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# Investigating Effects Of Amine-Based Modifier On Recycled Asphalt Shingles Blending Index

Govinda Sedhay North Carolina Agricultural and Technical State University

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Investigating Effects of Amine-based Modifier on Recycled Asphalt Shingles Blending Index

Govinda Sedhay

North Carolina A&T State University

A thesis submitted to the graduate faculty

in partial fulfillment of the requirement for the degree of

MASTER OF SCIENCE

Department: Civil, Architectural and Environmental

Major: Civil Engineering

Major Professor: Dr. Elham H. Fini

Greensboro, North Carolina

2014

The Graduate School North Carolina Agricultural and Technical State University This is to certify that the Master's Thesis of

Govinda Sedhay

has met the thesis requirements of North Carolina Agricultural and Technical State University

> Greensboro, North Carolina 2014

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#### Biographical Sketch

Govinda Sedhay was born on 27<sup>th</sup> of November, 1981 in Lamjung, Nepal. He completed his Schooling from Earthly Paradise School, Besishahar, Lamjung. He received undergraduate in Civil Engineering from Institute of Engineering, Western Regional Campus. During his undergraduate studies he developed interest towards the transportation related courses. He was interested in the asphalt technology and flexible pavement technology. In order to pursue his interest and desire to experience the world class education, he started his graduate studies at North Carolina A & T State University from fall 2012 under the guidance of Dr. Elham H. Fini. Based on his research results, Govinda has developed a manuscript which was submitted to Journal of *American Journal of Engineering and Applied Sciences*. He also presented his research results via a poster during energy day at NC A&T State University. Furthermore, he is planning to present his work at ASCE conference to be held in Charlotte, NC in April 2014.

Dedicated to my parents, Mr Tulasi Prasad Sedhain & Yam Kumari Sedhay and beloved

wife Susmita Poudel.

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#### Abstract

<span id="page-15-0"></span>This study was undertaken to investigate the effects of amine-based modifiers on the rheological characteristics of particle-filled viscous media such as recycled asphalt shingles (RAS). RAS are a recycled material that contains high concentrations of asphalt which has the potential for use in hot mix asphalt (HMA) when added to virgin asphalt. When using the RAS as a binder in HMA it is important to mix it with the virgin asphalt properly to achieve the best performance, which can also be enhanced by the incorporation of amine-based modifiers. Tear-off shingles were acquired from a roofing company and ground very fine so that 85% of the particles passed through sieve number 200. The virgin asphalt binder (PG  $64-22$ ) and three (20%, 30%, & 40%) percentages of grounded RAS were blended at a temperature of 180°C at a rotational speed of 400 rpm. These three mixtures were then blended with three different amine-based modifiers  $(1.5\%$  of Rediset®, 0.5% of Evotherm®, and 5% of bio-binder by weight of mixture) at 135 °C and a rotational speed of 400 rpm. The percentage of each modifier was selected based on recommendations of the manufacturers. The properties of the blended binder were studied using a rotational viscometer (RV) utilizing a Brookfield Viscometer DVIII-Ultra. Two different spindles were used to measure the viscosity of the binders at four different temperatures  $(105^{\circ}C,$ 120 $^{\circ}$ C, 135 $^{\circ}$ C, and 150 $^{\circ}$ C) and six (5, 10, 20, 25, 50, and 100) different rotational speeds. The analysis showed that viscosity increased with increasing percentages of RAS; however, the viscosities decreased after incorporation of the amine-based modifiers. Additionally, viscosity results were found to be different between the two spindles used. Viscosity measurement values were consistently higher when the vane spindle was used as compared to the smooth spindle. This can be attributed to incomplete blending of the RAS particles with asphalt matrix. However, the viscosity difference between the two spindles was reduced as the temperature was increased

and when modifiers were present. This, in turn, indicates an improvement of blending due to the addition of modifier and an increase in blending temperature. Furthermore, the coefficient of variation was significantly lower in cases where the vane spindle was used, indicating that the vane spindle could be more appropriate for measuring the viscosity of particle-filled viscous media.

 An empirical relation was proposed to measure the blending behavior of the amine-based modified binders. The blending index was calculated using an empirical relation for all temperatures and rotational speeds. It was found that the blending index was affected by changes in temperature and shear speed. The blending index increased with increasing temperature. In addition, the bio-binder modified binder showed higher blending index compared to the other modified binders. Therefore, bio-binder is effective in reducing binder viscosity and enhancing blending between aged asphalt in RAS and un-aged asphalt (PG 64-22) in the mixture.

#### **CHAPTER 1**

#### **Introduction**

#### <span id="page-17-1"></span><span id="page-17-0"></span>**1.1 Background**

 The United States has the largest road network system in the world comprising more than 4 million miles of pavement. Of this, 2.3 million miles are surfaced with hot-mix asphalt (FHWA, 2011); therefore, hot-mix asphalt is the predominant material in pavement construction, rehabilitation, and maintenance projects (National Asphalt Association, 2005). Due to increases in population and living standards there is a significant annual increase in traffic. It is important that the entire pavement surface have sufficient capacity to bear the rapid growth of traffic volume, high axle loads, and severe climatic conditions. To fulfill this increasing demand in the US, thousands of miles of road are constructed and thousands of miles of road are maintained and rehabilitated each year. All types of road construction work require sufficient amounts of materials such as aggregate, asphalt binder, fuel, coal, and so forth. Most of these construction materials occur naturally and can be found at various sources (e.g., mines, wells). Extraction of these materials leads to their gradual depletion. Additionally, extracting these materials from quarries and transporting them to job sites is difficult and costly, thereby increasing overall construction costs.

 Accordingly, transportation agencies are increasingly interested in investigating new technologies that will reduce the cost of asphalt pavement materials while maximizing long-term performance. The American Society of Civil Engineers (ASCE) 2009 Infrastructure Report Card revealed that 186 billion dollars is needed annually for rehabilitation and maintenance of the United States roadway system, but only 70.6 billion dollars is being invested annually. The cost of asphalt materials can be reduced by replacing the virgin asphalt (binder) with recycled

products obtained from construction waste or other byproducts that contain large amounts of asphalt. Adopting the use of recycled products not only reduces the cost of asphalt materials but also diverts construction waste away from landfills. Using recycled products to manufacture asphalt pavement also lowers the energy required to produce the pavement and minimizes the process's impact on the environment. Al-Qadi, Imad, Elseifi, and Carpenter (2007) reported that the performance of properly designed asphalt mixes containing recycled products exhibits no differences compared to asphalt mixes containing only virgin materials. When compared to conventional virgin mixes some recycled products even improve performance in certain applications.

 Recycled asphalt pavement (RAP), recycled asphalt shingles (RAS), and Crumb rubber are the most common sources of secondary recycled materials used in road construction. RAP is old pavement that has been milled from the roadway, crushed into smaller aggregate sizes, and stockpiled. At the end of an asphalt pavement's "service life," the pavement is still valuable because it contains mineral aggregates and asphalt cement that can be reheated and reincorporated into new hot mix asphalt (HMA). The Federal Highway Administration (FHWA) and the Environmental Protection Agency (EPA) reported that of the 100.1 million tons of asphalt pavement removed each year, 80.3 million tons is reused as part of new roads, roadbeds, shoulders, and embankments, making asphalt the United States' most recycled material (FHWA, 1993).

 Another source for secondary materials is recycled asphalt shingles (RAS) that are obtained by reprocessing old roofing asphalt sheet (shingles). Used roofing shingles are recyclable products and have been used in road construction as an aggregate or a binder (NAHB Research Center, 1998). The use of RAS has several benefits with respect to the environment

and the conservation of energy. The use of RAS in pavement construction is a more sustainable and eco-friendly approach. RAS are primarily used in new HMA for pavement surfaces; however, they have been increasingly used as coarse aggregate and binder materials for construction of new roads or the rehabilitation of existing roads. Used roofing shingles are available in large quantities. More than 11 million tons of waste asphalt roofing shingles are generated in the United States each year (Krivit, 2007). The cost of disposing waste shingles in landfills can be as high as \$90 to \$100 per ton (Malik, Teto and Mogawer, 2000). Additionally, disposing of shingles in landfills increases municipal disposal costs and pollutes the environment. When shingles are re-used in new construction these problems are minimized.

 RAS have been used in pavement construction since the early 1990s; at that time RAS were incorporated into hot mix asphalt (HMA). Pavement containing RAS was used in a trial section of roadway in the state of Pennsylvania (Pennsylvania Department of Transportation, 2011). The Pennsylvania Department of Transportation (2011) oversaw a project in July, 1991 in which 0.93 miles of four-lane highway was constructed with an asphalt mixture containing 5% RAS. This was the first commercial application of pavement formulated using RAS. In the same year, the Minnesota Department of Transportation (Mn/DOT), the Minnesota office of Environmental Assistance (Mn/OEA), and the University of Minnesota started a research project to investigate the effectiveness of using RAS in HMA (Janisch & Turgeon, 1996). In this study it was found that the use of up to 5% RAS was beneficial in that it caused a slight increase in asphalt cement hardness.

Janisch and Turgeon's (1996) findings, increases in energy prices, and the gradual depletion of natural resources all served to stress the compulsory need to adopt new methodologies that would benefit the environment, users, and the industry. Although recycling by-product materials is beneficial in most cases because of the reduced consumption of virgin materials, it is imperative that the performance of the highway is not compromised.

Benefits that may derive from the recycling of by-product materials in HMA include:

- 1. reduced consumption of virgin materials,
- 2. reduced emissions and energy consumption during processing and manufacturing as a result of using fewer virgin materials,
- 3. reduced amount of by-product material disposed in landfills,
- 4. diminished consternation of the public regarding emissions, and

5. improved economic competitiveness in the asphalt paving construction industry. Clearly, recycling asphalt shingles in HMA could be a valuable approach in the road

construction industry.

#### <span id="page-20-0"></span>**1.2 Problem Statement**

 Disposing of roofing shingles at the end of their service lives results in the accumulation of large quantities of old asphalt materials in landfills. If these materials are not properly treated, serious environmental problems can result. The fabrication of additional HMA from virgin aggregate and bituminous materials only compounds the problem. RAS have been considered a valuable construction material because they can be included in new hot mix asphalt for use in both the construction and maintenance of paved surfaces. According to the FHWA, the use of RAS in the production of new asphalt materials results in economic, environmental, and energy savings (Roof to rood).

 Recently, RAS have been increasingly used as a coarse material (dry process) and as a part of the fabrication of binder material (wet process). The latter method is more commonly used in the pavement industry. In this method RAS is blended with virgin binder using a

shearing mechanism. Blending of RAS with virgin asphalt decreases the quantity of virgin material required and thereby lowers construction costs. The new challenge is to use higher percentages of RAS in pavement construction without reducing performance. Elseifi, Salari, Mohammad, Hassan, Daly and Dessouky, 2012 demonstrated a method called the "wet process" in which they increased the percentage of RAS without depleting the required performance criteria. In this wet process, RAS were ground into a very fine form called "ground RAS." The ground RAS was then blended with virgin binder material at high temperatures and high shear rates prior to mixing with the aggregate so that the RAS mixture could act as a binder. Using the wet process facilitated better control of the chemical and physical reactions taking place in the binder blend. . Elseifi, et al., 2012 reported that using this method up to 20% RAS can be used in road construction materials without compromising the performance of the road. Although Elseifi, et al.'s (2012) wet process was based on manufactured recycled shingles. A search of the extant literature revealed that no research has been conducted focusing only on tear-off roofing shingles and their blending behavior with virgin asphalt; therefore, this research study focused on the blending behavior, performance, and characteristics of RAS with virgin asphalt in the presence of three different modifiers as reflected in the resultant blending index.

#### <span id="page-21-0"></span>**1.3 Research Objectives**

 The main disadvantage of using high percentages of RAS in construction is the increased stiffness of the mixture. This, in turn, can make the mixture hard to mix and compact. To address this problem, certain amine-based modifiers are added to the mixture to reduce the stiffness and enhance workability. To evaluate the effects of various modifiers, viscosity can be measured to determine the rheological behavior of the modified mixtures. The objective of this research was to evaluate the physical and rheological properties and performance of mixtures modified with

RAS with and without the presence of amine-based modifiers. In addition, this work evaluated the effectiveness of the designed modifiers in RAS-modified mixtures in terms of improving the blending between aged and un-aged asphalt. In this study the viscosity of samples was measured using two different types of spindles. The measured viscosity data obtained from this research has the potential to introduce new concepts that might be used to determine the blending index of mixtures, thereby providing a new approach for further research and development of new modifiers for flexible pavements.

#### <span id="page-22-0"></span>**1.4 Experimental Plan**

 This study was designed to investigate the effect of amine based modifiers on RAS modified asphalt rheological characteristics. The methodology used in this study was an extensive literature review on RAS and RAP modified Hot Mix Asphalt technology incorporation of various additives. To conduct the experiments, three different proportions (20%, 30%, and 40%) of RAS were blended with PG 64-22 in the first step. Then the RAS modified mixtures were blended with bio-binder, Redised®, and Evotherm® respectively in the second step. In total, 24 different specimens were prepared: (a) four specimens made from 20% RAS and virgin binder with or without modifiers, (b) four specimens made from 30% RAS with virgin binder with or without incorporation of modifiers, and (c) four made from 40% RAS with virgin binder with or without incorporation of modifiers. Each of these 12 specimens was processed using both spindle type SC4-27 and spindle type V73.

 In this research the proportion of Redised® was 1.5%, Evotherm® was 0.5%, and biobinder was 5% by weight total mixture. To make a homogeneous mixture, each combination of RAS and virgin asphalt were first blended for 60 minutes at 400 rpm and 180°C; similarly, each modifier was blended separately with RAS modified mixture (already prepared) at 400 rpm and

 $130^{\circ}$ C for 20 minutes. From each blended mixture 10.5 grams of binder was poured into an aluminum chamber. The chamber was then placed into a preheated Thermosel® for 20 minutes. To measure viscosity a Brookfield Engineering viscometer was chosen and tested. A dynamic shear rheometer (DSR) test was conducted to measure the viscoelastic properties of the binders.

 An experiment set up used in this study is shown in Figure 1.1; furthermore, in detail each mixture design and modification will described in chapter three in section 3.3 and 3.4.



<span id="page-23-1"></span>*Figure 1.1* Experiment set up used in this study

#### <span id="page-23-0"></span>**1.5 Organization of Thesis**

 This thesis is divided into five chapters including this introductory chapter. Chapter 1 is used to present background, the problem statement, research objectives, and research methodology. Chapter 2 contains an extensive review of the literature pertaining to the use of RAS in HMA and a discussion of past and ongoing studies of recycled asphalt materials. In Chapter 3 the materials, an evaluation of their properties, and test methodologies used in this study are described. Chapter 4 is used to present and discuss the test results and data analysis of the modified binders evaluated, and Chapter 5 contains the summary and conclusions of the research work conducted.

#### **CHAPTER 2**

#### **Literature Review**

<span id="page-25-0"></span>This chapter is designed to introduce RAS as a construction material for use in asphalt pavement. Some previous applications of RAS in HMA used in the pavement industry are discussed. A thorough literature review regarding the laboratory tests which are performed in this research are also included.

#### <span id="page-25-1"></span>**2.1 Past Studies of Recycled Asphalt Materials**

 Environmental measures are becoming more dominant factors in the decision-making process in infrastructure and construction projects. Additionally, global crude oil prices have increased rapidly in recent decades. The price of liquid asphalt has grown dramatically; the price of asphalt increased from \$235/ton in 2004 to more than \$635/ton in 2013 (New York Department of Transportation, 2013). As a product derived from petroleum distillation, asphalt is becoming less available because of improvements in cooking technologies that allow refineries to produce synthetic fuel from asphalt. This, in turn, reduces the supply of asphalt available for road construction (Cleveland, 1993). An increasing concern for sustainable development, in addition to the emphasis on material conservation, reuse, and recycling, has encouraged a number of government and highway agencies to commission research investigations to characterize, and optimize the production of pavement materials. The use of recycled materials can provide additional value. They have been used in applications that show performance similar to conventional materials and cost effectiveness has been demonstrated (Iswandaru & Wilson, 2006). These successes have driven researchers and pavement industry companies to address the issue of using more and more recycling materials derived from waste products. For example, biobinder, RAS, and RAP can be used as alternative asphalt resources while looking for substitutes

for virgin asphalt (Fini, Kalberer et al., 2011). Similarly, with regard to RAS, in the US more than 11 million tons of asphalt roofing materials are produced each year. Ten million tons are post-consumer (tear-off), and one million tons are pre-consumer manufacturing scrap. Asphalt roofing shingles have been used in paving practices since the early 1990s as a portion of aggregate, and more recently have also been used as a binder in hot mix asphalt. Due to the presence of large quantities of asphalt in RAS, most state agencies that regulate road construction have allowed RAS to be used with certain maximum percentages in hot mix asphalt. The maximum allowable percentage of RAS in most states is approximately 5% by weight of the total aggregate. Some states limit RAS type to manufacturing scrap only, while others allow for the application of tear-offs as well. For example, following the supplemental specification issued by the Ohio Department of Transportation in 2011, the state of Ohio allowed the use of either manufacturer's RAS or tear-off RAS depending on the particular pavement course (Ohio Department of Transportation, 2011). Figure 2.1 shows the states that currently allow the utilization of RAS in HMA.



<span id="page-26-0"></span>*Figure 2.1*. Allowable percentage of RAS in HMA (from Scholz, 2010)

 Several authors of previous studies mentioned that the introduction of shingles into an asphalt mixture can increase the stiffness of the mixtures which can, in turn, promote pavement resistance to rutting. Ali et al., 1995 and Burak et al., 2004 studied the effects on engineering properties that resulted from introducing roofing shingles into HMA. They found that the Marshall Stability values increased when adding 1% shingle into the mixture but that further increasing the percentage of shingle caused a decrease in stability values. However, they noted that at concentrations of up to 5% shingle the stability values of the mixtures were still higher than the minimum values called out in the superpave specification criteria. In addition, this and other studies showed that by incorporating 5% shingle in pavement construction contractors can reduce the construction cost by \$2.79/ton (Brock et al., 1989). In another study, Foo et al. (1999) reported that the introduction of shingles into virgin asphalt can cause a significant increase in the stiffness of the asphalt binder. The use of shingles in a HMA mixture will generally improve the rutting resistance; however, the mixture may show lower fatigue life and lower thermal cracking resistance. In such cases it was recommended that the use of an appropriate softener (bio-binder) in virgin binder could improve the fatigue and low temperature performance of the mixture (Fini, Al-Qadi et al., 2011). There are several studies and innovations related to facilitating the application of RAS without compromising the "workability" and mechanical properties of the mixture (e.g., application of softer binder, mechanical grinding, and wet processing). Recently, a bio-based additive was produced that is able to make the binder softer, thereby enhancing workability and mixing (Mogawer et al. 2012, Fini, Al-Qadi et al., 2011, Beale, 2011 and; Williams, 2013).

 Another innovation was grinding the RAS to ultra-fine particle size and blending it with asphalt binder through a wet process. This facilitated the incorporation of higher percentages of RAS in hot mix asphalt (Elseifi et al., 2012). In this wet process, the ground RAS is blended with the binder at a high temperature prior to mixing it with the aggregates. This method permits better control of the chemical and physical reactions which occur in the binder blend. Results of the rheological and stability testing for this wet process indicated that 20% RAS can be used successfully in HMA.

 Fini et al. (2011) studied the effects of bio-binders on mixtures containing RAS. A biobinder derived from swine manure was added to the base PG 52-28 asphalt binder at a concentration of 5% by weight of asphalt binder and a bio-modified binder was created. Due to the chemical and physical nature of the bio-binder, its introduction along with RAS allowed mixing at a lower temperature of 124ºC and compaction occurred at 113ºC. This study showed that the presence of bio-binder led to improved blending between the aged asphalt and the virgin asphalt. In addition, it was found that bio-binder improved the workability and compaction of the RAS content mixture. In another study by Fini, Al-Qadi et al. (2011) on the analysis of dynamic modulus of mixtures it was shown that incorporation of 40% RAP to the control mixture increased the mixture stiffness. The introduction of the bio-modified binder decreased the 40% RAP mixture's stiffness; therefore, it indicated that the bio-modified binder can effectively reduce the mixing and compaction temperatures and help to reduce the stiffness effect caused by the introduction of high percentages of RAP and RAS in the mixtures.

 Based on previous research studies it became clear that aged RAS are one of the constituents that help increase the viscosity of the mixture but may cause stress on the pavement during preparation, mixing, compaction, and during its life of operation. To address this issue different types of modifiers and additives are being used in construction according to their properties and design guidelines/specifications. In an attempt to establish a suitable design

method, a new method called warm mix asphalt (WMA) was introduced recently. Marisa et al. (2012) conducted a Marshall Stability Test, immersion compression test, and water sensitivity test on a warm recycled mix. They concluded that the temperatures for the production and compaction of the mixtures influenced the final results. The best result was obtained from mixtures compacted at 90 °C.

#### <span id="page-29-0"></span>**2.2 Recycled Asphalt Shingles (RAS)**

 Shingles are manufactured for 15–20 years of service. After their life service time they are replaced by new roofing shingles which produces a large quantity of waste/scrap shingles. Reuse of recycled asphalt shingles was identified by the U.S. Environmental Protection Agency (EPA) as a top priority. Constituents of typical asphalt shingle include 20-35% asphalt cement, 2-15% cellulose felt, 20-38% mineral granule/aggregates, and 8-40% mineral filler/stabilizer. Due to the high content of asphalt in shingles, the primary application of RAS is production of hot mix asphalt. Most states' departments of transportation (DOT) approved 5% (depends upon the type) RAS in HMA. Research by Button et al. (1995) and Grodinsky (2002) revealed that the use of more than 5% by weight RAS in HMA affected adversely the creep stiffness and tensile strength of HMA. Consequently, this 5 % RAS application uses only 10-20% of the total asphalt shingle waste generated (Turley, 2010). To make use of the additional waste another potential application of RAS could be incorporation into structural fill including highway embankment fills or backfill behind retaining walls.

 Asphalt shingles contain approximately 30% AC by mass (Foo, 1999); therefore, using RAS in HMA decreases the amount of virgin AC required, and decreases the costs to produce HMA. It can also enhance the properties of the HMA when small amounts of RAS are incorporated; however, this improvement may be dependent upon the source and quality of the RAS. The roofing application of shingles and the demolition of the roofing shingles are shown in Figure 2.2 and Figure 2.3.

<span id="page-30-0"></span>

*Figure 2.2* Asphalt shingles on a residential roof

<span id="page-30-1"></span>

*Figure 2.3* Tear-off shingles after service life

 The granular material in asphalt shingles is composed of coal slag and crushed rock coated with ceramic metal oxides. It is generally uniform in size, ranging from 0.3mm to 2.36 mm, and is hard and angular when powdered limestone (70% passing the No. 200 sieve) is also added as a stabilizer (Newcomb, 1993; Ross, 1997) which makes the mixture stiffer.

#### <span id="page-31-0"></span>**2.3 Types of Recycled Asphalt Shingles**

 Understanding the composition and properties of asphalt shingles is necessary to fully characterize asphalt mixtures in which they are incorporated. The American Society for Testing and Materials (ASTM) clearly specifies shingles according to their production in documents ASTM D225 and ASTM D3462. The specifications in ASTM D225 apply to asphalt shingles made with organic (cellulose or wood fiber) backing, and ASTM D3462 contains specifications for asphalt shingles made with fiberglass backing.

<span id="page-31-1"></span> **2.3.1 Organic shingles.** Organic shingles are made of paper (felt)-saturated asphalt cement (AC). These types of shingles are heavier and contain more AC. In cold regions, such as the northern USA and Canada, these shingles are used due to the higher flexibility conferred by the large AC content. The increased flexibility makes them less likely to crack in cold weather.

<span id="page-31-2"></span> **2.3.2 Fiberglass shingles.** Fiberglass shingles contain a base layer (mat) of fiberglass coating. These types of shingles are easier to work with and install because the fiberglass base makes the shingles lighter in weight. Fiberglass shingles provide greater resistance to moisture and fire than organic shingles.

#### <span id="page-31-3"></span>**2.4 Typical Asphalt Shingle Composition**

 The percentages of the individual component materials in asphalt are different in shingles manufactured with organic felt compared to shingles manufactured with fiberglass felt. Brock (2007) summarized the composition of each type of shingle and his reported data are presented in Table 2.1. Typical figures for each individual component are shown in Figure 2.4.

#### <span id="page-32-0"></span>Table 2.1

	<b>Organic Shingles</b>		<b>Fiberglass Shingles</b>		<b>Old Shingles</b>	
<b>Composition</b>	(Lbs. per	$(\%)$	(Lbs. per	$(\% )$	(Lbs. per	$(\%)$
	$100$ sq. ft.)		$100$ sq. ft.)		$100$ sq. ft.)	
Asphalt	68	30	38	19	72.5	31
Filler	58	26	83	40	58	25
Granules	75	33	79	38	75	32
Mat	$\overline{0}$	$\overline{0}$	$\overline{4}$	$\overline{2}$	$\mathbf{0}$	$\theta$
Felt	22	10	$\mathbf{0}$	$\mathbf{0}$	27.5	12
Cut-out	$\overline{2}$	$\mathbf{1}$	$\overline{2}$	$\mathbf{1}$	$\boldsymbol{0}$	$\boldsymbol{0}$
Total	225		206		233	

*Composition of Both Types of Asphalt Shingles (from Brock, 2007)* 

 Shingles are manufactured by saturating and coating both sides of organic or fiberglass backing felt with liquid asphalt. The asphalt used to coat the felt material is different from asphalt used in paving materials. The asphalt used in roofing shingles is much harder and stiffer because the manufacturers use an "air-blown" process to increase the viscosity of the asphalt. The air-blown process infuses oxygen into the asphalt, which changes the chemical make-up of the asphalt and makes it stiffer. The shingles are then covered with sand and crushed-stone granules to increase their durability and resistance to weathering. The individual components of asphalt shingles are shown in Figure 2.4.



*Figure 2.4* Composition of asphalt shingles (from Grzybowski, 2010)

#### <span id="page-33-2"></span><span id="page-33-0"></span>**2.5 Asphalt Cement Content in Tear-off Shingles**

 Weathering a portion of the surface granules on roofing shingles results in a greater overall percentage of AC compared to new shingles. Oxidation and volatilization of the lighter organic compounds in roofing shingles makes the AC in tear-off shingles stiffer. As a result, using higher percentages of RAS in HMA can lead to the mix being stiffer than a virgin mix. Tear-off shingles tend to include nails, paper, wood, and other debris that makes recycling a longer process (Mallick, 2000). Care and consideration should be taken when RAS is added to HMA to avoid this potential contamination.

#### <span id="page-33-1"></span>**2.6 Benefits of Recycled Asphalt Shingles in Hot Mix Asphalt**

 The benefits of using shingles in HMA include cost savings, environmental preservation, and the potential for improved performance. Recycling RAS in HMA avoids the expense associated with the disposal of shingle waste and reduces the amount of material entering landfill sites, thereby benefitting the environment. The amount of virgin AC required in HMA mixes can be reduced by incorporating RAS; this reduces costs. A relatively small number of shingles can

displace a large percentage of AC (Foo 1999) in hot HMA. Additionally, studies revealed increased resistance to high-temperature rutting in HMA that contained factory waste shingles (Foo, 1999). The benefits of using RAS include:

- 1. reduced consumption of virgin materials,
- 2. reduced emissions and energy consumption during processing and manufacturing of virgin materials,
- 3. reduced amounts of by-product materials disposed of in landfills,
- 4. diminished public consternation over emissions,
- 5. improved economic competitiveness in the asphalt paving construction field,
- 6. reduced or eliminated disposal costs for municipalities,
- 7. established attractive and wise solution for the use of waste materials, and
- 8. decreased dependency on virgin asphalt.

#### **CHAPTER 3**

#### **Materials Used and Experiment Methodology**

<span id="page-35-0"></span> In this chapter each of the materials that were used in this study are characterized. Asphalt binder PG 64-22 and modifiers (Rediset® and Evotherm®) are characterized based on the manufacturer or supplier specification. Tear-off shingles and Bio-binder are characterized based on the Fini et al (2011), Elseifi et al. (2012), Iswandaru & Wilson (2006), and Burak et al. (2004) research and findings.

#### <span id="page-35-1"></span>**3.1 Materials Characterization**

<span id="page-35-2"></span>**3.1.1 Evotherm®.** Evotherm<sup>®</sup> is a warm mix additive/modifier used successfully in warm mix technology in asphalt pavement construction. Evotherm<sup>®</sup> WMA is a comprehensive chemical additive system designed to allow the production and compaction of high quality asphalt pavements at temperatures much lower than those needed in conventional HMA. The benefit is the reduced consumption of energy when manufacturing the asphalt mixes. Various job sites studied by Michel, Frederic and Faucon (2003) achieved energy savings of approximately 40% percent, with measured gains ranging from 35% to 55% depending on the moisture content of the aggregate materials and the ambient weather conditions. Additionally, the reduction in processing temperatures caused a significant drop in the emission rates of stack gases and particulates at the mix plant. One study showed a 48% reduction in greenhouse gases, 58% reduction in nitrogen oxides, and 41% reduction in sulfur dioxide, which is responsible for acid rain (Michel et al., 2003).

<span id="page-35-3"></span> **3.1.2 Virgin asphalt binder.** Un-modified binder which was classified as PG 64-22 according to Superpave specifications was selected for this study. This bitumen is a petroleumbased refined product. Typical heating temperature of the bitumen is 177˚C with a flash point of
325˚C. Preferred storage temperatures range between 140˚C and 168˚C. The use of unnecessarily high temperatures results in increased hardening, oxidation, and heating costs. PG 64-22 is primarily used in paving for both new construction and pavement rehabilitation. (U.S. Oil & Refining Corporation, 2005). It was used in an attempt to offset the potential mixture stiffening resulting from the use of a high percentage of RAS in the mixture. Based on the viscosity of the binder, the mixing temperature was 180ºC. Table-3.1 shows properties of the virgin binder.

Table 3.1

<b>Property</b>	<b>Test Method</b>	<b>Value</b>
Flash Point, °C	ASTM D92,	Varies according to grade,
Cleveland Open Cup	EN 22592 (b)	Typically > $230^{\circ}$ C (445°F).
		$>$ 270°C (520°F) in ASTM D312,
		$>$ 250°C (482°F) in EN 13304
Loss on Heating, % m	ASTM D2872,	$0.5-1\%$ maximum depending upon
(Maximum)	EN 12607-1	the specification
Specific gravity	<b>ASTM D70</b>	$\geq$ 0.95, typically $>$ 1.0, not a specification
value	EN 15326	
Solubility, %	ASTM D2042,	$\geq$ 0.99% m by specification
(Minimum)	EN 12592	(Trichloroethylene, Toluene, or Xylene
		as specified)
Solubility in water		Negligible
Softening Point	<b>ASTM D86, EN 172</b>	$>$ 30°C (86°F, grade dependent
Vapor Pressure		Below detection limit at ambient temperature

*Typical Physical Properties of Asphalt Binder (ASTM International, 2013)* 

 **3.1.3 Recycled asphalt shingles (RAS).** The shingles used in this study were postconsumer (tear-off) type shingles which were acquired from a local roofing company in Greensboro, North Carolina. Further work (preparation) was undertaken in civil engineering labs at North Carolina A & T State University.

 **3.1.4 Bio-binder.** Bio-binder is derived from non-petroleum-based renewable resources such as wood or corn. Recent research efforts have suggested that using a bio-binder along with a petroleum-based asphalt can produce a bio-modified binder (Fini, Al-Qadi, Zada B. and Beale, 2011 and; Williams, 2013); therefore, the bio-binder could be an alternative to petroleum-based asphalts. In this study bio-binder used was produced by thermochemical liquefaction processing of swine manure under relatively high temperature ( $T = 340^{\circ}$ C) and pressure ( $P = 10.3$  MPa) for specific residence times  $(RT = 80 \text{ min.})$  is used to produce bio oil and utilizes the heavy residue remaining in this process as an asphalt modifier. Table 3.2 shows the chemical composition of bio-binder and asphalt.

Table 3.2





**3.1.5 Rediset®.** The warm mix asphalt at lower compaction temperature needs lower optimum binder content to conform to the mix design criteria; its stability is lower than mixture fabrication at high temperatures. The use of a lower temperature leads to less energy consumption and lower emissions production at asphalt mixing plants. Hamzaha, Golchina and Ching (2013) study showed that the optimum binder content (OBC) of warm mix asphalt (WMA) was slightly lower than the OBC for HMA. Furthermore, the higher Rediset® content slightly decreased the stability of the asphalt mixture. This implied that higher Rediset® content has a softening role in the asphalt mixtures (Hamzaha et al., 2013). Table 3.3 shows the recommended concentration of Rediset® for various applications.

Table 3.3

*Recommended Doses of Liquid Rediset® by Weight of Mixture (AkzoNobel Surface Chemistry, 2013)* 



#### **3.2 Preparation of RAS**

 The tear-off shingles used in this study were obtained from a local roofing company in Greensboro, North Carolina. Dirty particles like iron nails, wood, paper, pieces of glass, and other debris were separated from the shingles. The separated shingles were then ground utilizing an industrial Hamilton Beach grinder. Grounded RAS was then gradated to isolate the required

particle size samples using sieve analysis. The grounded RAS were put on the top of the sieve and shaken for 20 minutes using an automatic shaking mechanism. A typical Hamilton Beach grinder is shown in Figure 3.1. The various sieve sizes are shown in Table 3.4.

Table 3.4





# **3.3 Specimens Preparation**

PG 64-22 was placed in a typical bench oven set to 135°C until it reached a homogeneous liquid phase. To prepare the samples

- 1. 150 grams of heated PG 64-22 was poured into three cans; 20%, 30%, and 40% RAS by weight of total mixture was blended with heated PG 64-22; and
- 2. 5% of bio-binder, 1.5% of Rediset®, and 0.5% of Evotherm® by weight of total mixture were blended separately.

 The details of the mixing proportions and titles given to each modified binder are shown in Table 3.5. In the title of each modified binder, first two letters (MB) stands for modified binder; followed by two digits which stand for percentages of recycled asphalt shingles (RAS) and a single letter (E, R, B) which stands for the type of amine-based modifiers: "E" for Evotherm**®**, "R"for Rediset**®**, "B" for Bio-binder from swine manure and "N" for no modifiers. Table 3.5

<b>Binders</b>	<b>Percentage Content (%)</b>					
<b>Name</b>	<b>RAS</b>	PG 64-22	<b>Evotherm®</b>	<b>Rediset®</b>	<b>Bio-binder</b>	
Control	$\boldsymbol{0}$	100	$\mathbf{0}$	$\mathbf{0}$	$\mathbf{0}$	
$MB-20-N$	20	80	$\mathbf{0}$	$\overline{0}$	$\overline{0}$	
$MB-20-E$	20	79.5	0.5	$\boldsymbol{0}$	$\boldsymbol{0}$	
$MB-20-R$	20	78.5	$\mathbf{0}$	$\overline{1.5}$	$\mathbf{0}$	
$MB-20-B$	20	$\overline{75}$	$\overline{0}$	$\overline{0}$	$\overline{5}$	
$MB-30-N$	30	$70\,$	$\overline{0}$	$\overline{0}$	$\boldsymbol{0}$	
$MB-30-E$	30	69.5	0.5	$\mathbf{0}$	$\boldsymbol{0}$	
$MB-30-R$	30	68.5	$\overline{0}$	1.5	$\overline{0}$	
$MB-30-B$	30	65	$\boldsymbol{0}$	$\boldsymbol{0}$	5	
$MB-40-N$	40	60	$\overline{0}$	$\overline{0}$	$\theta$	
$MB-40-E$	40	59.5	0.5	$\boldsymbol{0}$	$\boldsymbol{0}$	
$MB-40-R$	40	58.5	$\boldsymbol{0}$	1.5	$\theta$	
$MB-40-B$	40	55	$\mathbf{0}$	$\boldsymbol{0}$	5	

*Description of Proportion of Test Materials* 



*Figure 3.1* A typical grinder used to prepare the tear-off shingles

# **3.4 Mixing (Blending) Process**

 To perform the mixing, 12 aluminum cans were taken and filled by preheated asphalt binder of 150 grams. Among those 12 cans, four cans were blended with 20% grinded recycled asphalt shingles, four cans were blended with 30% of RAS, and the last four cans were blended with 40% of RAS at high temperature and shear speed. Single cans representing each percentage (a total of three cans) were kept separate and used as control samples. Three sets of cans were made from the rest of the cans (a total of nine cans) with each of the three groups containing 20%, 30%, and 40% RAS. The first set of cans was blended with 0.5 % Evotherm®. Similarly, the second set of cans was blended with 1.5% Rediset®, and the third set of cans was blended with 5% bio-binder at high temperature and rotational speed. Each blended mixture was then poured into a small aluminum chamber. Altogether there were 12 specimens for spindle SC27 and an equal number of specimens for the V73 spindle. Modification details are presented in the next section.

**3.4.1 RAS modification.** For this modification three cans were filled with 150 grams of PG 64-22 asphalt binder. These cans were then placed on a heating plate set to  $180^{\circ}$ C in preparation for blending. To make a 20% RAS mixture 37.5 grams of RAS was gradually poured into 150 grams of PG 64-22making 187.5 grams of binder. An electric drill with a mixing attachment was used for 60 minutes of blending. This procedure was then repeated for the remaining two mixture designs: 30% RAS was formed by gradually adding 64.30 grams of RAS to 150 grams of PG 64-22 to form 214.28 grams of binder, and 100 grams of RAS was gradually added to 150 grams of PG 64-22 to make 250 grams of 40% RAS binder. All samples were then heated to  $180^{\circ}$ C until the blending process finished. This process was adopted for all cans of each percentage of RAS.

**3.4.2 Bio-binder modification.** After RAS modification, the samples were treated with modifiers. For bio-binder modifications, each percentage of RAS-modified binder was treated with a 5% bio-binder. The mixture including 5% bio-binder (9.86 grams) was poured into the 20% RAS modification (187.50 grams). To prepare for blending this 196.87 grams of mixture was then placed on a heating plate set to  $135^{\circ}$ C. Once again an electric drill with a mixing attachment was used to blend for 20 minutes during blending. This procedure was then repeated for the remaining mixture designs. Five percent bio-binder (10.71 grams) was gradually added to 214.28 grams of 30% RAS-modified binder over a heating plate at  $135^{\circ}$ C and blended for 20 minutes. Similarly, 5% bio-binder (12.5 grams) was gradually added to 250 grams of 40% RASmodified binder and blended. It was assumed that the mixtures were homogenous after undergoing this process.

**3.4.3 Rediset® modification.** The proportion of Rediset® was 1.5% by weight of binder for the Rediset® modifications. The mixture including 1.5% Rediset® (2.81 grams) was slowly

poured into the 20% RAD-modified (187.50 grams) mixture and then placed on a heating plate set to 135<sup>o</sup>C. An electric drill with a mixing attachment was used to blend. Blending time was set to 20 minutes. This process was repeated for the remaining two mixture designs: 1.5% Rediset® (3.21 grams) was gradually added to the 30% RAS-modified (214.28 grams) mixture, and 1.5% (3.75 grams) Rediset® was added gradually to the 40% RAS-modified (250 grams) binder.

**3.4.4 Evotherm® modification.** The proportion of Evotherm® was 0.5% by weight of total mixture for Evotherm® modification. The mixture including 0.5% Rediset® (0.94 grams) was poured into the 20% RAD-modified (187.50 grams) mixture and then placed on a heating plate set to  $135^{\circ}$ C where the blending takes place. An electric drill with a mixing attachment was used for 20 minutes of blending. This process was then repeated for the remaining two mixture designs: 0.5% Evotherm® (1.07 grams) was gradually added to the 30% RAS-modified (214.28 grams) binder and 0.5% Evotherm® (1.25 grams) was added to the 40% RAS-modified (250 grams) binder. In all blending processes the shearing (rotational speed) was 400 rpm.

#### **3.5 Experiment Method**

**3.5.1 Viscosity measurements.** The viscosity of the prepared specimens was measured at different temperatures and shear rates using a Brookfield viscometer (RV-DVIII Ultra) followed by the ASTM D4402 test procedure. To prepare the test specimens; after blending, two specimens were prepared from each modification by pouring 10.5 grams of blended binder into the tiny aluminum chambers shown in Figure 3.2. Altogether twenty-four specimens were prepared for viscosity measurements: one set (twelve) of specimens was for smooth spindle (SC 27) and the second set (twelve specimens) was for vane spindle (V73) and these tubes (aluminum chambers) were then placed in the preheated Thermosel® for 30 minutes to reach thermal equilibrium.



*Figure 3.2* Aluminum chambers used in this study

 To investigate the properties of the modified binders, the viscometer was set to temperatures of 105°C, 120°C, 135°C, and 150°C at speeds of 5, 10, 20, 25, 50, and 100 rpm. The samples were then preheated by putting them into the Thermosel® set to the designated temperatures for an additional 20 minutes to ensure the achievement of thermal equilibrium. The test was run and the results recorded three times at 1-minute intervals to ensure the viscosity measurements were consistent. In this study two spindles (SC4-27 and Vane Spindle V73) were used to measure the mixture viscosity. Figure 3.3 shows a conventional oven used to preheat the samples in this study and Figure 3.4 shows the two different types of spindles used. Figure 3.5 is a typical blending mechanism used for blending (mixing) in this study.



*Figure 3.3* Conventional oven used to preheat the samples



*Figure 3.4* (a) Smooth spindle *SC 4-27* and (b) Vane spindle *V73*





**3.5.2 Temperature susceptibility.** Temperature susceptibility is a measure of how fast binder properties change with changes in temperature (Claudy & Martin, 1998). The temperature susceptibility of the RAS-modified asphalt blends was evaluated by developing temperatureviscosity plots for the specimens prepared. If an asphalt binder has a high susceptibility to

temperature, its viscosity changes rapidly as the temperature changes. Asphalts with high temperature susceptibility are undesirable as they are more prone to undergo thermal and UV oxidation (Firoozifar & Foroutan, 2011). Therefore, it is important to quantify numerically the temperature susceptibility of the binders. The following equation has been commonly used to calculate temperature susceptibility (VTS; Rasmussen, Lytton, & Chang, 2002).

$$
VTS = \frac{LogLog(\eta 2) - LogLog(\eta 1)}{Log(T2) - Log(T1)}
$$
................. Equation 3.1

where

 $T_1$  and  $T_2$  are the temperatures of the binder at known points in Rankin units (R), and

 $\eta_1$  and  $\eta_2$  are the viscosities of the binder at the known points (cp).

The magnitude of the VTS is directly proportional to the temperature susceptibility of the asphalt binder.

**3.5.3 Shear susceptibility.** Shear susceptibility is defined as the rate of change in viscosity with the shear rate (Roberts et al., 1996). The shear susceptibility, also known as the shear index, is determined by calculating the slope of the line formed by a log of rotational speed versus the log viscosity graph using Equation 3.2 (Raouf & Williams, 2010 a).

$$
SS = \frac{Log(Viscosity)}{Log(Shear Rate)}
$$

where

 viscosity is the measured deformation by shear or tensile stress, and speed is the rate at which shear is applied to the material.

 Prior studies showed that binder with relatively small shear susceptibility (low gains in shear susceptibility relative to the increase in viscosity) result in better overall pavement performance (Roberts et al., 1996).

**3.5.4 Blending index.** Blending index is an indication of the degree of blending achieved between the oxidized binder in RAS and virgin binder. The blending index of the RAS-modified binder was evaluated using viscosity variation versus temperature. Using the difference between the two measurements at the same temperature and speed rate, a blending index was defined as follows:

BX = LogLog(η(SC27)) Loglog(η(V73)) ∗ LogLog(T) ∗ 100% ...........................Equation 3.3

where

T is the temperature of the binder at known points expressed in degrees Celsius  $(°C)$ , and  $\eta_{SC27}$  and  $\eta_{V73}$  are the viscosities of the binder at known points (cP).

# **3.6 Dynamic Shear Rheometer (DSR) Test**

 The dynamic shear rheometer (DSR) is an instrument used to characterize the viscous and elastic behaviors of asphalt binders at medium to high temperatures. This characterization is used in the superpave PG asphalt binder specification. Due to the viscoelastic nature of asphalt it behaves partly like an elastic solid (deformation due to loading is recoverable; is able to return to its original shape after load is removed) and partly like a viscous liquid (deformation due to loading is non-recoverable; it cannot return to its original shape after the load is removed). DSR measures an asphalt's complex shear modulus  $(G^*)$  and phase angle  $(\delta)$ .

 The complex shear modulus is the ratio of total shear stress (ηmax-ηmix) to the total shear strain (γmax-γmin) and is considered to be the asphalt's total resistance to deformation when repeatedly sheared. The phase angle is a measure of the response time between the applied shear stress and the resulting shear strain. If asphalt was purely elastic, the phase angle would be zero degrees. If asphalt was purely viscous, the phase angle would be 90 degrees. Figure 3.6 illustrates the relationship between the phase angle and time factor.



*Figure 3.6* Relationship between phase angle and the time. (Pavement interactive, 2011)

The complex shear modulus  $(G^*)$  consists of two components: one is the storage modulus (G' the elastic component), and the other is the loss modulus (G'' the viscous component). Their relationship is illustrated in Figure 3.7.



*Figure 3.7* Relationship between strong modulus and loss modulus. (Pavement interactive, 2011)

 For asphalt binder to have rutting resistance, it must have high stiffness and elastic properties at high temperatures. Elasticity is defined as the property of being able to recover its original shape after being deformed by a load. The higher the G\* value, the stiffer the asphalt binder is. Similarly, the lower the  $\delta$  value is, the greater the elastic portion of  $G^*$  is. Therefore, as part of the PG binder specification system, the parameter  $G^*/\text{Sin}(\delta)$  is specified to be a minimum value (1.0 kPa for un-aged binders and 2.2 kPa for RTFO-aged binders).

 The methods in ASTM D7175-(2008) were followed to test the asphalt binder in the DSR. Eight mm diameter samples of each modification were inserted to create a sandwiched structure between two plates that load in a sinusoidal pattern at a rate of 10 radians/second (1.59 Hz) while submerged in water. The specified DSR oscillation rate of 10 radians/second (1.59 Hz) was used to imitate the shearing action related to a traffic speed of about 55 mph (Pavement interactive, 2011). The composition of the samples for this test were:

- $\bullet$  20% RAS with PG 64-22,
- 20% RAS with PG 64-22 and 5% Bio-binder,
- 20% RAS with PG 64-22 and 1.5% Rediset®, and
- 20% RAS with PG 64-22 and 0.5% Evotherm®.

To prepare the samples, each blended binder was heated at  $130^{\circ}$ C for 10 minutes until it reached the liquid phase. Five grams of liquid binder was then poured into a round silicon mold (radius of 25 mm) and left 45 minutes at room temperature to solidify. Then specimen was placed between the two plates of the rheometer and the oscillatory Dynamic Shear Rheometer test was conducted. The data obtained from DSR test was analyzed and presented in section 4.7 of chapter Four.

#### **CHAPTER 4**

#### **Results and Discussion**

 In this chapter data from the experiments conducted in the large-scale viscosity tests are analyzed. First, the viscosity tests and results using spindle SC 27 will be explained. The viscosity tests and results measured using spindle V73 will then be explained and a comparison made between the rheological properties revealed with the  $SC27 \& V73$  spindles. Using the empirical relationships of the blending index, all modified binders blending indices are discussed.

# **4.1 Rheological Characterization of Binders Utilizing Spindle SC27**

 These experiments were designed to characterize the rheological properties of RASmodified binders with or without the incorporation of amine-based modifier. The experiments were conducted using a Brookfield rotational viscometer following the ASTM D4402 test procedure (ASTM International, 2013). To complete the test the following test combinations were made and run at different temperatures and shear rates (rpm):

- recycled asphalt shingles (RAS) and virgin asphalt binder (PG 64-22);
- recycled asphalt shingles (RAS), virgin asphalt binder (PG 64-22), and Rediset®;
- recycled asphalt shingles  $(RAS)$ , virgin asphalt binder (PG 64-22), and Evotherm®; and
- recycled asphalt shingles (RAS), virgin asphalt binder (PG 64-22), and bio-binder.

 The RAS was incorporated as 20%, 30%, and 40% of the mixture by weight and blended with PG 64-22 separately for each percentage. Rediset<sup>®</sup>, Evotherm<sup>®</sup>, and Bio-binder were then blended with these mixtures according to the pre-determined proportions. The temperatures were varied in this test to 105<sup>o</sup>C, 120<sup>o</sup>C, 135<sup>o</sup>C, and 150<sup>o</sup>C and shear rates of 5, 10, 20, 25, 50 and 100 rpm were applied as per the experimental design.

**4.1.1 Rheological characterization of RAS modified binders.** The influence of RAS in virgin binder at different temperatures and shear rates was investigated and is shown graphically in Figures 4.1 and 4.2. The viscosity increased as RAS were incorporated into virgin binder. The viscosity of RAS-modified binder was found to be higher than the virgin binder or unmodified binder. When the temperature was increased the viscosity was decreased in all cases (either RAS-modified or non-modified). As seen in Figure 4.1, at a temperature of  $105^{\circ}$ C the viscosity of RAS-modified binders was higher than the control sample, and at a temperature of 150°C, the viscosity of the RAS-modified binders was still higher than the control but the viscosity value was less than the viscosity measured at a lower temperature. These results suggest that viscosity decreased when mixing temperature was increased.



*Figure 4.1* Viscosity vs. Temperature for RAS-modified Mixtures at 20 rpm

Figure 4.2. Shows the viscosity of the RAS-modified binders measured at a rotational speed of 25 and at different temperatures with spindle SC 27. In this figure, all modified mixtures showed higher viscosities than the non-modified (control) mixtures at all temperatures. Comparison of the values measured at 20 rpm and 25 rpm shows that the viscosity values at 25 rpm are less than the values at 20 rpm. This reveals that when rotational speed was increased the viscosity decreased. Therefore, it can be said that the RAS modification can increase binder viscosity. Furthermore, the viscosity increased with increasing RAS percentages and decreased with increases in the mixing temperature and the shear rate of the spindle.



*Figure 4.2* Viscosity vs. Temperature for RAS-modified mixtures at 25 rpm

**4.1.2. 20% RAS-modified binder with and without modifiers.** In this study three amine-based modifiers were used to improve the properties of the RAS modified binder. Rediset® and Evotherm® are commercial modifiers and the doses used were those specified by the manufacturers. In contrast, bio-binder is a modifier produced in the lab by the author by thermo chemical liquefaction of swine manure, and doses used were those specified in past research. The rheological properties of the binders prepared by incorporation of the modifiers were characterized. In Figure 4.3, all modified binders are shown to have lower viscosities than the non-modified binders. At lower temperatures each binder had a higher viscosity value than that found at the higher temperatures. At each temperature bio-modified binder had a lower viscosity than Rediset®-modified and Evotherm®-modified. Therefore, it can be said that biobinder can effectively reduce the binder viscosity. This trend was consistent for all other speeds tested.



*Figure 4.3* Viscosity vs. Temperature of 20% RAS-modified binder with and without modifiers at 20 rpm by SC 27

In Figure 4.4, all binders' viscosities are decreased compared to the values in Figure 4.3. For example, for the bio-binder at 105°C the value dropped from 3600 to 3500 cP because of the change in shearing rate. This phenomenon was found in all modified and non-modified binders.



*Figure 4.4* Viscosity vs. Temperature of 20% RAS-modified binder with and without modifiers at 25 rpm by SC27

**4.1.3. 30% RAS-modified binder with and without modifiers.** Rheological properties were studied for 30% RAS-modified mixture with and without incorporation of Rediset®, Evotherm®, and Bio-binder at different temperatures and shear rates. The results are shown in Figures 4.5 and 4.6. In each figure it can be seen that the viscosities decreased with incorporation of modifiers in RAS-modified binders. The viscosities were decreased by increasing the temperature and shear rate in all modified and non-modified binders. This change of viscosity can be seen clearly in Figure 4.5 for shear rate 20 and Figure 4.6 for shear rate 25, respectively.



*Figure 4.5* Viscosity vs. Temperature of 30% RAS-modified binder with and without modifiers at 20 rpm by SC27

Additionally, in Figure 4.6 it can be seen that the viscosity decreased with increasing temperature and shear rate but the rate of viscosity decreases is decreased when temperature was increased. At a temperature of  $105^{\circ}$ C the changes in viscosity between the binders are high compared to those found at a temperature of 150 °C.



*Figure 4.6* Viscosity vs. Temperature of 30% RAS-modified binder with and without modifiers at 25 rpm by SC27

**4.1.4. 40% RAS-modified binder with and without modifiers.** Similar studies were

conducted for 40% RAS-modified binder with Rediset®, Evotherm®, and Bio-binder incorporated. The results are shown in Figure 4.7 and Figure 4.8 at 20 rpm and 25 rpm, respectively.



*Figure 4.7* Viscosity vs. Temperature of 40% RAS-modified binder with and without modifiers at 20 rpm by SC27



*Figure 4.8* Viscosity vs. Temperature of 40% RAS-modified binder with and without modifiers at 25 rpm by SC27

 In both figures it can be seen that the viscosities decreased with incorporation of modifiers into the mixtures. Based upon all of the figures 4.3 to figure 4.8 it was determined that bio-binder-modified binders have the lowest viscosity at all tested temperatures and rotational speeds compared to the other modified or non-modified binders.

# **4.2 Rheological Characterization of Binders Utilizing Spindle V73**

 In this study the entire experiment was repeated for spindle V73 following the same procedures and using the same machine (Brookfield viscometer). The only difference was the spindle used.

**4.2.1. 20% RAS-modified binder with and without modifiers.** The influence of modifiers Rediset®, Evotherm®, and Bio-binder in 20% RAS mixtures at different temperatures and shear rates was investigated. Figures 4.9 and 4.10 reveal the viscosity changes measured at various temperatures at shear rates of 20 and 25 rpm, respectively. It can be seen that the viscosity of all specimens decreases with increases in temperature. This trend was consistent for all designated rotational speeds.



*Figure 4.9* Viscosity vs. Temperature of 20% RAS-modified binder with and without modifiers

at 20 rpm by V73



*Figure 4.10* Viscosity vs. Temperature of 20% RAS-modified binder with and without modifiers at 25 rpm by V73

When spindle V73 was used, all measured viscosities were higher than those measured using spindle SC27. As shown in Figure 4.9, all modified binders have lower viscosities than the non-modified (MB-20-N) binders. The bio-modified binders showed the lowest values compared to the other binders. The bio-binder can effectively lower the binder viscosity from 12900 to 5000; Rediset® then lowered the binder viscosity to 8100, and, similarly, Evotherm® lowered

the viscosity to 9000 at 105 $\degree$ C. This same trend is also seen in Figure 4.10 at a shear rate of 25 rpm.

**4.2.2. 30% RAS-modified binder with and without modifiers.** Increases in the RAS percentage caused increased viscosity in binders. Thirty percent RAS content binders had higher viscosity values than those measured below 30%. In Figures 4.11 and 4.12 it can be seen that higher viscosities were found compared to the values shown in Figures 4.9 and 4.10.

As shown in Figure 4.11 the non-modified (MB-30-N) binder has a higher viscosity than the modified binders. In addition of modifiers into mixture helps to decrease the viscosity of the mixture. In all cases the Bio-binder reduced viscosity more than the mixtures of Rediset® and Evotherm® seen in Figure 4.11; at a temperature of  $105^{\circ}$ C Bio-binder reduced binder viscosity 57%, Rediset® reduced binder viscosity by 30%, and Evotherm® reduced binder viscosity by 15%. However, compared to the values measured at a temperature of  $120^{\circ}$ C, these percentages are low. At  $150^{\circ}$ C all modifiers showed approximately equal changes in percentage of viscosity because at higher temperatures all binders are in equal liquidity phases.



*Figure 4.11* Viscosity vs. Temperature of 30 % RAS-modified binder with and without modifiers at 20 rpm by V73

The same trend is shown in Figure 4.12, but the values are lower than the values seen in Figure 4.11 because the shear rate also affects the viscosity of the binders. Higher shear rates result in lower values and vice versa.



*Figure 4.12* Viscosity vs. Temperature of 30% RAS-modified binder with and without modifiers at 25 rpm by V73

**4.2.3. 40% RAS-modified binder with and without modifiers.** The rheological properties of the binder prepared with 40% RAS and incorporation of all modifiers were investigated utilizing spindle V73. Figures 4.13 and 4.14 show the changes in viscosity as a function of temperature and shear rate. Concentration of 40% RAS increases the binder viscosity but incorporation of other modifiers led to decreases in viscosity.

As seen in Figure 4.13, at a temperature of  $105^{\circ}$ C the binder without modifiers (MB-40-N) measured 18000 cP at a shear rate of 20 rpm, and 15000 cP at a shear rate of 25 rpm (Figure 4.14), but at  $150^{\circ}$ C, viscosity was 825 cP at 20 rpm and 800 cP at 25 rpm, values which are very close compared to the differences measured at other temperatures. Therefore, it can be said that the temperature and the shear rate are the main components affecting the rheological properties of the binders or mixtures.



*Figure 4.13* Viscosity vs. Temperature of 40% RAS-modified binder with and without modifiers at 20 rpm by V73



*Figure 4.14* Viscosity vs. Temperature of 40% RAS-modified binder with and without modifiers at 25 rpm by V73

# **4.3 Comparative Rheological Characterization of 20% RAS-modified Binder Measured by two Spindles.**

To determine the effectiveness of the instrument (spindle), a comparative study was conducted. For this comparison only mixtures containing 20% RAS and only shear rates of 20 rpm were used. The graphical representations are shown in Figures 4.15 through 4.18. Each of the binders is shown separately at 20% RAS content and a shear rate of 20 rpm.

 In Figure 4.15, it is seen that the viscosity measured from V73 was very high compared to the viscosity measured with SC 27. At a temperature of  $105^{\circ}$ C, the viscosity measured with the V73 spindle is 2.26 times greater than that measured with the SC 27 spindle. Similarly, at  $120^{\circ}$ C V73 measurements are 2.25 times greater than the values obtained with the SC 27 spindle, at  $135^{\circ}$ C V73 values were 1.9 times greater than the values obtained with the SC 27 spindle, and at  $150^{\circ}$ C V73 measurements were 1.8 times greater than the values obtained with the SC 27 spindle. As temperature increased the rate and magnitude of change both decreased.



*Figure 4.15* Measured viscosities of RAS-modified binder at 20 rpm using two spindles

Figure 4.16 is a graph for the Evotherm®-modified binder at a shear rate of 20 rpm for two different spindles. The results show that the values measured with the V73 spindle were higher than the values measured with the SC27 spindle at all temperatures. The values were 2, 1.9, 1.5, and 1.3 times greater than those obtained with spindle V73 when measured at temperatures of 105 $\degree$ C, 120 $\degree$ C, 135 $\degree$ C, and 150 $\degree$ C, respectively.





Here it can be seen that the difference between the two spindle values measured decreased as the temperature was increased in steps from  $105^{\circ}$ C to  $150^{\circ}$ C.

The graph shown in Figure 4.17 is the viscosity measured using two different spindles for the same specimen modified by the amine-based modifier Rediset®. The results show that at all designed temperatures the vane spindle measured higher values than the smooth spindle.



*Figure 4.17* Measured viscosities of Rediset®-modified binder at 20 rpm by two spindles

At a temperature of 105 $\rm{^{\circ}C}$  V73 measured 2 times greater than SC27; similarly, at 120 $\rm{^{\circ}C}$ 

it was also 2 times greater; at  $135^{\circ}$ C it was 1.75 times greater; and at  $150^{\circ}$ C it was 1.8 times greater.

The same trend can be seen in Figure 4.18 in the case of Bio-modification. In this modified binder both spindles measured less viscosity at all temperatures compared to the other modified binders. However, higher values of viscosities were recorded using the vane spindle than the smooth spindle. At a temperature of  $105^{\circ}$ C the vane spindle recorded viscosities 1.35 times greater than the values obtained with the smooth spindle. Similarly, at  $120^{\circ}$ C,  $135^{\circ}$ C, and  $150^{\circ}$ C the vane spindle measured viscosity values 1.3, 1.4, and 1.2 times higher than the smooth spindle values. Analysis revealed the difference in rates between RAS-modified binders with and without amine-based modifiers. The difference in the rates of viscosity measured with the two spindles was highest in RAS-modified binder, followed by the Evotherm®-modified binder, the Rediset®-modified binder, and lowest in the Bio-modified binders. Among the binders tested, the Bio-modified binder showed the best results.





After studying the relative effectiveness of the two spindles it is clear that the vane (V73) spindle could measure higher viscosity values than the smooth spindle in all kinds of binders.

Therefore, the vane spindle is more effective when evaluating the rheological properties of the binders because of it measured more significant value than smooth spindle.

# **4.4 Viscosity Temperature Susceptibility (VTS)**

The viscosity temperature susceptibility of all modified and non-modified binders was evaluated separately from the data gathered using the two different types of spindles at 20 rpm and calculated values are summarized in table 4.1 and table 4.2 separately.

Table 4.1

<b>Temperature</b>	Viscosity Temperature Susceptibility of all binders at 20 rpm by SC27				
	<b>RAS</b> concentration	<b>Modifiers</b>			
		N	${\bf E}$	$\mathbf R$	$\bf{B}$
105	20		$\overline{\phantom{0}}$	$\overline{\phantom{0}}$	-
120		$-1.01$	$-0.97$	$-1.05$	$-1.02$
135		$-1.12$	$-1.14$	$-0.93$	$-1.14$
150		$-1.14$	$-0.95$	$-1.17$	$-0.98$
105	30		$\overline{\phantom{0}}$		۰
120		$-1.02$	$-1.01$	$-0.85$	$-1.02$
135		$-1.13$	$-0.93$	$-1.16$	$-1.08$
150		$-1.14$	$-1.22$	$-1.14$	$-1.16$
105	40	$\blacksquare$	$\overline{\phantom{0}}$	$\overline{\phantom{0}}$	$\blacksquare$
120		$-2.05$	$-0.94$	$-1.02$	$-0.92$
135		$-2.11$	$-1.13$	$-1.04$	$-1.13$
150		$-2.15$	$-1.12$	$-1.21$	$-1.15$

*VTS Values of all Modified and Non-Modified Binders at 20 rpm Measured by SC27 Spindle* 

 In table 4.1, the Viscosity Temperature Susceptibility values obtained utilizing spindle SC27 are summarized. From the table 4.1 it can be cleared that all modified binders have lower VTS than non-modified binder in addition among them, Bio-modified binder showed the lowest VTS indicating that Bio-binder is less susceptible in temperature. Same result can be seen in case of the viscosity temperature susceptibility obtained by utilizing vane spindle (V73) which is shown in table 4.2.

Table 4.2

<b>Temperature</b>	Viscosity Temperature Susceptibility of all binders at 20 rpm by V73					
	<b>RAS</b> concentration		<b>Modifiers</b>			
		${\bf N}$	$\bf{E}$	$\mathbf R$	$\bf{B}$	
105	20	$\overline{\phantom{0}}$			-	
120		$-0.92$	$-0.99$	$-0.98$	$-1.02$	
135		$-1.20$	$-1.00$	$-1.29$	$-0.99$	
150		$-0.92$	$-1.02$	$-1.12$	$-1.14$	
105	30	$\overline{\phantom{0}}$	$\overline{\phantom{0}}$	$\overline{\phantom{0}}$	$\overline{\phantom{a}}$	
120		$-0.95$	$-0.94$	$-0.89$	$-0.88$	
135		$-1.03$	$-1.03$	$-1.03$	$-1.10$	
150		$-1.12$	$-1.01$	$-1.23$	$-1.21$	
105	40	$\overline{\phantom{0}}$	$\blacksquare$	$\overline{\phantom{a}}$	$\blacksquare$	
120		$-1.11$	$-0.90$	$-1.06$	$-0.81$	
135		$-0.98$	$-1.05$	$-1.07$	$-1.02$	
150		$-1.10$	$-1.12$	$-1.11$	$-1.48$	

*VTS Values of all Modified and Non-Modified Binders at 20 rpm Measured by V73 Spindle* 

In this study the viscosity temperature susceptibility was evaluated using two set ups; in the first configuration, the VTS of the same binder incorporated into three different percentages (20, 30, and 40) of RAS content was evaluated. In the second setup, the VTS of different binders incorporated into the same percentage of RAS content was determined.

**4.4.1 VTS of same binder in different percentages of RAS.** Figures 4.19 and 4.20 are graphical plots of the VTS for non-modified and Bio-modified binders at 20%, 30%, and 40% RAS content. Figure 4.19 shows the VTS plot for the two different spindles for the same binder without amine-based modifiers. The plot shows that the values of VTSs measured with the vane spindle are higher than those obtained using the smooth spindle. VTS values increased as the RAS percentage in the mixtures increased. In Figure 4.19, 20% RAS-modified binder was less susceptible to temperature. However, in both cases, as temperature increased the temperature susceptibility decreased in each combination of RAS. Therefore, it can be said that out of the three RAS concentrations the lowest concentration has the lowest temperature susceptibility compared to the highest concentration of RAS in the binder/mixtures.



*Figure 4.19* VTS for all RAS-modified binders without modifiers at 20 rpm

The VTSs were evaluated for all amine-based modifiers separately. All modified binders showed a similar trend in that the Bio-modified binder showed lower VTS values than the Rediset®- and Evotherm®-modified binders. In Figure 4.20, the VTSs of Bio-binder modified with three percentages of RAS is shown. The Bio-binder modified with 20% RAS was less susceptible to temperature than the mixes containing 30% and 40% RAS. Furthermore, the VTSs were lower in data obtained with the smooth spindle compared to the vane spindle measured at all temperatures. Therefore, it can be concluded that low concentrations of RAS in the mixture were less susceptible to temperature for cases with and without incorporation of amine-based modifiers.



*Figure 4.20* VTS for all RAS modified binder with 5% of bio- binder at 20 rpm

**4.4.2 VTS of different binder in same percentage of RAS.** The VTS for mixes with and without incorporation of amine-based modifiers at the same concentration of RAS were evaluated using two different spindles and the results are shown in Figure 4.21 through 4.23. To study the viscosity temperature susceptibility of all binders at 20% RAS content at different temperatures, all log (log (viscosity)) versus Log (temperature) data measured with the two

spindles were plotted in Figure 4.21. It was observed that vane spindle measured data have higher VTS values than the smooth spindle measured data. The common finding in the data measured with both spindles was that the binders without incorporated amine-based modifiers have higher VTS values than all other modifiers' binders. Evotherm® showed the second highest VTS among the others at 20% RAS content binders. The Rediset® had lower values and the lowest VTS values were observed in Bio-modified binder with 20% RAS in both spindle cases. This result was true for all rotational speeds evaluated with the same modification (concentration of RAS). The Rediset®-modified and Evotherm®-modified binders showed closer values across the temperature range used. It can be said, therefore, that 20% RAS with PG 64-22 modified by 5% bio-binder was less temperature susceptible than those modified with 1.5% Rediset® and 0.5% Evotherm®. As RAS percentages increased the VTS of the binders increased and can be justified by evaluation of Figure 4.21.



*Figure 4.21* VTS for all 20% RAS content binders with and without modifiers at 20 rpm

In Figure 4.22, all amine-based modifiers were added to a mixture containing 30% RAS with PG 64-22. VTS values at every temperature went up faster than the values found in Figure

4.19 (modification with 20% RAS plus PG 64-22), but the trend was similar, as increases in temperature decreased the temperature susceptibility. Among all of the binders the Bio-modified binder showed the lowest viscosity temperature susceptibility in both the smooth- and vanespindle cases. Furthermore, VTS was evaluated for the amine-based modifiers modified and unmodified binder with 40% RAS content binders by utilizing smooth and vane spindles. The results are shown graphically in Figure 4.23. Similar trends were found in 20% and 30% RAS content binders. All vane spindle measurements produced higher VTS values compared to the smooth spindle measurements of VTS at all temperatures. However, the temperature susceptibility was decreased as the temperature increased for all binders. Bio-modified binder was found to be less temperature susceptible than the other binders.



*Figure 4.22* VTS for all 30% RAS content binders with and without modifiers at 20 rpm

Based on these VTS results it can be said that temperature susceptibility varies with the concentration of the impurities (in this case recycled asphalt shingles). Higher RAS content mixtures were more temperature susceptible and lower RAS content mixtures were less temperature susceptible. In this study, 20% RAS content binders were less susceptible than the 30% RAS, and then 40% RAS, content binders. Additionally, incorporation of additives/ modifiers in the mixture helped make the mixture less temperature susceptible at all temperatures and rotational speeds.



*Figure 4.23* VTS for all 40% RAS content binders with and without modifiers at 20 rpm

 In this study, Evotherm® made the mixture less temperature susceptible than the nonmodfied samples, Rediset® made the mixture less temperature susceptible than Evotherm®, and, finally, Bio-binder made the mixture less temperature susceptible than other binders. Therefore, it can be said that Bio-binder seemed to be a good modifier in the sense of viscosity temperature susceptibility for all percentages of recycled asphalt shingles content binders.

# **4.5 Shear Susceptibility**

 Shear susceptibility of all the binders was evaluated by utilizing the smooth spindle measured data. The shear susceptibility was plotted as log (shear rate) versus log (viscosity) at temperatures of 105 $^{\circ}$ C, 120 $^{\circ}$ C, 135 $^{\circ}$ C, and 150 $^{\circ}$ C. Results obtained at a temperature of 135 $^{\circ}$ C are illustrated in table 4.3 and graphically shown in Figures 4.24 through 4.27.

 The Shear Susceptibility of the modified and non-modified binders obtained using spindle SC4-27 at 135°C are summarized in table 4.3. All modified binders have lesser Shear Susceptibility than non-modified binders. Among them Bio-modified binder has lowest Shear Susceptibility.

Table 4.3



*Shear Susceptibility of all Modified and Non-Modified Binders at 135<sup>o</sup>C*
An analysis of Figure 4.24 reveals that the shear susceptibility for 40% RAS-modified binder (MB-40-N) was more consistent than other percentages of RAS-modified binders. However, the values are in decreasing order as the shear rate increased, and none of the binders showed momentary fluctuations in value which suggests stability. A gradual decrease in shear susceptibility can occur at all other temperatures.





Shear susceptibility was studied for the same RAS content binder influenced by three modifiers and is plotted separately in Figures 4.25 through 4.27 for data gathered at 135 °C. In Figure 4.25 a plot of shear susceptibility of Bio-binder mixed with 20% RAS and PG 64-22 at  $135^{\circ}$ C is shown. It also followed the same trend as increasing the shear rate decreased the shear susceptibility. Here, 30% RAS (MB-30-B) showed a more consistent result than 20% (MB-20-B) and 40% RAS content mixtures (MB-40-B). In Figure 4.26 it is shown that the shear susceptibility of different RAS content mixtures influences the 0.5% Evotherm® formulations at 135<sup>o</sup>C. The plot showed the consistent results in 30% (MB-30-E) and 40% RAS content

mixtures (MB-40-E) than the 20% RAS content mixture (MB-20-E). However, the shear susceptibility decreased with increasing shear rate.



*Figure 4.25* Shear susceptibility of all RAS-modified mixture with 5% bio-binder at 135 °C



*Figure 4.26* Shear susceptibility of all RAS-modified mixtures with 0.5% Evotherm® at 135°C

Figure 4.27 shows the shear susceptibility plot of Rediset®-modified binder with different RAS contents at 135°C. This showed that the Rediset® blend with 30% RAS (MB-30-R) was more consistent than the 20% (MB-20-R) and 40% RAS content (MB-40-R). Therefore,

from all of these figures it can be concluded that all the modified binders follow a similar trend: increases in the shear rate decrease the shear susceptibility. However, the 30% RAS content mixture blended with modifiers showed the most consistent results shear rates were changed.





As a part of the investigation of the properties of the binders, the blending index (Bx) of each mixture was calculated using Equation 3.3. The results for each modified binder at 20%, 30%, and 40% RAS and 20 rpm are summarized in table 4.4 and graphically plotted in Figures 4.28 to 4.30.

In figure 4.28 the blending indices of the modified binders incorporating 20% RAS at 20 rpm and four different temperatures are shown. From the plot, it is clear that all three modified binders incorporating modifiers have higher blending indices than the mixture containing only RAS-modified binder. Bx values increased with increasing temperature. Among them, Biomodified binder showed higher Bx values than Evotherm®- and then Rediset®-modified binders. In the case of the Bio-modified binder (MB-20-B) the blending index values increased

from 29.7 to 32.5 when temperature was changed from  $105^{\circ}$ C to  $150^{\circ}$ C, which is a 2.8% difference. Similarly, in Evotherm®-modified binder (MB-20-E) a 3.4% increase was measured, and in Rediset®-modified binder (MB-20-R), Bx values increased 1.7% between data points acquired at temperatures of 105°C to 150°C.

### *Table 4.4*



## *Blending Indices of all Modified and Non-Modified Binders at 20 rpm*



*Figure 4.28* Bx for all 20% RAS contain modified binders at different temperatures

 In Figure 4.29 the blending index of all amine-based modified and non-modified binders incorporating 30% RAS at different temperatures and 20 rpm were plotted. The plot showed that the blending index of modified binders is higher than the Bx of unmodified binder. The blending index at  $150^{\circ}$ C is higher than the other temperatures for Evotherm®, Rediset®, and bio-modified binders.



*Figure 4.29* Bx for all 30% RAS modified binders at different tmperatures

The increment in Bx from 105<sup>o</sup>C to 150<sup>o</sup>C for Rediset®-modified binder is 1.18%, in Evotherm®-modified binder it is 2.0%, and for Bio-binder modified binder it is 2.3%. The change in the Bx values in the case of Bio-binder is higher than the other two cases; therefore, comparatively, bBio-binder showed better results in this study.



A similar trend was seen in the 40% RAS content binder (see Figure 4.30).



As a part of this study, the blending index of all binders at  $135^{\circ}$ C was calculated to determine the best percentage of RAS in hot mix asphalt. The results showed that 20% tear-off RAS would be the best percentage to use in HMA because in all cases it showed higher Bx values than 40% RAS content binders and then 30% RAS content binders. The values are plotted in Figure 4.31. The graph shows that the blending values were higher in 20% RAS than in 40% and 30% RAS. This finding indicates that lower RAS content results in more homogeneity in the binder because adding less impurity containing fluid lets it act more like a Newtonian fluid. Actually, the idea behind this study is based on the concept that Newtonian fluids (homogeneous mixtures) ultimately have a higher blending index. Furthermore, it was expected that levels

below 20% RAS should have higher blending indices which can be justified by evaluating the trend in Figure 4.31.



*Figure 4.31* Change of Bx of all modified binders at  $135^{\circ}$ C

#### **4.7 Dynamic Shear Rheometer (DSR) Test**

A dynamic shear rheometer (DSR) was utilized to measure the binders' viscoelastic properties. Twenty-percent recycled asphalt shingles, containing mixtures with or without modifiers, were used to measure viscoelastic properties. The results are plotted in Figures 4.32 through 4.34. In figure 4.32, the plot shows the changes in the complex modulus  $(G^*)$  values at different frequencies. The complex modulus was increased with increases in the reduced frequency or decreases in temperature. It can be seen that the G\* of all modified binders is greater than the control binder. This indicates that the RAS makes binder stiffer, as higher G\* values indicate more stiffness and lower G\* values indicate less stiffness.

 Furthermore, incorporation of amine-based modifiers improved the softness of the binder. As seen in Figure 4.32, at all reduced frequencies RAS-modified binders (MB-20-N) showed higher complex modulus values than the other modified binders. The Rediset<sup>®</sup> (MB-20-R) and

Evotherm® (MB-20-E) content binders showed similar results at all reduced frequencies; however, in the case of Bio-binder (MB-20-B), higher G<sup>\*</sup> values occurred at lower frequencies, and lower G\* values at higher frequencies than the other modified binders.



*Figure 4.32* Master curve of 20% RAS-filled mixture for unmodified and modified binders

Figure 4.32 was evaluated in more detail by plotting it on an expanded scale. Figures 4.32(a) and 4.32(b) are separate plots of the master curve at the higher and lower reduced frequencies or it can say higher and lower temperature. It was observed that at higher frequencies all modified binders showed higher values of G\*, but comparatively bio-modified binders showed lower G\* values, indicating that the stiffness of the binder is reduced. Similarly, the master curve was plotted for all modified binders at lower reduced frequencies (higher temperature) and is shown in Figure 4.32(b). At lower reduced frequencies the Bio-modified binder showed higher G\* values indicating that the binder made the material stiffer. At lower reduced frequencies or high temperature incorporation of RAS may not cause distress (cracking) on the pavement, but it may be the cause of the distress on the pavement at higher reduced frequency or lower temperatures. This distress can be reduced by introducing the bio-binder in

RAS-modified hot mix asphalt because Bio-binder showed best performance (result) at lower temperature to reduction of the stiffness of the mixture by lowering the complex modulus  $(G^*)$ value.



*Figure 4.32(a)* Master curve of 20% RAS-filled mixture for non-modified and modified binders





*Figure 4.32(b)* Master curve of 20% RAS-filled mixture for unmodified and modified binders at lower reduced frequencies

And Figure 4.33 shows plots of the complex shear modulus  $(G^*)$  of all binders at a temperature of  $64^{\circ}$ C and a frequency of 1.67E+00 as specified in the superpave criteria.  $G^*$  can be considered the sample's total resistance to deformation when repeatedly sheared and also indicates the binders' stiffness; in Figure 4.33 Bio-binder modifications showed the lowest modulus (G\*) compared to the other modifications.



*Figure 4.33* Complex modulus of the binders at  $64^{\circ}$ C and a frequency of 1.67E+00

 Therefore, it can be said that Bio-binder improved the softness of the binder. In Figure 4.34 the phase angle of all binders at a temperature of  $64^{\circ}$ C and a frequency of 1.67E+00 are shown. Phase angle  $(\delta)$  is the log between the applied shear stress and the resulting shear strain and indicates whether the binder is more viscous or elastic. Higher values indicate a material is more viscous and lower values indicate more elasticity.

As shown in Figure 4.34, all modified binders have lower phase angle values than the non-modified binders. This indicates that the amine-based modifiers made the binders more elastic. However, among these, the Bio-binder modified binder has the lowest phase angle value.



*Figure 4.34* Phase angle of the binders at 64°C and a frequency of 1.67E+00

It can be said that elasticity improved means there is good blending of the ingredients in the mixtures/binders. Therefore, Bio-binder can enhance blending in a mixture.

#### **CHAPTER 5**

#### **Conclusion and Future Research**

#### **5.1 Summary**

This research was undertaken to evaluate the rheological characteristics of mixtures prepared by the addition of various percentages of recycled asphalt shingles into virgin asphalt with or without the incorporation of amine-based modifiers. The effectiveness of the spindles used to measure the viscosity of the modified mixtures was also investigated. Three (20%, 30%, and 40%) RAS-filled viscous media were prepared with or without incorporation of amine-based modifiers. A Brookfield viscometer was utilized to measure the viscosity of these binders using two different spindles.

In total, 24 specimens were made and the viscosity of each specimen was measured at four different temperatures and six different shearing rates. In this study a dynamic shear rheometer (DSR) test was also conducted on four specimens which were prepared with or without the incorporation of three amine-based modifiers at 20% RAS-filled media. All tests were conducted at the Civil Engineering Lab at North Carolina A & T State University.

The RV test was used to measure viscosity, which is the rate of deformation due to an applied shear or tensile stress. For each sample, an RV test was run three times to ensure accuracy with a fixed temperature and a fixed shear rate. These three readings were then tabulated, and the mean and coefficient of variation were calculated. The temperature was kept constant for five different shear rates, each of which was measured three times. This test was used to determine the rheological properties (temperature susceptibility, shear susceptibility, and blending index) of the samples.

The DSR test was used to measure viscoelastic properties, shear modulus  $(G^*)$ , and the phase angle (δ) of the mixtures prepared with and without amine-based modifiers. A small sample with an 8 mm diameter was prepared from each binder and placed ("sandwiched") between the two plates of the rheometer. The test specimens were kept at near constant temperatures by heating and cooling a surrounding environmental chamber. The top plate oscillated at 10 rad/sec (1.59 Hz) in a sinusoidal wave form while the equipment measured the maximum applied stress, the resulting maximum strain, and the time lag between them. The software then automatically calculated the complex modulus  $(G^*)$  and phase angle ( $\delta$ ). Much of the procedure is automated by the test software.

 An empirical relationship between viscosity and temperature was proposed to measure the blending index of the mixtures based on the measured viscosity using two different spindles at the same temperature. The results of this portion of the research study are listed below.

#### **5.2 Observation and Conclusions**

 The purpose of this study was to evaluate the effect of specific amine-based modifiers in partially filled viscous media on the basis of changes in rheological properties. Based on the test results for amine-based modifiers modified asphalt, the following conclusions can be made:

- Viscosity increased with the addition of recycled asphalt shingles into virgin asphalt binder (PG 6.4-22) and the increasing viscosity correlated with increases in the percentage of recycled asphalt shingles added.
- The viscosity of the binder was decreased with increases in the mixing temperature and increases in the shear rate. Furthermore, the viscosities were decreased with incorporation of the amine-based modifiers into RAS-filled mixtures.
- Among the three modifiers (Bio-binder, Rediset®, and Evotherm®), Bio-binder can effectively reduce the viscosity of the binders at all temperatures ( $105^{\circ}$ C,  $120^{\circ}$ C,  $135^{\circ}$ C, and  $150^{\circ}$ C) and all rotational speeds.
- $\bullet$  In all modified and non-modified binders, use of the vane spindle (V73) resulted in higher measured viscosities than those measured using the smooth spindle (SC27).
- The coefficient of variation of the measured viscosities was significantly lower in the case of the vane spindle versus the smooth spindle, indicating that the vane spindle was more appropriate for measuring the viscosity of the mixtures/binders.
- The viscosity temperature susceptibility (VTS) of the binder was increased by increasing the percentage of RAS added to the virgin asphalt. Using either spindle, 20% RASmodified binder was less temperature susceptible than 30% and 40% RAS-modified binders at all temperatures and rotational speeds.
- Rediset®, Evotherm®, and Bio-binder modifiers reduced the VTS of the binders. Among them, Bio-binder reduced the VTS effectively when using either spindle. Overall, use of the vane spindle resulted in higher measured VTS values than use of the smooth spindle.
- The shear susceptibility for 40% RAS-modified mixture was more consistent than the 30% and 20% RAS-modified mixtures.
- The overall shear rate dropped when 5% Bio-binder, 1.5% Rediset®, or 0.5% Evotherm® was added into mixtures (PG 64-22 and RAS) tested at 135°C.
- The shear susceptibility of the Bio-modified binder was found to be more consistent in all percentages of RAS compared to the Rediset®- and Evotherm®-modified binders.
- The blending index was measured by using an empirical relation. Results indicated that the blending index increased as temperature increased. All modified binders showed

higher blending indices at a temperature of  $150^{\circ}$ C compared to samples measured at 135<sup>o</sup>C, 120<sup>o</sup>C, and 105<sup>o</sup>C.

- Overall blending index was higher in amine-based modifier's modified binder compared to only RAS-modified binders at all temperatures.
- Comparing the blending index of the Redist®-, Evotherm®-, and Bio-binder modified binders, the Bio-binder showed the best results at all temperature tested at a rotational speed of 20 rpm.
- Among the blending indices evaluated at 135 °C and 20 rpm for 20%, 30%, and 40% RAS-filled medium, the highest value was found in Bio-binder modified mixtures. Additionally, 20% RAS-filled media showed higher results than 30% and 40% RASfilled media (mixtures) indicating that Bio-binder most effectively increases mixing between aged and unaged asphalt in the mixture.
- The dynamic shear rheometer test was conducted for all modified and unmodified binders at 20% RAS content viscous media. The complex moduli (G\*) for modified binders were higher than the control (PG 64-22) binder. Furthermore, incorporation of amine-based modifiers into the control decreased the G\*.
- The phase angle  $(\delta)$  was found to be lower in modified binders compared to the nonmodifiers content binder indicating that the amine-based modifiers make binder more elastic, which is only possible when thorough mixing of the ingredients occurs in the mixture.
- At higher temperatures (lower frequencies) Bio-modified binders show higher values of  $G^*$  than the others but at lower temperatures (higher frequencies) it showed lower  $G^*$ than the other binders. This finding indicates that incorporation of Bio-binder into the

RAS-modified mixture at lower temperatures is more beneficial in terms of reduction of the mixture stiffness.

• Among the three modifiers, the Bio-binder reduced the  $G^*$  and  $\delta$  in the mixture effectively and enhanced the mixing between RAS and virgin asphalt in the mixture. In summary, the addition of Bio-binder to partially RAS-filled viscous mixtures reduces the viscosity, temperature susceptibility, shear susceptibility, complex modulus, and phase angle and enhances the blending index of the asphalt binders tested.

#### **5.3 Future Research**

 This study focused primarily on three amine-based modifiers and their application to enhance rheological characteristics of asphalt binder. Further research is needed to specify interaction mechanisms between each of these modifiers and asphalt molecules. In addition, determining the optimum percentage of each additive should be determined in order to maximize the blending of the modified binder. As such, the following recommendations are made for future studies:

- study molecular interactions between modifiers and asphalt (aged as well unaged),
- improve or predict the most appropriate relation to measure the blending behaviors of the mixture,
- study the accuracy of the proposed empirical relation to calculate the blending index of the mixtures, and
- undertake the study needed to determine the appropriate proportions of the modifiers in hot mix asphalt in terms of blend indices.

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## **Appendix A**

## **Tabulated results from RV tests for RAS modified binder Measured by SC 27**

Table A-1

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table A-2

*RV results for 20% RAS at 105°C, 25 rpm.* 



Table A-3

*RV results for 20% RAS at 120°C, 20 rpm.*



## *RV results for 20% RAS at 120°C, 25 rpm.*



#### Table A-5

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table A-6

*RV results for 20% RAS at 135°C, 25 rpm.* 



## *RV results for 20% RAS at 150°C, 20 rpm.*



## Table A-8

*RV results for 20% RAS at 150°C, 25 rpm.*



Table A-9

*RV results for 30% RAS at 105°C, 20 rpm.* 



# *RV results for 30% RAS at 105°C, 25 rpm.*



### Table A-11

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table A-12

*RV results for 30% RAS at 120°C, 25 rpm.* 



# *RV results for 30% RAS at 135°C, 20 rpm.*



### Table A-14

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table A-15

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



### Table A-17

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table A-18

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



### Table A-20

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table A-21:

*RV results for 40% RAS at 135°C, 20 rpm.* 



# *RV results for 40% RAS at 135°C, 25 rpm.*



### Table A-23

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table A-24

*RV results for 40% RAS at 150°C, 25 rpm.* 



# **Measured by spindle type V73**

Table A-25

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table A-26

*RV results for 20% RAS at 105°C, 25 rpm.* 



Table A-27

*RV results for 20% RAS at 120°C, 20 rpm.* 



## *RV results for 20% RAS at 120°C, 25 rpm.*



### Table A-29

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table A-30

*RV results for 20% RAS at 135°C, 25 rpm.* 



# *RV results for 20% RAS at 150°C, 20 rpm.*



### Table A-32

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table A-33

*RV results for 30% RAS at 105°C, 20 rpm.* 


# *RV results for 30% RAS at 105°C, 25 rpm.*



## Table A-35

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table A-36

*RV results for 30% RAS at 120°C, 25 rpm.* 



# *RV results for 30% RAS at 135°C, 20 rpm.*



## Table A-38

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table A-39

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



### Table A-41

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table A-42

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



#### Table A-44

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table A-45

*RV results for 40% RAS at 135°C, 20 rpm.* 



# *RV results for 40% RAS at 105°C, 25 rpm.*



#### Table A-47

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table A-48

*RV results for 40% RAS at 150°C, 25 rpm.* 



# **Appendix B**

# **Tabulated results from RV tests for Rediset® modified binder Measured by SC 27**

Table B-1

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table B-2

*RV results for 20% RAS at 105°C, 25 rpm.* 



## Table B-3

*RV results for 20% RAS at 120°C, 20 rpm.* 



# *RV results for 20% RAS at 120°C, 25 rpm.*



## Table B-5

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table B-6:

*RV results for 20% RAS at 135°C, 25 rpm.* 



# *RV results for 20% RAS at 150°C, 20 rpm.*



## Table B-8

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table B-9

*RV results for 30% RAS at 105°C, 20 rpm.* 



### Table B-10:

# *RV results for 30% RAS at 105°C, 25 rpm.*



### Table B-11

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table B-12

*RV results for 30% RAS at 120°C, 25 rpm.* 



# *RV results for 30% RAS at 135°C, 20 rpm.*



## Table B-14

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table B-15

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



### Table B-17

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table B-18

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



### Table B-20

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table B-21

*RV results for 40% RAS at 135°C, 20 rpm.* 



# *RV results for 40% RAS at 135°C, 25 rpm.*



## Table B-23

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table B-24

*RV results for 40% RAS at 150°C, 25 rpm.* 



# **Measured by V73 type spindle**

#### Table B-25

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table B-26

*RV results for 20% RAS at 105°C, 25 rpm.* 



Table B-27

*RV results for 20% RAS at 120°C, 20 rpm.* 



# *RV results for 20% RAS at 120°C, 25 rpm.*



## Table B-29

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table B-30

*RV results for 20% RAS at 135°C, 25 rpm.* 



# *RV results for 20% RAS at 150°C, 20 rpm.*



## Table B-32

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table B-33

*RV results for 30% RAS at 105°C, 20 rpm.* 



# *RV results for 30% RAS at 105°C, 25 rpm.*



### Table B-35

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table B-36

*RV results for 30% RAS at 120°C, 25 rpm.* 



# *RV results for 30% RAS at 135°C, 20 rpm.*



## Table B-38

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table B-39

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



### Table B-41

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table B-42

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



### Table B-44

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table B-45

*RV results for 40% RAS at 135°C, 20 rpm.* 



# *RV results for 40% RAS at 135°C, 25 rpm.*



### Table B-47

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table B-48

*RV results for 40% RAS at 150°C, 25 rpm.* 



# **Appendix C**

# **Tabulated results from RV tests for Evotherm® modified binder Measured by SC 27**

Table C-1

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table C-2

*RV results for 20% RAS at 105°C, 25 rpm.* 



Table C-3

*RV results for 20% RAS at 120°C, 20 rpm.* 



# *RV results for 20% RAS at 120°C, 25 rpm.*



## Table C-5

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table C-6

*RV results for 20% RAS at 135°C, 25 rpm.* 



# *RV results for 20% RAS at 150°C, 20 rpm.*



## Table C-8

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table C-9

*RV results for 30% RAS at 105°C, 20 rpm.* 



# *RV results for 30% RAS at 105°C, 25 rpm.*



# Table C-11

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table C-12

*RV results for 30% RAS at 120°C, 25 rpm.* 



# *RV results for 30% RAS at 135°C, 20 rpm.*



### Table C-14

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table C-15

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



# Table C-17

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table C-18

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



### Table C-20

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table C-21

*RV results for 40% RAS at 135°C, 20 rpm.*



# *RV results for 40% RAS at 135°C, 25 rpm.*



## Table C-23

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table C-24

*RV results for 40% RAS at 150°C, 25 rpm.* 



## **Measured by V73 type spindle**

# Table C-25

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table C-26

*RV results for 20% RAS at 105°C, 25 rpm.* 



Table C-27

*RV results for 20% RAS at 120°C, 20 rpm.* 



# *RV results for 20% RAS at 120°C, 25 rpm.*



## Table C-29

## *RV results for 20% RAS at 135°C, 20 rpm.*



Table C-30

*RV results for 20% RAS at 135°C, 25 rpm.* 



# *RV results for 20% RAS at 150°C, 20 rpm.*



## Table C-32

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table C-33

*RV results for 30% RAS at 105°C, 20 rpm.* 



# *RV results for 30% RAS at 105°C, 25 rpm.*



## Table C-35

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table C-36

*RV results for 30% RAS at 120°C, 25 rpm.*



# *RV results for 30% RAS at 135°C, 20 rpm.*



## Table C-38

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table C-39

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



#### Table C-41

## *RV results for 40% RAS at 105°C, 20 rpm.*



Table C-42

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



## Table C-44

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table C-45

*RV results for 40% RAS at 135°C, 20 rpm.* 


### Table C-46

## *RV results for 40% RAS at 135°C, 25 rpm.*



## Table C-47

## *RV results for 40% RAS at 150°C, 20 rpm.*



Table C-48

*RV results for 40% RAS at 150°C, 25 rpm.* 



## **Appendix D**

# **Tabulated results from RV tests for Bio-binder modified binder Measured by SC 27**

Table D-1

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table D-2

*RV results for 20% RAS at 105°C, 25 rpm.* 



## Table D-3

*RV results for 20% RAS at 120°C, 20 rpm.* 



## *RV results for 20% RAS at 120°C, 25 rpm.*



## Table D-5

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table D-6

*RV results for 20% RAS at 135°C, 25 rpm.* 



## *RV results for 20% RAS at 150°C, 20 rpm.*



## Table D-8

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table D-9

*RV results for 30% RAS at 105°C, 20 rpm.* 



# *RV results for 30% RAS at 105°C, 25 rpm.*



#### Table D-11

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table D-12

*RV results for 30% RAS at 120°C, 25 rpm.* 



## *RV results for 30% RAS at 135°C, 20 rpm.*



## Table D-14

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table D-15

*RV results for 30% RAS at 150°C, 20 rpm.* 



## *RV results for 30% RAS at 150°C, 25 rpm.*



#### Table D-17

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table D-18

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



## Table D-20

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table D-21

*RV results for 40% RAS at 135°C, 20 rpm.* 



## *RV results for 40% RAS at 135°C, 25 rpm.*



## Table D-23

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table D-24

*RV results for 40% RAS at 150°C, 25 rpm.* 



# **Measured by V73 type spindle**

#### Table D-25

*RV results for 20% RAS at 105°C, 20 rpm.* 



Table D-26

*RV results for 20% RAS at 105°C, 25 rpm.* 



Table D-27

*RV results for 20% RAS at 120°C, 20 rpm.* 



# *RV results for 20% RAS at 120°C, 25 rpm.*



## Table D-29

*RV results for 20% RAS at 135°C, 20 rpm.* 



Table D-30

*RV results for 20% RAS at 135°C, 25 rpm.* 



# *RV results for 20% RAS at 150°C, 20 rpm.*



## Table D-32

*RV results for 20% RAS at 150°C, 25 rpm.* 



Table D-33

*RV results for 30% RAS at 105°C, 20 rpm.* 



## *RV results for 30% RAS at 105°C, 25 rpm.*



## Table D-35

*RV results for 30% RAS at 120°C, 20 rpm.* 



Table D-36

*RV results for 30% RAS at 120°C, 25 rpm.* 



## *RV results for 30% RAS at 135°C, 20 rpm.*



## Table D-38

*RV results for 30% RAS at 135°C, 25 rpm.* 



Table D-39

*RV results for 30% RAS at 150°C, 20 rpm.* 



# *RV results for 30% RAS at 150°C, 25 rpm.*



#### Table D-41

*RV results for 40% RAS at 105°C, 20 rpm.* 



Table D-42

*RV results for 40% RAS at 105°C, 25 rpm.* 



# *RV results for 40% RAS at 120°C, 20 rpm.*



## Table D-44

*RV results for 40% RAS at 120°C, 25 rpm.* 



Table D-45

*RV results for 40% RAS at 135°C, 20 rpm.* 



# *RV results for 40% RAS at 135°C, 25 rpm.*



## Table D-47

*RV results for 40% RAS at 150°C, 20 rpm.* 



Table D-48

*RV results for 40% RAS at 150°C, 25 rpm.* 

