into RAP binder using finite element modelling. He concluded that only diffusion rate cannot be responsible for proper blending of RAP binder and rejuvenator. There are also other factors that contribute to the blending such as homogenous dispersion and mechanical mixing.

There has been a concern regarding the completion of the diffusion process. Carpenter et al. (1980) have reported that the diffusion process may not be completed after the construction. Thus, higher rutting rates were initially detected on roadways and on test sections subjected to accelerated pavement testing (Potter et al., 1997). Compared to virgin mixtures (when insufficient rejuvenator is added), properly rejuvenated asphalt mixtures also showed higher susceptibility to low-temperature cracking (Tam et al., 1991). According to Dekold et al. (1992) and Terrel et al. (1978) the moisture susceptibility of recycled mixtures remained similar to or improved over that of virgin mixtures. However, recycled stripping-susceptible mixtures may increase moisture susceptibility, and antistripping additives should be used properly (Dekold et al., 1992). The type of rejuvenator used had minute effect on moisture susceptibility (Epps et al., 1980).

2.6. Previous studies on using Rejuvenators

The process of aging of binder is divided into two phases- short term aging and long-term aging. Short-term aging of asphalt aging occurs during mixing, silo storage, transportation and laying processes due to exposure to high temperatures and long-term aging occurs for the rest of the service life (Zaumanis et al, 2014). Moderation of the stiffness the aged binder is done by using softer grade binder in the mixtures. But production of softer binder is not cost effective and environment friendly. Using rejuvenator in the mixtures along with aged recycled material and a virgin binder has been found to be effective in terms cost reduction and sustainability. But the procedure of adding the rejuvenator to the asphalt mixtures is not that simple. Softening effect might be good for mitigating the stiffness of aged recycled material but in terms rutting resistance pavement will wither sooner than expected. There are several factors that should be taken into consideration while selecting an optimum dosage for rejuvenators. The factors include amount of aged binder, type of rejuvenators and effect of rejuvenators on binder and mixture. All the factors lead to the inevitability of obtaining an optimum dosage of rejuvenators.

2.6.1. Higher Percentage of RAP & Type of Rejuvenator

Reuse of old asphalt pavement material is a valuable approach in terms of technology, economy and environment (Kennedy et al, 1998). Many state agencies have also reported significant savings when RAP is used (Page et al.,1987). Current practice allows use of RAP only 10-20%. Haghshena et al. (2016) conducted a study where he explored the effect of three rejuvenators (*petroleum, green and agriculture*) on 65% RAP containing mixtures. It was concluded *that petroleum-tech* and *agriculture-tech* rejuvenator restored the chemical composition of aged binder whereas the *green-tech* showed opposite trend. It was also reported that the influence of *green-tech* rejuvenator on softening of binder is more significant than the *petroleum-tech* and *agriculture-tech* rejuvenators.

Zaumanis et al. (2014) carried out experiments and studied the effect of six different rejuvenators 100% recycled HMA laboratory samples and recycled binder. In this *study Waste Vegetable Oil, Waste Vegetable Grease, Organic Oil, Distilled Tall Oil, Aromatic Extract* and *Waste Engine Oil* were used. From rheological analysis of binder, it was evident that organic products performed similar to petroleum products at much lower dose. Except Waste Engine Oil (WEO), the rest of the rejuvenators changed the Superpave Performance Grade (PG) from 94-12 of extracted Binder to 64-22 at similar dose. All six products reduced the binder viscosity close to the virgin binder at intermediate temperature (25^oC), but at higher temperature as measured by softening point and kinematic viscosity tests, the binder viscosity remained higher than that of a virgin binder.

Porot et al. (2016) conducted a study on using a bio-based rejuvenator, *SYLVAROAD*TM*RP1000*, to treat aged RAP binder. Based on conclusions of a preliminary study in laboratory on the asphalt binder based on penetration, softening point and viscosity, a full-scale trial in Taiwan was made, paving a secondary road for which the properties of the binder were monitored over time. The section with the bio-based rejuvenator showed major improvement over the section without. The impact of another bio-based rejuvenator, *BituTech RAP*, on the viscoelastic properties of high RAP content (15% and 50%) mixtures were investigated (Hajj et al., 2013). It was concluded that as long as the high-temperature properties of the mixtures were not risked, *BituTech RAP* could save cost without having to introduce softer binder in the mix to compensate for stiffness of RAP.

Ali et al. (2016) examined the effect of five rejuvenators (*a Naphthenic Oil, a Paraffinic Oil, an Aromatic Extracts, a Tall Oil, and an Oleic Acid*) on mixtures containing 25% and 45% RAP. The statistical analysis revealed that the rejuvenator type had a significant impact on the true high and low temperature performance grades. *Paraffinic oil*-based rejuvenator showed consistent improved performance over other rejuvenators. But overall all the rejuvenators provided a viable option to using high percentages of RAP materials.

There have been several researches on using waste oil as rejuvenators as oil prominently affects the properties of binder. *Used frying oil (UFO)*, when blended with

Trinidad lake asphalt and Trinidad Polymer Bitumen, decreased stiffness value gradually with increasing dosage (Singh-Ackbarali et al., 2017). This study demonstrated the possible recycle of *UFO* as an asphalt modifier capable of producing *UFO* modified asphaltic blends for special applications. The waste vegetable oil has greatest effect to the penetration and softening point value when added to the heated RAP binder. It can be emphasized that the temperature, amount of waste oil and RAP are notable to give momentous influence on the performance properties (Kamaruddin et al., 2014)

2.6.2. Selection of Rejuvenator Content

Incorporation of rejuvenators or recycling agents in a blend of aged binder and softer binder should be at an optimum dosage. Higher dosage makes the asphalt mixtures susceptible to rutting and lower dosage is responsible for stiffness of mixture. Normally, the rejuvenator dosage is selected based on experience or the producer recommendation (Arámbula-Mercado et al., 2018). The optimum dosage can be selected using blending charts which is based on the viscosity and/or penetration of the blends of the recycled binder with various amounts of rejuvenators (Zaumanis et al., 2015). According to Shen et al., (2002) and Zaumanis et al., 2014, the Superpave Performance Grade (PG) system can also be used to determine the optimum dosage needed to restore the performance properties of the recycled binder. A minimum dosage guarantees sufficient cracking resistance (intermediate- and low-temperature PG), while a maximum dosage ensures adequate rutting resistance (high temperature PG) (Zaumanis et al., 2014). In addition to PG, Δ Tc, which is the difference in the bending beam rheometer (BBR) test temperatures when the

creep stiffness (S) and stress relaxation rate (m-value) reach the PG specification limits of 300 MPa and 0.30, respectively, is a parameter used to quantify the brittleness of the recycled blend at a given rejuvenator dosage (Arámbula-Mercado et al., 2018)

Zaumanis et al., (2014) conducted a study where he determined the optimum dosage using six rejuvenators based on test results of two dosages, high and low. It was reported that performance grade of blend of the aged and soft binder was adjusted back to softer binder linearly with an increase in the rejuvenator dose and intermediate PG parameter reduced linearly up to the Superpave G*Sin\delta requirement of maximum 5000 kPa which is an indication of fatigue resistance He also concluded that Organic rejuvenators require smaller dose compared to petroleum rejuvenators to cause similar softening effect on aged RAP binder (Turner et al., 2015)

In NCAT report (2012), the effects of the rejuvenator (*Cyclogen* \circledast *L*) contents on the performance properties of the RAP and RAS binders were studied. Based on the linear relationship between rejuvenator contents and critical temperatures of the RAP and RAS binders, a content of 12% by the total weight of recycled binders was selected. Again in 2015 National Center for Asphalt Technology (NCAT) explored the benefits of *SYLVAROAD* TM *RP 1000* in a two-phased study. In first phase, 5% and 10% contents of rejuvenator were used to explore the changes in rheological performance of RAP binder. Based on Superpave Performance Grade Specifications a dose of 6.8% was selected which restored the continuous grade of the RAP binder.

E. Arámbula-Mercado et al. (2018) evaluated three dosage selection methods of recycling agent based on the PG and ΔTc of recycled blends. The methods were 1.

Restoring Lower Performance Grade (PGL) and Verifying Higher Performance Grade (PGH), 2. Achieving critical temperature of -5^oC after 40 hour of PAV aging and 3. Restoring Higher Performance Grade (PGH). Among these methods, restoring higher performance grade (PGH) provided better results.

2.6.3. Incorporation of rejuvenator

The most common practice for incorporating rejuvenator in mixtures is to follow the producer recommendation based on dosage and percentage of the recycling agent with respect to the total binder content. In the method of addition, rejuvenator is added to the mixture without altering total binder content. In replacement method, the total binder content is reduced by the recycling agent amount.

Another method is to spread the rejuvenator on the road surface to restore the original properties of the pavement. The problem with this method is that the rejuvenator may not reach deep into the asphalt layer. Reduction of skid resistance and releasing carcinogenic aromatic compound of rejuvenator in the environment are major safety concerns. García et al. (2010) introduced encapsulated rejuvenator which is free of aforementioned problems. These microcapsules can withstand high mixing temperature and stress due to their plastic-elastic-deformation ability the capsule containing rejuvenator is embedded into asphalt concrete and breaks when the stress on the pavement surpasses the threshold limit. Microcapsules containing rejuvenator is considered a promising product for the evolution of smart pavement construction.

Laboratory studies on the effect of rejuvenator are done in two steps. First several tests are performed on the rejuvenated binder. The outcomes from binder studies are used in rejuvenated mixture tests. Previous studies on the rejuvenated binder and mixtures are discussed below:

Shen et al. (2007) studied the effects of rejuvenator on performance-based properties of rejuvenated asphalt binder and mixtures. At first the performance-based properties of aged asphalt binder containing rejuvenator at various percentage were investigated at high, intermediate temperatures using DSR and low temperatures using BBR. Using these data an optimum concentration of rejuvenator using the blending chart was determined that will help the aged binder to reach a target PG grade. The second part of the study explored rutting resistance using Wheel Tracking Device and fractures properties under low temperatures using Thermal Stress Restrained Specimen (TSRS) on both rejuvenator added hot mix asphalt (HMA) and a virgin HMA as a control mix. It was concluded that rejuvenators improved the aged binders and mixtures containing rejuvenated aged binder significantly.

Zaumanis et al. (2014) carried out experiments and studied the effect of six different rejuvenators at 12% dosage on 100% recycled HMA laboratory samples and recycled binder. In this study Waste Vegetable Oil, Waste Vegetable Grease, Organic Oil, Distilled Tall Oil, Aromatic Extract and Waste Engine Oil were used. From rheological analysis of binder, it was evident that organic products performed similar to petroleum products at much lower dose. Except Waste Engine Oil, the rest of the rejuvenators changed the Superpave Performance Grade (PG) from 94-12 of extracted Binder to 64-22 at similar dose. The results obtained from the experiments confirmed excellent rutting resistance along with longer fatigue life compared to virgin mixtures. All rejuvenated mixtures showed better resistance at low temperature cracking by lowering the critical temperature. Though the rejuvenators improved the workability of RAP mixtures but at 12% dose none of them ensured workability equal to virgin mix. This study only reflects the results based on 12% dose, it was suggested that an optimization of rejuvenator dosage will improve performance of mixes in most cases.

In a laboratory evaluation Lu and Saleh. (2016) combined warm mix asphalt with high RAP content by using a chemical warm mix additive, Evotherm and a rejuvenator, Sylvaroad. The viscosity tests of the binder and mechanical performance tests of WMA-RAP mixtures were carried out and compared to a control HMA. Both the additives reduced the binder's viscosity and the reduction of viscosity increased with the increment of dosage. Evotherm improved moisture resistance in WMA-RAP mixtures than Sylvaroad. WMA mixture with Sylvaroad showed a higher number of cycles to fatigue failure than the control HMA. The authors had drawn two conclusions based on this study. Firstly, for mixtures with Evotherm, the maximum proportion of RAP adding into WMA should be kept at 25%. Secondly, for Sylvaroad mixtures, RAP proportion can be added up to 70% if the moisture resistance of the mixture is satisfied and the binder content increased to maintain good fatigue resistance. Also, by combining Evotherm into the Sylvaroad mixtures the moisture resistance issue of can be solved.

To study the effects of rejuvenators on the nano-mechanical properties of the interfacial blending zone that develops between RAP and virgin asphalt binder in a high RAP content mixture Nazzal et al. (2015) implemented Atomic Force Microscopy (AFM) techniques. In this study, rejuvenators did not have a significant effect on the modulus of the virgin binder but the nano-indentation modulus of the interface blending zones was significantly reduced when rejuvenators were used. The Dynamic Shear Rheometer (DSR) and AFM results indicated that the RAP-virgin binder interfacial blending zone moduli are different than those of the blends prepared by manually mixing of the RAP and virgin binders. The Hamburg Wheel Tracking Device test results showed adverse effect on the rutting performance of the RAP mixtures due to rejuvenators. Correlation between the AFM indentation modulus of interfacial blending zone and the macro-scale rutting performance of high RAP content mixtures was found well. Fatigue life was found to be improved in high RAP content mixtures when added with rejuvenators. The authors concluded that the interfacial blending zone bonding energy might be one of the main factors dictating fatigue performance of high RAP mixtures.

Ali et al. (2016) examined the ability of five rejuvenators (a Naphthenic Oil, a Paraffinic Oil, an Aromatic Extracts, a Tall Oil, and an Oleic Acid) to restore low and high temperature true performance grades of aged binders. Several sets of asphalt mixtures containing different percentages (i.e. 25% and 45%) of RAP materials were prepared using PG 76-22 polymer-modified asphalt binder. After adding rejuvenators at manufacturers' recommended dosage, the loose mixes were aged for 2h and 6h. the extracted binder from the mixes were considered RTFO aged. The true low and high temperature grades of the extracted binders were determined using the Dynamic Shear Rheometer (DSR) and the Bending Beam Rheometer (BBR) tests. The test results were analyzed using DSR shear modulus master curves and multi factor Analysis of Variance. The analysis indicated that among all rejuvenators the Paraffinic Oil rejuvenator was the most effective in lowering the PG grade of the aged binder contained in the RAP than that of the control binder without rejuvenation. In addition, rejuvenator's efficacy was not affected by aging and increasing the amount of RAP materials (up to 45%). The rejuvenators were also found to improve the fatigue resistance without substantially influencing rutting performance.

Elkashef et al. (2017) used a new soybean derivative rejuvenator at 0.75% by the weight of bitumen. The authors explored rheological properties after adding the rejuvenator to a PG 64-28 and a PG 58-28 bitumen using a dynamic shear rheometer, a bending beam rheometer and a rotational viscometer. Fourier Transform Infrared-Attenuated Total Reflection was implemented to assess the aging characteristics of rejuvenated binders, results of which indicated similar aging in both modified and control asphalt binders. It was observed that at such a low concentration, the rejuvenator lowered the viscosity, improved the fatigue and low-temperature properties of the tested asphalt binders significantly. Dynamic modulus specimens for both the control and modified blends were prepared using a mixing and compaction temperature of 120°C, as well as a temperature of 140°C. the mixing and compaction temperature of 120°C had the effect of the mix performing better at lower test temperatures than the specimens produced using a mixing and compaction temperature of 140°C.
Li et al. (2014) studied the effect of aged modified asphalt in reclaimed asphalt pavement (RAP) mix. A polymer emulsion, Styrene–butadiene rubber (SBR) latex, was used to blend 7 modified asphalt with conventional asphalt. Experiments were conducted in the research laboratory to measure the outcome of SBR latex on RAP mix. SBR latex boosts the low temperature characteristics of RAP binder efficiently without causing any noticeable negative impact on RAP mix compaction. SBR latex enhanced the viscoelastic properties and other performances of RAP mix, including the resistance to low-temperature cracking, rutting, and moisture damage.

Mogawer et al. (2016) explored the effect of introducing five asphalt rejuvenators into 50% RAP surface-layer mixture and evaluated the performance in terms of rutting and cracking. It was found that the rejuvenators deteriorated the rutting resistance of the 50% RAP mixture. But the use of polymer modified asphalt (PMA) binders improved these degradations. The rejuvenators were found to augment the fatigue cracking resistance of the 50% RAP mixture to a level higher than that of all-virgin control mixture and also the 50% RAP mixture with softer binder. The authors concluded that a blend of an asphalt rejuvenator and a PMA binder was required to yield a high RAP mixture with similar or better performance than a similar conventional mixture.

Im et al. (2014) conducted various laboratory tests including Hamburg test, overlay test, dynamic modulus test, and repeated load test to compare the performance and engineering properties of HMA mixtures without rejuvenators to those of mixtures incorporated with rejuvenators. The objective of the study was to evaluate the impacts of various rejuvenators on the performance and engineering properties of hot-mix asphalt (HMA) mixtures containing recycling materials (i.e., RAP and RAS). It was found that the use of rejuvenators improved cracking resistance of the recycled mixes and incorporation of rejuvenators in the recycled materials enhanced moisture susceptibility and rutting resistance of the blends. The authors concluded that the performance of the rejuvenators depend on degree of blending between the binder of the recycled materials and the virgin binder, aggregates, and the rejuvenator dosage.

2.6.5. Compound Rejuvenation

The performance of polymer modified asphalt pavement is better than nonmodified asphalt pavement in terms of rutting, fatigue resistance. But just like any other conventional asphalt binder, polymer modified binder ages due to weathering. The rejuvenators that are being used commonly are developed for non-modified asphalt binder rejuvenation. Ma et al. (2010) compared two cases of rejuvenation of SBS modified binder, one with rejuvenator alone and the second one with combination of a rejuvenator and a modifying additive. He recommended compound rejuvenation for polymer modified binders as rejuvenator alone did not reverse the effect of aging according to test results.

In a different study Lu and Saleh (2016) performed a laboratory evaluation of warm mix asphalt incorporating high RAP proportion by using a chemical warm mix additive, Evotherm and a rejuvenator, Sylvaroad. They concluded that, by combining Evotherm and Sylvaroad, the use of RAP in the mixture can go up to 70% and moisture resistance will also be improved to a great scale.

CHAPTER III

RESEARCH METHODOLOGY

3.1. Introduction

This chapter discusses the research plan to obtain the optimum dosage of three rejuvenators to restore the performance grade of the blend of the new binder and the recycled binder close to the new binder. The conclusion will be drawn based on the binder test results. This study is divided into two parts. First part consists of extraction and recovery of recycled binder from Recycled Asphalt Pavement. The second part consists of experiments on the blend of recycled binder and new binder with a low and a high dosage of the selected rejuvenators and without any rejuvenator.

3.2. Research Plan Flow Chart

The new binder in consideration is PG 64-22 and the rejuvenators are SylvaroadTM RP 1000, Hydrolene (H90T) and Industrial Soybean Oil. First step is to extract and recover the recycled binder from RAP. The amount of RAP that will be used in this study is 40% by its asphalt content. In the second part, the same tests are run on PG 64-22, PG 64-22 with 40% RAP and PG 64-22 with 40% RAP and two dosages of the selected rejuvenators. The selected percentage of rejuvenators is by total weight of recycled binder. Figure 3.1

represents the complete laboratory testing plan flow chart to obtain optimum rejuvenator content.



Figure 3.1: Laboratory Testing Plan with Control Binders



Figure 3.2: Laboratory Testing Plan with Recycled Blend with Rejuvenators

3.3. Sample Preparation

Extraction is done by dissolving the RAP material in Toluene for a period of time until it extracts most of the binder from RAP. The recycled binder was recovered by the Abson method.

Each of the binder blends undergoes the simulation of short-term and long-term aging. Unaged condition is right after the preparation of blend has been made. Rolling Thin Film Oven (RTFO) is used to simulate short term aging of the binder. Both Unaged and RTFO aged binder are tested in Dynamic Shear Rheometer (DSR) at high temperatures. For long-term aging, Pressure Aging Vessel (PAV) is used. Residue from PAV aging is used for DSR tests at intermediate temperatures and in Bending Beam Rheometer at low temperature.

The method of preparing blend with rejuvenators starts with mixing the rejuvenator with the new binder (PG 64-22). The rejuvenators are heated at 140^oF for 40 minutes. PG 64-22 and RAP binder are heated at 325^oF to make them flowable. Rejuvenator is added to the PG 64-22 first and mixing procedure is continued for one minute using a glass rod. Finally, RAP binder is added to the blend and mix it again for one minute. After thorough mixing, samples for DSR test are collected and residues are used for short-term and long-term aging simulation tests. RTFO aged residue was used in DSR tests at high temperatures and PAV aged residues are collected and stored to use in DSR tests at intermediate temperature and BBR test at low temperatures.

CHAPTER IV

LABORATORY TESTING PLAN

4.1. Introduction

This chapter discusses the laboratory experiments that are done in this study. Three types of experiments were done in this study. Extraction and Recovery test to obtain recycled binder, DSR test and BBR test were performed on the binder without rejuvenators and with rejuvenators binder and Dynamic Modulus test was done on mixtures. To simulate the aging of binder Rolling Thin Film Oven test and Pressure Aging Vessel test were also performed.

4.2. Extraction Process

In this study recycled binder is obtained from RAP by centrifuge extraction process. The centrifuge method is done in accordance with AASHTO T-164. In this method, a solvent is used to remove asphalt binder from aggregates in HMA mixtures. Prior to extraction presence of moisture is made sure. The HMA mixtures are dried in oven to get rid of the moisture. Based on the desired quantity of binder a minimum weight of the sample is

spooned into the sampling bowl. The extracting solvent is then introduced into the bowl until the mixture is fully submerged. A filter ring must be placed on the top of the sampling bowl prior to closing it with the lid for determining the asphalt content. The extracted asphalt binder flows through the holes on the lid of the sampling bowl. After placing the lid and closing it tightly, the mix must be immersed in the solvent for a period of time not more than an hour to dissolve as much binder as possible. Another lid has to be placed on the extraction bowl which contains the sampling bowl. The extraction bowl has an outlet which lets the extracted solvent to flow through the hose connected to it.



Figure 4.1: Extraction Centrifuge

After a reasonable period of time the centrifuge is switched on. The speed of the sampling bowl is increased gradually until it reached 3600 r/min and the centrifuge should be kept running until all solution is drained out. The same process is repeated to get most of the binder extracted from the aggregate. The color of aggregate will turn to grey once most of the binder is removed. The residue from extraction procedure is then recovered by Abson method or Rotary Evaporator method.

4.3. Recovery Process

Recycled binder is recovered from the extracted binder-solvent solution. In this study Abson method has been used for the recovery process which is done in accordance with AASHTO R 59. The process is mainly a condensation procedure under prescribed conditions to a point where most of the solvent has been distilled. Carbon Dioxide gas is introduced into the distillation process to remove all traces of the extraction solvent.

4.3.1. The Abson Method

The basic principal of the Abson method includes heating of the extracted at a certain temperature until the solvent starts to evaporate and leaves the flask that had the solution in it, leaving only the recovered asphalt binder inside the flask after the procedure is complete. The recovery set consists of a heating mantle, a temperature controller, source of water and carbon dioxide gas cylinder. A three-neck flask, filled with extracted solution, placed inside of a heating mantle that is connected to the temperature controller. The temperature controller has a temperature probe that is inserted through one neck of the flask into the solution to monitor the solution temperature. A hose with an aeration tube is attached to the carbon dioxide tank's end to provide agitation and avoid foaming. A kinked glass tube attached of the flask allows the solvent vapor to come out of the flask and gets distilled via the condenser.



Figure 4.2: Abson Method Recovery Setup

After carefully examining all the element in the recovery set up the process starts by heating up the solution in the flask by setting the temperature in the controller to the solvent's boiling point. The carbon dioxide is introduced at a low rate 100 mL/min. The distillation should be continued until it reaches temperature range 157°C to 160°C and at that point carbon dioxide flow should be increased to 900 mL/min To ensure that all the solvent has left the flask, continuous stirring of the aeration tube should be adequate. The test is completed when there are no more drops of solvent is coming out of the condenser. Figure 4.2 shows the recovery setup used in this research.

4.4. Short Term Aging using RTFO

Short-term aging of the asphalt binder is simulated by using Rolling Thin Film Oven and it's done in accordance to AASHTO T 240. In RTFO asphalt binder is exposed to elevated temperatures to simulate manufacturing and placement aging. The RTFO also gives a quantitative measure of the volatiles lost during the aging process. The basic RTFO procedure takes unaged asphalt binder samples in cylindrical glass bottles and places these bottles in a rotating carriage within an oven. The rotating rack can hold up to 8 bottles inside an environmental chamber. The RTFO oven is equipped with an air nozzle which oxidize the asphalt binder along with high temperature. Specifications in AASHTO T 240 state that each bottle should be filled with 35 g of unaged binder and let to cool down in a horizontal position for at least 60 min and not more than 180 min. After cooling down at room temperature the sample is introduced to the $325^{\circ}F(163^{\circ}C)$ in the oven's rotating rack and the test is conducted for 85 minutes while the air nozzle is pumping air at a rate of 4000 +/- 300 mL/min while the rotating rack is rotating at 15 +/- 0.2 r/min. After placing the cooled bottles to the RTFO oven temperature of the oven drops down. According to AASHTO T 240, the temperature in the oven should rise to 325°F in less than 10 minutes, otherwise the test shall be terminated. Figure 4.3 shows the RTFO used in this study.



Figure 4.3: Rolling Thin Film Oven

4.5. Long-Term Aging using PAV

The pressurized aging vessel (PAV) is done in accordance with AASHTO R 28. PAV is used to simulate long term aging of the asphalt binder (5 to 10 years in pavement life) and the simulation is done by introducing pressurized air and elevated temperatures. The test uses the RTFO residue to simulate long-term aging. The setup consists of an environmental chamber with a rack inside that can hold up to 10 PAV pans, each pan is filled with 50 g of the RTFO residue binder. According to AASHTO R 28, regulated pressurized air of 2.1 ± 0.1 MPa is introduced to the PAV machine through an air tank which stands next to the PAV machine. The set temperature in the environmental chamber is between 90°C and 110°C based on the area this test is made for, but usually a temperature of 100°C is used. The vessel is preheated with the empty sample rack to prevent temperature dropping. After preheating the rack is taken out of the chamber and the 10 pans are placed into the rack regardless whether the pans are full or empty. Then the sample with the rack is inserted into the chamber and seal top cover tightly. The aging procedure continues for 20 hours. Figure 4.4 shows a picture of the PAV used in this study which was designed by Applied Test Systems (ATS).



Figure 4.4: PAV Machine

4.6. Binder Testing

Two tests are required to perform on the blends of binder to determine the performance grade. To determine the continuous grade for high temperature, Dynamic Shear Rheometer (DSR) tests are run. To find out the continuous grade for low temperature, Bending Beam Rheometer test is performed. Both of the tests are essential to define and compare the rheological properties of different blends of the binder with or without the rejuvenators.

4.6.1. Dynamic Shear Rheometer (DSR).

For better understanding of the rheological properties of the asphalt binder DSR test is conducted. DSR gives two important parameters, dynamic shear modulus and the phase angle, and it's done in accordance with AASHTO T 315. G*, the complex shear modulus, is defined as the ratio between the absolute value of the shear stress to the absolute value of the shear strain. The phase angle is defined as the angle in radians between a sinusoidal applied strain and the resultant sinusoidal stress in a controlled-strain testing mode. The complex shear modulus and phase angle are used to understand the rheological properties and a comparison can be conducted among the corresponding blends of binders under each rejuvenator used to observe the effect it has on the binder.

Sample sizes for DSR tests change based on the aging process and grade of the binder. For unaged and short-term aging (RTFO), specifications for DSR test demand for a 25 mm mold to be used for preparing the DSR samples and a gap of 1 mm is kept between the spindles holding the binder sample in between. Strain level is governed by the sample size and aging procedure that the binder goes through. For unaged binder strain level is 12% and for RTFO 10% is used. But the sample size and strain level is completely different for PAV aged sample. PAV binders requires an 8 mm mold to be used for preparing the samples, a 2 mm gap is kept between the spindles, and a strain of 1% is used for the test. Figure 4.5 shows the DSR machine that was used in this study.

For a known binder grade DSR test is conducted by performing a temperature/frequency sweep test, in which a software is set to test the binder at different frequencies at different temperatures. G* and phase angle value at a frequency of 10 rad/s at each temperature is the governing values which is required to analyze and determine the grade of the binder. These parameters at 10 rad/s simulates a shearing action of a traffic speed of 55 mph. To determine the performance grade of an unknown grade binder a strain sweep test is performed prior to performing a temperature/frequency sweep. The strain sweep test is used to determine the appropriate strain level to use in order not to surpass the maximum limit of the torque readings which might result in ruining the torque sensor, and at the same time maintaining a minimum torque value of 2 gm.cm.



Figure 4.5: DSR Machine

4.6.2. Bending Beam Rheometer (BBR)

Bending beam rheometer provides two important parameters to measure low temperature stiffness and relaxation properties of an asphalt binder. These parameters are an indication of a binder's ability to prevent low temperature cracking. Bending beam rheometer test is conducted in accordance with AASHTO T313. The basic principal of this test involves a small simply supported asphalt beam. The beam is prepared carefully using a mold of known dimensions. The mold is left to cool down first in room temperature for 45 mins to one hour and finally in a freezer for 5-10 minutes. After demolding, the asphalt beam is submerged inside the device's-controlled temperature fluid bath for 60 minutes at desired test temperature. After 60 minutes, a seating load is applied to make sure that the loading piston is touching the beam, a constant load of 980 +/- 50 mN is then applied. The asphalt beam is loaded with a constant load for 240 seconds. During this period of time the midpoint deflection of the asphalt beam is monitored versus time. The stress relaxation properties of an asphalt binder are determined by recording creep stiffness calculations at 8, 15, 30, 60, 120, and 240 seconds. A higher creep stiffness value indicates higher thermal stresses. So, a maximum creep stiffness value (300 MPa) is required. As a lower m-value indicates a lesser ability to relax stresses so a minimum m-value (0.300) is specified. The decisions are made based on creep stiffness value and m value at 60 seconds. Figure 4.6 shows the bending beam rheometer test machine.



Figure 4.6: BBR Equipment

4.7. Superpave Performance Grading (PG) System

The Performance Grading(PG) system is a method of categorizing an asphalt binder based on its performance at different temperatures. The concept behind the system is that asphalt binder properties should be related to the conditions under which the binder is used in specific climate and region. Performance grade of any asphalt binder is stated as in the following form (PG xx-yy) where xx represents the average 7-day maximum pavement design temperature at which this binder meets all the criteria, and yy is the minimum pavement temperature this binder can reach and still meets all criteria. Up to the stated high temperature in performance grade of the binder, the pavement can resist rutting and the minimum temperature indicates the resistibility against thermal cracking. DSR test results contribute in determining the high temperature and BBR test results are used to determine the low temperature. The superpave performance grading (PG) system is done in accordance to AASHTO MP1.

To grade an asphalt binder a series of tests is required to determine the rheological and physical properties. The tests primarily include DSR (which is done on unaged, short-term aged and long-term aged binder) and BBR (which is done on PAV residue). DSR is usually done on high temperatures for the unaged binder and RTFO aged binder. For both unaged and RTFO aged binder, G*/Sinδ value at 10 rad/s is evaluated at different test temperatures. According to the superpave PG grade system the threshold value at unaged is 1.00kPa for unaged and 2.2kPa for RTFO aged binder. The temperature at which unaged and RTFO aged binder attain the threshold value, the high temperature grade of the particular binder is set to that temperature. Other important tests are flash point temperature and viscosity which are usually done using the Brookfield Viscometer in accordance to ASTM D4402. The minimum flash point temperature for all binders is 230°C all binders must pass in order to be qualified for the Superpave PG grading. The maximum viscosity for all binders must be 3 pa.s at a temperature.

DSR tests on PAV residue is conducted to assess the fatigue cracking ability of a binder. At intermediate temperature G*Sin\delta value should not be more than 5000kPa at 10 rad/s. To determine the intermediate temperature of certain binder, first step is to know the high temperature and low temperature grade of that binder. All the threshold values are listed in PG grading sheet provided by the AASHTO MP1.

The bending beam rheometer (BBR) tests are performed on PAV residue binder to determine the low temperature grading. According to Superpave PG grade system,

maximum creep stiffness of 300 MPa and a minimum m-value of 0.3 are required for a test temperature to be identified as passing low temperature. A method has been developed to save some time since going to extremely low temperatures can be time consuming. A temperature at which the resistance to thermal cracking parameters are satisfied, another - 10°C must be added to it. In case of the binder has not pass the BBR test parameters, another test can be introduced which is the Direct Tension Test.

4.8. Solvent

In this study Toluene has been used in solvent extraction and recovery process. Toluene is an aromatic hydrocarbon. It is colorless and water-insoluble. Toluene is primarily used as a solvent in paint thinners. The chemical formula is CH₇H₈. Toluene has a clear appearance and boils at 231°F(111°C). It is a highly flammable liquid. Cautions must be taken while handling as it can cause sever neurological damage. If swallowed it may cause skin irritation, drowsiness (Safety Data Sheet-Toulene).



Figure 4.7: Toluene Molecular Structural Formula

4.9. Rejuvenators

In this study three rejuvenators have been used. The rejuvenators are SylvaroadTM RP 1000, Hydrolene (H90T) and Industrial Soybean Oil.

4.9.1. SylvaroadTM RP 1000

SylvaroadTM RP 1000 is soluble in any grade of bitumen. It has clear and bright appearance and yellow to amber as color It is derived from Crude Tall Oil (STO), a renewable raw material that is a byproduct of the paper industry. For being a bio-based additive, it makes effective use of resources to ensure the road construction is more sustainable. SylvaroadTM RP 1000 is designed to fully restore the binder properties of recycled asphalt. Previously reclaimed asphalt pavement (RAP) was used in mixture as a "black rock". But Sylvaroad makes road construction sustainable by allowing higher amount of RAP binder in to the mixtures which also reduces the use of virgin materials. With the help of it reclaimed asphalt can be used in top layers of the pavement. This performance additive does not release any known detrimental components during production and application. It is non-hazardous and safe to handle. The additive is registered as a non-labeled product under REACH registration It also leads to lower energy consumption by allowing a lower drying temperature in the drum.

4.9.2. Hydrolene® H90T

Hydrolene has a high aromatic content that enables penetration and rejuvenation of asphalt quickly and efficiently. It improves fatigue performance by increasing aromatic resins lost during oxidation or aging. Hydrolene does not cause continuous age softening of the RAP asphalt binder which could be contributing factor for increased rutting. It offers greater workability and compaction in the RAP mix design because low viscosity. Hydrolene is well-matched with warm-mix foaming and additive packages. Hydrolene has a dark appearance and slight odor. Although Hydrolene allows higher amount of recycled binder in asphalt mix design but it is categorized as a hazardous material. It falls under carcinogenicity category 1B. It is suspected of damaging the unborn child and can causes damage to organs through prolonged exposure. Handling of Hydrolene strongly requires appropriate protective equipment in a well-ventilated environment. Flash point being greater than 200⁰F makes Hydrolene a combustible liquid. Storage of Hydrolene should be in original container protected from direct sunlight in a dry, cool and well-ventilated area, away from incompatible materials. Disposal of this additive must be according to federal, state and local regulations.

4.9.3. Industrial Soybean Oil

For past couple years soybean oil has been emerged as a potential recycling agent for rejuvenation of recycled asphalt. The soybean oil that has been used in this study is industrial grade which is distributed by RAP Technologies. Previous studies have concluded that a small amount of soybean oil can improve the fatigue resistance and low temperature cracking. Soybean oil offers excellent workability making the mixing easy with asphalt binder. It adjusts the ratio of asphaltene to maltene in aged binder to ensure better performance during service years of the pavement. For being a vegetable derived recycling agent, soybean oil is easy to handle and non-hazardous.

CHAPTER V

RESULTS & DISCUSSION

5.1. Introduction

This chapter discusses the results from the laboratory tests that were performed for this research. Primarily the test results from DSR and BBR are used in determining the optimum dosage for each of the three rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene). Moreover, test results are used observe the effect of rejuvenators on the blend of new binder and RAP binder. A statistical analysis is also conducted to find out which rejuvenator performs better.

5.2. Effect on viscoelastic parameters at different temperatures

To observe the effect of the selected rejuvenators (Sylvaroad, Industrial Soybean Oil and Hydrolene) on the viscoelastic properties, values of G*, δ and G*/Sin δ at different test temperatures had been plotted in the graphs. All the values of viscoelastic parameters correspond to the frequency of 10rad/sec which simulates the shearing action equivalent to a vehicle going 55 mph.

5.2.1. Stiffness comparison at unaged condition

Using initial low and high dosage for each of the rejuvenators two different blends had been prepared. Just after complete mixing, samples were collected to perform DSR test at high temperature. Samples that were collected right after mixing were at unaged condition. G* values were obtained from DSR tests on PG 64-22, recycled blend (PG 64-22 and 40% of RAP) and rejuvenated recycled blend with low and high dosage. Figure 5.1-5.6 represents the effect of rejuvenators at different temperatures at frequency of 10 rad/sec.

From figure 5.1-5.3 it can be observed that, at unaged condition, each rejuvenator shows great potential in reducing the stiffness of the recycled blend. Based on figure 5.1 and 5.3, Sylvaroad and Hydrolene appear to have shown same effect in terms of dropping the stiffness of the recycled blend at both low and high dosage. According to figure 5.2, higher dosage of Soybean Oil definitively had shown very little effect in lowering the stiffness from recycled blend with low dosage. However, adding high dosage of Soybean Oil is expected to decrease the stiffness of recycled blend significantly.



Figure 5.1: G* Comparison for Sylvaroad at Unaged Condition



Figure 5.2: G* Comparison for Soybean Oil at Unaged Condition



Figure 5.3: G* Comparison for Hydrolene at Unaged Condition

5.2.2. Stiffness comparison at short-term aged condition

Rolling thin film oven test was run on the recycled blend and rejuvenated blends to evaluate the effect of rejuvenators after short term aging. In the RTFO test, unaged rejuvenated blend of binder was used to simulate the aging effect. DSR tests were performed with RTFO aged samples and G* were obtained at 10 rad/sec frequency.

Figure 5.4-5.6 support affirmatively the effect of rejuvenators in restoring the G* value of recycled blends close to the stiffness of PG 64-22. Among the three rejuvenators Soybean oil showed great potential in terms of softening capability even after aging. However, it may be concluded that lower dosage than 12% is sufficient for Soybean oil to get the desired effect. Between, Hydrolene and Sylvaroad, the latter showed substantial restoring capability at higher dosage.



Figure 5.4: G* Comparison for Sylvaroad after RTFO aging



Figure 5.5: G* Comparison for Hydrolene after RTFO aging



Figure 5.6: G* Comparison for Soybean Oil after RTFO aging

5.2.1. Stiffness comparison at long-term aged condition

RTFO aged recycled blend and rejuvenated blend of binders were used in PAV machine to simulate long term aging effect. PAV aged binders were tested in DSR machine at intermediate temperatures. In this section effect of rejuvenators on recycled blend will be discussed after going through long term aging process.

From figure 5.7-5.9 it can be observed that Hydrolene had gradually lost its ability to soften the recycled binder while going through aging process for longer period. It is safe to conclude that among the three rejuvenators, Soybean oil is capable of keeping the recycled blend soft at significant degree after long service years.



Figure 5.7: G* Comparison for Sylvaroad after PAV aging



Figure 5.8: G* Comparison for Hydrolene after PAV aging



Figure 5.9: G* Comparison for Soybean Oil after PAV aging

5.2.2. Effect on phase angle and $G^*/Sin\delta$ at unaged condition

Phase angle is defined by the lag between applied shear strain and resulting shear stress. Larger phase angle value is an indication of viscous nature of the liquid. The larger the phase angle the more viscous the liquid. For being a viscoelastic material, asphalt's phase angle value ranges from 0 to 90 degrees. If the phase angle value is 90 degrees then the liquid is purely viscous and for 0 degree the liquid will be considered as pure plastic.

The effect of rejuvenators at different dosage on the phase angle of the recycled binder blend will be discussed in this section. Particularly this section will evaluate the effect on phase angle at unaged condition. The significance of the variation in phase angle relates to the Sin value of the angle, which is used to determine the PG grading of a binder. For high temperatures G*/Sinδ needs to meet specific value that is stated in Superpave PG binder specification for unaged and RTFO aged binder. So higher phase angle will give lower $G^*/Sin\delta$ value. For intermediate temperatures, at which PAV aged binders are tested in DSR, G* Sin\delta value is used to satisfy the threshold value listed in the Superpave PG binder specification. In this case, higher phase angle value will contribute to the larger G*Sin\delta value.

From figure 5.10 it can be noticed that at higher dosage Sylvaroad gave higher phase angle value with increasing temperature. It can be observed from the trends in the figure 5.10 that with increasing temperatures rejuvenators are capable of increasing the phase angle regardless of dosage rate. Therefore, the difference between phase angle values at low and high dosage is not significant. Thus figure 5.11 affirms that at unaged condition the G*Sin\delta value distribution over a range of high temperatures is governed by the stiffness, G*. The same conclusion can be drawn for Hydrolene and Soybean Oil based on figure 5.12-5.15.



Figure 5.10: Phase Angle Comparison for Sylvaroad at Unaged Condition



Figure 5.11: G*/Sino Comparison for Sylvaroad at Unaged Condition



Figure 5.12: Phase Angle Comparison for Hydrolene at Unaged Condition



Figure 5.13: G*/Sino Comparison for Hydrolene at Unaged Condition



Figure 5.14: Phase Angle Comparison for Soybean Oil at Unaged Condition



Figure 5.15: G*/Sino Comparison for Soybean Oil at Unaged Condition

5.2.3. Effect on phase angle and $G^*/Sin\delta$ at short-term aged condition

After going through aging process for short period the phase angle value is expected to be reduced than it was at unaged condition. This phenomenon occurs due to exposure to high temperatures and air pressure. The lower phase angle value contributes to binder's higher viscosity and higher value of stiffness. Each of the rejuvenators were capable of increasing the phase angle value. The difference between phase angle value at low and high dosage of rejuvenators found to be insignificant at unaged state. After RTFO aging, recycled blend with Sylvaroad and Soybean Oil demonstrated a distinctive pattern at increasing phase angle value. From figure 5.16 and 5.18 it is also observed that after aging, higher dosage (8%) of Sylvaroad restored the phase angle close to PG 64-22 whereas low dosage of Soybean Oil was capable of leaving the same effect at lower dosage (6%). Comparison between G*/Sin\delta distribution over a range of temperatures, from figure 5.17 and 5.19, proves that a high dosage of Soybean Oil can lower the recycled blend close to that of PG 64-22.



Figure 5.16: Phase Angle Comparison for Sylvaroad after RTFO aging



Figure 5.17: G*/Sino Comparison for Sylvaroad after RTFO aging


Figure 5.18: Phase Angle Comparison for Soybean Oil after RTFO aging



Figure 5.19: G*/Sino Comparison for Soybean Oil after RTFO aging

From figure 5.20 and 5.21 it can be concluded that although Hydrolene did not show clear pattern in the change due to different dosage but it was capable of increasing the phase angle value and lowering the $G^*/Sin\delta$ value to a significant extent.



Figure 5.20: Phase Angle Comparison for Hydrolene after RTFO aging



Figure 5.21: G*/Sinð Comparison for Hydrolene after RTFO aging

5.2.1. Effect on phase angle and $G^*/Sin\delta$ at long-term aged condition

Pressure aging vessel is used to simulate the long-term aging effect on binder's viscoelastic properties. All the rejuvenators are capable of softening the recycled blend even after going through long period of aging procedure. Figure 5.22-5.27 compare the phase angle value and the variation of stiffness parameter (G* Sinδ) from the data obtained from DSR tests on PAV aged blend of binders with different rejuvenators and recycled blend and PG 64-22. All the DSR tests on the PAV aged binder had been conducted at intermediate temperatures.

In fatigue parameter calculation, higher phase angle value contributes to higher stiffness. According to Superpave PG binder specification the threshold stiffness parameter, G*Sinô, is desired to be lower than 5000 kPa at intermediate temperatures. Based on the observations presented in the figure 5.22-5.27 each of the rejuvenators showed excellence in increasing phase angle value of the recycled blend. However, the G*Sinô value for Sylvaroad had no distinctive difference between high and low dosage. Among the three rejuvenators, Hydrolene showed poor rejuvenation effect after going through long term aging. From the data presented in figure 5.24-5.25, it can be concluded that Soybean Oil may demonstrate pronounced resistance against fatigue cracking.



Figure 5.22: Phase Angle Comparison for Sylvaroad after PAV aging



Figure 5.23: G*Sino Comparison for Sylvaroad after PAV aging



Figure 5.24: Phase Angle Comparison for Soybean Oil after PAV aging



Figure 5.25: G*Sino Comparison for Soybean Oil after PAV aging



Figure 5.26: Phase Angle Comparison for Hydrolene after PAV aging



Figure 5.27: G*Sinð Comparison for Hydrolene after PAV aging

5.2.2. Effect on stiffness and relaxation parameter at low temperature

Thermal cracking in asphalt pavement is a distress which occurs at low temperature. As surrounding temperatures gets low below 0^{0} C, pavements shrink and develop internal stresses. If this shrinkage occurs quickly the pavement may crack because it does not get the time to relax these stresses. As a result, thermal cracking develops on HMA layer. Asphalt binders that do no get hardened at low temperature and can easily relax the internal stresses are desirable. The two parameters, stiffness (S) and relaxation parameter (m value), are measured in Bending Beam Rheometer test system. PAV aged binders are used in BBR to determine these parameters.

In this section, BBR test data of PAV aged recycled blend (PG 64-22 and 40% RAP) and recycled blend with two dosages of each of the three rejuvenators is evaluated.

According to Superpave PG binder requirements stiffness value cannot exceed 300 MPa and m value should be minimum 0.3.







Figure 5.29: m value comparison for Sylvaroad at low temperatures

It was observed that stiffness value at tested temperatures for recycled blend did not exceed 300 MPa. However, m value at -22^{0} C is lower than 0.3. Therefore, attaining minimum threshold value for relaxation parameter of the binder is of utmost importance.

Comparing the observations presented in the figure 5.28-5.33, it can be concluded that with declining temperature, stiffness will increase and m value will decrease. Among the three rejuvenators, Sylvaroad and Soybean Oil was capable of improving the relaxation properties significantly.

















5.3. Effect on viscoelastic properties at different loading frequency

In this section asphalt binder's viscoelastic parameter G^* and δ are evaluated at a wide range of loading frequency for recycled blend and rejuvenated recycled blend by three rejuvenators (Sylvaroad, Soybean Oil and Hydrolene). Asphalt pavement is designed based on passenger vehicle for which the loading frequency is considered 10 rad/sec. DSR test results give G^* and δ value at different frequencies which range from 0.1 rad/sec to 100rad/sec. G^* and δ values at lower frequencies are taken into account for a pavement design where traffic volume is considerably small. Viscoelastic parameters at higher frequencies are used in pavement design for highways and interstate roadways.

In this section G* and δ values are plotted for each loading frequency obtained from DSR test results. Since the virgin binder in this research was PG 64-22 so the reference temperature for this assessment was 64°C for high temperatures and 25°C for intermediate temperatures according to Superpave PG binder requirements. G* and δ values for all the blend of binders were obtained at the reference temperature from the DSR tests on unaged, short-term aged and long-term aged binder.

5.3.1. G* and δ comparison at unaged condition

The figure 5.34-5.36 represent the dataset where $G^*/Sin\delta$ value at each loading frequency for recycled blend and recycled blend with the three rejuvenators at low and high dosages are plotted at unaged state. Similarly,

The figures representing phase angle values at corresponding loading rate showed that at certain range of lower frequency δ value continued to rise to higher than 90 degrees. Asphalt binder is a viscoelastic material. Phase angle greater than 90 degrees supports binder's pure viscous state. Higher phase angle value contributes to lower G* value. At lower frequency lower G* value indicates reduction in rutting resistance. Among the three rejuvenators, Soybean Oil contributed to higher phase angle value at lower frequency at both low and high dosages. This result suggests that thorough assessment should be performed prior selecting a dosage for Soybean Oil in pavement design consideration for low traffic volume road.



Figure 5.34: G*/Sinδ at different loading frequency for Sylvaroad (Unaged at 64⁰C)



Figure 5.35: G*/Sinδ at different loading frequency for Soybean Oil (Unaged at 64⁰C)



Figure 5.36: G*/Sinδ at different loading frequency for Hydrolene (Unaged at 64⁰C)

5.3.2. G*/Sinδ comparison at short-term aged condition

Resistance against rutting is a major concern in first few weeks after laying top surface of pavement containing asphalt mixture. Asphalt mixture starts to get aged from the time of manufacturing in the plant. After placement of hot mix asphalt, pavement is opened to traffic. During first week rutting may distress the pavement due to large traffic volume. Since the asphalt binder goes through certain amount aging during mixing and placement the threshold value for G*/Sin\delta is no longer considered 1kPa. The minimum value of G*/Sinδ for short term aged binder is 2.2 kPa according to Superpave PG binder requirement.

 $G^*/Sin\delta$ value at each loading frequency is also presented in the figure 5.37-5.39 at RTFO aged state. Response pattern in terms of rutting parameter at loading frequencies were found to be similar for each of the rejuvenator. However, recycled blend with high Sylvaroad content (8%) seemed to be the most effective in lowering $G^*/Sin\delta$ compared to other two rejuvenators.



Figure 5.37: G*/Sinδ at different loading frequency for Sylvaroad (RTFO at 64⁰C)



Figure 5.38: G*/Sinδ at different loading frequency for Soybean Oil (RTFO at 64⁰C)



Figure 5.39: G*/Sinδ at different loading frequency for Hydrolene (RTFO at 64⁰C)

5.3.3. G*Sin\delta comparison at long-term aged condition

In this research PAV aged binders were used to evaluate the effect of rejuvenators at intermediate temperatures. Stiffness at intermediate temperature indicates resistance against repeated loading. The pavement distress due to repeated loading at intermediate temperature is termed as fatigue cracking. In this section, G*Sinð value at a wide range of loading frequency was discussed for the selected rejuvenators. According to Superpave PG binder requirements fatigue resistance parameter, G*Sinð, should be less than 5000 kPa. High viscosity and stiffness makes the binder brittle in nature, which facilitates the process of fatigue cracking. The fatigue parameter (G*Sinð) of the recycled blend at 25⁰C is much lower than the threshold value. Therefore, having rejuvenator in the recycled blend would reduce the stiffness further. Differences between the G*Sinð value for recycled blend with high (8%) and low (4%) content of Sylvaroad were not much. Hydrolene, however showed marginal distinction in values of fatigue parameter at low (4%) and high (10%) content. Based on figure 5.41, it can be assumed that in long-term service year Soybean Oil with high content (\approx 12%) will provide better resistance against fatigue cracking at intermediate temperature.



Figure 5.40: G*Sinδ at different loading frequency for Sylvaroad (PAV at 25⁰C)



Figure 5.41: G*Sinδ at different loading frequency for Soybean Oil (PAV at 25⁰C)



Figure 5.42: G*Sinδ at different loading frequency for Hydrolene (PAV at 25⁰C)

5.4. Effect of rejuvenators on rutting parameter at reference temperature

The main objective of using rejuvenator is to maximize the use of RAP in mixtures and substitute the new binder to minimize the initial production cost. But optimizing the dosage is a matter of great concern as higher dose can decrease the rutting resistance and lower dose can make asphalt the mixture vulnerable against thermal cracking. In this study, to determine the effective dosage of rejuvenators which will improve the resistance against rutting and thermal cracking, a low and a high dose for each rejuvenator has been evaluated. The effect of the three rejuvenators on high temperature parameters and low temperature parameters at different aging procedure have been investigated. To understand the influence better this evaluation has been focused on the temperatures of target binder which is PG 64-22. The target high temperature is 64°C, the intermediate temperature is 25°C and low temperature is -22°C, according to AASHTO MP1.

5.4.1. Effect on rutting parameter at high temperature

The G*/Sin δ values have been plotted for the target binder, the recycled binder with and without rejuvenators at unaged condition in the figure 5.43, 5.45 and 5.47 and for RTFO aged condition in the figure 5.44, 5.46 and 5.48.

The change in $G^*/Sin\delta$ value at $64^{\circ}C$ confirms the expected effect of adding rejuvenator in the recycled blend as the they contribute to softening of the aged binder. The

change in drop in stiffness from low dosage to high dosage is minimal. Therefore, it can be concluded that softening ability of the rejuvenator is dependent on dosage rate.

It is also observed that rate of change in stiffness seemed proportionate in the both of the unaged and RTFO aged binders. With regard to resistance against rutting, performance of Soybean Oil was of concern as it softened the recycled blend significantly compared to Sylvaroad and Hydrolene.



Figure 5.43: $G^*/Sin\delta$ value comparison for Sylvaroad (Unaged at $64^{\circ}C$)



Figure 5.44: G*/Sinδ value comparison for Sylvaroad (RTFO aged at 64⁰C)



Figure 5.45: G*/Sinð value comparison for Soybean Oil (Unaged at 64⁰C)



Figure 5.46: G*/Sinδ value comparison for Soybean Oil (RTFO at 64⁰C)



Figure 5.47 G*/Sinδ value comparison for Hydrolene (Unaged at 64⁰C)



Figure 5.48: G*/Sinδ value comparison for Hydrolene (RTFO at 64⁰C)

Anova tests had been conducted to determine the effect of dosage on the recycled blend in terms of rejuvenation capability. Table 5.1 and table 5.3 presents Anova test results on unaged and RTFO aged binders respectively. $G^*/Sin \delta$ values had been analyzed against dosage rate in the aforementioned tables. The results confirmed that the rejuvenation capability of the rejuvenators is significantly dosage rate dependent. Table 5.2 and 5.4 represents Tuckey comparison between the rejuvenated recycled blends for unaged and RTFO aged binders respectively. Low dosage rates range from 4% to 6% and high dosage rates range from 8% to 12%. At unaged state both Sylvaroad and Hydrolene at low dosage lowered the G*/Sin δ value. Rejuvenation performance of Hydrolene and Sylavaroad at higher dosage is equivalent to Soybean Oil at low dosage (6%). The same rejuvenation effect was seen RTFO aged binders containing rejuvenators.

Table 5.1: ANOVA analysis (dosage dependent) for $G^*/Sin\delta$ Values at unaged state

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Dose	6	18.542	3.09034	2999.06	0
Error	14	0.0144	0.00103		
Total	20	18.5564		-	

Table 5.2: Tukey Pairwise comparison (dosage dependent) for $(G^*/Sin\delta)$ at unaged state

Blend	N	Mean	Grouping
Recycled blend	3	4.33595	А
RB_Hydrolene_low	3	2.33394	В
RB_Sylvaroad_low	3	2.31565	В
RB_Soybean Oil_low	3	1.81064	С
RB_Hydrolene _high	3	1.64712	С
RB_Sylvaroad _high	3	1.63666	D
RB_Soybean Oil _high	3	1.2986	E

Table 5.3: ANOVA analysis (dosage dependent) for $G^*/Sin\delta$ Values at RTFO aged state

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Dosage	6	179.321	29.8868	781.47	0
Error	14	0.535	0.0382		
Total	20	179.856			

Table 5.4: Tukey Pairwise comparison (dosage dependent) for $G^*/Sin\delta$ RTFO aged state

Blend	Ν	Mean	Grouping
Recycled blend	3	12.5707	А
RB_SYL_low	3	6.236	В
RB_HYD_low	3	6.1901	В
RB_ISO_low	3	4.629	С
RB_HYD_high	3	4.2202	CD
RB_SYL_high	3	3.8587	DE
RB_ISO_high	3	3.3538	Е

5.4.2. Effect on fatigue parameter at intermediate temperature

According to Superpave PG binder requirement fatigue parameter value G*Sinð should not exceed 5000 kPa. In this section all the observation corresponded to intermediate temperature of 25^oC.Recycled blend itself was expected to perform well in fatigue resistance as G*Sinð value is less than the critical value at intermediate temperature (25^oC) Therefore, conclusion can be drawn based on the figure 5.49-5.51 that all of the three rejuvenators would give excellent resistance against fatigue cracking. However, Hydrolene did not lowered G*Sinð value and might lose its rejuvenation effect over time. Sylvaroad gave agreeable G*Sinð value with the increment of dosage. Among the three rejuvenators, Soybean Oil had lowered the fatigue parameter significantly over long period of time.

Anova test results showed significant effect dosage rate (table 5.5) on the fatigue parameter ($G^*Sin\delta$) of the recycled blend after PAV aging process. Based on the table 5.6 it can be concluded that Soybean Oil at low dosage demonstrated dominance over Hydrolene and Sylvaroad at high dosage.

Tukey comparison among the rejuvenators showed that Hydrolene did not continue to lower the stiffness of the binder significantly after going through long period of aging process. Among the three rejuvenators Soybean Oil showed excellent softening efficiency than the other two rejuvenators.



Figure 5.49: G*Sinδ value comparison for Sylvaroad at 25^oC



Figure 5.50: G*Sinδ value comparison for Soybean Oil at 25⁰C



Figure 5.51: G*Sinδ value comparison for Hydrolene at 25⁰C

Table 5.5: Anova analysis (dosage dependent) for G* Sinδ at intermediate temperatures

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Dosage	6	6421358	1070226	40.59	0
Dobuge	Ũ	0.21000	10,0220	10.07	0
Error	14	369108	26365		
Total	20	6790466			

Dosage	N	Mean	Grouping
Recycled blend	3	3250.39	А
RB_HYD_low	3	3138.23	А
RB_HYD_high	3	2380.86	В
RB_SYL_low	3	2260.77	В
RB_ISO_low	3	2102.21	В
RB_SYL_high	3	2067.94	В
RB_ISO_high	3	1591.53	С

Table 5.6: Tukey Pairwise comparison (dosage dependent) for G^* Sin δ at intermediate temperatures

5.4.3. Effect on stiffness and relaxation parameter at low temperature

Stiffness value will increase and relaxation capability will decrease with declining temperature. Introducing higher dosage of rejuvenators will improve the relaxation properties.

Observations presented in the figures 5.52-5.57 revealed that, at reference temperature -22^oC, all the rejuvenated binder blends gave m value greater than 0.3. Sylvaroad was capable of improving relaxation property at lower dosage than Soybean Oil. Hydrolene did not show promising effect in increasing m value.

Anova test results are presented to assess the effect of dosage rate on the stiffness and relaxation properties at low temperature. The reference temperature for this assessment is -22^{0} C as it is corresponding temperature of PG 64-22. At low temperature, when binder's stiffness increases its relaxation ability decreases. The observations presented in the table 5.7-5.8 proved significant effect of dosage rate on stiffness (S) and relaxation parameter (m value) respectively.

Based on dosage rate, Tuckey comparisons among the rejuvenators were presented in the table 5.9-5.10. The observations reaffirmed the fact that Soybean Oil was the most effective in reducing stiffness and increasing relaxation ability of recycled blend at low temperature. With regard to performance Sylvaroad was the second best among the three rejuvenators.



Figure 5.52: Stiffness comparison for Sylvaroad (at -22⁰C)



Figure 5.53: m value comparison for Sylvaroad (at -22⁰C)



Figure 5.54: Stiffness comparison for Soybean Oil (at -22^oC)



Figure 5.55: m value comparison for Soybean Oil (at -22° C)



Figure 5.56: Stiffness comparison for Hydrolene (at -22^oC)



Figure 5.57: m value comparison for Hydrolene (at -22° C)

Table 5.7: Anova analysis (dosage dependent) for Stiffness (S) at low temperatures

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Dose	6	16086	2680.96	30.24	0
Error	14	1241	88.66		
Total	20	17327		-	

Table 5.8: Tukey Pairwise comparison (dosage dependent) for Stiffness (S) at low temperatures

Dose	N	Mean	Grouping
Recycled blend	3	186.67	А
RB_HYD_low	3	164.67	AB
RB_SYL_low	3	143.67	BC
RB_HYD_high	3	142	BCD
RB_ISO_low	3	130.67	CD
RB_SYL_high	3	116	DE
RB_ISO_high	3	96.47	Е

Table 5.9: Anova analysis (dosage dependent) for m value at low temperatures

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Dose	6	0.007095	0.001183	116.04	0
Error	14	0.000143	0.00001		
Total	20	0.007238			

Table 5.10: Tukey Pairwise comparison (dosage dependent) for m value at low temperatures

Dose	N	Mean	Grouping
RB_ISO_high	3	0.34367	А
RB_SYL_high	3	0.33433	В
RB_ISO_low	3	0.315	С
RB_HYD_high	3	0.314	С
RB_SYL_low	3	0.312	С
RB_HYD_low	3	0.30167	D
Recycled blend	3	0.28367	Е

5.5. Drop in Stiffness due to increment of rejuvenator dosage

To better understand the relationship between dosage rate and aging condition relative comparison have been made with $G^*/Sin\delta$ value for 64^0C at unaged and short term aged recycled binder blends with rejuvenators and without rejuvenators.

In the Table 5.11 the drop of stiffness due to each of the rejuvenators at 1% dosage increment has been calculated. For both of Hydrolene and Sylvaroad, the change in stiffness due to aging at same rejuvenator dosage rate remains unchanged. However, Industrial Soybean oil has been affected greatly by the aging procedure at higher dosage.

It might be emphasized that dosage rate for Industrial Soybean Oil is critical as it continuously softens the binder while going through aging process.

Table 5.11: Drop in stiffness due to 1% increment of d	osage
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Recycled Binder	G*/Sinð	Drop in	G*/Sinð	Drop in
Blends	(Unaged)	Stiffness	(RTFO)	Stiffness
		(Unaged)		(RTFO)
Recycled Blend	4.3		12.67	
Recycled Blend	2.32	11.65%	6.24	12.7%
+4% Sylvaroad				
Recycled Blend	1.63	7.78%	3.86	8.69%
+8% Sylvaroad				
Recycled Blend	2.33	11.54%	6.2	12.79%
+4% Hydrolene				
Recycled Blend	1.65	6.2%	4.22	6.67%
+10% Hydrolene				
Recycled Blend	1.81	9.71%	4.63	10.58%
+6% Soybean Oil				
Recycled Blend	1.3	5.84%	3.35	12.26%
+12% Soybean Oil				

Table 5.12: Drop in stiffness due to 1% increment of dosage at intermediate temperature

Recycled Binder Blends	G*Sinð (kPa)	Drop in Stiffness (PAV)
Recycled Blend	3250.388	
4% Sylvaroad	2260.772	7.612%
8% Sylvaroad	2067.945	4.547%
4% Hydrolene	3138.233	0.863%
10% Hydrolene	2380.864	2.675%
6% Soybean Oil	2102.213	5.887%
12% Soybean Oil	1591.529	4.253%

In the table 5.12 drop in stiffness based on two dosages for each of the rejuvenator has been presented. The data correspond to 25^oC and 10 rad/sec. Drop in stiffness value corresponds to 1% increment dosage increment. With increment of dosage Sylvaroad demonstrates inverse effect due to long term aging. Additionally, aging does not affect the softening capability of Industrial Soybean Oil with increased dosage. However, Hydrolene had performed poor in the long-term aging process.
5.6. Dosage Selection Method

Dosage of rejuvenators should be selected carefully as excessive amount of rejuvenator will significantly soften the recycled binder which contributes to rutting. Contrariwise, a low dosage may reduce the brittleness of the recycled binder but not improve the cracking resistance. Arámbula-Mercado et al. (2017) has conducted a study where he evaluated three methods of recycling agent dosage selection. These three methods are restoring PGL and verifying PGH, achieving $\Delta T_c = -5^{\circ}C$ and restoring PGH. He concluded that restoring PGH provided with the better results and ΔT_c values compared to the other two methods. This method restored the stiffness and phase angle value to an acceptable range after aging. Optimum dosage selection method of rejuvenator, based on the conclusion drawn by Arámbula-Mercado et al. (2018), that has been used in this research is restoring PGH.

5.7. Determining Optimum Dosage of Rejuvenators

To determine the amount of recycling agent required to match the PGH of the blend of PG 64-22 and RAP binder to the grade of the target binder, which is PG 64-22, the continuous grade high and low temperatures were calculated for each of the rejuvenators from the DSR and BBR test results. The dosage percentages (no rejuvenators, low dose and high dose) were plotted against the continuous grade temperatures. From these graphs, with the help of regression equation, optimum dosage for the three rejuvenators were found out restoring the PGH or higher performance grade temperature.

5.7.1. Dosage Rate of SylvaroadTM RP 1000

For SylvaroadTM RP 1000, low dosage and high dosage were selected 4% and 8% respectively by weight of RAP binder. To restore the PGH, the dosage was increased until the PGH reached a value of 67.5^oC, which rounds to a PG 64-XX per AASHTO M 320. In this approach the dosage is found out to be 8.0% which is marked by point A (8,67) in figure 5.58. At this rejuvenator dosage the PGL of the blend is estimated from regression equation for PAV m-controlled line and yielded a value of -25.74^oC as marked by point B (8, -25.74) in figure 5.58. This value of PGL meets the target PGL of the target binder based on AASHTO M 320. So, a dosage of 8% for Sylvaroad is selected to restore to a continuous performance grade of PG 67-26, which corresponds to the PG 64-22 target binder after rounding by 6^oC increments per AASHTO M 320.



Figure 5.58: PG blending chart with selected dosage for Sylvaroad

5.7.2. Dosage Rate of Industrial Soybean Oil

Low dosage and high dosage for Industrial Soybean Oil were selected 6% and 12% respectively by weight of RAP binder. The dosage was increased until the PGH reached a value of 67.5^oC, which rounds to a PG 64-XX per AASHTO M 320. In this approach the dosage is found out to be 9.6% which is marked by point B (9.6,67.5) in figure 5.59.



Figure 5.59: PG blending chart with selected dosage for Industrial Soybean Oil

5.7.3. Dosage Rate of Hydrolene H90T

In this research, for Hydrolene, the low dosage and high dosage were used 4% and 10% respectively by weight of RAP binder. As described before, the dosage was increased until the PGH reached a value of 67.5° C. For Hydrolene, the PGH restoring dosage is found out to be 10% which is marked by point A (10,67.5) in figure 5.60. Corresponding PGL of the blend, at 10% dosage rate, is estimated from regression equation for PAV m-controlled line and yielded a value of -23.7° C as marked by point B(10, -23.7) in figure 4.3. Based on AASHTO M 320 the estimated value of PGL meets the target PGL of the target binder.

So, a dosage of 10% for Hydrolene is selected to restore to a continuous PG of PG 67-24, which matches to the PG 64-22 target binder after rounding by 6⁰C increments per AASHTO M 320.



Figure 5.60: PG blending chart with selected dosage for Hydrolene

5.8. Effect of Rejuvenators on the Binder's Continuous Grade

Introducing the rejuvenator in asphalt binder changes the continuous grade temperature at unaged, short term and long term aged state. These Continuous grading temperature values will be used in developing the regression equation for each of the rejuvenators. Continuous grading done in this research were provided as seen in table 5.13. The formulas that were used for interpolation for G*/Sin∂, Stiffness (S) and m-value in this study is based on ASTM D7643-16.

The data provided in the table 5.13 and figure 5.61-5.62 clearly show that rejuvenators have significant effect on the blend of PG 64-22 with 40% RAP binder compared to the blend with no rejuvenator. Based on the criteria to determine the performance grade of a binder per AASHTO M 320, among the three rejuvenators Industrial Soybean Oil has shown the most softening effect with a low dosage to restore the performance grade of the blend same as the target binder.

Table 5.13 Continuous Grade of The Binders

	Tc for	Tc for	Tc for	Tc for
Binder Blend	Unaged	RTFO	Stiffness	m Value
RAP	92.8	101.4	-20.7	-14.3
Recycled Blend	76.1	76.1	-26.3	-19.8
Recycled Blend +4%Sylvaroad	70.9	72.1	-27.6	-23.3
Recycled Blend +8%Sylvaroad	68.1	68.4	-29.4	-25.5
Recycled Blend +6%ISO	68.9	69.8	-27.9	-23.7
Recycled Blend +12%ISO	66.3	67.4	-30.0	-26.1
Recycled Blend +4%Hydrolene	70.8	72.0	-26.4	-22.2
Recycled Blend +10%Hydrolene	68.1	69.0	-27.2	-23.4



Figure 5.61: True performance grade of the binders for high temperature



Figure 5.62: True performance grade of the binders for low temperature

5.9. Verifying $\Delta T_c = -5^0 C$

 ΔT_c defined as the numerical difference between the low continuous grade temperature determined from the Bending Beam Rheometer (BBR) stiffness criteria (which is the temperature at which stiffness, S is equal to 300 MPa) and the low continuous grade temperature determined from the BBR m-value (which is the temperature at which m equals 0.300). This parameter is used to measure the ductility loss of aged asphalt binder, particularly for explaining block cracking. Block cracking is a non-load related cracking phenomenon comparable to thermal cracking that causes cracks to develop in both longitudinal and transverse directions. According to previous studies with aging of asphalt binders, the low temperature relaxation parameter, as measured by the BBR m-value, deteriorate significantly faster than the low temperature stiffness increases. ΔT_c helps to address the issue of explaining the effect of ductility of the aged binders more accurately. To limit the risk of thermal cracking, Anderson et al. (2011) recommended a maximum ΔT_c threshold of -5^o C after 40-hour PAV aging. However, establishing this ΔT_c value would contribute in selecting high rejuvenator dosage which would increase the cost and decrease the rutting resistance of mixture. Thus, the standard 20-hour PAV aging is used evaluate $\Delta T_c = -5^0 \text{ C}$ at selected optimum dosage for the three rejuvenators.

In the following table 5.14 ΔT_c values have been listed for the three rejuvenators:

Pland	Dosage	T _c	T _c	۸T
Біена	Rate	(Stiffness)	(m Value)	
Recycled Blend	0%	-26.3	-19.8	-6.6
Recycled Blend+ Sylvaroad	8%	-29.27	-25.74	-3.534
Recycled Blend+ Industrial Soybean Oil	10%	-29.33	-25.1	-4.23
Recycled Blend+ Hydrolene	10%	-27.13	-23.68	-3.45

Table 5.14: ΔT_c value for the blends at selected dosage

 ΔT_c value for the blend of the binders with no rejuvenators shows proneness to brittleness. With the addition of rejuvenators, the blend significantly improves its susceptibility to brittleness by satisfying $\Delta T_c \leq -5^0 C$. ΔT_c values of the three rejuvenators are close to each other which do not show any superiority over one another.

CHAPTER VI

CONCLUSION AND RECOMMENDATION

6.1. Introduction

After proper treatment of RAP and appropriate mix design procedure along with the modern technology and research will support the asphalt plants to increase RAP use. To fully utilize the RAP material rejuvenators have garnered attention in asphalt industry. Higher amount of RAP material in mix design offer many unique benefits to pavements that are difficult to match with the use of softer virgin binders.

In this study effect of three rejuvenators were evaluated. The rejuvenators were Sylvaroad, Soybean Oil and Hydrolene. A low and high dosage were recommended by manufacturer. The selected performance grade binder was PG 64-22 and selected RAP percentage was 40% of total binder weight. Each of the rejuvenated blend was aged for short term (RTFO) and long term (PAV). Both unaged and aged asphalt blends were tested in DSR for high and intermediate temperatures to find out true high temperature grade. PAV aged samples were also tested in BBR to determine true low grade of the blends. Based on the DSR and BBR test results optimum dosage content was found out

6.2. Conclusion

Based on the experimental test results, the following relevant observations and conclusions were made:

- Effect of rejuvenators on viscoelastic parameters at high temperature
 - Sylvaroad and Hydrolene were similarly effective in lowering stiffness and increasing phase angle at same amount of low dosage. However, to restore viscoelastic properties of PG 64-22, Hydrolene will need higher dosage comparing to both Sylvaroad and Soybean Oil.
 - Among the three rejuvenators, Industrial Soybean Oil managed to restore the viscoelastic property of the recycled blend as close to PG 64-22.
 - Sylvaroad, at higher dosage of 8%, was as effective as Soybean Oil at higher dosage of 12%. Rejuvenation capability of Sylvaroad had increased upon aging. This outcome indicates that Sylvaroad might be more effective than Soybean Oil.
 - Although Soybean Oil had demonstrated great potential at lowering stiffness but it can be detrimental for the asphalt pavement due to rutting problem. The dosage selection for Soybean Oil needs to be evaluated prior mix design.
- Effect of rejuvenators on viscoelastic parameters at intermediate and low temperature
 - Sylvaroad was reliable in lowering stiffness effectively. Soybean Oil was the best at reversing the age of the recycled blend to great extent. Fatigue cracking

will less likely to be developed if Soybean Oil is used in pavement containing higher amount of RAP. However, Hydrolene had lost the rejuvenating capability while going through aging procedure.

- Recycled blend had at low temperature had stiffness lower than the specified maximum value according to Superpave PG binder specification. Monitoring stiffness at low temperature was not a concern. The relaxation capacity of the recycled blend was however altered. Soybean Oil and Sylvaroad had facilitated to get higher m value at both high and low dosage.
- Effect of rejuvenators on viscoelastic parameters at different loading frequency
 - In DSR tests recycled blend with different rejuvenators were tested at different loading frequency at both unaged and aged state. All the rejuvenators showed similar trend as it was seen for viscoelastic parameters at different temperatures.
- Effect of rejuvenators on performance grade temperature
 - Soybean Oil followed by Sylvaroad, effectively lowered the true low and high temperature performance grade of the recycled blend.
- Optimum rejuvenator content
 - Optimum dosage for Sylvaroad, Soybean Oil and Hydrolene was found to be 8%, 10% and 10% respectively. These dosages will allow higher percentage of RAP (40%) in the asphalt mix design. Additionally, ΔT_c values for these dosages were found to be less than -5^o C.

- Statistical Analysis
 - The Anova test results revealed that the dosage content had a significant impact on the rutting parameter at high temperature, fatigue parameter at intermediate temperature, stiffness and relaxation property at low temperature.
 - Tukey comparison based on the dosage content supported the conclusions presented in the previous discussions.

6.3. Recommendation

The following additional studies are recommended to assess the possibility of using higher percentage of RAP (more than 40%) and the rejuvenator efficiency:

- This study considered only 40% (of total binder weight) of RAP binder. Different higher percentages can be considered to study the effect on the recycled blend.
- Instead of using different high and low dosages, a fixed rate can be applied for each rejuvenator to evaluate the effect on same variable.
- DSR tests at a wide range of high and intermediate temperatures will aid in developing master curves for complex shear modulus for a wide range of loading frequencies. These master curves can be used in assess the effect of rejuvenator with high amount of RAP at any loading frequency.
- Further assessment can be done using optimum dosage of rejuvenators in mix design.

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