

DOKUZ EYLÜL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**IMPROVING THE PROPERTIES OF
RECLAIMED ASPHALT USED IN ROAD
CONSTRUCTION BY REJUVENATING AGENTS**

by

Peyman AGHAZADEH DOKANDARI

October, 2018

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IMPROVING THE PROPERTIES OF RECLAIMED ASPHALT USED IN ROAD CONSTRUCTION BY REJUVENATING AGENTS

**A Thesis Submitted to the
Graduate School of Natural and Applied Sciences of Dokuz Eylül University
In Partial Fulfillment of the Requirements for the Degree of Doctor of
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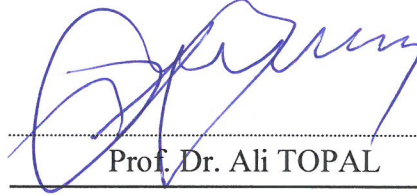
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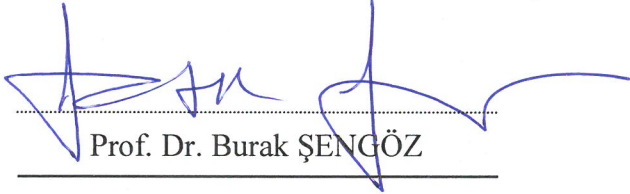
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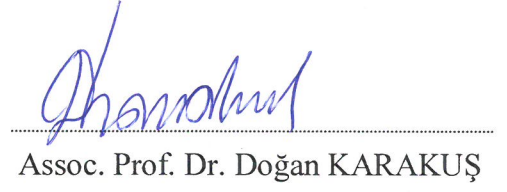
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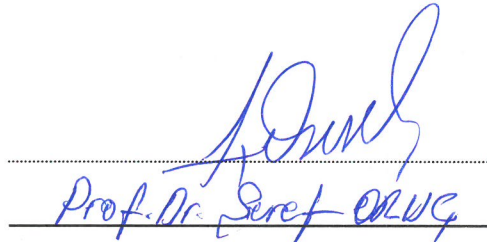
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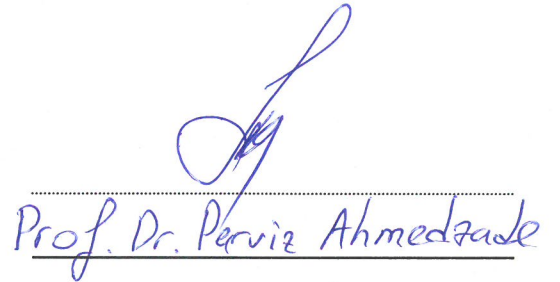
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IMPROVING THE PROPERTIES OF RECLAIMED ASPHALT USED IN ROAD CONSTRUCTION BY REJUVENATING AGENTS

ABSTRACT

Recycling is the inevitable part of many industries. Reserving and effective use of natural resources is a must for today's human being. Recycling in the asphalt industry is one of the most concerned areas and Reclaimed Asphalt Pavement (RAP) is amongst the most recycled materials in the world. Utilization of RAP in new asphalt pavements may lead to severe quality problems mostly due to asphalt aging phenomenon.

Within the scope of this study, the use of rejuvenators to improve the properties of RAP has been investigated. Two different kinds of waste oils together with a commercial rejuvenator were used. The rejuvenated bitumen samples were investigated from rheological, morphological and chemical aspects. In addition, mixture testing was also performed to study the effectiveness of used rejuvenators.

Experimental studies on bitumen samples mainly included conventional and Dynamic Shear Rheometer (DSR) testing for rheological studies, Atomic Force Microscopy (AFM) imaging for morphological studies and Fourier Transform Infrared Spectroscopy (FTIR) spectra analysis for chemical studies. Mixture testing consisted of Marshall and Indirect Tensile Test (IDT) investigation.

The results unveiled that the utilization of rejuvenators can significantly improve the quality of recycled asphalt. Binder and mixture testing showed high consistency and conformity with each other. The results proved that many oily based materials including waste oils can be used as rejuvenating agents. Rejuvenators can significantly restore the properties of aged RAP bitumen and increase the amount of RAP in bituminous mixtures.

Keywords: Reclaimed Asphalt Pavement, rejuvenator, bitumen Aging, Dynamic Shear Rheometer, Atomic Force Microscopy, Fourier Transform Infrared Spectroscopy



YOL İNŞAATINDA KULLANILAN GERİ KAZANILMIŞ ASFALT ÖZELLİKLERİNİN GENÇLEŞTİRİCİ KATKILAR KULLANILARAK İYİLEŞTİRİLMESİ

ÖZ

Geri dönüşüm, birçok endüstrinin kaçınılmaz parçasıdır. Doğal kaynakların rezerve edilmesi ve etkin kullanımı günümüz insanı için kaçınılmaz hale gelmiştir. Geri Kazanılmış Asfalt Kaplaması (RAP), dünyanın en çok geri dönüştürülmüş malzemelerinden biridir. Ancak yeni asfalt kaplamalarda RAP kullanımı doğru yöntemler ile yapılmadığı sürece, ciddi kalite sorunlarına yol açabilir.

Bu çalışma kapsamında, RAP özelliklerini iyileştirmek için asfalt gençleştirici katkılarının kullanımı araştırılmıştır. RAP bitümünü yeniden kullanmak üzere ticari bir gençleştirici ile iki farklı tipte atık yağ kullanılarak yaşlanmış bitümün özellikleri iyileştirilmeye çalışılmıştır. Çalışma kapsamında yeniden ortaya çıkan gençleştirilmiş bitüm örnekleri, reolojik, morfolojik ve kimyasal açıdan incelenmiştir. Bitüm örnekleri üzerinde yapılan incelemelere ek olarak, karışım üzerinde de çeşitli deneyler uygulanarak RAP kullanımı araştırılmıştır.

Deneysel çalışmalar kapsamında bitüm numuneleri üzerinde geleneksel deneylerin yanı sıra, reolojik çalışmalar için Dinamik Kesme Reometresi (DSR), morfolojik açıdan incelemek üzere Atomik Kuvvet Mikroskopi (AFM) görüntüleme yöntemi ve kimyasal açıdan incelemeler için ise Fourier Dönüşümlü Kızılötesi Spektroskopisi (FTIR) spektrum analizi kullanılmıştır. RAP içeren karışımlar üzerinde yapılan incelemelerde ise Marshall ve İndirekt Çekme Testi (IDT) deneylerinden yararlanılmıştır.

Sonuçlar, uygun gençleştiricilerin kullanımının, geri dönüştürülmüş asfaltın kalitesini önemli ölçüde artırdığını ortaya çıkarmıştır. Bitüm örnekleri ve karışımlar üzerinde yapılan deney sonuçlarına bakıldığında, tüm bitüm örnekleri için reolojik, morfolojik ve kimyasal sonuçlar ve karışım sonuçları birbirine paralellik

göstermektedir. Sonuçlar, bitüm gençleştiricisi olarak kullanılan yağ bazlı malzemelerin seçimi doğru yapılırsa şayet, birçok atık yağ dahil, yağ bazlı ürünlerin yaşlanmış bitüm için gençleştirici katkı olarak kullanılabileceğini kanıtlamıştır. Gençleştirici katkılar, bitümlü karışımlarda RAP özelliklerini önemli ölçüde iyileştirerek daha yüksek oranlarda RAP kullanımı için olanak sağlayabilmektedir.

Anahtar Kelimeler: Geri Kazanılmış Asfalt Kaplaması, asfalt gençleştirici katkı, yaşlanmış bitüm, Dinamik Kesme Reometresi, Atomik Kuvvet Mikroskopi, Fourier Dönüşümlü Kızılötesi Spektroskopisi



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CHAPTER ONE

INTRODUCTION

Flexible pavements are built to serve for a specific traffic load combination during the service life. A new built flexible pavement performance can be evaluated based on its stability and durability. As a flexible pavement gets old, its characteristics change in the course of time. In case of flexible pavements, aging mainly causes durability issues rather than stability problems. Aged binder is expected to exhibit higher stability values since it is stiffer than its original state. Durability can be defined as how bitumen characteristics change with age and thus it is directly related to time.

Pavements are subject to different kinds of failures. These failures may have various reasons. Depending on the type of failure, it may be associated with stability and/or durability. For instance, rutting is more likely to be related to stability rather than durability, since fatigue cracking is a type of failure which is directly attributed to durability. Failures associated with durability cause serious problems in flexible pavements which consequently necessitate the renewing of flexible pavement.

Highway authorities oblige a new flexible pavement to satisfy local specifications limits. In the design phase of flexible pavements, a virgin binder has to successfully pass a series of performance tests. The same is true for aggregates. An old flexible pavement contains aged binder and more likely crushed aggregates. Granular characteristics of aggregates change significantly under heavy traffic loads during the service life. Thus, an old flexible pavement cannot be used over again without essential modification and/or readjustment. The old flexible materials are called Reclaimed Asphalt Pavement (RAP). It can be gained through different methods such as cold milling or ripping.

RAP materials can be used in a wide range of applications in road construction. RAP can be utilized as backfill material in embankments or can be reutilized in various courses of flexible pavements. Depending on the area of application, re-utilizing of RAP can provide different levels of savings in terms of costs. Reasonably, the highest

level of saving can be provided when RAP is reutilized within a wearing course of a flexible pavement. However, re-utilizing RAP within the surface courses requires high levels of attention since these courses are supposed to be more qualified compared to other sub-courses. On the other hand, the upper courses are more subject to durability issues. It is important to have a correct evaluation regarding the overall quality of existing RAP on hand and reutilize it within the optimum area of application. Recycling of bituminous materials has generated considerable discussion and development during the last decades. Although it is not a new idea, recent studies appear to be in response to the need of many countries to reduce their dependency on imported crude oil and the derivative product known as bitumen.

Many recent technologies introduce innovative methods for recycling of RAP. It is important to designate the most valuable field of recycling for RAP. Many studies have addressed many areas of application for RAP and the optimum amount within the mentioned area (Recycling & Reclaiming Association, 2001). Worldwide in addition to science world, within the high petroleum prices period, the private sector has developed many methods in order to use RAP with high possible amounts (Hansen & Copeland, 2013). Although private sector technologies are controlled and confirmed by authorities in accordance with specifications, these technologies are not accessible to the public due to know-how issues.

Allegedly the United States of America and many other countries have led the technological development of modern recycling methods for RAP. While recycling is not practiced nationwide in the USA, now it has become a common practice in many states. Other countries which seem to be interested in developing of recycling processes include Germany, France, Finland, India, South Africa and Turkey (Sonmez et al., 2011).

RAP contains both aggregates and bitumen, and hence it saves natural resources while it is an eco-friendly technology (Tao & Mallick, 2009). The use of RAP provides an economical solution for pavement industry since it helps to save both aggregates and bitumen (Tao & Mallick, 2009).

Recycling methods can generally be categorized as Cold and Hot recycling (Recycling & Reclaiming Association, 2001). Cold recycling of bituminous mixtures involves RAP, water and a recycling agent in place without applying heat generally by use of emulsions since hot recycling implements heat and generally recycling agents in presence of heat. Both methods can be conducted in production plant or in-place. The common point in most recycling methods is the presence of recycling agents which pave the way for easier application.

Use of rejuvenators also called recycling agent is one of the most famous methods introduced to facilitate the use of RAP especially within the upper courses of flexible pavements such as binder and surface courses (Sonmez et al., 2011). Various kinds of rejuvenators have been introduced to asphalt recycling industry so far, some commercialized and some still remained within the literature. These kinds of rejuvenators mostly are used within the hot recycling applications. Many of rejuvenators have an oily base since it is believed that aged asphalt mainly lacks the oily components of bitumen which disappear during the service life of bituminous pavements (Fernández-Gómez et al., 2013).

After all, the demand for sustainable pavements increases day by day in pavement industry. Efficient utilization of raw materials along with the economic issues is the main reason to increase the amount of RAP in bituminous mixtures. The objective of this study is to seek for innovative recycling agents mainly originated from waste oils. In this regard, two kinds of plentiful waste oils in Turkish market were evaluated and compared to a commercial recycling agent. Waste Engine Oil (WEO) and Waste Vegetable Oil (WVO) were utilized and compared to a Commercial Recycling Agent (CRA).

Within the experimental studies, the optimum contents of these additives were determined, and test samples were prepared mostly at these desired dosages. In general, the experimental plan of the study can be divided into two basic categories. The first category includes the evaluation of these recycling agents with aged RAP binder mainly called bitumen tests. In this category, only the tests applicable to binder

were employed. The second category involves the utilization of these additives for recycling purposes through mixture preparation and specimen testing.

Within the binder testing category, the effects of these additives on aged RAP binder were evaluated through three technical aspects: Rheology, Morphology, and Chemistry. Conventional and modern binder tests were applied on the virgin binder, aged RAP binder and rejuvenated binder samples. The objective was to obtain a binder sample through rejuvenating which is similar to a virgin binder in terms of rheology. In the morphological evaluation, all binder samples were evaluated by means of Atomic Force Microscopy. Finally, some bitumen samples were tested for their chemical constituents using Fourier Transform Infrared Spectroscopy (FTIR) device.

Following the binder testing, the mixtures containing different rejuvenated and non-rejuvenated RAP contents were prepared using these rejuvenating agents. The specimens were tested for different mixture testing to ensure Turkish specification limits. It is found that these rejuvenating agents are capable of increasing the possible amounts of RAP materials within bituminous mixtures without major performance drawbacks.

This dissertation consists of five main Chapters. In addition to this chapter (Introduction), the reader will encounter four other different chapters. Within the following chapters, a comprehensive literature review is presented regarding the recycling of RAP materials by use of rejuvenating agents in Chapter 2. Chapter 3 includes the properties of materials utilized in this study and the outline of the methodology used. The results are given using charts and graphics together with the numerical analysis in Chapter 4. At the end of the dissertation, the study reaches a conclusion on the use of waste oils and commercial rejuvenating agent as in Chapter 5.

CHAPTER TWO

LITERATURE REVIEW

“Asphalt” is a highly viscous hydrocarbon residue of refinery process in which the input substance is crude oil. The color of asphalt can vary from dark brown to black. The main process which takes place in refinery is called distillation which helps to separate the hydrocarbons with different molecular weights from each other through selective evaporation and condensation also called fractional distillation. A typical asphalt production scheme is presented in Figure 2.1 (Nicholls, 1998). There are also natural distillation examples in the nature which has led to create asphalt lakes. Trinidad Lake Asphalt, shown in Figure 2.2, is one of the most famous examples of the World’s natural asphalt lakes. Other types of natural asphalts are Gilsonite and rock asphalt (Nicholls, 1998).

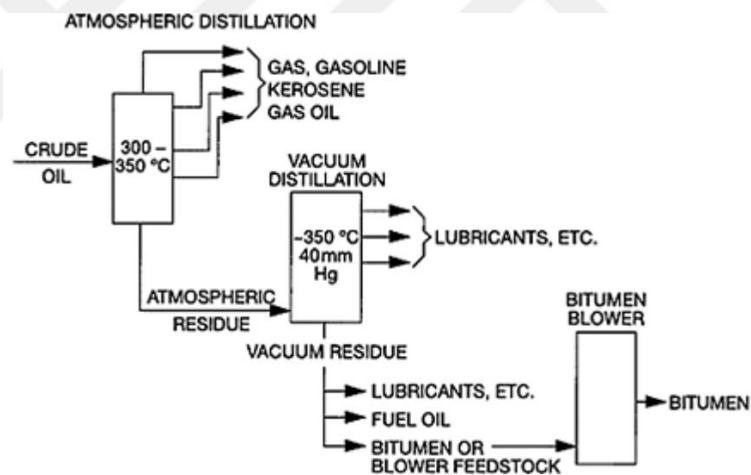


Figure 2.1 Typical bitumen production scheme (Nicholls, 1998)



Figure 2.2 Trinidad Lake Asphalt (Pavement Interactive, 2012a)

2.1 Asphalt Binder (Bitumen)

The terms “Asphalt Binder” and “Bitumen” are used alternatively in flexible pavement technologies. According to the Cambridge Advanced Learner’s Dictionary & Thesaurus, binder means a substance added to a mixture to make all the parts stick together (Dictionary, 2017). “Asphalt binder” refers to a cementitious asphaltic substance which is used in bituminous mixtures in road constructions. The pre-dominating constituent of asphalt binder is bitumen. So, the term “Bitumen” mainly means asphalt binder in civil engineering literature. According to ASTM, bitumen is *“a hydrocarbon material of natural or pyrogenous origin, gaseous, liquid, semi-solid, or solid which is completely soluble in carbon disulfide”*.

Bitumen can be classified according to its physical properties and/or chemical structure. In the following sections, the physical properties and the chemical structure of bitumen are presented briefly.

2.1.1 Physical Properties and Grading Systems of Bitumen

In the pavement industry, it is the physical properties of bitumen which is used for performance characterization. The main physical properties of bitumen which are used for characterization of bitumen are rheology and durability. Durability in civil engineering means the ability of a specific material to withstand the conditions in the course of time. In this context, the durability (also called age hardening) of bitumen is described as how the bitumen physical properties change during the service life. Generally, the bitumen becomes more stiff and brittle when ages.

On the other hand, the rheology is the science of studying the deformation and flow behavior of materials under different conditions. The rheology of bitumen plays a very important role in pavement performance. For instance, if the bitumen used within a flexible pavement deforms and flows very easily, it would lead to poor rutting resistance since if it is stiff and resistant to deformation and flow, the resulting flexible pavement would be less flexible. The stiff bitumen can be affected adversely in low-temperature conditions and eventually can cause fatigue cracking.

In consequence, most grading systems take these two important physical characteristics into account in the characterization of bitumen. These grading systems are based on the test results which are directly attributed to the rheological properties of bitumen. The most important grading systems used worldwide are presented briefly within the following subsections.

2.1.1.1 Penetration Grading

The consistency of bitumen is measured by the penetration test using a standard needle. In the refinery industry, the penetration grading refers to a range of penetration units in which one penetration unit equals 0.1 mm of penetration by a standard needle. The penetration value is the depth to which the standard needle penetrates into the bitumen sample under a specified load (100 gr) for 5 seconds at 25 °C (ASTM D5 or EN 1426). Figure 2.3 shows a standard penetration test device. In addition to penetration test, softening point is also measured and indicated in this grading system.



Figure 2.3 Standard penetration test device (Personal archive, 2012)

The softening point is the reference temperature at which a specific ball (with specific size and weight) passes through the bitumen sample and flowingly touches the bottom of a specific frame while being heated at a certain rate (ASTM D36 or EN 1427). A standard softening point test apparatus is presented in Figure 2.4. Typical bitumen binders used in Turkey are 50/70 and 70/100 penetration grade. In the Aegean region 50/70 pen. grade are used according to Turkish General Directorate for Highways (Karayolları Genel Müdürlüğü).



Figure 2.4 Standard softening point test apparatus (Personal archive, 2012)

2.1.1.2 Viscosity Grading

Another common grading system in refinery industry is the viscosity grading system in which the bitumen viscosity is referenced in poises ($\text{cm-g-s} = \text{dyne-second/cm}^2$) or poises divided by 10 after being measured by a rotational viscometer at 60 °C and 135 °C. Viscosity is defined as the measure of a fluid resistance to flow. It's calculated as the ratio between the applied shear stress and the rate of shear strain. These values are measured by means of viscometers. In the case of bitumen the most commonly used viscometer is rotational viscometer. A rotational viscometer and sample containers are presented in Figure 2.5.



Figure 2.5 Major RV equipment and sample containers (Personal archive, 2017)

Although the viscosity grading is an acceptable grading system it is not widely used since it does not characterize the low-temperature behavior of bitumen.

2.1.1.3 Performance Grading

Both penetration grading and viscosity grading have limitations in characterizing of bitumen since these grading systems do not fully evaluate the rheological characteristics of bitumen. The need for a new grading system which is able to more precisely characterize the bitumen for use in flexible pavements lead the researchers to develop a new grading system which is based on new tests and specifications. With the use of these tests and specifications, it is more possible to address bitumen behavior and performance in flexible pavements.

In Superpave performance grading system, the main principle is to select a bitumen grade in order to achieve the best performance in the conditions under which it is used. When we talk about the conditions under which the bitumen is supposed to be used, it

involves traffic load combinations, climatic conditions and also aging concerns. For this reason, like penetration grading and viscosity grading systems the PG system uses several tests to identify that if a particular bitumen is able to satisfy test limits at certain temperatures or not. The tests temperatures are chosen based on the particular climatic conditions in which that the bitumen is supposed be used.

Choosing the right bitumen based on conditions of use is more reliable in Superpave PG system compared to penetration grading and viscosity grading systems. The design criteria are to choose a bitumen to tolerate the maximum and minimum temperatures (°C) that the pavement would encounter within the service life. The maximum temperature is obtained as the average seven day maximum pavement temperature (°C) since the minimum temperature is the temperature (°C) that the pavement would potentially experience in cold weather conditions. Thus, the Superpave PG is indicated using two numbers separated with a dash (-). For instance, a PG 64-22 is supposed to be used within a climate in which the maximum seven day average temperature is 64°C and the minimum pavement temperature is 22°C. The PG can be changed by using bitumen modifiers. Therefore, if a higher PG is required, an existing source can be modified to achieve the desired PG before being used.

A series of tests are run in performance grading systems. These tests include Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), Rotational Viscometer (RV), Direct Tension Tester (DTT), Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel (PAV).

These tests are performed on the original binder, RTFO residue and PAV residue. Based on these tests results it is possible to entirely characterize the bitumen for specific conditions. The test specifications can be found in AASTO M320 standard. Table 2.1 contains the specifications for DSR test results.

Table 2.1 Performance grading routine values and design criteria

Performance Grade	PG 58					PG 64					PG 70					PG 76					PG 82						
Average seven day maximum pavement design temperature, °C	<58					<64					<70					<76					<82						
Minimum pavement design temperature, °C	>(-16) >(-10) >(-16) >(-22) >(-28) >(-34) >(-40)					>(-10) >(-16) >(-22) >(-28) >(-34) >(-40)																					
Original binder																											
Dynamic shear (T315), G*/sin δ, min 1 kPa, test tempearture @ 10 rad/s, °C	58					64					70					76					82						
RTFO treated binder (T240)																											
Dynamic shear (T315), G*/sin δ, min 2.2 kPa, test tempearture @ 10 rad/s, °C	58					64					70					76					82						
Pressure ageing vessel residue (R28)																											
PAV aging tempearture, °C	100					100					100 (110)					100 (110)					100 (110)						
Dynamic shear (T315), G*/sin δ, max 5000 kPa, test tempearture @ 10 rad/s, °C	25	22	19	16	13	31	28	25	22	19	16	34	31	28	25	22	19	37	34	31	28	25	40	37	34	31	28

2.1.2 Chemical Structure of Bitumen

According to shell bitumen handbook, Asphaltenes are black or brown amorphous solids which are insoluble in n-heptanes. In addition to carbon and hydrogen, Asphaltenes also contain trace quantities of nitrogen, sulfur and oxygen atoms. Asphaltenes are complex aromatic materials with high polarity and rather high molecular weight in comparison to maltenes. On the other hand, Maltenes are made up of other subgroups such as saturates, aromatics and resins. Bitumen classification is based on chromatographic techniques that make it possible to identify these four categories commonly known as SARA (Saturates, Aromatics, Resins and Asphaltenes) (Whiteoak & Read, 2003). Figure 2.6 shows the schematic representation of chromatographic method.

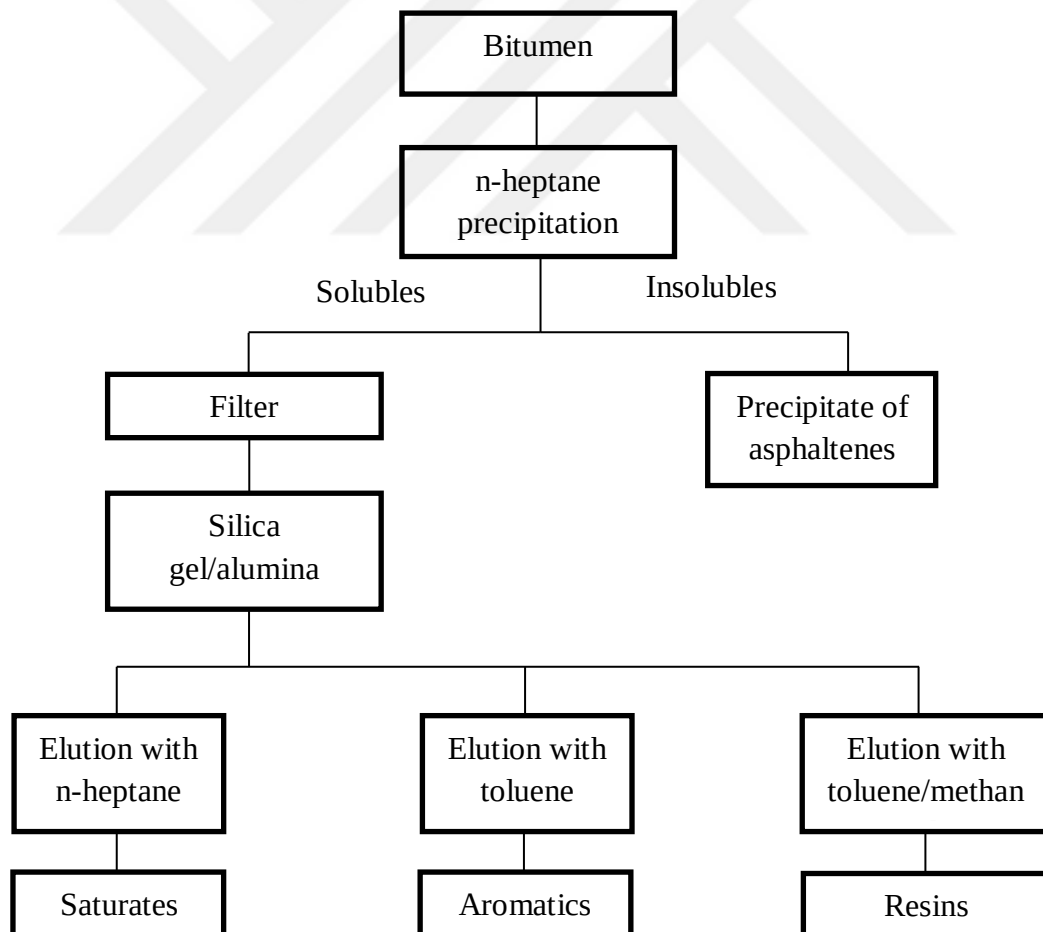


Figure 2.6 Schematic of the SARA chromatographic method (Whiteoak & Read, 2003)

2.1.2.1 Saturates

Saturates are generally consisted of branched-chain and straight aliphatic mixed with cycloaliphatic compounds. The color of saturates has been reported to vary from pale straw to colorless in some cases. Saturates are non-polar viscous oils. A study has reported the H/C (hydrogen-to-carbon) molar ratio of saturates approximately as two. The same study also claims that the molecular weight of saturates changes between 470 and 880 g/mol (Lesueur, 2009). Depending on the type of bitumen, saturates comprise approximately 5 to 20% of bitumen.

A study reported that the increase in the content of saturates can ease many negative aspects of the rheological concerns in bitumen. They also reported that although bitumen becomes less elastic, less viscous and less resistant to plastic deformation by increasing in the saturates content, it gains resistance against fatigue cracking (Hermadi et al., 2012). Studies have proved that the saturates content decreases as the bitumen ages (Zhao & Machel, 2012).

2.1.2.2 Aromatics

The aromatic are viscous liquids which are made up of non-polar carbon chains attached to unsaturated rings. According to Lesueur (2009), the main fraction of the dispersion medium in bitumen colloidal system is comprised of the aromatics. The aromatics have been reported to be the lowest molecular weight naphthenic aromatic compounds. The aromatics comprise about 40 to 65% of the total bitumen. The color of aromatics has been described as dark-brown. The H/C ratio of the aromatics has been reported to vary from 1.4 to 1.6. The average molecular weight range has been reported to be between 570 and 980 g/mol (Lesueur, 2009).

Aromatics are considered to be more volatilizing as compared to asphaltenes and resins due to their lower molecular weight. Thus the aromatics content may significantly decrease when bitumen is heated. This phenomenon is one of the major causes of hardening of bitumen during the aging process (Peralta et al., 2014).

2.1.2.3 Resins

Mainly composed of hydrogen and carbon, resins also involve minor amounts of oxygen, sulfur and nitrogen atoms. Resins have been reported to be soluble in n-heptane. Their color has been described as dark brown. They can be found in solid or semi-solid forms. Resins are strongly adhesive materials with high polarity. Resins play the peptizer role for the asphaltenes. The solution (sol) or gelatinous (gel) feature of the bitumen is highly dependent on the proportion of resins to asphaltenes. Reports claim the molecular weight of resins varies between 780 and 1400 g/mol. H/C atomic ratio of resins have found to be between 1.4 and 1.7 (Lesueur, 2009).

It is known that when bitumen ages the aromatic fraction decreases since the resin and asphaltene fractions (Whiteoak & Read, 2003)

2.1.2.4 Asphaltenes

Unlike other bitumen fractions, asphaltenes are insoluble in n-heptane. In addition to carbon and hydrogen, Asphaltenes also contain trace quantities of nitrogen, sulfur and oxygen atoms. Asphaltenes are complex aromatic materials with high polarity and rather high molecular weight in comparison to maltenes.

Various studies have reported several molecular weights values, varying extensively from 800 to 3500 g/mol. The H/C ratio of asphaltenes has been reported ranging from 0.98 to 1.6 (Lesueur, 2009). The size of asphaltene particles has been reported approximately as 2 to 5 nm. The content of asphaltenes plays an important role in the rheological behavior of bitumen. The high asphaltene contents results in stiffer, more viscous bitumen. It also affects the conventional properties of bitumen such as penetration value, softening point and the viscosity of bitumen. Asphaltenes comprise about 5 to 25% of the bitumen. The asphaltenes are much larger in particle size in comparison to other fractions such as maltenes. Asphaltenes are influential in final form and structure of the bitumen.

2.1.3 Microstructure of Bitumen

Studies related to bitumen microstructure with Atomic Force Microscopy (AFM) micro-topographic structures similar to bees on the top surface of bitumen. Loeber et al. (1996) used AFM to study the microstructure of bitumen. They indicated that they used AFM to investigate bitumen microstructure because it is capable of scanning the surface in micro size without damaging the original surface of the bitumen. The authors observed the micro bee body like structures on the surface of bitumen for the first time and called it bee-structures as they are much similar to bodies of bees (Loeber et al., 1996). Following the study of Loeber et al. (1996) many other studies focused on the bee structures on the surface of bitumen. Figure 2.7 shows the view of a bee structure on the top surface of bitumen. The bright and dark colors represent the indentation in micro size topography of bitumen surface.

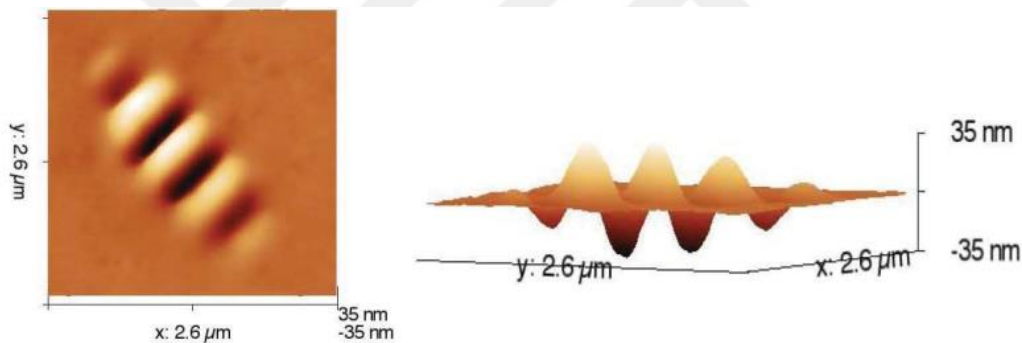


Figure 2.7 Bitumen AFM bee-structure top and side view (Masson et al., 2006)

As mentioned above, other researchers also conducted extensive research on the bee structures and also on the microstructures around the bee shapes. In addition to bee structures, other shapes rarely were reported on the surface of bitumen by the researchers who also used AFM to investigate the surface of bitumen in micro dimensions (Yu et al., 2015). These shapes were reported to be more like flakes, dendrite branches and flowers (Yu et al., 2015). Masson et al. (2006) studied the bee-structure in more details using AFM as well. They defined four different regions in microtopographic characterization of bitumen surface. These regions are shown in Figure 2.8.

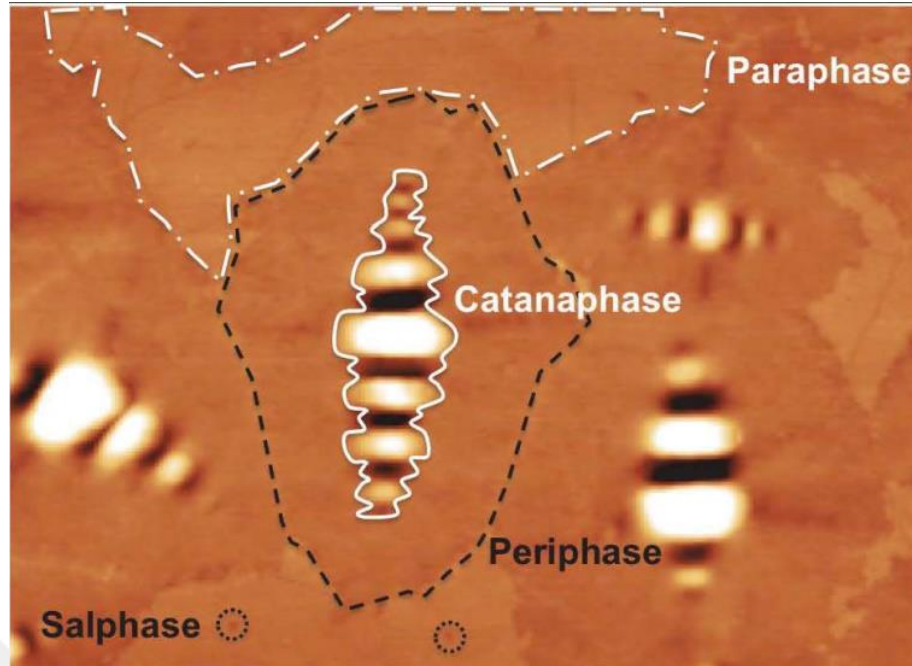


Figure 2.8 Bitumen microstructures region (Masson et al., 2006)

Loeber et al. (1996) tried to explain the bee-structures on the bitumen surface by the theory of polarity born by resins and asphaltenes in the bitumen (Loeber et al., 1996). Later on, Pauli et al. (2001) conducted similar research by investigating eight various SHRP (Strategic Highway Research Program) bitumen binders with different asphaltene and wax contents using AFM (Pauli et al., 2001).

The theory of Loeber et al. (1996) about the bee structures origin was confirmed which previously was attributed to asphaltenes since they observed the bee-structures mostly on the surface of bitumen samples with more asphaltene contents rather than fewer asphaltene contents. They simply came to the conclusion that the bee structures can be attributed to the asphaltenes in bitumen. However, they couldn't clearly clarify the absence of bee-structures on the surface of some bitumen samples with relatively high asphaltene contents. The authors endorsed that the absence of bee structures in some bitumen samples would be due to lower viscosity of these samples which prevents the formation of micro-topographic structures on the surface of these samples (Pauli et al., 2001).

Ganeeva et al. (2010) studied the compliance of two oxidized bitumen samples from different sources (Central Asia and Russia) with olefinic thermo-elasto-plastics. They managed to observe bee structures on both oxidized bitumen samples. However, the size of bee structures in one of the samples decreased significantly after being modified with thermo-elasto-plastics since the bee structures completely disappeared in the other sample after modification and irregular globules of different sizes were observed instead of bee structures. The authors didn't clarify the disappearance of bee structures after modification (Ganeeva et al., 2010). Further studies of these bitumen samples only confirmed that the increase in asphaltene-resin composition lightly increased the bee structures for both bitumen types and did not significantly affect the bee structures (Oldham, 2015).

Another study on the issue was directly focused on the effect of asphaltenes and maltenes on the bee structures. Jäger et al. (2004) tried to precipitate the asphaltenes by use of n-heptane which is non-polar solvent in order to determine the chemical composition of the bee structures. The authors found out that the bee structures completely disappeared after the precipitating of asphaltenes. The authors came to the conclusion that the bee structures are attributed to the polarity of bitumen on the surface born by the asphaltenes and also resins (Jäger et al., 2004).

A very similar study conducted by Zhang et al. (2011a) proved the fact that the bee structures are attributed to the existence of asphaltene in bitumen. The authors observed the surface of different samples with and without asphaltenes mostly composed of maltenes. They reported that the samples including mostly maltenes have smooth surfaces without any bee structures and in some parts, they only reported some dents on the surface of these samples since the samples with high asphaltene amount the existence of the bee structures were reported to be obvious (Zhang et al., 2011a).

In another study conducted by the same researchers, the authors also suggested that microcrystalline waxes which are also eliminated with the precipitating of asphaltenes may have an efficient role on the formation of bee structures (Zhang et al., 2011a). After the suggestion of Zhang et al. (2011a) on the probable role of waxes on the

formation of the bee structures further studies were conducted to clarify the theory of the formation of bee structures on the surface of bitumen.

The wax content of a bitumen sample can significantly affect the microstructure of the samples and therefore the other characteristics of the bitumen sample. The asphalt binder typically has three main types of wax. The first type is paraffin wax which is found in n-alkanes that are almost free of side branches. The second type which was also referred to by Zhang et al. (2011b) known as microcrystalline wax are crystals found to be in flat, large plate forms (Dorset, 2005). The last type with the polar functional group is described to be from special aromatics and molecules which may crystallize after being cooled (Carbognani et al., 2000).

De Moraes et al. (2010) used an AFM device with the feature to control the temperature of the samples while testing. Through conducting several tests and observation with AFM and also with help of the results obtained from DSC testing, the authors concluded that the bee structures are more likely to be attributed to the existence of paraffin was since they were able to observe the bee structures at the temperature at which the crystallization of paraffin wax occurs since they couldn't observe the bee structures at temperatures over 56 °C the temperature at which the crystal structure of the paraffin wax is destroyed (De Moraes et al., 2010).

Das et al. (2013) conducted a similar research to investigate the effect of heating on the microstructure of bitumen. They also observed the same phenomenon on the bitumen samples modified with Sasobit® a warm mix asphalt additive which is also known to be a type of paraffin wax. The authors observe the bee structures on the surface of Sasobit ® modified samples at the temperatures which were in the crystallization range of this specific paraffin wax. Thus, the authors concluded that the bee structures are more likely attributed to the paraffin waxes rather than asphaltenes (Das et al., 2013).

Soenen et al. (2014) conducted a study on four bitumen samples with the same penetration grades at three different libraries aiming to investigate the microstructures

of the samples by use of AFM imaging and DSC testing. Although the authors observed various microstructural characteristics on the surfaces of the samples with the same penetration grades due to the variety of chemical compositions, they also draw the same conclusion with the previous studies indicating that the bee structures are disappeared at the temperatures at which the waxes lose their crystallized structures (Soenen et al., 2014).

Schmets et al. (2010) also studied the effects of waxes on the microstructure of the standard SHRP bitumen binders. Based on DSC results in correlations with AFM imaging, the authors reported that the bee structures are more related to wax content rather than asphaltene content. By high-resolution AFM imaging, the authors were able to observe the microstructure of bitumen samples in more details compared to previous studies. The authors indicated that the bee structures can mostly be attributed to the existence of wax inside the bitumen chemical composition (Schmets et al., 2010).

Although these recent studies suggest that the bee structures on the surface of bitumen in microscale are more likely due to the crystalline waxes in the chemical composition of bitumen, there is no certain evidence that these structures are only attributed to these waxes since some other studies indicate that the asphaltenes are the polarity born by the asphaltenes and resins is responsible for the formation of the bee structures on the surface of bitumen. This led the researchers to a combined idea that both asphaltenes and waxes may be responsible for the formation of bee shape topography on the surface of bitumen.

Within the scope of another study conducted by Pauli et al. (2011), the morphological characteristics of different asphalt core samples were investigated using AFM imaging (Pauli et al., 2011). Following the determination of crystalline materials content using DSC testing, the authors applied some series of heating and cooling cycles on different samples. They observed that the samples with less crystalline material showed little changes in their microstructure since the changes in the microstructure characteristics of the samples with over 1% of crystalline materials

were significant. The authors made an inference on that not only the crystalline paraffin waxes are responsible for the microtopography of the bitumen samples, but also non-wax materials especially the asphaltenes causes roughness on the surface of the samples. Similar assumptions were also made by other authors as well indicating that the cooperation of asphaltenes and waxes are the main reason for the formation of bee structures on bitumen samples in micro dimensions (Sourty et al., 2011; Fischer et al., 2013).

There are also other theories suggesting different reasons for the formation of the bee structures rather than the theories of asphaltenes and waxes. For instance, a study done by Masson et al. (2006), AFM imaging was used to examine different bitumen samples in terms of the microstructure. The authors reported that the asphaltene content and the bee size demonstrate poor correlation with each other. The authors indicated that the other materials such as nickel and vanadium which can form some bonds with the free electrons in nitrogen, oxygen, and sulfur presented within the chemical composition of the bitumen which composes aromatics piles in the bitumen are responsible for the bee structures (Masson et al., 2006).

Some studies have used AFM imaging in order to investigate the effect of aging on the microstructure characteristics of bitumen samples. Such studies have mainly suggested the disappearance of the bee structures on the surface of aged bitumen. Within a study done by Nahar et al. (2013) virgin and extracted RAP bitumen (aged binder) were studied using AFM and DSR analysis. The authors found strong correlations between the existence of the bee structures and the complex modulus of the samples.

Since the aged RAP bitumen was reported to have less amplitude than virgin bitumen in terms of the bee structures than the virgin bitumen, the blended bitumen sample was interestingly reported to show higher amplitude compared to the even virgin binder. Thus, the authors suggested that the bee structures would be simple wrinkles born from different thermal expansion coefficients of different sources of bitumen. Although other studies do not support this idea on the formation of the bee

structures, this idea was found to be quite different within the literature (Nahar et al., 2013).

There are studies within the literature which address the effects of the aging phenomenon on the microstructure of bitumen. In this regard, Wu et al. (2009) tested different types of SBS modified bitumen and PAV aged samples. Although no bee structures were observed on the micro surface of a type of un-aged bitumen sample, in the case of PG 76 all bee structures were observed before and after aging. It was reported that aging causes the decreasing in size or even disappearing of bee structures (Wu et al., 2009).

In another study conducted by Zhang et al. (2012) the same concerns were addressed. The authors used a cycle of RTFOT aging followed by UV aging instead of PAV aging. Although the bee structures were clearly observable on the surface of un-aged binders, they completely disappeared after getting aged by UV process (Zhang et al., 2012).

Nahar et al. (2014) employed artificial aging processes to obtain aged bitumen. They used Belgian Rotating Cylinder Ageing Test (RCAT) which is similar to RTFOT. 500 grams of bitumen binder samples are heated at 163°C for 18 hours while being rotated at 5 rpm with the gradual direct flow of oxygen. The authors reported that the number of bee structures decreased completely while their sizes were increased after being aged (Nahar et al., 2014). However, Yu et al. (2014) reported very small changes in the microstructure of aged bitumen samples. They used RTFO and PAV aging tests to obtain aged bitumen (Yu et al., 2014).

There are studies within the literature which address the effects of rejuvenators on the microstructure of bitumen and aged RAP bitumen. These studies are presented in the following sections.

2.2 Asphalt Pavement

Asphalt pavement or asphalt concrete is composed of mainly bitumen and aggregates. Other than bitumen and aggregates, there may be various additives inside an asphalt concrete in order to improve its characteristics either in terms of stability or durability. Asphalt pavement requires special design. Thus, there are design methods developed so far to make the best and the most optimum mixture ready to use within specific climatic conditions. The most famous mix design methods are the Marshall mix design method, the Superpave mix design method, and Hveem mix design method.

On the other hand, independent from mix design method there are different types of asphalt pavement technologies based on various gradation types. These technologies are dense graded asphalt, open-graded asphalt, gap graded asphalt and also stone mastic asphalt pavements.

Asphalt concrete also can be categorized on the basis of the layer it is supposed to be used within. There are mainly two categories in this regard, the asphalt concrete for surface courses and the asphalt concrete for base courses. The gradation and design measures for asphalt concrete mixtures vary on this context.

2.3 Asphalt Pavement Failures

There are types of failures in asphalt pavements. These failures may be occurred due to many reasons. Failures can be categorized based on the factors leading to the distresses of pavements. These failures might occur due to either lack of stability or durability. The durability induced failures mainly occur within the course of time especially when the asphalt pavement gets older and becomes aged. Nevertheless, it is important to note that, in many cases the distresses on asphalt pavements have several factors affecting in conjunction with each other. The significant asphalt pavement failures are presented within the following sections.

2.3.1 Permanent Deformation

Permanent deformation best known as rutting is one of the major distresses of asphalt concretes. Rutting is simply surface depression in the wheel path. Rutting may be caused by several reasons such as unstable HMA, densification of HMA and settlement in subgrade (FHWA, 2006). Rutting may cause several inconveniences such as vehicle hydroplaning and pulling vehicles toward ruts. Figure 2.9 shows a typical rutting.

To prevent rutting, sufficient compaction of the layers should be applied exactly. Beside this, pavement design, especially in terms of drainage ability and layers thickness, should be adequate.



Figure 2.9 Shallow rutting (vertical indentation) is observable in this street (Pavement interactive, 2014)

Rutting typically occurs during the summer times under higher temperatures. This might be considered that rutting would be solely a bitumen problem, but it is more correct to indicate that rutting is the problem of the mixture and the structure (Asphalt Institute, 1995).

2.3.2 Fatigue Cracking (Alligator)

Fatigue cracks also known as alligator cracks because of their special shape are a series of longitudinal and interconnected cracks mainly caused by repeated loads. These cracks normally initiate as short longitudinal cracks in the wheel path developing to an alligator pattern (FHWA, 2006).

Since the alligator cracks occur due to repeated traffic loads, there should be a distinctive care about traffic loads repetition during the design process. On the other hand, when determining the mix design of the pavement, tensile stresses should be considered carefully. Figure 2.10 demonstrates an advanced stage of fatigue cracking.



Figure 2.10 Severe alligator cracking near the stop line at an intersection (Hawai Asphalt, 2012)

2.3.3 Thermal Cracking

Thermal cracking also called low temperature cracking or transverse cracking due to its transversal shape, is a type of pavement distress that occur as the temperature decreases in adverse environmental conditions. Since the asphalt concrete is strained from movement owing to the friction with underlying films, tensile stresses progress within the material. In case that these stresses exceed the tensile strength of the

mixture, cracks start progressing in the transverse direction (FHWA, 2006). A typical thermal cracking has been shown in Figure 2.11.

The width of the cracks may differ in different seasons. Depend on the width of the cracks; moisture infiltration may occur within the cracks. Thermal cracks also cause an inconvenience due to roughness while driving (FHWA, 2006).



Figure 2.11 Transverse (thermal) cracking (Pavement interactive, 2012b)

2.3.4 Stripping

Moisture can be described as the first factor of distress in asphalt concrete. Asphalt concrete may be deemed at risk to water if the bitumen-to-aggregate adhesion deteriorates in the existence of water or water vapor (Hicks, 1991).

Moisture susceptibility is a complicated event. It is hard to say with confidence whether a specific feature will be the overriding issue in determining moisture susceptibility. Normally, moisture susceptibility is increased when moisture content raises in the mixture, This phenomenon reduces the bond between bitumen and aggregate surface or actually scours the bitumen (McGennis et al., 1984). Moisture damage eventually results in loss of performance in asphalt concretes.

To prevent moisture induced damage, it is suggested to use good aggregate. In addition to this, aggregate type and the bitumen used should provide the bond needed. Many studies also present some additives which are helpful in terms of anti-stripping. Drainage conditions should be designed carefully as well. During application period mixing temperature must be concerned and there should be a distinctive care about adding completely dried aggregates.

Initial objections on the WMA mixtures were that the aggregates are not heated enough to overcome moisture problems. Recent studies show that WMA pavements present enough strength against moisture induced damages as well as the HMA pavements (Kim et al., 2012). Figure 2.12 demonstrates a weak moisture susceptibility at a bottom of a core.



Figure 2.12 A small pavement core showing stripping at the bottom of the pavement section (Pavement interactive, 2012b)

2.3.5 Bleeding

Bleeding is a thin layer of bitumen (asphalt binder) on the gathered on the upper surface of asphalt pavement. It forms a polished film on the surface which reflects the lights giving it a smooth display on the surface. The bled film may be stick in hot

weather conditions and slippery in rainy weather conditions. The probable risk is the lack of skid resistance when the surface is wet. The main reason of bleeding is poor mix design and/or errors in manufacturing process.

An excessive amount of bitumen within the asphalt pavement or a wrong type of bitumen both can cause bleeding problems. If the bleeding problem is a minor problem, it can be temporarily repaired by applying proper amount of sand over it since it is not adequate solution and the bleeding problem may continue in various parts in hot seasons.

Sometimes, the excessive traffic loads during the summer may cause bleeding problems when the asphalt pavement surface reaches high temperatures. Selection of right PG or penetration grade of bitumen with higher softening point in the regions where the temperature differences are significant between cold and hot seasons is important in terms of bleeding. Figure 2.13 shows a surface with partial bleeding problem.



Figure 2.13 Severe bleeding (Pavement interactive, 2012b)

2.3.6 Longitudinal Cracking

Longitudinal cracks are somehow types of fatigue cracking which are parallel to the pavement's centerline. During the construction phase, there may be poor joint construction which can cause longitudinal cracks. It is important to place the construction joint somewhere out of the wheel path on the pavement surface to avoid these joints been overloaded during the service life of the pavement. Additionally, the other causes of longitudinal cracks may be the problems imposed from underlying surfaces.

As aforementioned, the foremost problem in formation of longitudinal cracks is fatigue. The longitudinal cracks if not treated at early stages, can cause permeability problems and consequently main problems at lower courses. A typical longitudinal cracking formed due to weak construction joints is presented in Figure 2.14.



Figure 2.14 Longitudinal cracking (Pavement interactive, 2012b)

2.4 Aging of Bitumen

The durability of asphalt concrete is a measure of its level of resistance to hardening (also called aging) over time. Generally, when bitumen ages, it becomes more brittle and harder. Beside this, the viscosity of aged bitumen is higher than virgin bitumen. Durability plays a considerable role on asphalt concrete mixture performance and considerably changes the other qualities (e.g. rutting and fatigue).

Bitumen content and air voids are significant factors in control of durability. The main factors of aging can simply be described as follows (Roberts et al., 1991):

- Oxidation: Is the reaction between bitumen and oxygen gas.
- Volatilization: is the evaporation of the less heavy components of the bitumen over time. It is mostly due to temperature and happens mainly during mixing process.
- Polymerization: is the process of combining monomers together in a chemical reaction to form polymer chains. These chains are considered to cause a progressive hardening.
- Thixotropy: is the property of the bitumen when it sets where not physically disturbed or set in motion. In bitumen, thixotropy is assumed to be a consequence of hydrophilic suspended particles that form a lattice structure. This triggers an increase in viscosity and hence, hardening. This phenomenon can somewhat be reversible by heat and agitation. Roads with less traffic loads are largely at risk of thixotropy.
- Syneresis: can be defined as the separation of less viscous liquids from the more viscous liquids throughout the bitumen. This may cause hardening in the asphalt mixture. Due to either physical or chemical changes, shrinkage or rearrangement of the bitumen occurs in this case. Syneresis is considered to be a kind of bleeding distress.

- Separation: is the absorption of the oily components, resins or asphaltenes from the bitumen by some aggregates with high level of porosity. This causes the bitumen hardening due to less oily components.

Aging is mainly contains of two components. First component is mainly associated with loss of volatilization in asphalt during the construction and mixing phase also called short-term aging, and the second component which is associated with gradual oxidation of the in-place material in the field called long-term aging (Bell, 1989).

2.4.1 Aging Simulation of Bitumen

Promising methods for laboratory simulating of asphalt concretes should consider short-term aging simulation conditions as well as the long-term aging conditions. Most simulation methods such as Thin Film Oven Test (TFOT), Rolling Thin Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) test try to subject bitumen itself to aging conditions whereas some methods subject mixture to aging conditions as AASHTO R 30 (Standard Practice for Mixture Conditioning of Hot Mix Asphalt) Method. Brief principles and test procedures for these methods are as follows:

2.4.1.1 Thin Film Oven Test (TFOT)

Welborn (1984), mentions Dow (1903), representing the use of an early extended heating test. Years later, Lewis and Welbom (1940), introduced the TFOT for making comparison between asphalts in terms of aging characteristics. Further work was described by Lewis & Halstead (1946) . This test is a simulation of short-term aging process.

In initial practices of this test, a 50 ml of asphalt sample was heated in a 3 mm film inside a 140 mm diameter flat container for 5 hrs. at 163°C. AASHTO (American Association of State Highway and Transportation Officials,) and ASTM (American Society for Testing and Materials) then adopted this test as an aging simulation. Residue of the aged bitumen then is tested for penetration, softening point and ductility (Bell, 1989). Loss in mass is also calculated after test by weighing sample residue.

2.4.1.2 Rolling Thin Film Oven Test (RTFOT)

The California division of highways developed this test as an improvement to the TFOT in order to age bitumen in thinner films than the 3mm which is used in TFOT (Bell, 1989). Like TFOT, the RTFOT aging method is also used for simulating aging during mixing and placement (Short-term aging).

Hveem et al. (1963) adjusted the procedure for this test. 35 grams of un-aged bitumen is located in a cylindrical specific glassy bottle, which is then placed in a carousel inside the RTFOT device (Hveem et al., 1963). This helps bitumen thickness to decrease to 1.25 mm in comparison with TFOT. The oven is heated to 163°C and the carousel continues rotating for 85 minutes at 15 rpm rate. The carousel rotation continuously exposures new bitumen to the heat and air blown by a compressor (Bell, 1989). This test is adopted by both AASHTO and ASTM.

Samples residues are then tested for conventional bitumen tests such as penetration, viscosity, softening point and ductility to evaluate the effect of aging characteristics and to gain aging indices. Mass loss should also be calculated after extruding bottles from RTFOT oven. For further investigation, sample residues can be tested for Pressure Aging Vessel (PAV) to investigate long-term aging characteristics of the sample.

2.4.1.3 Pressure Aging Vessel (PAV) Test

This test is used for long-term aging simulation. Bitumen sample is then ready for Physical characteristics test. Heat and pressure are the conditions applied to bitumen sample to simulate aging during service life of the pavement. Bitumen sample residue of RTFOT which has been subjected to short-term aging is taken by PAV. Test procedure contains placing sample inside a steel pan and heated inside pressurized (305 psi) vessel to apply aging. This test has been adopted by AASHTO and the details can be found in AASHTO R 28.

2.4.1.4 Rolling Cylinder Ageing Test (RCAT)

This test has developed by the Belgian Road Research Centre in conformance with EN 15323. Both short term and long-term aging conditions can be simulated using RCAT through changing test temperature and duration. In case of short aging, the test adopts similar conditions to the RTFOT (163°C), since the duration is a bit longer (235+5 minutes). For long term aging, in order to get similar results as PAV, the suggested aging conditions are 176+16 hours RCAT at 85°C or 125+11 hours RCAT at 90°C on RTFOT or short term aged RCAT residue (Airey, 2003).

2.4.1.5 UV (Ultraviolet) Radiation Oven Test (UROT)

Use of UV radiation for aging simulation of bitumen has been addressed in some recent studies (Wu et al., 2010; Zeng et al., 2015). Although there is no proved standard for this test, it has gained popularity among academicians and industry world at recent years. Mainly, the system is consisted of a temperature-controlled cabin and an UV lamp and in some cases, there is also an air blowing unit to obtain better results. The test sample usually is prepared as a very thin bitumen sample to get fully exposed to UV radiation.

2.4.1.6 Aging of Bituminous Mixture (AASHTO R 30)

AASHTO has associated a standard test method for aging simulation of asphalt mixtures. So far described methods for aging simulation, all take bitumen samples and not bituminous mixtures. This standard takes bitumen – aggregate mixtures for further short- and long-term aging processes. Performance tests which take compacted Marshall or Gyratory compactor specimens can be applied after aging process. A brief instruction for this standard (for samples intended to be compacted with Marshall Compactor) is as follows:

- For short-term aging simulation, asphalt mixtures are placed in a pan (in loose mix state) and spread to an even thickness ranging between 25 to 50 mm, and

then the pan is placed inside a forced-draft oven set for 135°C for $4\text{ h} \pm 5$ minutes. The mix should be stirred every hour for providing a uniform conditioning. The mixture is then ready for compaction and curing process.

- For long-term aging simulation, short-term aging process should be done in advance. After compaction, specimens should be cooled in an oven set for 60°C . Cooling may approximately take 2 hrs. for appropriate sized specimens. After the specimens are cooled in room temperature for 16 hrs. they can be extruded from molds. Specimens are then placed in an oven set for $85 \pm 1^{\circ}\text{C}$ for 120 ± 0.5 hrs. after this period, the oven should be turned off and the specimens are allowed to cool in room temperature inside the opened door oven. Cooling may take approximately 16 hrs.

2.4.2 Evaluation of Aged Bitumen

There are significant differences between the behaviors of virgin bitumen and aged bitumen. It is also true for new constructed asphalt pavements and old aged pavements. Distresses in an asphalt pavement gain speed as the asphalt ages. As the asphalt concrete ages, the higher it is likely to get affected by the environmental conditions. To evaluate aging characteristics of bitumen or an asphalt concrete, conventional performance tests can be used. Although many of these tests results may show differences due to aging, some tests can give us more proper evaluation.

Normally, aged bitumen can be tested for a set of tests including Penetration, Softening Point, Rotational Viscosity (RV), Ductility, Bending Beam Rheometer (BBR), Direct Tension Test (DTT) and Dynamic Shear Rheometer (DSR) (Pavement Interactive, 2012c). Each test is used to evaluate a specific characteristic of the bitumen after being aged. Rather than bitumen conventional test, BBR and DTT for instance, are used to investigate low temperature cracking behavior of the bitumen. All these tests are applied on aged samples in order to investigate aging characterization of bitumen and bituminous mixtures.

2.4.2.1 Rotational Viscosity

Brookfield rotational viscometer is the most common device which is used to determine bitumen viscosity. The temperature range for this test is between 135°C and 165°C which is acceptable temperature range for mixing and compaction of the asphalt-aggregate mixtures (National Cooperative Highway Research Program., 2010).

The Rotational Viscosity test helps to certify if the bitumen viscosity is sufficient for pumping and mixing or not (Roberts, Brown, & Kennedy, 1996). The graphics for viscosities vs. temperature are then plotted. acceptable temperature for mixing is the range matching 0.17 ± 0.02 Pa·s and acceptable temperature for compaction is the range matching 0.28 ± 0.03 Pa·s (Mo et al., 2012).

As the bitumen ages, it becomes stiffer, therefore the viscosity of an aged bitumen is rather more than the viscosity of a virgin bitumen in a specific temperature. ASTM (1973) presented the aging index as the ratio of the viscosity of the bitumen after aging to the viscosity of the bitumen before aging process. Although this index doesn't consider the conditions of mixing, but it gives out a good evaluating index (ASTM, 1973) .

2.4.2.2 Dynamic Shear Rheometer (DSR)

Asphalt is a viscoelastic material. It means that asphalt can behave like an elastic solid in certain conditions and like a viscous liquid in some other conditions depending on the test temperature. In terms of rheology, DSR is a device which is capable of characterizing rheological behavior of bitumen samples in different temperatures. This characterization is used in the Performance Grading (PG) bitumen. The device can measure a bitumen sample's complex shear modulus (G^*) and phase angle (δ).

Complex shear modulus (G^*) is the resistance of a bitumen sample to resulting shear strain when subjected to repeatedly shear stress since the phase angle (δ) is the

interval between the strain caused by shear stress and the shear stress itself. The phase angle (δ) value for purely elastic materials is 0° since this value is 90° for purely viscous materials. This means that the higher the phase angle (δ), the more viscous is the material (Pavement Interactive, 2012d). DSR uses a thin bitumen sample, sandwiching it between two circular steel plates. To create shearing, the upper plate oscillates across the sample which is placed on the fixed upper plate.

DSR test can be conducted on un-aged and aged (by TFOT, RTFOT & PAV) samples. Aged samples show different rheological behavior in comparison with un-aged samples since they have become stiffer after getting aged. The complex shear modulus (G^*) and the phase angle (δ) values for an aged sample, helps to understand how an asphalt concrete may behave after being aged during service life of the pavement. This helps to predict rutting and fatigue cracking performance of asphalt concretes.

The dynamic shear rheometer (DSR) is usually used for characterizing the viscoelastic behavior of bitumen binder and evaluate its rutting and cracking potential. The primary principle for the usage of DSR testing is that the binder behaves like elastic solids at lower temperatures and viscous fluid at high operating temperatures. These behaviors can be witnessed by measuring the complex modulus (G^*) and phase angle (δ) of a bitumen sample under a predetermined temperature and frequency of loading. The G^* and δ parameters are measured by the DSR as a result of applying a torque to the binder sample that is sandwiched between fixed and an oscillating plate and measuring the resultant strain. Figure 2.15 illustrates the mechanism by which the DSR operates.

The G^* is basically the ratio of shear stress to shear strain, while δ is the time lag between the stress and the strain in the binder sample. These properties are determined by taking the average of maximum HMA pavement field temperature over a 7-day period of time during the summer season, the data for which is attained from the local weather stations.

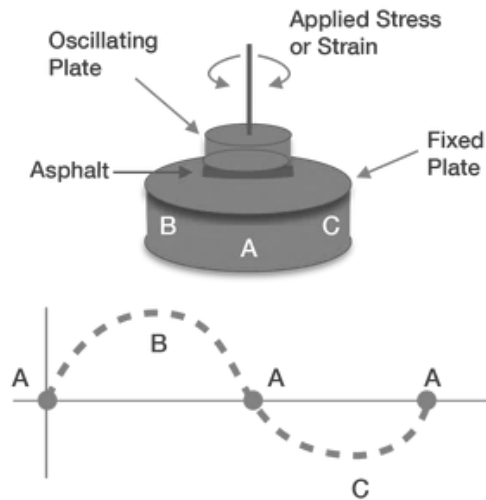


Figure 2.15 DSR operational mechanism and the resultant generated curve (Majeska, 2016)

The intermediate testing temperature is determined from an average of the 7-day average maximum and minimum design temperatures experienced in the field. As can be seen in Figure 2.16, the δ values range from 0 to 90, the higher the value of δ the more viscous the sample would be (Mallick & El-Korchi, 2017).

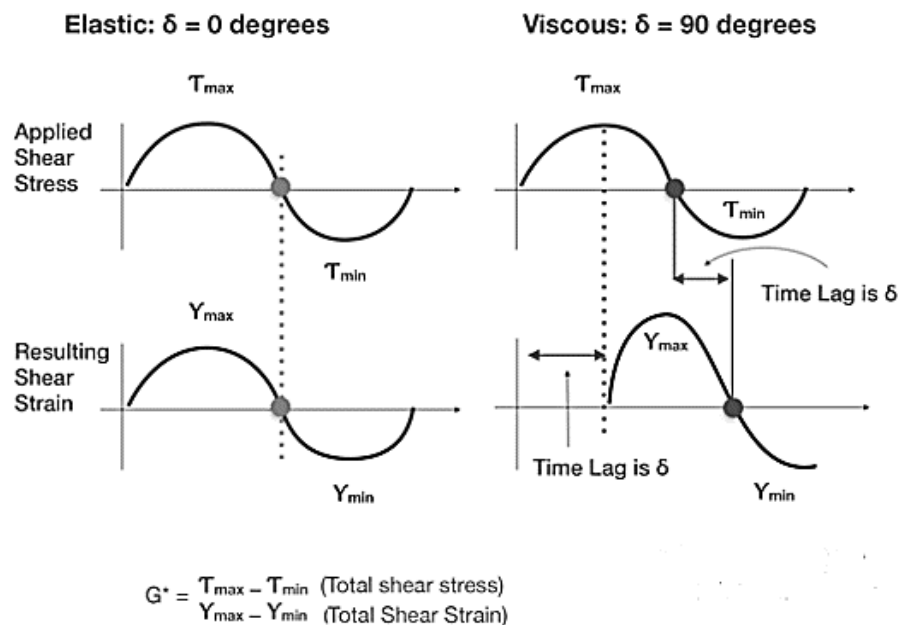


Figure 2.16 Phase angle curve variations (Majeska, 2016)

A conventional DSR accommodates two different sample sizes depending on the testing conditions and temperature ranges for that particular test. For the temperatures

greater than 46°C, a 1 mm thick and 25 mm in diameter sample is considered. However, if the temperature is in between 4°C and 40°C, in this case, a sample that is 2 mm thick and 8 mm in diameter is used (Figure 2.17).

The bitumen samples that undergo the DSR tests are usually unaged, short-term aged (RTFOT aged) and long-term aged (PAV aged) samples. With the help of DSR one can evaluate the rutting and fatigue characteristics of binders. The rutting potential is assessed by the $G^*/\sin \delta$ value which is termed as the rutting factor, of which G^* and δ can be obtained from DSR testing. As it is believed that higher G^* value infers greater resistance to rutting, and lower δ means highly elastic bitumen sample, therefore, higher G^* and lower δ are desirable to eventually improve the rutting performance of HMA.

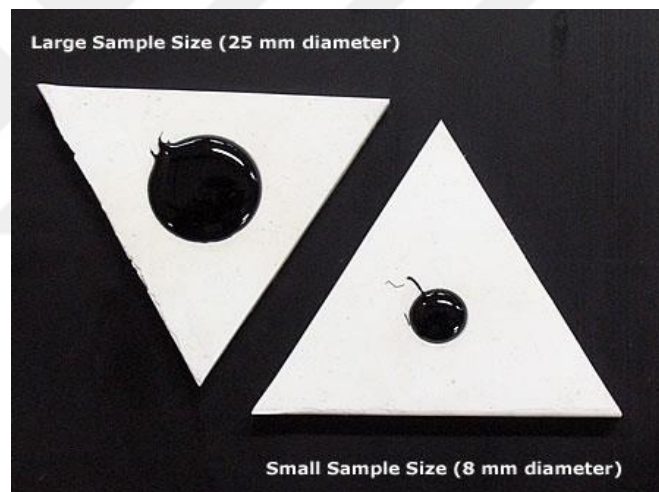


Figure 2.17 DSR sample molds (Pavement Interactive, 2012d)

Conducting this test on binder sample for the rutting factor prior to subjecting it to the long-term aging condition is very crucial, because the stiffness of the sample increases and resistance to rutting also improves (Bahia & Anderson, 1995). Therefore, the DSR test for rutting potential is carried out over the RTFOT condition samples. It is also conducted on unaged binder samples because sometimes RTFOT might not obtain proper information regarding the actual aging of the bitumen samples during the production and placing of the mix. The bitumen samples can also be tested with the DSR to determine the fatigue cracking of hot mix. The fatigue potential is

measured by the fatigue factor, defined as $G^* \sin \delta$. The bitumen sample becomes stiffer due to aging throughout its service life and becomes more cracking susceptible. The DSR test for fatigue potential evaluation is performed on PAV-aged samples (Mallick & El-Korchi, 2017).

During a study, the asphalt binder was first recovered from loose mix obtained during construction and from cores obtained after first, second and third years. Both dynamic shear rheometer (DSR) tests were performed on the binder extracted from the mix and on bitumen binders subjected to RTFO test and PAV conditioning method.

By comparing the DSR test results of the binder extracted from the loose mix and the DSR tests results of RTFOT conditioned binder samples indicated that the binder samples get relatively more aged in the plant mix process than in the RTFOT process carried out in the lab. However, the statistical analyses prove that the RTFOT conditioning method appears to accurately simulate the short-term aging of the binder sample. G^* and δ values from the extracted binder samples were used in order to develop the times versus property models. G^* values were observed to increase and δ values decreased with the time elapse.

Dynamic shear rheometer (DSR) tests were conducted on the recovered binder and on asphalt binders subjected to RTFO test (RTFOT) and PAV method. Comparison of DSR test results of asphalt binder recovered from loose mix and DSR test results of RTFOT conditioned asphalt binder indicated that asphalt binders are aged more in the plant mix process than in the RTFOT process. However, statistical analyses indicate that the RTFOT method seems to be capable of simulating short-term aging of asphalt binder. Complex shear modules (G^*) and phase angle (δ) values from asphalt binder extracted from in-place mixes were used to develop time versus property models. G^* values were found to increase and δ values were found to decrease with time (Mallick & Brown, 2004).

A study was conducted for assessing the effects of the solvent on the bitumen samples. Unaged, RTFOT-aged and PAV-aged samples underwent the DSR test. The

results inferred that by incorporating the Toluene solvent into the bitumen sample, the sample doesn't alter the rheological properties of the neat binder. The results also exhibited an increment in the G^* and decrement in δ . This infers that using of solvent can potentially enhance the performance of the modified binder sample without affecting the physical properties of bitumen for short and long-term aging (Ismail et al., 2012).

In a different study, the investigation of the properties of different bitumen binder samples was executed at different conditions of unaged, RTFOT-aged, PAV-aged, UV radiation and natural exposure aged (NEA) using DSR. The conclusion drawn from the investigation suggested that the UV radiation oven test has a strong corresponding relationship with NEA. The short-term NEA may affect the bitumen's properties more significantly than the conventional PAV aging (Wu et al., 2008).

Another research focused on the establishment of molecular dynamic simulation to simulate the actual movement of rejuvenators in the aged bitumen samples and to characterize their diffusion coefficient. Thereafter, the DSR tests were carried out to further investigate the rejuvenating effect. Based on the results, it was concluded that the molecular simulation and experimental results from the DSR test were quite similar. This infers that the molecular simulation proposed for the study could be successfully used to characterize the diffusion coefficient of rejuvenators in aged bitumen samples (Xiao et al., 2017).

2.4.2.3 Bending Beam Rheometer (BBR)

The vulnerability of asphalt mixture to low-temperature cracking is profoundly dependent on the behavior of bitumen binder becoming prone to aging and brittleness, thus understanding the properties of the binder is an inevitable step to be taken. The properties of aged bitumen binder at these low temperatures were usually predicted with the Fraass fracture temperatures for so many years. However, the Fraass breaking point test method was associated with a number of shortcomings. As a result, the

Bending Beam Rheometer (BBR) method was introduced by incorporating new important parameters such as stiffness and creep rate (Turk & Tušar, 2016).

BBR, as shown in Figure 2.18, is basically a device used to characterize the low-temperature properties of the bitumen binder. The BBR test (AASHTO T313) is a flexural stiffness test that is intended to measure the stiffness and relaxation properties of binders at below freezing temperatures after field aging and extraction or after the RTFOT/PAV conditioning is applied. The objective of the test is to make sure that the stiffness and brittleness of the binder are not exceeding the limit so that the binders are resistible to the transverse cracking occurring in HMA pavements in low temperatures (Salmans et al., 2015).

BBR can also determine the low-temperature Performance Grading (PG) of asphalt binders. The BBR method has been standardized (EN 14771) but is still not preferred in most of the European regions due to its limitation of only investigating the aged bitumen binder and not examining the fresh binder samples. Besides, the standard limit values for the evaluation of the quality of bitumen binder are still not set (Turk & Tušar, 2016).



Figure 2.18 Bending Beam Rheometer (BBR) (Pavement Interactive, 2012e)

For performing the BBR test, a small beam of bitumen sample (Figure 2.19), prepared with the dimensions of 125 millimeters long, 12.5 millimeters wide, and 6.25 millimeters thick that is simply supported and immersed in a cold liquid bath, is loaded for the duration of 60 seconds and the resultant deflection is measured. The test is generally run at -36, -30, -24, -18, -12, -6 and 0°C (Advanced Asphalt Technologies, 2011). The creep stiffness of the sample is then calculated based on the applied load and resulting deflection. Creep stiffness is used to evaluate the potential for high thermal stress development (Salmans et al., 2015).

Another parameter considered in the process of data evaluation of the BBR test is the m-value which is the log-log slope of the creep curve at a given loading time. The m-value of a bitumen binder provides an indication of the binder's ability to relax stress. During the low-temperature Performance grading of the bitumen binder, the BBR test is run at 10°C higher than the low grading temperature. For an instance, a PG 64-22 binder sample would be tested using the BBR at -12°C.

According to the standards, the maximum allowable stiffness in the BBR test is 300 Mpa at 60 seconds, and the minimum m-value is 0.300 Mpa is typically specified for laboratory RTFOT/PAV aged binder samples at the same loading time (Advanced Asphalt Technologies, 2011).

A study focused on the comparative analysis and selection of the best method amongst the three low-temperature test methods of binders (Fraass breaking point, ABCD cracking temperature, and BBR criteria) was performed. The BBR, specifically the m-value criterion, was suggested as the most precise and appropriate method to adequately predict the evolution of binder due to aging at low temperature in terms of the performance of mixtures (Dreessen et al., 2010).

The same study also indicated that the BBR test method allows distinguishing the binder characteristics considering the aging state. It also classifies binders according to their relevant ability to resist aging. As per the study results, the crosslinked PMB binder exhibited impressive performance in terms of both lower critical temperature

and the reduction of aging effect. These trends were specifically noticed after the application of PAV (long-term) aging conditioning. In addition, BBR critical low-temperature variation during the PAV aging process, in other words, the aging susceptibility, was reduced in the case of crosslinked PMB binder samples with the gap values substantially higher than the standard deviation of the BBR test method ($\pm 2^{\circ}\text{C}$). This is true particularly from the m-value criterion point of view (Hung et al., 2016).

Kanabar, in his study, implemented the extended bending beam rheometer (BBR) method in which the bitumen binder was conditioned for 1 hour, 24 hours and 72 hours prior to the specification grading. This method enabled a much-improved ranking of performance compared to the regular BBR method in which the binder was only cooled for 1 hour prior to testing. However, this method was also not entirely successful since it came up with some drawbacks associated with current laboratory chemical aging protocols (RTFOT and PAV aging).

Generally, these artificially aging methods cannot age the samples that can simulate the in-service bitumen binders. Therefore, it is really important to identify the types of chemical changes that take place in order to develop an improved chemical aging process (Kanabar, 2010).



Figure 2.19 BBR beam on its supports (Pavement Interactive, 2012e)

Some researchers at the University of Minnesota suggested that the BBR can also be used to measure the creep properties of asphalt mixtures as well, as shown in Figure 2.20. However, the main hindrance in implementing this into current practice is the use of small size BBR specimens that may not represent the mechanical behavior of the actual asphalt pavement especially when the mixtures containing larger aggregate particle sizes than the smallest dimension of the beam specimen (Falchetto et al., 2014).

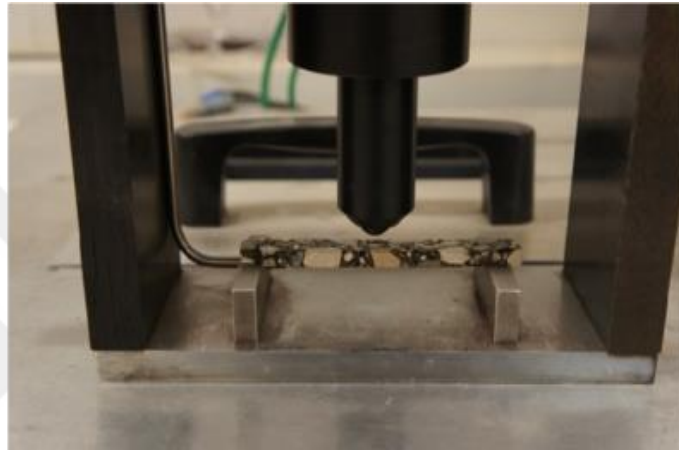


Figure 2.20 Bending Beam Rheometer with the asphalt beam mixture (Falchetto et al., 2014)

2.4.2.4 Indirect Tensile Test (IDT)

IDT can be used to define the creep compliances and the ITS of asphalt mixtures at low and normal temperatures. The ITS results can give us worthy evaluation keys in low temperature and fatigue cracking of asphalt pavements. Some studies introduce ITS result as a good indicator in predicting laboratory rutting potential of asphalt mixtures (Anderson, Christensen, & Bonaquist, 2003). This test is widely used in the investigation of moisture induced damages of bituminous mixtures. AASHTO has associated a standard for assessment of moisture susceptibility of asphalt mixtures using ITS test results (AASHTO T283).

When evaluating the aging characteristics, as the bitumen ages, it becomes more brittle and stiffer, thus the ITS results of an aged mixture are rather more than the results of an un-aged mixture. This can provide us aging indices to investigate aging

characterization of asphalt mixtures. Sengoz (2003) has implemented ITS results of mixtures with various air voids, to assess aging and moisture susceptibility characteristics of HMA mixtures.

Another study on short- and long-term aging behavior of rubber modified asphalts conducted by Liang and Lee (1996) has also proved the fact that the short-term and long-term aging increased the measured tensile strengths. Sengoz and Topal (2008) investigated the effect of SBS polymer modified bitumen on the aging properties of asphalt mixtures using ITS test results. They calculated aging indices as the ratio of short and the long-term aged specimen's ITS values to the values of un-aged control specimens prepared with the same additive content. Hurley and Prowell (2005) used ITS results to check the rutting potential after application and the short- and long-term aging characterization of WMA mixes containing Sasobit®.

Test method details are described in the standard developed by ASTM (2012). The second section of Chapter five includes a brief procedure of this test.

2.4.2.5 Atomic Force Microscopy

The atomic force microscope (AFM) is primarily a combination of the principles of the scanning tunneling microscope and the stylus profilometer. It comprises a probe that does not cause the damage of the sample surface (Binnig et al., 1986). The AFM works in a manner that a sharp tip probes the sample surface and collects the data. Usually, the cantilever tip is located at the free end of the cantilever and is only some microns in length, having a low spring constant of 100 to 200 μm in length (Benatar & Howland, 2000). The vertical displacement and lateral distortion in the cantilever are measured and mapped with the tip probing the surface of the sample (Allen et al., 2012).

In the ideal conditions, the resolution limit of a generic optical microscope is around 200 nm (Morris et al., 1999). However, AFM can ensure the atomic and molecular resolution (Binnig et al., 1986; Mou et al., 1996).

AFM is intended to investigate the surface topography, phase separation and other properties of materials like adhesion and stiffness. The investigation is commonly based on the mode of operating and stiffness of the material sample under study (Allen et al., 2012).

The first study devoted to using AFM for the investigation of the graphite structure was conducted by Binnig et al. which was then followed by many other studies investigating biological samples and other materials including metals (alloys, semiconductors, catalysts) or organic materials (polymers and biomaterials) but the investigation of asphalt binder structure using AFM was not adequately researched (Binnig et al., 1986; Allen et al., 2012).

Atomic force microscope ensures the structural study of asphalt binders without any pre-preparation. However, some studies stated that the morphological variation observed in asphalt binders are not done with ease due to its colloidal behavior. The existence of oil phase hinders the normal observations when using either electron microscopy or AFM (Loeber et al., 1996).

For the first time, AFM was employed in asphalt applications by Loeber and his coworkers for observing a heat-cast film to preserve the solid-state morphology. A dispersed phase with a “bee-like” appearance was attributed to asphaltenes based on a hypothetical assessment which was thereafter approved and supported by Pauli et al. as a result of their study (Pauli et al., 2001).

By combining various AFM modes and experimental conditions, the basis for a comprehensive assessment of asphalt binder’s micromechanical properties is provided (Allen et al., 2012). Figure 2.21 depicts the schematics for the lateral force technique of AFM applications. Some researchers used AFM to characterize asphalt binders and their properties, by correlating the surface morphology with the constituting elements of the asphalt binder (Pauli et al., 2001).

In a different study, the phase detection mode of AFM was employed to assess the morphology of asphalt binder and suggested a system through which the binder was categorized into three different groups, depending on the distinct domains or visible phases (Masson et al., 2006). Masson et al. also reported that the stiffness of the binder changed spatially with the microstructures. However, there is a small number of research present in the literature over the rheology of the microdomains observed using AFM, or the impact of factors like aging on these specific properties (Masson et al., 2007).

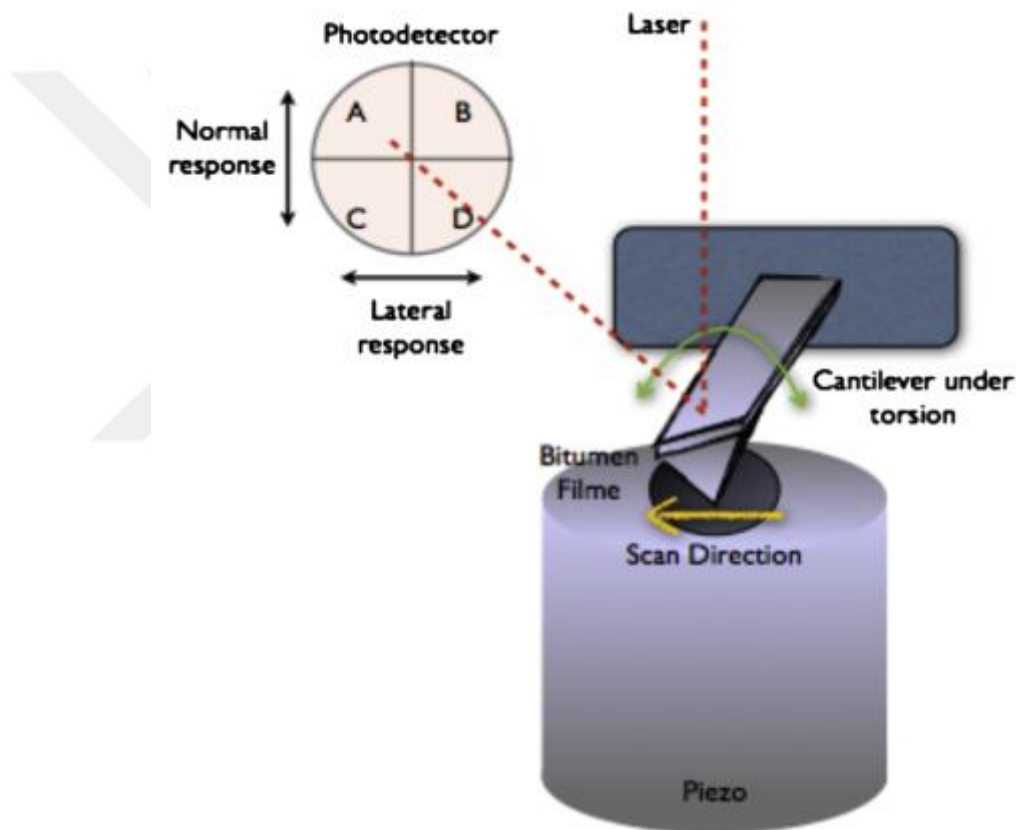


Figure 2.21 Schematics of the atomic force microscopy operated in lateral force mode (LFM) (Rebelo et al., 2014a)

The influence of aging on the asphalt binder is assessed by various researchers. A study concluded that the aging effect leads to the surface stiffness of asphalt binders, however, the variations were dependent on the aging conditions (Zhang et al., 2012). A study showed that before the occurrence of the aging phenomenon, asphalt binder exhibits two different domains; continuous and disperse domain. The micro-

mechanical properties of the domains (stiffness, adhesion, and viscoelastic behavior) were measured by the means of nanoindentation. The properties were utilized to exhibit that the oxidative aging causes the changes in the microstructure of binder in terms of phase dispersion, grouping, and materialization. These changes confirm that the presence of “bee-structures” is the result of the latter (Allen et al., 2012).

By investigating the microstructure and microrheology of asphalt binders before and after aging with RTFOT and PAV with the means of AFM, it was found out that asphalt binder films are primarily composed of asphaltene micelles dispersed in a hydrocarbon matrix. In original binder samples, the micelles structures could not be resolved in the topographic images. For the sample that was aged, the micellar structure was readily detected in both topographic and phase imaging (Rebelo et al., 2014b). Another study was conducted based on the comparison of various aging techniques to monitor the formation of carbonyl groups. The study reported that the aging leads to the variations in the microstructure of asphalt binders mainly associated with aromatics (Lamontagne et al., 2001).

A study covered the investigation of laboratory and field-aged binder samples in which it was inferred that the differences between laboratory and field-aged samples are relatable to the variations in the oxidation compounds produced during the aging process: the occurrence of double bonds and aromatization of molecules are also capable of determining the aged binder behavior. In general, the growth in molecular weight, molecular interaction and the resultant increase in binder stiffness may lead to a state of fragility negatively affecting the long-term performance of asphalt mixture (Aguiar-Moya et al., 2017).

In another study, the influence of thin film oven test (TFOT) and ultraviolet (UV) aging on the morphology of neat and OMMT modified asphalt binder was investigated. The dimension and the amount of ‘bee-like’ structures were decreased after the aging. The reason for this reduction was the increase in solubility of microcrystalline waxes (>C40) and waxy molecules in asphalt binder during aging which may cause the obstruction to their crystallization. In addition, the comparison

between the matrix and the disperse domains got decreased and the single-phase trend occurred in the asphalt binder during both TFOT and UV aging. However, these negative variations can be tackled very effectively with the incorporation of OMMT into a binder by considering the barrier properties of the OMMT layer to heat, oxygen and UV radiations, indicating its better anti-aging performance characteristic. This property of OMMT comes along with the changes in physical properties of neat and OMMT modified asphalt binder before and after TFOT and UV aging (Zhang et al., 2011b).

2.4.2.6 Fourier Transform Infrared Spectroscopy

Aging of asphalt binder is one of the most common causes of failures in the flexible pavement that leads to the formation of carbonyl compounds and sulfoxides, transforming the generic fractions of binder (maltenes) into rather larger molecules (asphaltenes) consequently resulting in relatively higher molecular weight and polydispersity (Lu & Isacsson, 2002). In recent years, researchers and engineers have adopted various approaches for assessment and quantification of the aging effect on the asphalt binders (Singh et al., 2017). Some of these approaches included the Corbett chromatography, Gas or Gel Permeation Chromatography and Fourier Transform Infrared Spectroscopy (FTIR). Amongst these, FTIR is the widely preferred technique to assess the variations in the chemical composition of binders due to oxidative aging (Hofko et al., 2018). Many studies have utilized it to study aging characteristics of asphalt binders (Lu & Isacsson, 1998; Lamontagne et al., 2001; Wu et al., 2009).

FT-IR is basically designed in a manner that it measures the infrared (IR) absorption of the sample material. The FT-IR spectrometer generally consists of an IR source that causes the generation of IR radiation that passes through the sample material causing the bonds in the molecules to vibrate and rotate at discrete frequencies, as a consequence, some of the radiation is absorbed by the sample and the rest passes through it.

The transmitted IR radiation is absorbed by the detector which gives an absorbance spectrum corresponding to the wavelength. This spectrum is different for each given molecular structure by using which the functional groups present in the sample material can be identified. Figure 2.22 exhibits the operational manner of FT-IR spectroscopy (Singh et al., 2017).

FT-IR spectroscopy is able to identify the chemical evolution in bitumen binder and can indicate the severity of oxidation experienced by the sample due to aging. It has come up as a major analytical technique to analyze the aging mechanism in binder sample through the characterization of oxygen-containing functional and hydrocarbon groups. The FT-IR is also capable of yielding rapid qualitative and quantitative results that are highly reproducible and very accurate. The technique can differentiate stretching vibration of carbonyl mode with ease which is up to a large extent dominated by asphaltene compound following the aging process (Toteva et al., 2009).

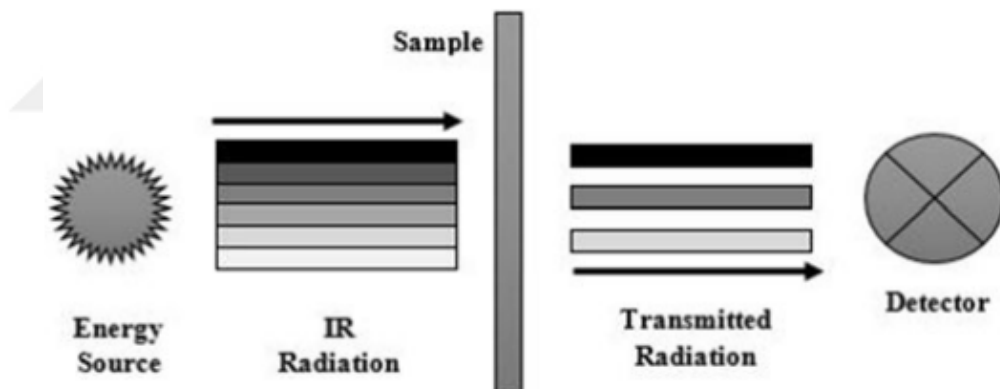


Figure 2.22 Operational manner of FTIR spectroscopy (Singh et al., 2017)

During a study, the FTIR spectroscopy analysis was conducted using an integration method that considers the shaded area below the absorbance spectrum around a maximum band using baseline and tangential methods. A statistical investigation for approving the reproducibility of FTIR spectra analysis based on the accumulated data was carried out. The study concluded that the FTIR technique is the most reproducible than all the other methods in the situation where more than one laboratory is providing the FTIR data (Hofko et al., 2018).

The primary objective of a spectroscopy absorption, as in FTIR technique, is to attain the information on how much a sample under study may absorb the light (i.e. infrared radiation) at a certain range of wavelength (Hofko et al., 2018). As can be seen in Figure 2.23, carbonyl and sulfoxide peaks are usually located at the wavenumbers of 1700 and 1030 cm^{-1} in an FTIR spectrum, respectively (Dony et al., 2016). While in general, for mid-FTIR, the wavelength ranges from 2.5 to 25 μm , corresponding to the wavenumbers from 4000 to 400 cm^{-1} , respectively (Hofko et al., 2018).

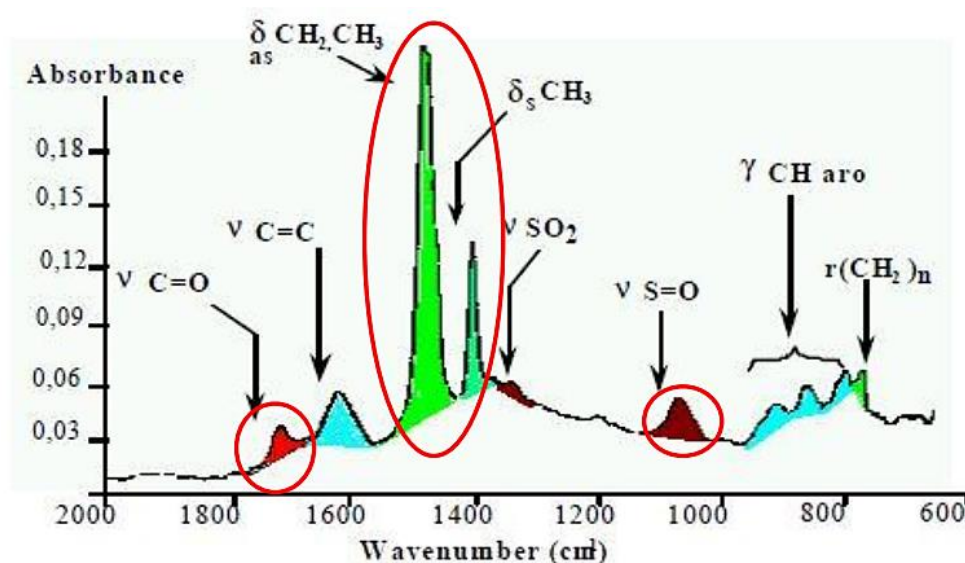


Figure 2.23 FTIR bitumen spectrum – presentation of carbonyl, sulfoxide and reference peaks (circled in red) (Dony et al., 2016)

The main band wavenumbers are shown in Table 2.2 (Larsen et al., 2009). FTIR relies on the fact that the bonds in molecules absorb infrared light at frequencies that are characteristic for their vibrations, i.e. resonant frequencies. The selection rules for IR active bands totally depend on the symmetry of the molecules or molecular groups, therefore, they are related to the material's structure. Various molecular bond groups can be unambiguously distinguished at various, well-defined wavelengths of an absorbance spectrum by characteristic bands (local maxima in the spectrum) (Smith, 2011).

In the case of bituminous binders, especially those structures associated with oxidative aging, i.e. uptake of oxygen, two areas have been the focus of research: A) the carbonyl functional group has been utilized to characterize the severity of bitumen oxidation and a linear relation between the increase of the log of viscosity and the increase in carbonyl concentration during the binder aging process has been established (Petersen & Glaser, 2011). B) The sulfoxide group is mostly produced in higher contents than carbonyl, specifically in bitumen binder containing high sulfur contents. Variations in these chemical groups can be possibly correlated to the variations in the rheological properties and allow for a better understanding of chemo-mechanical coupling (Liu et al., 1998).

Table 2.2 Designation of the main bands of the FT-IR spectra (Larsen et al., 2009)

Wavenumber (cm ⁻¹)	Assignations ^a
3594, 3735	ν O—H
2924, 2853	ν C—H aliphatic
1735	ν C=O
1700	ν C=O conjugated
1600	ν C=C aromatic
1460	δ C—H of $-(CH_2)_n-$ (aliphatic index)
1376	δ C—H of CH_3 (aliphatic branched)
1030	ν S=O sulfoxide
966	δ C—H trans disubstituted $-CH=CH-$ (butadiene block)
748, 690	δ C—H aromatic monosubstituted (styrene block)

^a ν = Stretching, δ = bending

Recently, a research was performed to look into the reproducibility and repeatability of various methods for assessing the ATR FTIR spectra of bituminous binder samples at their different aging states from unaged to RTFOT and PAV aged samples. The findings suggested that the integration-based methods exhibit a repeatability in terms of coefficient of variation (CV) of 3.0 to 5.5% (Hofko et al., 2018).

2.4.2.7 Saturates-Aromatics-Resins-Asphaltenes (SARA) Analysis

A bitumen binder of any crude source is a complex system of various components (Loeber et al., 1998). It is mainly composed of more than thousand types of hydrocarbons having different molecular weights and also contains very small amounts of heterogeneous atoms including oxygen, nitrogen, Sulphur, and some traces of metals. It is nearly impractical to analyze them individually thus the fractionation of the chemical composition of the bitumen binder is suggested very often (Lesueur, 2009; Hermadi et al., 2012). The separation of the complex bitumen chemical composition into generic fractions considering their different solubilities and polarities is a widely adopted approach. This approach is based on the classification of bitumen chemical constituents into SARA (Saturates-Aromatics-Resins-Asphaltenes). Asphaltenes are insoluble in aliphatic solvents but can dissolve in aromatic solvents. Maltenes, on the other hand, are separated with the means of chromatographic column separation – based on polarity – into saturates, aromatics, and resins (Lesueur, 2009; Redelius & Soenen, 2015).

Bitumen is known to be substantially susceptible to the temperature variations causing the alteration in its aging behavior and rheological characteristics. The alterations are commonly attributed to the interactions of the individual bitumen chemical components (Loeber et al., 1998). Aging is a phenomenon experienced in bitumen binder which is arisen due to oxidation and/ or loss of the volatile constituents leading to the changes in the chemical composition and structural variations of bitumen (Mastrofini & Scarsella, 2000).

As a result of aging, the bitumen initially experiences a reduction in its aromatic content followed by an increment in the resin content and also yielding a higher asphaltene content. Hence a wide consensus is present on the aromatic induce resins which in turn generate asphaltenes (Lesueur, 2009). The resulted chemical variations can potentially affect and change the rheological properties as well causing the bitumen to become more brittle and solid-like (Lu & Isacson, 2002). The saturates exhibit lower chemical reactivity which indicates the saturates remaining constant and

unchanged (Read & Whiteoak, 2003; Wright, 1965). This entire variation occurring lead to a slightly higher but almost unaltered glass transition temperature. Asphaltenes subjected to aging might be slightly different from its initial (Lesueur, 2009).

Corbett in his study reported the increment in molecular weights of the asphaltenes due to air-blowing which suggest the existence of polymerization reactions (Corbett & Swarbrick, 1960). A similar conclusion was also reported in a research by Kats et al. in which the formation of carbenes and carboids from the asphaltenes was confirmed with the application of photooxidation. However, the results were true for one of the two types of bitumen binders used in their study (Kats et al., 1976).

In another study conducted on the assessment of bitumen oxidation, the increment in asphaltenes was correlated to the increase in the molecular weight from 5600 to 8000 g/mol as a result of blowing and Athabasca bitumen for 120 minutes duration at 260 °C temperature. Based on the achieved results, the molecular weight could only jump up to 7000 g/mol as the temperature was raised to 290 °C and remained constant even when the temperature reached up to 320 °C. Similarly, the ratios of H/C, N/C, and S/C were also computed resulting in their reduction. On the other hand, the O/C ratio further increased. This infers the growth in the grasping amount of oxygen by asphaltenes concurrently with the release of sulfur and nitrogen (Moschopedis & Speight, 1977).

The rate of asphaltenes produced was observed in a study to be linear when correlated with the time in RTFOT laboratory tests at 163°C with 6– 7 wt.% asphaltenes that were produced in 340 minutes (Siddiqui & Ali, 1999). The same linear increment was also seen for the in-situ aging of bitumen obtained from field pavement samples, with a growth from 2 to 10 wt.% in a 90 months duration from a site under French southern climate (located near Marseilles) (Lesueur, 2009).

The aging specifically caused the production of sulfoxides and regardless of its slower rate ketones were also formed that eventually yields anhydrides and carboxylic acids. Sulfoxides are formed quickly however they are not very promising in terms of

its thermal stability. Consequently, they reach a relatively steady state which is attributed to the initial Sulphur content of the bitumen and also the oxygen dispersion into the bitumen (Petersen, 1993).

In another research work performed on investigating the aging behavior of bitumen and its relationship with the SARA constituents exhibited that the aged bitumen samples were converted into a highly oxygenated and highly saturated compound whereas the original bitumen samples showed an abundant degree of unsaturation of the compounds. The condensed aromatic constituent of lower polarity appeared to be degraded in mass due to the application of oxygenation procedures (Handle et al., 2017).

In a different study, an attempt was made to cover the investigation of the influence of the adopted approach and the impact of oxidation on the molecular weight of bitumen, the comprehensive ultimate analysis, and the chemical structure of bitumen. The study concluded that asphaltenes from the continuous oxidation bitumen yield greater molecular weights, densities, aromatic contents, and also a higher number of rings in their molecule composition when compared to those achieved from the batch-wise oxidation bitumen.

The results from the study also suggested that the densities of maltenes raise with the degree of oxidation. Relatively lower molecular weights and C/H ratios were attained for maltenes in the continuous oxidation as compared to the batch-wise oxidation bitumen. Various oxidation conditions cause the different degrees of molecule conversions leading to the production of bitumen having different qualities. As a consequence of the more selective conversions of asphaltenes in the continuous oxidation bitumen, the latter exhibit lower temperature susceptibility (Glozman & Akhmetova, 1970).

2.5 Asphalt Mixture Design

The asphalt mixture is a paving material that comprises the bitumen binder, mineral aggregate, and air voids. The unmodified or modified bitumen binder functions as a

binding agent to bond aggregate particles into a cohesive mass. Mineral aggregate acts as a stone framework that receives the applied traffic loads (McGennis et al., 1995). The primary goal of asphalt mix design is to determine a unique bitumen content that is introduced into the aggregate of a specific gradation in order to prepare an economical asphalt mixture finish product that meets the specified mix type specification (Pavement Interactive, 2012f).

The state-of-the-art asphalt mix design technology is actually an evolution of ideas that have been assessed through the years of research. Bitumen film thickness and void in the mineral aggregate have been the two primary parameters for controlling the minimum amount of bitumen binder in a mixture since the early days of asphalt mix design (Huber, 2013b).

The need for establishing a method to design asphalt mixture dates back to the late 19th century when the first mixes were placed. In the 1880s, asphalt mixes for road construction in the U.S. were marketed as patented products made primarily from natural bitumen binder attained from the lake deposited of Trinidad, British West Indies. The binder was transported in barrels and blended with local petroleum fluxes and subsequently mixed with local aggregates using proportions determined based on the trial-and-error performed by the patent holder. At the beginning of the 20th century, the availability of manufactured asphalt started to increase, and the traffic volume also witnessed a gradual growth. Therefore, the public works engineers were obligated to think of developing the specifications to describe the properties of low-cost road mixtures purchased by their organizations (Roberts et al., 2002).

Hubbard-Field method was the first ever formal design method which was originally developed for designing sand-asphalt mixtures and later on it was modified for incorporating aggregates (Asphalt Institute, 1965). This approach had some drawbacks in terms of aggregate particle sizes being larger than about ½ inch if the 4:1 ratio of mold diameter to maximum particle size is to be maintained. Due to the likewise limitations existed in this approach, other new approaches like Hveem and

Marshall methods for designing of asphalt mixtures were developed and came into practice very rapidly (Roberts et al., 2002).

In the year 1927, Francis Hveem was assigned to his first oil mix job as a project engineer for the California Division of Highways. Based on the field experience and practice, the oil content for the oil mixes was determined based on the specific color of asphalt mixture that would contain the proper amount of oil (Stanton, 1927). Since Hveem himself was new to it and had no experience, he decided to measure everything about the mix that he thought might relate to establishing the right oil content (Vallerga & Lovering, 1985).

The negative aspect of the Hveem method is that the testing apparatus, especially the kneading compactor and Hveem stabilometer, are comparatively more expensive than Marshall equipment and also not easily portable. In addition, some important volumetric properties of mixtures related to the durability are not always determined as part of the Hveem design process. Some engineers pose the objection against the determination of bitumen binder content in the Hveem method may result in non-durable asphalt mix with insufficient binder content.

There are other mix design methods namely the Texas gyratory method that is currently practiced by the state DOTs in Texas, Oklahoma, and Colorado. This method retains volumetric design elements of the Marshall method and the stability determination from the Hveem method. The Texas gyratory compactor is a laboratory compactor that is considered to be a better-simulating means of traffic densification. However, it is believed that the operational characteristics of the compactor need to be refined in order to become suitable for a wider variety of design applications (McGennis et al., 1995).

Bruce G. Marshall proposed the Marshall asphalt mixture design method prior to World War II to establish a standard procedure in which the available laboratory equipment is utilized for determining the binder content of asphalt mixtures (Roberts et al., 2002). Marshall design method is actually the outgrowth of the Hubbard-Field

method. Hubbard-Field method utilized two different sized hand-tamp rammers for the compaction of specimens, however, Marshall employed a single rammer dropped from a specific height to compact specimens (Huber, 2013a)

Marshall mix design method is by far the most widely adopted method in most of the agencies worldwide. Some features were added to this method by the U.S. Army Corps of Engineers that resulted in formalizing it as a proper ASTM D 1559 standard. The Marshall method designs an optimal asphalt mixture by means of stability flow and density voids analyses. The plus point of Marshall method is that it facilitates the mix design by ensuring the important volumetric proportions of mix components to be at their proper levels in order to achieve a durable asphalt mixture (McGennis et al., 1995).

Another reason this method is preferred is the employment of the relatively inexpensive equipment and being easily portable that enables the remote quality control operations on the site as well. However, it is still believed that the laboratory compaction impact doesn't simulate the mixture densification occurring under the traffic.

The Marshall stability parameter also cannot adequately predict the shear strength of the asphalt mixture. These two flaws make this method conceptually biased towards addressing the rutting distress. Furthermore, both Marshall and Hveem methods are exhibiting the inability to measure fatigue cracking, low-temperature cracking, asphalt binder aging or mixture resistance to moisture damage (McGennis et al., 1995; Malunga et al., 2014; (Huber, 2013b). The Hveem and Marshall mix design methods served as the primary procedures for the design of asphalt mixtures until the mid-90s when the Superpave method was introduced (Huber, 2013a).

In the year 1987, the Strategic Highway Research Program (SHRP) started to develop the new system as Superpave which stands for Superior Performing Asphalt Pavements. The Superpave system specifies the constituting materials, asphalt mixture analysis and design, and pavement performance prediction. The system also includes

the specifications for test equipment, test methods, and criteria (McGennis et al., 1995). Superpave design procedure is essentially based on four major steps: 1) materials selection; 2) design aggregate gradation and structure; 3) design bitumen binder content; and 4) moisture-susceptibility assessment.

During the mix design, a gyratory compactor is utilized for the compaction of specimens. The effort for the compaction of specimens is specified in terms of the number of gyrations (Roberts et al., 2002). A significant drawback of the Superpave method is that it sets no specific provision for the utilization of reclaimed asphalt pavement (RAP) in the process of asphalt mix design. This has caused an obstacle and discouraged many of the road agencies to adopt the Superpave mix design method when incorporating RAP (McDaniel & Anderson, 2001).

2.5.1 Mix Design Involving RAP

The recycled mix design process adopted for incorporating RAP is quite similar to the designing process of virgin materials. The design process is carried out in a manner that RAP, after characterizing it, is blended at a predetermined content with the virgin aggregate and bitumen binder to produce the recycled mixture (Copeland, 2011).

A few changes have been made in the recycled mix design process since the emergence of hot-mix recycling method as a widely adopted method in the mid-1970s. Despite these changes, the mix design methods developed during the 1970s have facilitated reasonable guidance. The designing of a desirable recycled mix is ensured as a result of maintaining the properties of combined aggregate and blended binder similar to those required for designing conventional HMA and that the volumetric properties of the mixtures remain well within the specification (Newcomb et al., 2007).

The most important step in the designing process of recycled HMA is to determine the properties of RAP aggregate and RAP binder. The basic required properties of RAP materials are the viscosity of the binder, the bitumen content and the particle size distribution (gradation) and specific gravity of aggregate (Izaks et al., 2015).

A stepwise procedure for designing the mixtures involving RAP is presented in NCHRP Report No. 452 which relies on the Superpave method of mix design. However, it could be easily adapted to Marshall and Hveem mix design procedures as well. Some of the small changes in the RAP involving mix design as compared to the conventional mix design are stated as follows (McDaniel & Anderson, 2001):

- ✓ Heating the RAP in a gradual manner and to a lower temperature as compared to the conventional aggregate heating.
- ✓ Estimation of the specific gravity of RAP aggregate.
- ✓ Taking the RAP binder into account during the aggregate batching.
- ✓ Facilitating the reduction of the fresh binder content to account for the RAP binder.
- ✓ Possibly using a lower fresh binder grade to account for the RAP binder aging.

Presently, AASHTO guidelines for incorporating RAP materials also follow the recommendations McDaniel and Anderson (2001). With the increase in RAP content, the specified binder grade is varied. These guidelines were developed under the guidelines of the Superpave™ Mixture Expert Task Group (Newcomb et al., 2007).

A study inferred that the evolution of recycled mixture should be based on a three-tier system (Kandhal & Foo, 1997). The first tier is based on the RAP content lower than 15 percent which can be used without affecting the virgin binder grade that is selected for the project location and the mix design is carried out in its conventional manner. Second tier is based on the mixes involving the RAP content varying from 15 to 25 percent, in which the high and low-temperature grades of the virgin binder are both reduced by one grade to account for the stiffening effect of the aged binder (i.e. a PG 58-28 would be utilized instead of a PG 64-22). The third tier is based on the RAP content above 25%. The blending charts are developed and recommended for mixes involving above 25 percent of RAP in HMA (Cooley & Williams, 2013; Newcomb et al., 2007). These blending charts are intended to determine the percentage of RAP content that can be incorporated into the virgin mix by blending with the virgin binder (McDaniel and Anderson 2001). In the case of using blending charts, it is really

important to extract, recover, and also test the RAP binder by characterizing it (Newcomb et al., 2007).

For the RAP contents higher than 25%, the design and production process would also require additional effort, but the savings would substantially outweigh the added costs with utilizing higher RAP contents. Blending to meet gradation and the suitable binder grade in the final product are keys to executing successful mix design, production, and performance (Newcomb et al., 2007). The most critical parameter considered in the recycled mix design is the aging of RAP binder. In order to compensate for the incorporation of aged (stiffer) binder and also to ensure the adequate pavement performance, the introduction of rejuvenating or softening additives, using of softer virgin binder grade, and also an increase in total mixture binder content have emerged as some of the most prominent methods in the asphalt recycling industry (Izaks et al., 2015).

A number of researchers support the idea that HMA mix design with low RAP contents (up to 15%) is not considerably affected by variation in RAP (Bukowski, 1997; Huang et al., 2004a; Shah et al., 2007); however, higher RAP percentages can potentially change the overall performance of the HMA mixture. A study indicated that the use of a higher content of RAP did not affect the densities as much as it affected the bitumen binder content of the plant mix. Within some projects, higher variation in the binder content of the RAP material also had higher variation in binder content of the plant mix. Likewise, projects with higher variations in stiffness of RAP binder also exhibited higher variation in stiffness of the plant mix binder (Solaimanian & Savory, 1996). In addition, various studies conducted on RAP have inferred that the incorporation of RAP in asphalt mixes changes the physical behavior of the mix. The increment in RAP binder stiffness is believed to be the cause of increased modulus of asphalt mixes. Similarly, RAP also influences the fatigue behavior and low-temperature cracking of the mixes (McDaniel et al., 2000; Shu et al., 2008).

Another crucial parameter considered in the design of mixtures involving RAP is the variation in the voids of mineral aggregate (VMA). A study indicated that in one

source of material, the VMA decreased with increasing the RAP content, while for another source of material, the VMA increased with increasing the RAP content (Al-Qadi et al., 2009). In a different study, the same findings were obtained by demonstrating the similar results, that is, a decrease in optimum binder content and VMA with an increase in RAP content (West et al., 2009; Kim et al., 2009). The Illinois Department of Transportation (IDOT) identified, quantified, and correctly adjusted the assumed aggregate (dry) bulk specific gravity (G_{sb}) for RAP in the early 2000s. This result eliminated the use of effective specific gravity (G_{se}), which was leading to lower than desired optimum binder contents (Al-Qadi et al., 2012).

2.5.2 Indirect Tensile Test (IDT)

IDT can be used to define the creep compliances and the ITS of asphalt mixtures at low and normal temperatures. The ITS results can give us worthy evaluation keys in low temperature and fatigue cracking of asphalt pavements. Some studies introduce ITS result as a good indicator in predicting laboratory rutting potential of asphalt mixtures (Anderson, Christensen, & Bonaquist, 2003). This test is widely used in the investigation of moisture induced damages of bituminous mixtures. AASHTO has associated a standard for assessment of moisture susceptibility of asphalt mixtures using ITS test results.

When evaluating the aging characteristics, as the bitumen ages, it becomes more brittle and stiffer, thus the ITS results of an aged mixture are rather more than the results of an un-aged mixture. This can provide us aging indices to investigate aging characterization of asphalt mixtures. Sengoz (2003) has implemented ITS results of mixtures with various air voids, to assess aging and moisture susceptibility characteristics of HMA mixtures. Another study on short- and long-term aging behavior of rubber modified asphalts conducted by Liang and Lee (1996) has also proved the fact that the short-term and long-term aging increased the measured tensile strengths. Sengoz and Topal (2008) investigated the effect of SBS polymer modified bitumen on the aging properties of asphalt mixtures using ITS test results. They calculated aging indices as the ratio of short and the long-term aged specimen's ITS

values to the values of un-aged control specimens prepared with the same additive content. Hurley and Prowell (2005) used ITS results to check the rutting potential after short- and long-term aging characterization of warm mix asphalt specimens.

Zaumnis et al. (2014) used IDT to investigate the Influence of six rejuvenators on the performance properties of Reclaimed Asphalt Pavement (RAP) binder and 100% recycled asphalt mixtures. Through IDT and multiple testing, the authors reported that the rejuvenators improve mixture cracking resistance (Zaumnis et al., 2014a).

In another study, mixtures involving RAP were rejuvenated with rejuvenating agents and a softer binder. The volumetric analysis, Indirect Tensile Strength (ITS) of specimens as well as the rutting properties were investigated using an Asphalt Pavement Analyzer (APA). The authors indicated that the ITS values of specimens containing rejuvenators were better than those specimens in which no rejuvenator was included (Shen et al., 2007).

Gracia et al. (2011) used IDT in their study to investigate the utilization of capsules containing rejuvenators in asphalt concrete. The main idea was the brittleness of aged asphalt at low temperatures as proven by IDT testing and breaking up the capsules through cracking of asphalt concrete which would lead the rejuvenator released. The released rejuvenators would help asphalt regaining its original properties.

2.6 Reclaimed Asphalt Pavement

Recycled or reclaimed asphalt pavement (RAP) is referred to the removed and/or recycled hot mix asphalt material that contains both aged bitumen binder and aggregates. In the last few decades, the usage of RAP in various road related applications has become very common. RAP is basically obtained as a result of milling the surface of asphalt pavements which is done for the purpose of reconstruction, resurfacing, or to access the underground utilities. These materials are subsequently reprocessed in special plants (modified plants prepared for accommodating the RAP) for the future usages in the shape of its incorporation into virgin asphalt mixtures.

When RAP is properly crushed and screened, it consists of high-quality, well-graded aggregates coated by bitumen binder (Chesner et al., 1998).

The earliest application of RAP dates back to 1915 (Kandhal & Mallick, 1997). However, the proper RAP introduction to the asphalt industry initiated in the mid-1970s due to the substantial increase in the costs of bitumen binder as a result of the oil embargo (Zhou et al., 2013).

Recycling of asphalt mixture is proved to be an essential technique from technical, economic, and environmental aspects (Kennedy et al., 1998). The preference given to RAP materials over virgin mixture is due to the increase in the asphalt cost, the scarcity of quality aggregates, and the need of taking solid steps towards environmental preservation. Using old and aged bitumen binder in the newly blended mixtures and thus decreasing the required new bitumen content makes the use of RAP in HMA economically attractive (Huang et al., 2004b). A number of state agencies have also supported the fact that the introduction of RAP can result in substantial savings (Page & Murphy, 1987). By taking material and construction costs into consideration, reclaimed HMA pavement was estimated to ensure a saving ranging from 14 to 34% with a RAP content varying in between 20 to 50% (Kandhal & Mallick, 1998). The most economical use of RAP is idealized as its application in the intermediate and wearing layers of flexible pavements since relatively lesser expensive RAP binder could potentially replace a portion of the more expensive virgin binder (Copeland, 2011).

The percentage of RAP (typically expressed by total mass of the asphalt mixture) that is introduced into asphalt mixtures generally depends on the process of production (i.e. plant type, mixture production temperature, duration of mixing, and the discharge temperature), paving technology, the allowed emission proportion, and the source of RAP as well as its inherent properties. The source of RAP can vary as it might be obtained from the road strips that carry heavy traffic (highways or freeways). The RAP materials from these strips are more likely to contain polymer modified bitumen (PMB) that prevents the binder oxidation posing a positive influence over the RAP-

blended asphalt mixture (Dondi et al., 2016). These elements emerge as crucial factors that affect the interaction between RAP and virgin binders that exert significant influence over the performance of asphalt mixtures (Mogawer et al., 2012).

The researchers and highway agencies are forced to find the ways for incorporating RAP in rather higher contents into HMA to deal with the environmental and economic challenges. However, the increase in stiffness of the mix is considered to be the main barrier to achieving the goal of a higher RAP content application (Hussain & Yanzun, 2013). Since blending RAP with virgin materials eventually make the designed mixes stiffer and thus become vulnerable to cracking subsequently leading to durability-induced problems, that is considered as the main concern on RAP mixes. It is really crucial to address the premature cracking distresses in order to comprehensively use these recycled materials (Zhou et al., 2013).

The maintenance of the asphalt quality beyond a certain percentage of RAP, typically above 70%, in the asphalt mixture, has emerged as a serious challenge for the road agencies. Some types of additives, termed as the rejuvenating agents, are employed to rejuvenate the bitumen characteristics in the RAP. These agents are intended to somehow restore the original characteristics of binder ensuring the incorporation of the high content of RAP in the asphalt mixture (Qiu et al., 2013).

During the production operation of RAP asphalt mixture in hot in-plant recycling, most conventional drum plants can accommodate almost 50% RAP, while the percentage of RAP in batch plant ranges from 10 to 30% (Kandhal & Mallick, 1997). With the advancements in the production technologies, the supplying of up to 100% recycled HMA is also made possible (Zaumanis et al., 2014b).

The introduction of RAP into asphalt mixtures also helps in decreasing the amount of waste generated and resolve the problem of disposing of the highway construction materials, especially in large urban cities. Based on a survey conducted in the year 1996, it was reported that about 33% of all asphalt pavements across the United States were recycled and transformed into HMA (Sullivan, 1996). In the year 2001, the

Illinois Department of Transportation utilized about 623,000 tons of RAP in the construction of highways and showed strong willing to increase in its amount in the near future (Griffiths & Krstulovich, 2002). After passing over 30 years from the initial trial in Nevada and Texas states, it was approved that the utilization of RAP turned out to be the best choice and also would be the first priority to ensure the economic competitiveness of asphalt pavement construction (Al-Qadi et al., 2007). RAP incorporation has also proved to enhance the fatigue, rutting, thermal resistance and durability when the pavements are constructed with quality recycled asphalt (Al-Qadi et al., 2007; Asphalt Institute, 2003).

The level of interaction between aged and virgin bitumen binders is the main factor that is still not clearly explained. If RAP starts acting like a black rock, the aged and virgin binder would not interact. It is therefore assumed that RAP does not substantially affect the virgin binder properties. During the designing process, it is generally assumed that RAP doesn't act as a black rock and that the aged asphalt blends with the virgin binder during the mixing process (Al-Qadi et al., 2007).

2.6.1 Advantages and Disadvantages of Recycling

Asphalt pavement recycling has emerged as the most widely practiced approach worldwide mostly associated with many benefits. The pros and cons of asphalt recycling are discussed in the following sections.

2.6.1.1 Advantages of Recycling

The followings are some of the benefits of recycling the asphalt pavements (Koch et al., 2011; Aravind & Das, 2006):

- lower construction costs,
- Conservation of aggregate and bitumen binder,
- preservation of existing pavement geometrics,
- being environmentally friendly by reducing the emission, and
- conservation of energy.

The recycling of pavement is a rational and practical approach in order to meet the demand of construction materials, in the sense that the same materials used to construct the original road system can be potentially re-used to repair, reconstruct and maintain them (Harder, 2014). It is also claimed that the recycled mix has the potential to exhibit higher resistance to shearing and scuffing, which in turn improves the resistance to rutting (Betenson, 1979). The reflective cracking occurrence possibility is also very low with the usage of the recycled mix (Aravind & Das, 2006).

In the U.S., approximately 80 million tons of HMA are recycled each year. This amount of recycling proves the environmental friendliness of HMA when recycled (Alabama Asphalt Pavement Association, 2018). The comparison of asphalt pavement recycling with other products is exhibited in Figure 2.24. If the pavement is properly constructed with recycled materials, the consensus is present on the fact that the recycled pavement would perform as better as the pavement built with virgin asphalt (Santucci, 2007).

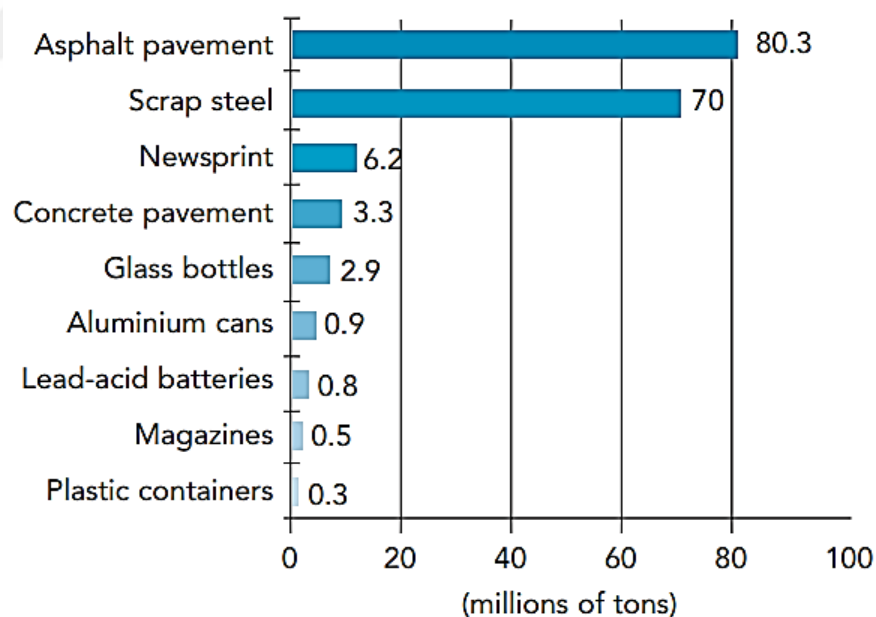


Figure 2.24 Comparison of various recycling products (Alabama Asphalt Pavement Association, 2018)

Currently, the recycling of asphalt pavements is the widespread single recycling approach practiced in the United States and most other parts of the world. In 2002, over 30 million tons of RAP were utilized in HMA with savings exceeding \$300

million, accomplished by lowering materials costs for the newly constructed asphalt and thus eliminating the disposal cost of the RAP (Putman et al., 2005).

Asphalt recycling facilitates percolation thus it is considered as the best choice for the places that always experience a lot of rain or snowfall throughout the year. Since recycled asphalt holds up well in wet seasons and winter season. It, therefore, allows water to pass through, making it easy to block the flooding and pooling in the driveway (Estates Paving, n.d.).

2.6.1.2 Disadvantages of Recycling

There could be hardly any drawback associated with the recycling of asphalt pavement. However, a couple of defects like the need for extra heavy machines and equipment for recycling operations, the color and also the quality of the roadway paved with recycled asphalt are rated as the undesirable characteristics of recycling (Estates Paving, n.d.).

From the color aspect, recycled asphalt is not as deep in color as the conventional virgin asphalt. Some locals dislike this shortcoming of recycled asphalt pavements and mostly prefer fresh, deep black non-recycled asphalt roadways in that sense. In addition, some recycled asphalt pavements may not have as good quality as other recycled asphalt or the traditional virgin asphalt. This is due to the fact that the recycled asphalt would highly depend on its recycling method and the source from where it is recycled (Estates Paving, n.d.).

2.6.2 Asphalt Recycling Methods

Recycling is defined as the reuse of a material generally associated with some processing that has already served its initial intended objective. In the context of asphalt pavements, the material obtained from the deteriorated bounded layers of pavements, known as the reclaimed asphalt pavement (RAP), is either partially or fully reused by incorporating it into virgin asphalt for fresh construction (Aravind & Das, 2006).

Since the 1970s Oil Embargo, the HMA industry has started recycling on a very large scale. The plants were modified to accommodate the RAP and also a vast research on mix properties was initiated in response to the lacking of the asphalt at that time. The practicing of asphalt recycling was transformed into a commonplace approach in most parts of the world (Newcomb, 2006).

Asphalt materials can be recycled by adopting the cold, warm or hot production approaches. In addition, the blending of the new binder, asphalt mixture, and mineral aggregate with the old asphalt (RAP) is performed either in the plant or in on the project site (Jacobson, 2002). Some researcher claim that there is the possibility of recycling 100% RAP (Hansen & Newcomb, 2011) from the pavements by practicing advanced techniques like hot recycling in the asphalt plant, hot in-place recycling, cold in-place recycling and full depth reclamation methods (Zaumanis & Mallick, 2015).

In-place recycling ensures the reduction in the use of new materials. This recycling method also decreases the emissions, traffic, and energy associated with the transport and production of these materials (Santucci, 2007). These techniques have pretty much evolved over the past four decades (Recycling & Reclaiming Association, 2001).

The selection of a specific approach for the purpose of recycling depends on the existing pavement condition, existing pavement material types and their sizes, recycled pavement structural requirement as well as the availability of recycling additives; rejuvenators.

The Asphalt Recycling and Reclaiming Association (Recycling & Reclaiming Association, 1992) defined some of the most common recycling methods practiced in the asphalt industry such as hot in-place recycling, cold in-place recycling, hot central plant recycling and full-depth reclamation methods (Kandhal & Mallick, 1997). Each of these methods is briefly explained in the following section.

2.6.2.1 Hot Mix Asphalt Recycling Method

This recycling method is the most commonly practiced method for the recycling of asphalt pavements. In the process of this method, reclaimed asphalt pavement materials (RAP) are mixed with virgin aggregates, new bitumen binder, and or along with a recycling additive (e.g. rejuvenating agents) in order to produce new recycled HMA. Two types of hot mix plants namely batch and drum plants are employed for this purpose. RAP can be obtained by milling or ripping and crushing operations performed over old asphalt pavement layers. The rest of the recycled mix laying and rolling process as similar to the regular HMA procedure.

RAP contents suggested in the guidelines for Superpave mix design are as followings (Santucci, 2007; McDaniel & Shah, 2003):

- 15% RAP content or less: PG binder is the same as that used in a virgin mix.
- 15-25% RAP: PG binder should be one grade lower on both high and low-temperature end, i.e. PG 64-16 rather than PG 70-10.
- >25% RAP: Test and blend the recovered asphalt from RAP with virgin asphalt as part of the design process to determine the amount of RAP to use.

2.6.2.2 Hot-in-Place Recycling Method

In the hot-in-place recycling method, initially, the pavement under operation is heated to a higher temperature using a proper heating arrangement to ease the removal process of the surface materials. After the heat application, the pavement surface is further cut out to the desired depth. Subsequently, the virgin aggregate and binder are added as per requirement followed by thorough mixing and also compacting the surface until the optimum layer thickness and mix density is achieved (Aravind & Das, 2006).

Since the process of this method takes a relatively lesser time, shorter disruption for traffic is caused. Furthermore, the transportation cost is less by avoiding the hauling and taking away of materials from the site. Relatively wider corridor width; right-of-way, should be considered to accommodate heavy and bulky machinery that operates for executing this operation. This need, however, creates problematic situations for in-place recycling operations especially within the urban areas (Jones, 1979).

2.6.2.3 Cold-in-Place Recycling Method

This method recycles on site and usually utilizes 100% RAP produced from the existing pavement (Santucci, 2007). In this recycling process, initially, the pavement is scarified to a predetermined depth with the mean of a scarifier. The obtained material is then crushed to the required gradation and the required amount of virgin aggregate and bitumen binder is added in the cold form (either emulsion or cutback).

Generally, the treatment depths are 2 to 4 inches when the conventional type of emulsified asphalt or recycling agents are used. It is then compacted and left for aeration. During this process, some additive types namely cement, quicklime, fly ash may also be added to enhance the early strength and moisture resistance of the recycled mix. In this case, the treatment depths can be up to 5 to 6 inches (Santucci, 2007; Aravind & Das, 2006). The cold mix recycling process takes some parameters like local geometric correction, correction of pavement distresses; surface cracks into consideration (Mosey & Defoe, 1979).

This method has considerable advantages, for instance, having low hauling cost of materials due to be an in-situ process. Besides, the amount of emission is almost negligible when compared to hot mix process (Aravind & Das, 2006).

During the process of recycling with this method, the machinery employed is bulky and would thus require adequate space for free maneuvering to operate during the recycling. In addition, the lane underwork would be closed to traffic for a relatively long duration in order to make sure that the newly laid layer is sufficiently cured. The

moisture content is really important when the emulsified bitumen binder is used since it would influence the gradation, mixing, and workability of recycled mix significantly (Mosey & Defoe, 1979).

2.6.2.4 Full Depth Reclamation/ Recycling Method

In this method of recycling a predetermined amount of the underlying base or subbase materials are blended with the entire thickness of the old asphalt pavement to produce an upgraded, uniform base material. Depending on the thickness of the existing pavement course, the treatment depths generally vary from 4 to 12 inches. It is basically a cold mix recycling process in which various types of additives namely asphalt emulsions and chemical agents such like calcium chloride, Portland cement, fly ash and lime are added to obtain a well-produced base layer (Santucci, 2007; Aravind & Das, 2006).

Full depth recycling/ reclamation is performed with the means of reclaiming bulky machines. These reclaimers are basically gigantic rototillers that are equipped with a pulverizing/ mixing drum rotating in an “up-cut” direction. The duration needed for curing the stabilized base material totally depends on the type of stabilizing additive used. The reclaimed and then compacted base layer is usually overlaid with HMA or some type of surface treatment right after it is cured (Santucci, 2007).

2.7 Recycling Agents (Rejuvenators)

Rejuvenating agents are special types of products intended to reverse the aged (oxidized) asphalt binder to relatively less-aged condition (TM, 1988). These agents generally contain a high concentration of maltene fraction namely lube extracts and extender oils, that are capable of restoring the maltenes/ asphaltenes balance that was unbalanced due to the aging phenomenon experienced either in the field or in the plant. This restoration leads to the improvement of the recycle bitumen binder in terms of the relaxation, ductile, cohesive and adhesive properties. Rejuvenating agents were first used as a pavement preservation treatment in the 1960s in order to ensure the

restoration of both chemical and physical properties of aged (recycled) asphalt binders in the field (Willis & Tran, 2015).

Numerous types of rejuvenating agent products are available in the market produced by different manufacturers for the incorporation into asphalt pavements to rejuvenate them and was developed as well as marketed as proprietary products (Shoenberger, 2003). The objective of using a specific type of rejuvenating agent (or recycling agent) is to reverse the stiffened (aged) asphalt binder closer to its original viscoelastic condition. The viscosity, flexibility, and stiffness of the asphalt binder can be enhanced with the penetration and diffusion of the rejuvenating agent in the surface of reclaimed asphalt pavement (Willis & Tran, 2015).

The rejuvenation agent works in a manner that it forms a layer the surrounds the binder-coated aggregate particle and starts to disperse into the outer layer. The rejuvenating agent itself begins penetrating into the aged asphalt binder layer subsequently a proper dispersion is ensured. As a result, the inner layer's viscosity decreases on the contrary to this, the outer layer's viscosity increases further up to the state when the balancing equilibrium is yielded (King et al., 2016).

The diffusion of rejuvenating agents in reclaimed asphalt occurs in a complicated mechanism and the diffusions patterns are not the same as those in liquids or in solids. The diffusion of rejuvenating agents in reclaimed asphalt is still a challenge for the agencies to accurately understand, predict and manage them. It is significantly relying on the content and swelling degree of asphaltenes. However, the existing theories and physical models of diffusion could be the ideal means to figure out the diffusion of rejuvenating agents in old asphalt systems (Cong et al., 2016).

A number of studies were devoted to binder rejuvenation which is a considered to be a sustainable and at the same time economic approach implemented for extending the service life of RAP surfacing layers. The application of rejuvenating agents in asphalt recycling is recommended by many for the purpose of enhancing the properties fundamental to the long-term performance of RAP, thereby enabling the higher

proportions of RAP to be practically used (King et al., 2016). In the recent time, studies have focused on incorporating the rejuvenating agents in specific concentrations into RAP and RAS for facilitating a comparatively high recycled binder (Willis & Tran, 2015).

The aging property and hardness degree (measured in terms of penetration and viscosity) of the asphalt binder residue to be recycled is considered as the main parameter when selecting a specific type of rejuvenating agent. The older the binder residual is, less consistent would be the rejuvenating agent type. In the recent time, proprietary recycling agents prepared from bio-based oils are widely preferred and available in the markets as rejuvenating agents for combining with asphalt mixtures containing higher proportions of RAP and RAS. A specific chemical composition of bio-based oils is not published yet since it is expected to find out more bio-based products and get supplied to the market in the near future. Table 2.3 summarizes a categorized, partial list of rejuvenating agents currently under usage in the asphalt industry (Willis & Tran, 2015).

Table 2.3 Types of rejuvenating agents used in the asphalt industry (Willis & Tran, 2015)

Category	Product Names	Description
Paraffinic Oils	Waste Engine Oil (WEO) Waste Engine Oil Bottoms (WEOB) Valero VP 165® Strobit®	Refined used lubricating oils
Aromatic Extracts	Hydolene® Reclamite® Cyclogen L® ValAro 130A®	Refined crude oil products with polar aromatic oil components
Naphthenic Oils	SonneWarmix RJ™ Ergon HyPrene®	Engineered hydrocarbons for asphalt modification
Triglycerides & Fatty Acids	Waste Vegetable Oil Waste Vegetable Grease Brown Grease Delta S*	Derived from vegetable oils *Has other key chemical elements in addition to triglycerides and fatty acids.
Tall Oils	Sylvaroad™ RP1000 Hydrogreen®	Paper industry byproducts Same chemical family as liquid antistripping agents and emulsifiers.

In a study, the influence of oil-based rejuvenating agents on Superpave mixtures involving specific contents of RAP was assessed. The mechanical properties of RAP

containing mixtures treated with Rejuvenating agents got enhanced. The results further indicated that additional RAP content could be accommodated within the Superpave mixtures by introducing the oil-based rejuvenating agents (Shen et al., 2007).

Waste vegetable oil and an aromatic extract were utilized in a study for the rejuvenation of aged asphalt binder. The usage of the agent was intended to improve the rheological properties of the binder. The results declared that the rejuvenating agent used could alter the chemical structure of the treated aged bitumen, as a consequence, the mechanical behavior of mixture also differed.

The researchers investigated the samples under study at both macro and micro scales and concluded that the characterization of rejuvenating influence over the aged binder could gain the advantage of enhanced recycling for asphalt binders. The researchers have evaluated the samples at both macro and micro scales and found out that the characterization of rejuvenating impact on the aged bitumen could gain the advantage of improved recycling for bituminous materials (Yu et al., 2014).

Several rejuvenating product types available in the markets are also coal tar-base agents and are termed as rejuvenators/sealers. These types of products facilitate a complete coating of the material surface thus playing the role of surface sealing. The coating is supposed to impart the qualities generally associate with seal coatings. The coal-tar based rejuvenating agents/ sealers ensures the protection against fuel spillages. Their long-term capability of these agents to remain as fuel spillage resistant would be associated with the traffic intensity and volume to which the pavement surface is subjected since the traffic is the main factor in terms of wearing off the surface material. The other expected distress related to coal-tar based agents would be cracking possibility that occurs very often when coal-tar sealers are used (Shoenberger, 2001).

A study performed recently on a coal-tar based rejuvenating agent/sealer by the Florida State Materials Office (SMO) found the product not applicable for highway applications (Sholar et al., 2000). The product went under study was a proprietary agent termed as SR-20. The assessment of this product by SMO spanned one month

after its laying, as a result, it was found out that the product was not effective and cannot be used as a rejuvenating agent. Despite being a suitable sealer, it was declared unacceptable due to having lower skid resistance values as compared to non-treated pavement (Shoenberger, 2003).

Skid resistance is a key factor in selecting a rejuvenating product when it is considered for the pavement surface. Skid resistance becomes even more crucial for the pavement surfaces that are exposed to a high-speed traffic. In a study, it was found out that the application of the rejuvenating agent to the asphalt pavement surface, especially for the shorter period of time (week or months), caused the reduction in the skid resistance of pavement (Brown & Johnson, 1976).

On a few occasions, the U.S. Military has utilized the rejuvenating agents to pavement surfaces. One of the spraying applications was executed for the U. S. Air Force on the runway and some taxiways of Lajes Field located in the Azores. The rejuvenating agent they sprayed was Reclamite, an emulsion of petroleum oils and resin in water, firstly in the year 1979 and subsequently in 1983. The applied rejuvenating agent was judged very influential, based on the results obtained from an increment in penetration and reduction in viscosity of the residual asphalt binder recovered after treatment (Shoenberger, 2003).

In recent times, another rejuvenating product termed as Bituminous Pavement Rejuvenator (BPR) has been introduced as a superior product. This product was developed by the Koppers Company in 1959. However, BPR is not an asphalt based-product it is rather a coal-tar based product with low viscosity blend of aromatic oils engineered to penetrate the reclaimed asphalt thus rejuvenate and plasticize the asphalt binder residue. The K. A. E Paving Consultants attained the rights of distributing the BPR in the year 1983. A study of four-year span conducted by the U.S Air Force inferred that both Reclamite and BPR can effectively rejuvenate the asphalt binder (Scofield & Wolfe, 1986).

CHAPTER THREE

EXPERIMENTAL

This chapter consists of experimental studies including the properties of materials, study methodology and the tests performed in line with the objective of the study. Generally, the tests conducted within the scope of the study can be divided into two main categories. The first category includes the tests targeting to investigate the properties of binder and the second category consists of the tests and investigation in regard with bituminous mixtures.

Within the scope of binder testing category, virgin bitumen, aged RAP bitumen and bitumen samples modified using the rejuvenating agents are discussed since the mixtures involving various contents of non-rejuvenated RAP and rejuvenated RAP were examined within the mixture tests category to ensure the mixture quality based on Turkish specifications.

3.1 Materials

In this section the information about the properties of material used within this study is presented. The materials used in this study can be summarized generally as the fresh bitumen, the aged bitumen (extracted from reclaimed asphalt pavement), aggregates, Reclaimed Asphalt Pavement (RAP) and rejuvenating agents also called rejuvenators.

3.1.1 Bitumen

The bitumen used in this study was chosen based on penetration grading system in line with Turkish highways specifications. In selection of the penetration grade, the region in which the study took place was taken into consideration. Thus, 50/70 penetration grade bitumen which was provided from TÜPRAŞ Aliğa Oil Terminal of Turkish Petroleum Refinery Corporation was used as the fresh bitumen within the study. This grade of penetration is imperatively used in Aegean region due to the climatic conditions. Conventional test results for virgin bitumen conducted at Dokuz Eylül University laboratory of bituminous materials are given in Table 3.1

Table 3.1 Laboratory test results for virgin bitumen

Test	Specification	Results	Specification limits
Penetration (25 °C; 0.1 mm)	TS EN 1426	63	50–70
Softening point (°C)	TS EN 1427	49.7	46–54
Viscosity at (135 °C)-Pa.s	ASTM D4402	0.425	–
Viscosity at (165 °C)-Pa.s	ASTM D4402	0.1	–
Rolling Thin Film Oven (163 °C)	TS EN 12607-1		
Change of mass (%)		0.05	0.5 (max)
Retained penetration after RTFO (%)	TS EN 1426	74	50 (min)
Softening point rise after RTFO (°C)	TS EN 1427	4.5	7 (max)
Specific gravity	ASTM D70	1.038	–
Flash point (°C)	TS EN 22592	+260	230 (min)

3.1.2 Aged Bitumen

To truly investigate the effects of rejuvenating agents on the properties of aged bitumen, samples of aged bitumen should be provided from a reliable and constant source. The aged samples should have represented the bitumen of an old pavement which was supposed to be rejuvenated by means of rejuvenators. It is essential to supply the aged bitumen from a reliable source in order to simulate the aged RAP binder.

The literature review on this issue suggests the use of artificial aging of bitumen can be a solution. The process to produce artificially aged bitumen or artificially aged asphalt mixture in order to simulate RAP has been a matter of debate in many studies. Some studies suggest that performing RTFOT and PAV tests on virgin bitumen is helpful.

Bowers and his friends have conducted extensive research to determine the most accurate artificial RAP bitumen production method to ideally simulate the genuine RAP binder. After performing FTIR analysis on genuine RAP binder extracted from RAP, the authors discovered that a combination of RTFOT aging followed by two times PAV aging can yield the best outcome (Bowers et al., 2014).

Acquiring homogenous aged bitumen either through extraction or artificial methods is a difficult job which requires special consideration. It is also a time-consuming process. Considering the objective of the study, the best way to obtain aged samples that are supposed to simulate RAP binder is extracting aged bitumen from a reliable RAP source. It is important to make sure that the RAP source has been subject to real field conditions in terms of durability and traffic loads. Not to mention that when it comes to application, any RAP should be separately investigated before putting to use in the field.

Within the scope of this study, extracted bitumen from a reliable RAP source was used. More information related to RAP source is presented in section 3.1.4. Conventional test results for extracted RAP bitumen are presented in Table 3.2

Table 3.2 RAP bitumen conventional test results

Test	Specification	Results
Penetration (25 °C; 0.1 mm)	TS EN 1426	38
Softening point (°C)	TS EN 1427	61
Viscosity at (135 °C)-Pa.s	ASTM D4402	0.538
Viscosity at (165 °C)-Pa.s	ASTM D4402	0.188

3.1.3 Aggregates

Within the scope of this study, limestone aggregates provided from Dere Madencilik Inc. (Quarry located in Belkahve – İzmir) were used in investigations related to mixtures. The physical properties of limestone that was used in preparing the laboratory asphalt mixture specimens are given in Table 3.3.

Limestone can be plentifully found in the region and many state roads have been constructed using mainly limestone as aggregates. A gradation chart was developed to provide the desired performance in conformity with Turkish specifications concerning the Type 1 wearing course. The adopted gradation is given in Table 3.4.

Table 3.3 Limestone physical properties

Test	Specification	Result	Specification limits
Specific Gravity (Coarse Agg.)	ASTM C 127		–
Bulk		2.704	–
SSD		2.717	–
Apparent		2.741	–
Specific Gravity (Fine Agg.)	ASTM C 128		
Bulk		2.691	–
SSD		2.709	–
Apparent		2.739	–
Specific Gravity (Filler)		2.732	–
Los Angeles Abrasion (%)	ASTM C 131	22.6	Max. 30
Flat and Elongated Particles (%)	ASTM D 4791	7.5	Max. 10
Sodium Sulphate Soundness (%)	ASTM C 88	1.47	Max. 10–20
Fine Aggregate Angularity	ASTM C 1252	47.85	Min. 40

Table 3.4 Limestone aggregate gradation

Sieve Size/No.	Gradation (%)	Specification	Specification Limits (%)
19 mm.	100	Type I Wearing Course (Turkish Specification)	100
12.5 mm.	92		83–100
9.5 mm.	73		70–90
No.4	44.2		40–55
No.10	31		25–38
No.40	12		10–20
No.80	8		6–15
No.200	5.3		4–10

3.1.4 Reclaimed Asphalt Pavement

Flexible pavements have a limited service life and have to be renewed when their service life is terminated. At this point, the conventional approach has been that if there is no elevation issue or serious flaking problems, the remaining pavement can act as an underlying course. Thus, a newer course could be constructed over it. Unfortunately, this approach associates serious concerns in terms of sustainability since the older materials are not used in an optimum way.

In recent years, with increasing awareness and developments in sustainable materials, the existing flexible pavement is removed following the end of its service life through reclamation using trimmer machines. These reclaimed materials are generally called reclaimed asphalt pavement or RAP (as an abbreviation) in highway engineering literature. These materials have high values either in terms of sustainability and or economy.

RAP can be used as raw material in the construction of newer roads or can be rejuvenated and used over and over again. The point is that during the service life of a pavement, asphalt binder and aggregates are subject to many physical and also chemical changes which can highly affect the quality of the whole mixture. Thus, the use of RAP requires paying particular attention and other extensive tests to ensure that the target mixture is sufficient in terms of specifications or not.

With regards to specifications, many specifications would not be adequate enough for use of RAP since many of them have not been designed to include the usage of old pavement materials. For this reason, highway authorities in so many countries have skeptical view on high recycling material contents. It is important to characterize RAP materials before putting it to use. In this section, the properties of RAP materials used in the study have been presented.

Reclaimed type I wearing surface course subjected to traffic loads for a period of 12 years was used as RAP material. 16 x 1000 gr. of batch samples were selected randomly using a random separator. The bitumen content and the gradation of RAP materials were determined. As mentioned in the previous section, the bitumen of the aged asphalt was extracted and distilled using a laboratory type extractor and distillatory.

According to the test results, the bitumen content of the RAP was found to be 4.30%. As a final step, the sieve analysis was performed on the extracted aggregates in order to define the target mixture contents in further steps. The gradation table for RAP is presented within the Table 3.5.

Table 3.5 RAP gradation table

Sieve No	Retained (%)	Passing (%)	Spec. Limits (%)
19 mm.	0	100	100
12.5 mm.	1.6	98.4	83–100
9.5 mm.	10.1	89.9	70–90
No.4	45.9	54.1	40–55
No.10	69.8	30.2	25–38
No.40	86.5	13.5	10–20
No.80	91.18	8.82	6–15
No.200	94.17	5.83	4–10

In determining the bitumen content of RAP materials, a centrifuge extractor with 1500 gr. capacity was employed. The centrifuge process was performed on each sample and the bitumen content of each sample was noted separately. The step by step process is presented below:

- As the first step, 1000 grams of re-graded RAP samples were heated in an oven to 150 °C temperature to be separated conveniently into grains.
- The samples were then placed inside the extractor container using a centrifuge paperboard.
- After being cooled at room temperature, the samples were weighted together with the extractor container and the centrifuge paperboard.
- 200 ml. Trichloroethylene as a solvent was poured into the container containing RAP sample letting the RAP material become loose gradually.
- The container was left for an hour in order to allow the RAP material to be solved inside of the solvent.
- The entire assembly was put in the extractor and the centrifuge process was started and continued until the drainage from the extraction tap was over.
- The above steps were repeated till the new solution color became crystal clear.

- The whole container involving paperboard and the RAP sample was then placed inside an oven set for 110 °C in order to allow the remaining solvent to be evaporated.
- Following the cooling down of the container, the whole assembly was weighted again and noted down.
- In order to determine the bitumen content of the RAP sample, the difference between the materials containing bitumen and the materials without bitumen was calculated.

It is very important to prevent filler materials from mixing with the extracted bitumen through being drained inside of the solvent during the centrifuge process. The process was carried out for ten different samples to determine the bitumen content as per test procedure. Eventually, the average of the whole samples was calculated and taken into consideration. The standard deviation of the samples was found out to be rational.

3.1.5 Rejuvenating Agents (Rejuvenators)

As aforementioned, oily based agents are capable of rejuvenating RAP materials. Three kinds of rejuvenators were used in this study. All rejuvenators were selected based on the literature review carried out on the rejuvenating agents. The approach in the selection of oily additives has mainly been the employment of waste oils as asphalt rejuvenators. Two of the agents used were selected from waste oils available in the market and the third rejuvenator was chosen from several commercial products being sold as asphalt rejuvenators.

In Turkey, any process regarding waste oils is subject to Waste Oil Control Regulation (WOCR). According to this law, the objective is to minimize the formation of waste oils in their source. In case that the formation of waste oils is inevitable, reduction and refinement for the recovery of waste oils is the first priority for the government. The collection of waste oils is only allowed for companies which hold a

collecting license from the ministry of environment and urbanization. The collector must allocate collected recoverable waste oils for facilities who hold the license for energy generation from the Ministry of Energy.

Waste oils which are hazardous and unrecoverable must be submitted to disposal facilities licensed by the Ministry as specified in WOCR sub-articles. Thus, any use of waste oils in other industry sectors rather than energy generation is not allowed according to WOCR. The disposal of waste oils through implementing them in the recycling of RAP materials can be submitted to the authorities to be evaluated. Within the scope of this study, only laboratory samples have been supplied from a licensed collector for research purposes. Within the following sub-sections, the rejuvenators used in this study are presented.

3.1.5.1 Waste Engine Oil

The waste engine oil (Figure 3.1), used as a rejuvenator, was procured from an authorized industrial collector. The collector claims that a blend of all waste engine oils was prepared and an adequate sample from this blend was dedicated for research purposes. The physical properties of waste engine oil from collectors Material Safety Data Sheet (MSDS) are presented in Table 3.6.

Table 3.6 Physical properties of waste engine oil (WEO)

Characteristic	Value
Color	Dark Brown
Density (gr/mL)	0.91
Viscosity @ 40°C, mm ² /s (centistokes)	90
Viscosity @ 100°C, mm ² /s (centistokes)	15
Flash Point (°C)	≥200
Boiling Point (°C)	≥280
Pour Point (°C)	≤-25
Freeze Point (°C)	N/A



Figure 3.1 Waste Engine Oil (Personal archive, 2017)

The Chemical properties of WEO gathered from various sources within the literature are given in Table 3.7.

Table 3.7 Chemical properties of waste engine oil (WEO) (Hidayah et al., 2013)

Substance	Symbol	Results (You	Maximum Allowable Level in the U.S.
		et al., 2011)	(You et al., 2011)
Arsenic	Zn	< 1.0 ppm	5 ppm
Ash	-	0.66%	NA
Cadmium	Pb	0.28 ppm	2 ppm
Chromium	Mn	< 4.0 ppm	10 ppm
Lead	Cu	14.0 ppm	100 ppm
Sulfur	S	0.19%	NA
Total Halogen	-	396 ppm	4000 ppm

3.1.5.2 Waste Vegetable Oil

A blend of waste vegetable frying oils (Figure 3.2) was employed in this study the rejuvenating agent. A mix of waste frying oils (approximately 70% Olive oil + 30%

sunflower seed oil) used for only once frying cycle was provided from a local Aegean food restaurant in order to use as a rejuvenating agent for extracted RAP bitumen.



Figure 3.2 Waste Vegetable Oil (Personal archive, 2017)

The physical properties of waste vegetable oil based on the Material Safety Data Sheet (MSDS) are presented in Table 3.8.

Table 3.8 Physical properties of waste vegetable oil (WVO)

Characteristic	Value
Color	Straw Yellow
Density (gr/mL)	0.92
Viscosity @ 40°C, mm ² /s (centistokes)	50
Viscosity @ 100°C, mm ² /s (centistokes)	10
Smoke Point (°C)	≥200
Flash Point (°C)	≥280
Boiling Point (°C)	≥250
Pour Point (°C)	≤10
Freeze Point (°C)	≤5

The chemical composition of waste cooking oils based on the literature regarding the asphalt recycling studies have been gathered and presented in Table 3.9.

Table 3.9 Chemical composition of waste cooking oils based on the literature about asphalt recycling studies (Asli et al., 2012)

Type of Free Fatty Acid	% Waste Cooking Oil
Oleic acid	43.67
Palmitic acid	38.35
Linoleic acid	11.39
Stearic acid	4.33
Myristic acid	1.03
γ - Linolenic acid	0.37
Lauric acid	0.34
Linolenic acid	0.29
Cis-11-Eicosenoic acid	0.16
Heneicosanoic acid	0.08
Total	100

3.1.5.3 Commercial Rejuvenating Agent

A Commercial Rejuvenating Agent (CRA) for RAP, as shown in Figure 3.3, was supplied by a private company which sells asphalt additives was also employed in the study. The results of comparisons with waste oils and commercial products.



Figure 3.3 Commercial Rejuvenating Agent (Personal archive, 2017)

The commercial rejuvenator is also an oily based additive which is sold to local and international asphalt companies for recycling purposes. Based on the information provided by the supplier, the optimum content of the commercial rejuvenator is 7.5% by weight of RAP bitumen.

The physical properties of the commercial rejuvenator according to the Material Safety Data Sheet (MSDS) that were procured from the manufacturing company are presented in Table 3.10.

Table 3.10 Physical properties of the commercial rejuvenator according to MSDS

Characteristic	Value
Color	Amber
Density (gr/mL)	0.9
Viscosity @ 40°C, mm ² /s (centistokes)	50
Viscosity @ 100°C, mm ² /s (centistokes)	15
Smoke Point (°C)	N/A
Flash Point (°C)	≥140
Boiling Point (°C)	≥350
Pour Point (°C)	≤0
Freeze Point (°C)	≤-10

The chemical composition of the commercial rejuvenator based on the information provided by the supplier is given in Table 3.11.

Table 3.11 Chemical composition of the commercial rejuvenator

Compound	EINECS No	CAS No	Content (%)
ME	203-992-5	112-62-9	30-40
FFA (Free Fatty Acid)	204-007-1	112-80-1	10-20
TRG (Triglyceride)	204-534-7	122-32-7	30-45

3.2 Experimental Plan

In this section, the experimental plan which was taken into consideration within the study is discussed. Like Materials section, basically, the experimental plan also can be divided into two main categories. The first category consists the experimental plan which is connected to the tests and procedures followed by within binder testing and the second category include the procedures and approaches in dealing with recycling of asphalt mixtures.

3.2.1 Binder Testing

Basically, to evaluate the rejuvenating of the aged binder, there are steps that should be followed. The first step is to determine the optimum content of rejuvenating agent which is supposed to enhance the properties of aged RAP bitumen. Besides, it is important to develop a blending process which has to be similar to the process which is going to be applied in the plant or field. In order to determine the optimum rejuvenator content, some evaluation tests have to be conducted both on aged RAP bitumen and rejuvenated RAP bitumen together with virgin bitumen.

Comparative study of binder tests results can help to understand the effect of the rejuvenating process on aged RAP bitumen. Thus, the next step is to design an experimental plan which is consisted of appropriate binder tests eligible to exhibit the differences between rejuvenated and non-rejuvenated bitumen samples. Within the study, both conventional and rheological evaluations of bitumen samples have been employed to fully understand the effect of rejuvenating agents on aged RAP bitumen. Additionally, morphological evaluation of binder samples through different microscopy methods was considered to investigate the impact of rejuvenators on aged RAP bitumen and also to understand the effect of aging and rejuvenating on bitumen samples. Finally, chemical evaluation of all samples by means of FTIR analysis could support the idea of the capability of oily based components as rejuvenating agents. Moreover, molecular changes through aging and rejuvenating were evaluated by considering the chemical evaluation of aged, rejuvenated and virgin bitumen samples.

3.2.1.1 Determination of Optimum Rejuvenators Content

The main approach in the determination of optimum rejuvenating agent content is to add an amount of additive in order to enhance the aged bitumen in an ideal way. The resulting rejuvenated bitumen is supposed to not only change into its original condition but also to meet any specification limit. However, it is very challenging to obtain a rejuvenated sample having the same characteristics as virgin bitumen. It is known that in its various aspects, the aging process is an irreversible phenomenon and it is near impossible to fully rejuvenate an aged sample. Since we know that any characteristics of bitumen are changeable by use of proper additives. The literature review tells us that the penetration grade of bitumen is the most used bitumen character which can be employed as a criterion in order to investigate the effect of rejuvenating agents. In this study the optimum rejuvenator content was determined based on target penetration value of 63 which was the penetration grade of base neat bitumen. It should be considered that for different bitumen types regarding aging rate and penetration grade required for region, the source bitumen should be taken into account carefully. The general directorate of highways still appoints the penetration grade in selection of proper bitumen within Turkish specification. In Turkey, if RAP is supposed to be used, the steps which should be followed in design of a flexible pavement consisting RAP include the determination of material properties of RAP and virgin materials, the selection of an appropriate percentage of RAP and virgin aggregate to meet gradation, the selection of appropriate binder content to satisfy viscosity and penetration requirements in terms of workability and to meet the specification requirements in terms of stability, flow, and air voids.. Thus, the penetration should be the first characteristic to be enhanced in implementing a rejuvenating agent.

Many studies have employed the penetration test in the determination of optimum rejuvenating agent (Asli et al., 2012; Servas et al., 1987; Zaumanis et al., 2014b). The optimum content of rejuvenators in the modified RAP bitumen was determined as the target content to obtain a rejuvenated bitumen sample having the same penetration value of virgin bitumen sample. In other words, when the RAP bitumen is modified with this rejuvenating additive content, the acquired sample will have the same

penetration value of virgin bitumen sample. To perform this task, RAP bitumen was modified with various dosages of each rejuvenator. The range was chosen based on literature review and preliminary studies (Jia et al., 2015). The modification was processed for 5 minutes at 140°C using a laboratory blender at normal shear rates (700 rpm) to obtain a homogenous rejuvenated bitumen sample. This modification method has been figured out as adequate in terms of time, temperature and shear rate since longer times and/or higher temperatures and shear rates can cause the volatilization of rejuvenating agents. However, other studies suggest different modification conditions for rejuvenating agents. These modification conditions have been summarized and presented in the Table 3.12.

Table 3.12 Modification conditions for rejuvenating agents discussed in the literature

Author	Type of Oil	Temperature (°C)	Time of Mixing (min)	Mixing speed (rpm)
(Dedene et al., 2011)	WEO	150	NA	NA
(Borhan et al., 2009)	WEO	NA	NA	NA
(A Zamhari et al., 2009)	WEO	NA	60 min / cycle	NA
(Villanueva et al., 2008)	WEO	160	60	500
(Asli et al., 2012)	WVO	130	30	200
(Zargar et al., 2012)	WVO	160	30	200

The literature review also reveals an exponential increase in penetration value of aged bitumen by use of oily based rejuvenators (Asli et al., 2012; Zargar et al., 2012). The same trend was recorded by the tests conducted within the scope of this study. The optimum rejuvenator contents were determined as 5%, 5% and 7.5 % by weight of aged RAP bitumen respectively for WEO, WVO, and CRA. The results will be discussed in Chapter 4. All the 17 types of bitumen binder samples that were tested within the scope of this study are presented in Table 13.

Table 3.13 Summary of the 17 types of binder samples tested within the scope of this study

Tested Samples																
Neat (Control Sample)	RAP Binder (Aged)	WEO					WVO					CRA				
		Rejuvenating Agent Content (%)														
		3	4	5	6	7	3	4	5	6	7	5.5	6.5	7.5	8.5	9.5

3.2.1.2 Conventional Evaluation of Rejuvenated Bitumen

Following the determination of optimum rejuvenator contents, samples were prepared with optimum rejuvenator contents and also with $\pm 1\%$ and $\pm 2\%$ variation from optimum contents by weight of extracted RAP bitumen. In this regard, samples containing 3%, 4%, 5%, 6% and 7% of WEO and WVO by weight of aged RAP bitumen together with samples containing 5.5%, 6.5%, 7.5%, 8.5% and 9.5% of CRA by weight of aged RAP bitumen were prepared and classified as rejuvenated bitumen samples.

All rejuvenated samples and aged RAP bitumen together with virgin bitumen were tested for the conventional bitumen tests (explained in Chapter 2). Conventional bitumen tests included penetration test, softening point tests before and after RTFO together with rotational viscosity test. All test results are presented and discussed in Chapter 4.

3.2.1.3 Rheological Evaluation of Rejuvenated Bitumen

Rheological tests to investigate the behavior of rejuvenated, non-rejuvenated samples and virgin bitumen were conducted using DSR testing. Within the scope of rheological testing, PG tests were conducted on samples containing optimum contents of rejuvenators.

Following the determination of performance grades, temperature and frequency sweep tests were performed on samples containing 3%, 4%, 5%, 6% and 7% of WEO and WVO by weight of aged RAP bitumen as well as on samples containing 5.5%, 6.5%, 7.5%, 8.5% and 9.5% of CRA by weight of aged RAP bitumen as well as on aged RAP bitumen and virgin bitumen. The results are demonstrated in Chapter 4 using graphs and tables. Temperatures on which the tests were conducted are 40°C to 70°C for better representations of region climate and the frequencies are 0.01 Hz representing standing vehicles, 0.1 for heavy traffic in urban conditions (6 km/hr), 1 Hz for state roads and highways and 10 Hz for high speeds at runways and racetracks.

3.2.1.4 Morphological Evaluation of Rejuvenated Bitumen

Within the scope of this study, two different microscopy methods were used to differentiate the rejuvenated and non-rejuvenated bitumen samples. The AFM microscopy was employed to investigate these differences on aged RAP bitumen, neat bitumen and rejuvenated RAP bitumen samples. All tests were conducted at Izmir Katip Çelebi University central laboratory.

Nanosurf ECS 204 electrochemistry stage imaging device, shown in Figure 3.4, with an isostage controller (for active vibration isolation) shown in Figure 3.5 were used. The system uses a software equipped to imaging technology. The whole system is presented in Figure 3.6. Multi 75E-G silicon probes with 75 Hz. resonant frequency and 3 N/m force constant coated with Cr/Pt conductive coating were used through the tapping method. This device allows performing simultaneous AFM imaging and electrochemical measurements on electrodes and various samples.



Figure 3.4 AFM imaging device (Personal archive, 2017)



Figure 3.5 AFM Isostage controller (Personal archive, 2017)



Figure 3.6 AFM imaging mechanism (Personal archive, 2017)

In addition to AFM, SEM was used to investigate the differences between aged RAP bitumen, neat bitumen and rejuvenated RAP bitumen samples. SEM imaging also was conducted at İzmir Katip Çelebi University central laboratory using a Carl Zeiss 300VP SEM device shown in Figure 3.7. By use of this device, samples can be viewed without coating, since the Quorum Q150 RES (Figure 3.8) coating device can also be used to obtain more detailed and clear images on non-conductive samples.

As bitumen is a non-conductive material, gold coating was used for all rejuvenated and non-rejuvenated samples. By these methods, it is possible to perform imaging analysis of the structures in the material micro and Nano dimensions. In addition, qualitative and quantitative elemental analysis can be done in the sample and the distribution of elements can be distinguished by mapping technique. Both dry of wet materials can be analyzed using this device.



Figure 3.7 Carl Zeiss 300VP SEM imaging device (Katip Celebi University Central Lab., 2016)

Morphological evaluations were carried on using two difference microscopy methods. In line with microscopic studies, SEM and AFM tests were employed. Unfortunately, the difference between rejuvenated and non-rejuvenated samples were not clearly identified using SEM method since using AFM could help to clearly differentiate aged bitumen sample with virgin and rejuvenated samples.

The samples containing 3%, 5%, and 7% of WEO and WVO by weight of aged RAP bitumen and the samples containing 5.5%, 7.5% and 9.5% of CRA by weight of aged RAP bitumen together with aged RAP bitumen and virgin bitumen were evaluated through AFM microscopy. The results are presented in Chapter 4 using microscopy images.



Figure 3.8 Quorum Q150 RES coating device (Katip Celebi University Central Lab., 2016)

3.2.1.5 Chemical Evaluation of Rejuvenated Bitumen

As mentioned in literature review, FTIR spectrometer helps to get an idea about the chemical content of different materials. To investigate the differences between non-rejuvenated (aged RAP bitumen) sample, rejuvenated samples and virgin bitumen samples in terms of their chemical compounds, FTIR test was employed. The spectrometric graphs containing different samples spectra are presented in Chapter 4.

3.2.2 Mixture Testing

The objective of mixing testing was to investigate the effectiveness of rejuvenating agents in terms of the amount of RAP used within flexible pavements with least deterioration at the course of time. We all know that the more the amount of RAP in a flexible pavement mix design, the more economical is the mix design. As by use of rejuvenating agents, the final objective is to increase the amount of RAP within flexible pavements, the mixture testing could help us understand how the rejuvenating agents used in this study could make it to use high amount of RAP within wearing courses of flexible pavements.

Within the study, the mixtures containing various amounts of non-rejuvenated RAP and rejuvenated RAP (treated with WEO, WVO and CRA), respectively representing control and rejuvenated specimens were prepared using a Marshall compactor. Following the preparation of the specimens, the mechanical performances of them were evaluated by means of a Marshall Stability and flow test device. Besides, volumetric analysis was performed and taken into consideration in selection of highest RAP contents.

Turkish specification limits were taken into account in determination of highest RAP content for both control and rejuvenated samples. Following the determination of highest RAP contents; the specimens containing highest RAP contents were prepared and tested for Indirect Tensile Strength (ITS) test. Based on ITS values; aging indices were calculated for these specimens and compared to each other.

3.2.2.1 Determination of Optimum RAP Content

Marshall Mix design method was employed to design virgin bituminous mixtures. The optimum bitumen content was determined 4.76 % by weight of aggregates. In order to implement various contents of RAP within the final mixture, the target bitumen amount supposed to be added to the total mixture was calculated separately for different RAP contents. Equation 3.1 was used to determine the amount of virgin binder to be added to the target mixture.

$$Pr = Pc - (Pa * Pp) \quad (3.1)$$

Where;

Pr: Percent of virgin binder to be added in the mix containing RAP

Pa: Percent of RAP binder in the mix

Pc: Percent of total binder in the mix

Pp: Percentage of RAP in the mix

Following the determination of virgin binder amounts to be added into the target mixtures with respect to the values given in Table 3.14, the asphalt concrete samples including various contents of non-rejuvenated and rejuvenated RAP respectively representing control and rejuvenated specimens were prepared taking the mixing and compaction temperatures into consideration.

Table 3.14 Binder contents to be added into the target mixes

RAP Content (%)	Pc (%)	Pa (%)	Pr (%)
10	4.76	4.30	4.33
20			3.9
30			3.47
40			3.04
50			2.61
60	4.76	4.30	2.18
70			1.75
80			1.32
90			0.89
100			0.46

The mixing and compaction temperatures were calculated using equiviscous temperature charts for virgin, RAP and rejuvenated binders plotted according to viscosity values determined at 135 °C and 165 °C. Viscosity values were measured by means of a Brookfield viscometer.

Following the determination of optimum rejuvenator contents; RAP mixtures were rejuvenated and stored. In order to perform the rejuvenation process, 4000 gr batches of RAP were heated to 140°C the same temperature in which binder blending took place. The bitumen content of RAP was calculated and taken into account in rejuvenation process. Within the process, the optimum amount of rejuvenating additive was gradually sprayed into the mixture while mixing was in process inside a laboratory mixer for 5 minutes. The derived rejuvenated RAP had a shining dark brown color compared to non-rejuvenated RAP as seen in Figure 3.9.



Figure 3.9 RAP before and after rejuvenation (Dokandari et al., 2016)

After preparing rejuvenated RAP materials, the mixtures containing various amounts of non-rejuvenated RAP and rejuvenated RAP (treated with WEO, WVO and CRA), respectively representing control and rejuvenated specimens were prepared using a Marshall compactor. Following the preparation of the specimens, the mechanical performances of all specimens were evaluated using a Marshall Stability and flow test device. Besides, volumetric analysis was performed by taking the selection of highest RAP contents into consideration. Turkish specification limits were considered for determining the highest RAP content for both control and rejuvenated samples. The results are argued within the mixture test results presented in Chapter 4.

3.2.2.2 Indirect Tensile Strengths and Aging Evaluation of Mixtures

Following the determination of high possible RAP contents for rejuvenated mixtures, specimens were prepared with this RAP content for both rejuvenated and non-rejuvenated mixtures and tested for Indirect Tensile Strength (ITS). To perform this task, ASTM D6931 -the standard test method for indirect tensile strength of bituminous mixtures was taken into account. The ITS test was conducted by Marshall stability and flow apparatus. The loading rate was set to 51(mm/min) in case for ITS. Figure 3.10 illustrates the true assembly of a specimen between loading strips. To be adequate and unbiased, three specimens for both non-rejuvenated and rejuvenated RAP involving mixtures and also control specimens with no RAP content were prepared and tested.

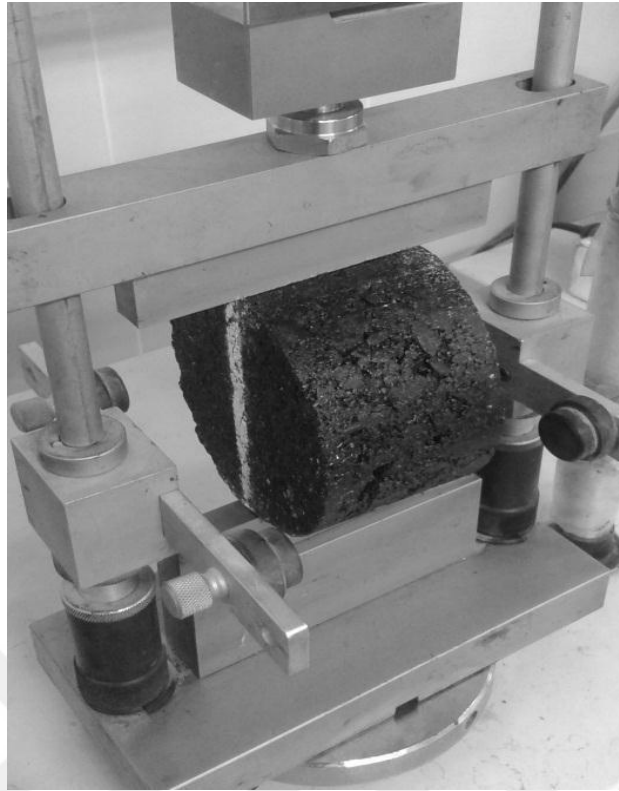


Figure 3.10 Specimen prepared for ITS test (Personal archive, 2012)

The ITS results give the evaluation keys in terms of low temperature and fatigue cracking of asphalt pavements. Some studies introduce ITS result as a good indicator in predicting laboratory rutting potential of asphalt mixtures (Dokandari et al., 2016). This test is widely used in investigation of moisture induced damages of bituminous mixtures.

It is known that, aged binder is more brittle and stiffer. In order to evaluate the aging characteristics of non-rejuvenated and rejuvenated RAP involving mixtures, the ITS results of RAP involving mixtures were compared to the results of control mixtures with no RAP. This comparison simply provides aging indices to investigate the aging characteristics of asphalt mixtures. Şengoz (2003) implemented ITS results of mixtures with various air voids, to assess aging and moisture susceptibility characteristics of HMA mixtures (Sengoz, 2003). Another study on short- and long-term aging behavior of rubber modified asphalts conducted by Liang and Lee (1996) claimed that the short-term and long-term aging increased the measured tensile strengths (Liang & Lee, 1996).

Şengoz and Topal (2008) investigated the effects of SBS polymer modified bitumen on the ageing properties of asphalt mixtures using ITS results (Topal & Sengoz, 2008). They calculated aging indices as the ratio of short- and long-term aged specimen's ITS values to the values of un-aged control specimens prepared with the same additive content. Hurley and Prowell (2005) used ITS results to check the rutting potential after application and the short- and long-term aging characterization of WMA mixtures (Hurley & Prowell, 2005).

The raw data recorded from the test device should be processed using the following equation to obtain the indirect tensile stresses:

$$S_t = \frac{2000 \times P}{\pi \times t \times D} \quad (3.2)$$

Where;

S_t = Indirect tensile strength (ITS), kPa

P = Maximum load, N

t = Specimen height immediately before test, mm

D = Specimen diameter, mm

Following the determination of ITS values, aging indices were calculated for non-rejuvenated and rejuvenated mixtures respectively as the ratio of ITS value of non-rejuvenated mixture over control mixture and the ratio of ITS value of rejuvenated mixture over control mixture. The results were then compared to each other.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

In this chapter, tests results regarding the experimental works which have been conducted within the scope of this study have been presented. The test results are divided into two main categories. The first category which constitutes the most part of the experimental works of the study is dedicated to the test results related to bitumen.

The virgin bitumen, RAP bitumen and other types of rejuvenated bitumen samples were studied from multi-disciplinary points of view including rheology, morphology and chemistry. The second category is consisted of the test results for mixtures including Marshall testing as per Turkish specifications and the ITS test results.

4.1 Bitumen Test Results

As discussed in Chapter 3, different types of binders were studied in this study. As control sample 50/70 Penetration grade was chosen as a reference bitumen grading. Extracted RAP bitumen samples were tested using the same tests conducted on the virgin bitumen. Then the other types of bitumen samples were prepared using extracted RAP bitumen samples and three different types of rejuvenating agents at different doses. The rehabilitated bitumen samples then were tested based on the same methodology to be compared with virgin bitumen.

The obtained bitumen samples were then tested to assure all qualifications indicated in Turkish specifications. By this methodology, it can be said that the newer types of bitumen samples were produced using the rejuvenating agents with qualifications like virgin bitumen. Although the new formulas may lack in some long term qualifications in comparison to virgin bitumen, but it can be attained that these novel types of bitumen samples can be used without major concerns at least in road sections with lower traffic combinations.

4.1.1 Conventional Test Results

The term conventional tests would be confusable to the reader. Thus, before referencing conventional tests, it should be clarified to the reader. The term conventional bitumen test is largely used in pavement industry to address the tests which were mostly used worldwide before the SHRP study. Although these tests are closely linked to the rheology of bitumen, but these tests were mostly used before the development of rheological testing such as DSR and BBR. Thus, the tests include Penetration Test (ASTM D5), Softening Point of Bitumen (ASTM D36), Thin Film Oven Test (ASTM D 1754), also the modified version of TFOT referred as Rolling Thin Film Oven Test (ASTM D 2872), Pressure Aging Vessel test (ASTM D6521), Ductility test (ASTM D 113), Flash Point test (ASTM D 92), and the Rotational Viscosity test (ASTM D 4402).

After SHRP, some of these tests were replaced with recent rheological testing since some such as RTFOT, PAV and RV are still in us. In fact, the RTFOT and PAV both are the tests which are used to evaluate the aging characteristics of bitumen and thus, these tests were continued to be employed at later years. As the study aims to introduce rejuvenating agents to Turkish industry, in addition to recent bitumen testing, the conventional testing is also considered in the study scope. The conventional tests which were employed in this study are Penetration test, Softening Point, RTFOT and RV. All bitumen samples including neat, extracted RAP bitumen and rejuvenated bitumen samples were tested for these conventional tests.

Table 4.1 represents the test results of neat bitumen employed within the study. The penetration value at 25 °C was recorded 63 for the neat bitumen. The softening point was 49.7 °C. The test results for the samples subjected to RTFO testing also are presented within this table. The test results meet the Turkish specification limits in terms of mass change, retained penetration and softening point after RTFO. The same tests were also applied on rejuvenated samples with optimum contents of rejuvenating agents to make sure that these samples also are capable of fulfilling the long term criteria as per Turkish specifications.

The basic conventional test results of extracted RAP bitumen are presented in Table 4.2 and the conventional test results for rejuvenated RAP bitumen are given in Table 4.3.

Table 4.1 Conventional test results of neat bitumen

Test	Specification	Results	Specification limits
Penetration (25 °C; 0.1 mm)	TS EN 1426	63	50–70
Softening point (°C)	TS EN 1427	49.7	46–54
Viscosity at (135 °C), Pa.s	ASTM D4402	0.425	–
Viscosity at (165 °C) Pa.s	ASTM D4402	0.1	–
Rolling Thin Film Oven (163 °C)	TS EN 12607-1		
Change of mass (%)		0.05	0.5 (max)
Retained penetration after RTFO (%)	TS EN 1426	74	50 (min)
Softening point rise after RTFO (°C)	TS EN 1427	4.5	7 (max)
Specific gravity	ASTM D70	1.038	–
Flash point (°C)	TS EN 22592	260	230 (min)

Table 4.2 Conventional test results for extracted RAP bitumen

Test	Specification	Results
Penetration (25 °C; 0.1 mm)	TS EN 1426	38
Softening point (°C)	TS EN 1427	61
Viscosity at (135 °C)-Pa.s	ASTM D4402	0.538
Viscosity at (165 °C)-Pa.s	ASTM D4402	0.188

As presented in Table 4.2, the results show that the penetration value of extracted RAP bitumen are rather lower than the penetration value of neat bitumen. As result of aging, the softening point is approximately 12 unit higher than the value recorded for neat bitumen. The viscosity values have also been recorded higher than the results for neat bitumen because of being naturally aged. In later stages, it was tried to restore all these values by use of rejuvenating agents and the results were surprising.

Table 4.3 Conventional test results for rejuvenated RAP bitumen

Test	Specification	WEO Content by Weight of Extracted RAP Bitumen						Specification limits
		0	3	4	5	6	7	
Penetration (25 °C; 0.1 mm)	TS EN 1426	38	49.0	53.5	59.5	68.0	77.4	50–70
Softening point (°C)	TS EN 1427	61	57.0	55.0	53.5	52.0	51.0	46–54
Viscosity @ 135 °C (Pa.s)	ASTM D4402	0.538	0.5	0.45	0.412	0.363	0.3	–
Viscosity @ 165 °C (Pa.s)	ASTM D4402	0.188	0.163	0.125	0.087	0.038	0.025	–
Test	Specification	WVO Content by Weight of Extracted RAP Bitumen						Specification limits
		0	3	4	5	6	7	
Penetration (25 °C; 0.1 mm)	TS EN 1426	38	50.3	55.1	62.0	69.7	78.8	50–70
Softening point (°C)	TS EN 1427	61	56.5	53.5	51.5	50.0	48.5	46–54
Viscosity @ 135 °C (Pa.s)	ASTM D4402	0.538	0.5	0.438	0.4	0.35	0.275	–
Viscosity @ 165 °C (Pa.s)	ASTM D4402	0.188	0.15	0.125	0.075	0.038	0.025	–
Test	Specification	CRA Content by Weight of Extracted RAP Bitumen						Specification limits
		0	5.5	6.5	7.5	8.5	9.5	
Penetration (25 °C; 0.1 mm)	TS EN 1426	38	56.1	61.2	67.0	73.7	81.0	50–70
Softening point (°C)	TS EN 1427	61	55.5	53.0	51.5	50.0	48.5	46–54
Viscosity @ 135 °C (Pa.s)	ASTM D4402	0.538	0.488	0.425	0.363	0.3	0.25	–
Viscosity @ 165 °C (Pa.s)	ASTM D4402	0.188	0.138	0.112	0.063	0.025	0.013	–

Each rejuvenated sample was tested for conventional test results and followingly was controlled to make sure if it meets the criteria indicated within the Turkish specification limits. As shown in Table 4.3, the results show that by choosing a proper rejuvenating agent content, it is possible to produce a new bitumen sample which acts like a fresh binder in terms of conventional test results.

The comparisons of test results were made by plotting the results of each conventional test for all samples.

The penetration values for all samples are plotted and shown in Figure 4.1.

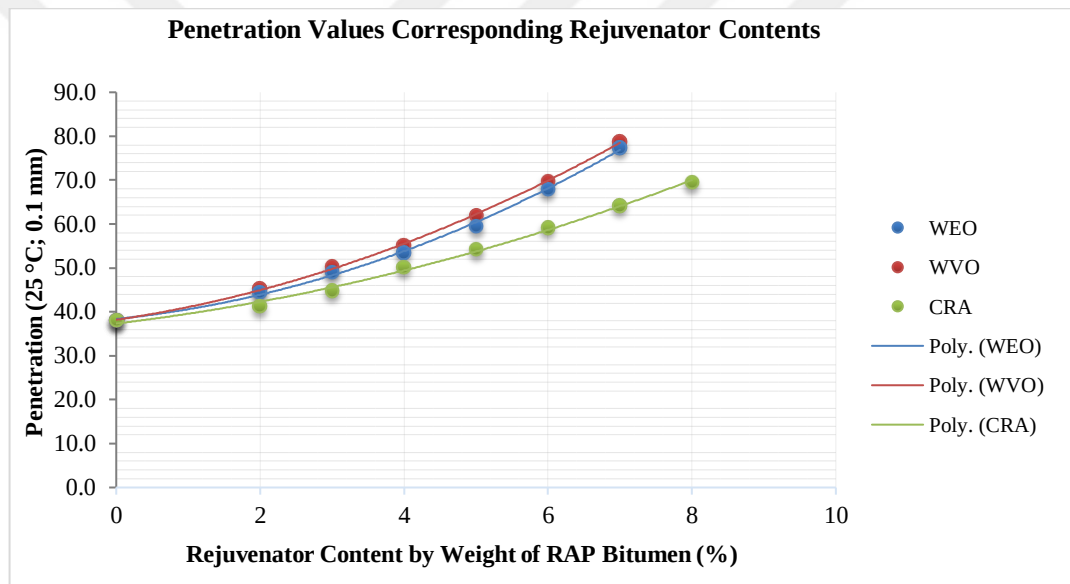


Figure 4.1 Penetration values corresponding rejuvenator contents

As shown on Figure 4.1, the penetration value increases slightly as result of rejuvenation. WEO and WVO additives represented very similar behavior in increasing the penetration value of aged RAP binder. The slope of increasing for CRA was detected to be slightly lower than WEO and WVO. All increasing trendlines track a polynomial trend in terms of penetration. It is possible to derive the desire penetration value by extracting the formula or through interpolation method. So it can be concluded that the desired penetration value can be achieved by making necessary alteration in additive content.

The results of softening point values corresponding different rejuvenator contents have been plotted and shown on Figure 4.2.

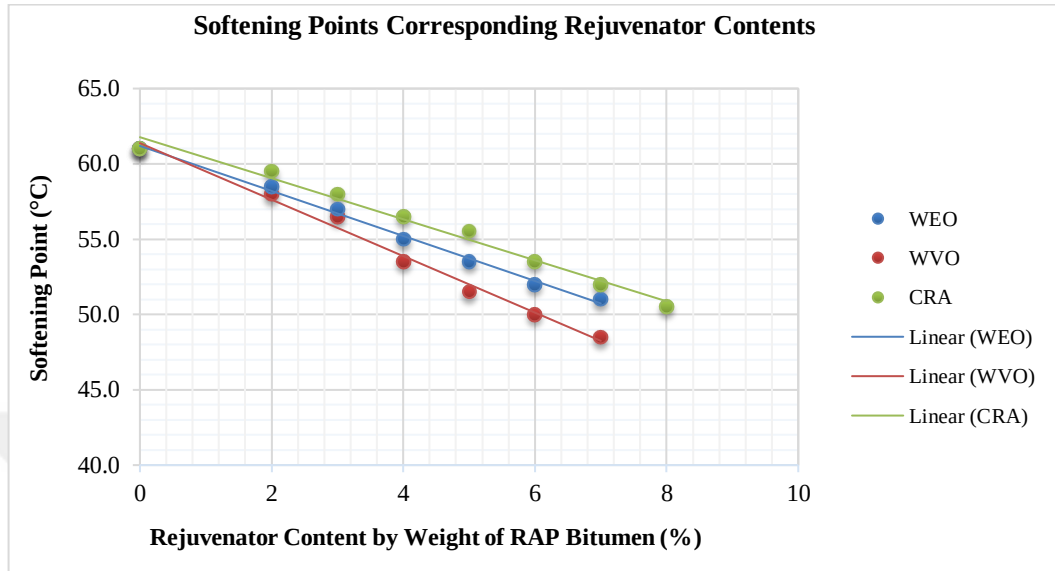


Figure 4.2 Softening point values corresponding rejuvenator contents

Figure 4.2 shows that the softening point values decrease by adding rejuvenating additives to aged RAP binder. According to Turkish specifications, only binders which have softening point values between 46 °C and 54 °C are allowed to be employed for 50-70 penetration grades regions. Each additive has its own trend line in decreasing of softening point values. As can be seen from the graph, the WVO sharply decrease the softening point values compared to WEO and CRA. Meanwhile, WEO can make more drop compared to CRA by using less additive content for modification of aged RAP bitumen. All trendlines have shown a linear drop in terms of softening point values. It is possible to calculate the desire softening point corresponding the additive content through interpolation method. So, the desired softening point can be achieved by adding the adequate rejuvenating agent content.

In addition to penetration and softening point values, the viscosity values at 135°C and 165 °C were also determined for rejuvenated bitumen samples. Table 4.3 contains the viscosity values for rejuvenated samples. Viscosities at 135°C and 165 °C corresponding various contents of each rejuvenating agents were plotted and presented in Figure 4.3 and Figure 4.4, respectively.

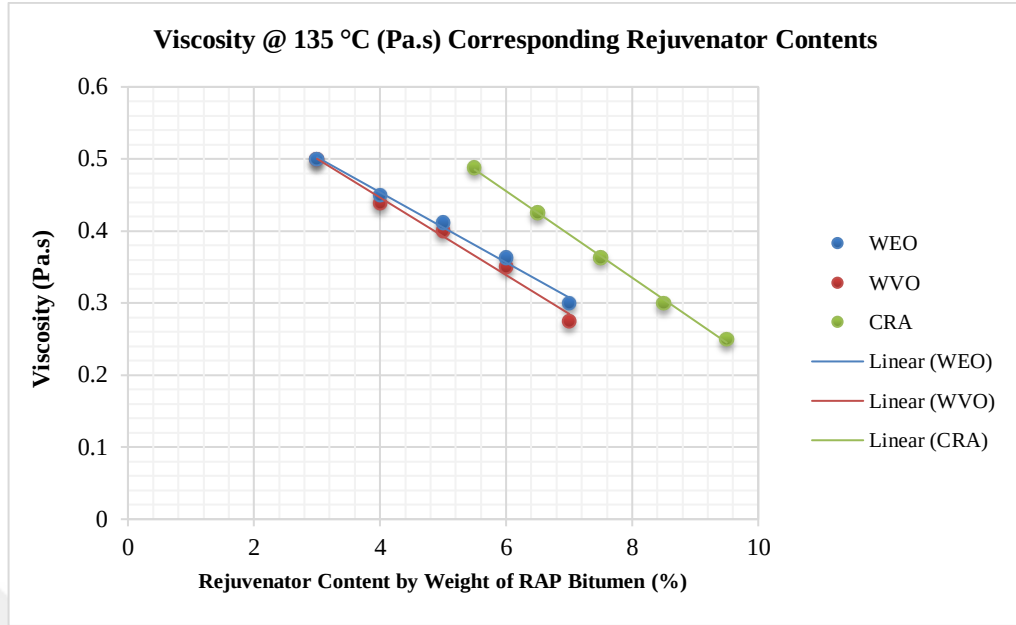


Figure 4.3 Viscosities at 135°C corresponding rejuvenator contents

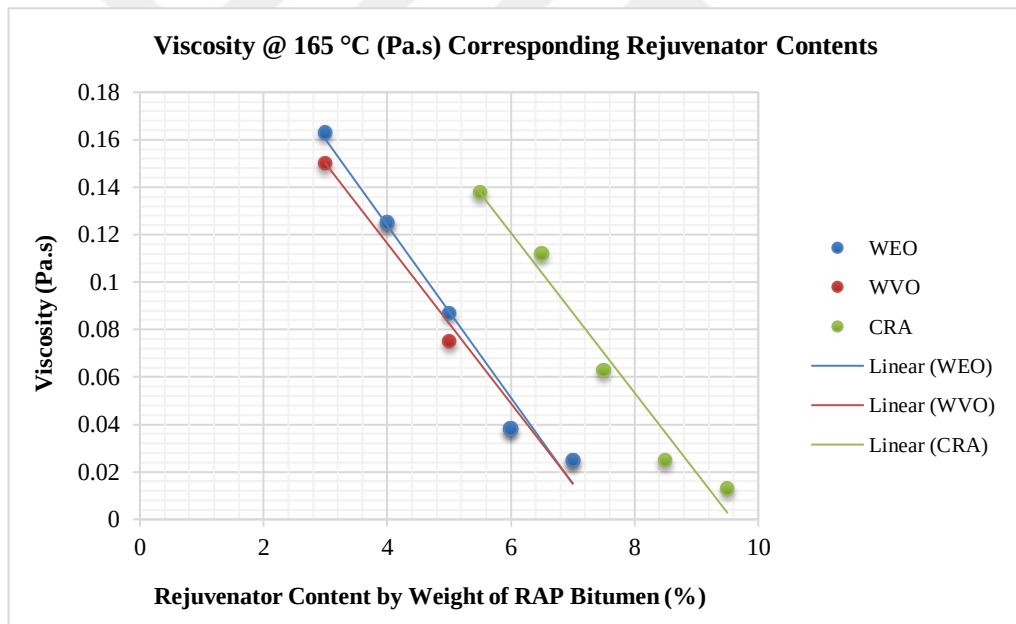


Figure 4.4 Viscosities at 165°C corresponding rejuvenator contents

Figure 4.3 and Figure 4.4 show that the viscosity values drop by adding more rejuvenating content into the aged RAP binder. The drop at 165°C temperature is sharper than the drops at 135°C. This can be attributed to the sharp viscosity changes in higher temperatures. It is possible to achieve the desired viscosity in term od mixing and compaction temperatures by adding the adequate amount of rejuvenating agent

directly into the mixture. The determination of mixing and compaction temperatures are explained within the section 4.2 where the mixture test results are presented. As can be seen in Figure 4.3 and Figure 4.4, the WVO additive can change the viscosity of aged RAP bitumen better than WEO and CRA at the same amount usage. It is due to the viscosity of WVO itself since it tends to be more fluent than two other rejuvenators. The WEO behaves like WVO in terms of changes in viscosities. Both WEO and WVO have viscosities like each other at similar contents since CRA is more viscous comparing WEO and WVO and thus, the amount of additive is relatively higher in CRA to achieve the same viscosity value needed in other agents. All viscosity drops track linear trends. It is possible to calculate the viscosity at any content at temperatures altering between 135°C and 165 °C for all rejuvenated bitumen samples.

As we know that the viscosity always increases by aging of bitumen, many studies have developed aging indices based on the ratio of aged bitumen sample viscosity to the viscosity of neat bitumen (Siddiqui & Ali, 1999; Apeagyei, 2011). These values are always bigger than unit and so, the bigger the aging index than 1, the more aged the sample is. According to this theory, it should be possible to define the rejuvenating indices based on the viscosity test results for rejuvenated samples. The rejuvenating indices are calculated as the ratio of the viscosity value of a rejuvenated sample over the viscosity of aged RAP bitumen at a defined temperature. On this regard, all viscosity values were calculated at 135°C. The results are presented in Table 4.4.

As mentioned before, the penetration test is used as a reference for calculation of optimum rejuvenating content for each rejuvenating agent. On this regard, the optimum rejuvenating contents were calculated as 5.4% for WEO, 5.1% for WVO and 6.8% for CRA. Following the calculations, the samples containing the optimum rejuvenating values were produced and tested for all conventional tests which are normally mandatory for neat binders as per Turkish specifications. The Table 4.4 contains the test results for all these conventional tests.

Table 4.4 Rejuvenating indices and conventional test results for optimum rejuvenating contents

Test	Specification	Bitumen Samples					Specification limits
		Neat	RAP Bitumen	Optimally Rejuvenated			
		50/70	Extracted	WEO (5.4%)	WVO (5.1%)	CRA (6.8%)	
Penetration (25 °C; 0.1 mm)	TS EN 1426	63	38	63	63	63	50–70
Softening point (°C)	TS EN 1427	49.7	61	50	51.5	50.5	46–54
Penetration Index		-0.72	0.57	-0.65	-0.27	-0.52	
Viscosity @ 135 °C (Pa.s)	ASTM D4402	0.425	0.538	0.412	0.4	0.325	–
Viscosity @ 165 °C (Pa.s)	ASTM D4402	0.1	0.188	0.088	0.075	0.05	–
Rejuvenation Indices	ASTM D4402	0.79	1.00	0.77	0.74	0.60	–
Rolling Thin Film Oven (163 °C)	TS EN 12607-1	RTFO Aged Samples Results					
Change of mass (%)		0.05	N/A	0.12	0.1	0.08	0.5 (max)
Retained penetration after RTFO (%)	TS EN 1426	74	N/A	53	59	61	50 (min)
Softening point rise after RTFO (°C)	TS EN 1427	4.5	N/A	6	5	5	7 (max)

The most concerned issue in bitumen rejuvenation is that if the newer samples can meet the long-term criteria or not. The after RTFO results showed that the rejuvenated samples can meet all Turkish criteria indicated within the specifications. Although the neat bitumen is better in quality, but it is possible to rejuvenate the bitumen at least for one more use. The results showed that it is not possible to directly use the RAP binder without making necessary change in all conventional properties. As mentioned, the resulting rejuvenated bitumen samples behave like fresh bitumen sample.

Figure 4.5 shows the rejuvenation indices for all rejuvenated samples produced using the optimum amounts of rejuvenating agents.

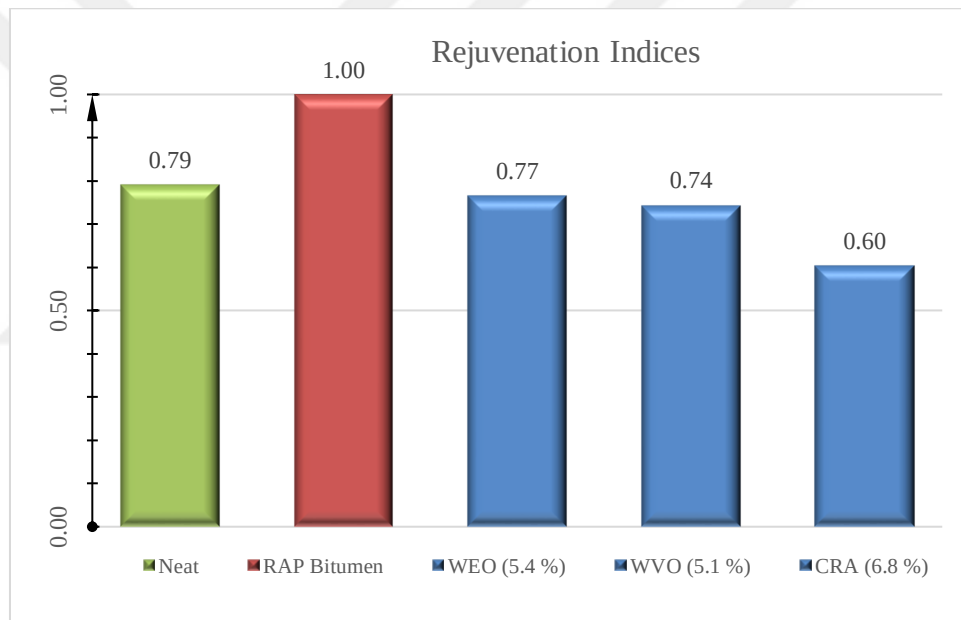


Figure 4.5 Rejuvenation indices for optimally rejuvenated samples

As presented in Figure 4.5, by using optimum amount of CRA, it is possible to achieve a bitumen sample which is more convenient to mix and compact compared to other samples including neat bitumen. Optimum WEO and WVO samples behave like each other in terms of viscosity. All rejuvenated samples have lower rejuvenation indices compared to neat bitumen.

The penetration index is a better index to evaluate the effect of aging and air blowing. Table 4.4 also contains the penetration indices calculated for each optimally

rejuvenated sample. Figure 4.6 shows the penetration indices for these samples and the aged RAP bitumen.

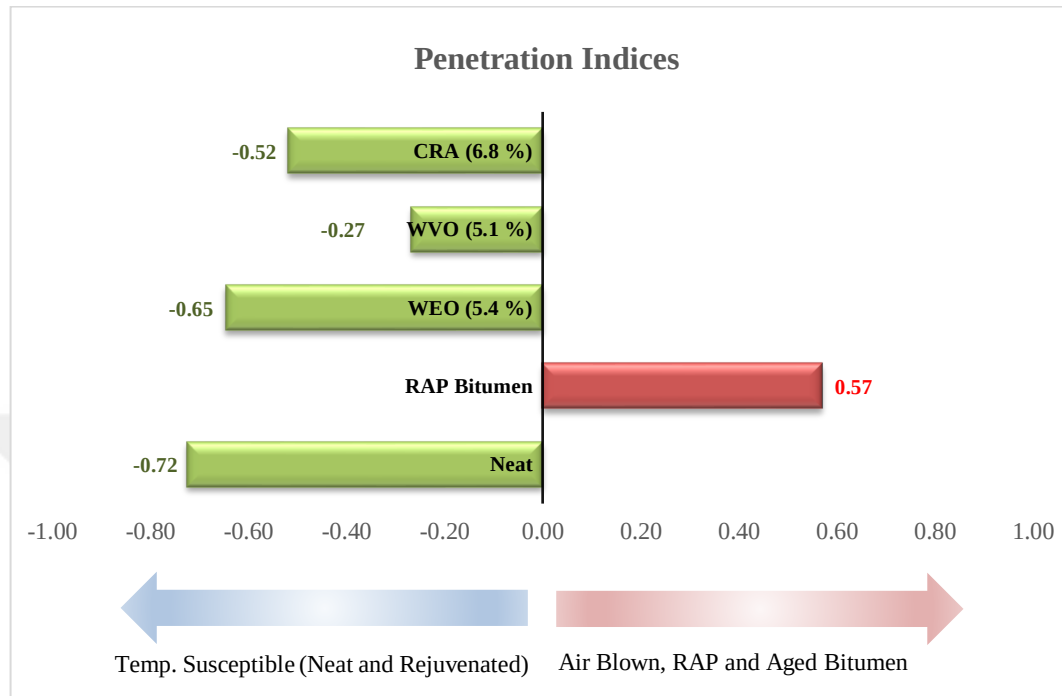


Figure 4.6 Penetration indices for optimally rejuvenated samples

As shown in Figure 4.6, the comparison between the penetration indices of aged RAP bitumen and the optimally rejuvenated samples show that the rejuvenation process helps the rejuvenated sample to act like neat bitumen. Although neat bitumen has the best penetration index values among all samples, the other samples also have acceptable values. The penetration indices on the other hand, show the temperature susceptibility of bitumen samples in terms of the region to be applied. The bars show that the WEO rejuvenated sample is more susceptible to temperature changes compared to other two samples.

The temperature susceptibility increases by rejuvenation process which is the opposite process of aging and air blowing. Neat bitumen remains more susceptible to seasonal temperature changes than all rejuvenated samples.

4.1.2 Rheological Results

This chapter includes the results of rheological studies conducted on all rejuvenated and non-rejuvenated bitumen samples. The rheological studies include results for DSR testing. The following chapters consist of the DSR results for each rejuvenating agent and the comparisons between the rejuvenated bitumen samples and non-rejuvenated bitumen samples in terms of rheology.

4.1.2.1 Dynamic Shear Rheometer Results of WEO

The selected additive contents for WEO were 3 to 7 percent of rejuvenating agent by weight of RAP aged bitumen. The extracted aged RAP bitumen was modified using the selected range of WEO. The rheological characteristics of all WEO rejuvenated bitumen samples were investigated using DSR testing. All samples were tested at temperatures ranging from 40°C, 50°C, 60°C and 70°C in various loading frequencies using frequency sweep. The results for all WEO rejuvenated samples are presented in the Appendix for 0.01 Hz, 0.1 Hz, 1 Hz and 10 Hz.

The table also includes the results for neat bitumen and aged RAP bitumen (non-rejuvenated samples) to make necessary comparisons between neat bitumen and non-rejuvenated samples to see the effects of rejuvenating agent on aged RAP bitumen. As the range of temperatures in this table are for rutting performance evaluation of rejuvenated and non-rejuvenated bitumen samples, $G^*/\sin(\delta)$ parameters were calculated for all samples at different temperatures and frequencies and presented in this table.

All test results are plotted for each frequency level and each temperature to understand the rheological behavior of the WEO rejuvenated bitumen samples in different frequencies and different temperatures. The results showed that WEO rejuvenating agent can change the extracted aged RAP bitumen sample behavior and characteristics in terms of rheology. The aged bitumen sample which has been hardened during the service life of the asphalt pavement, softens by use of WEO rejuvenating agent.

Figures 4.7 to 4.9, respectively, present the plotted test results for rutting parameters, complex viscosities and phase angles of WEO rejuvenated bitumen samples at 0.01 Hz frequency level. The results for rutting parameters and the complex viscosities have been plotted in logarithmic scale on Y axis to help better demonstration of the results.

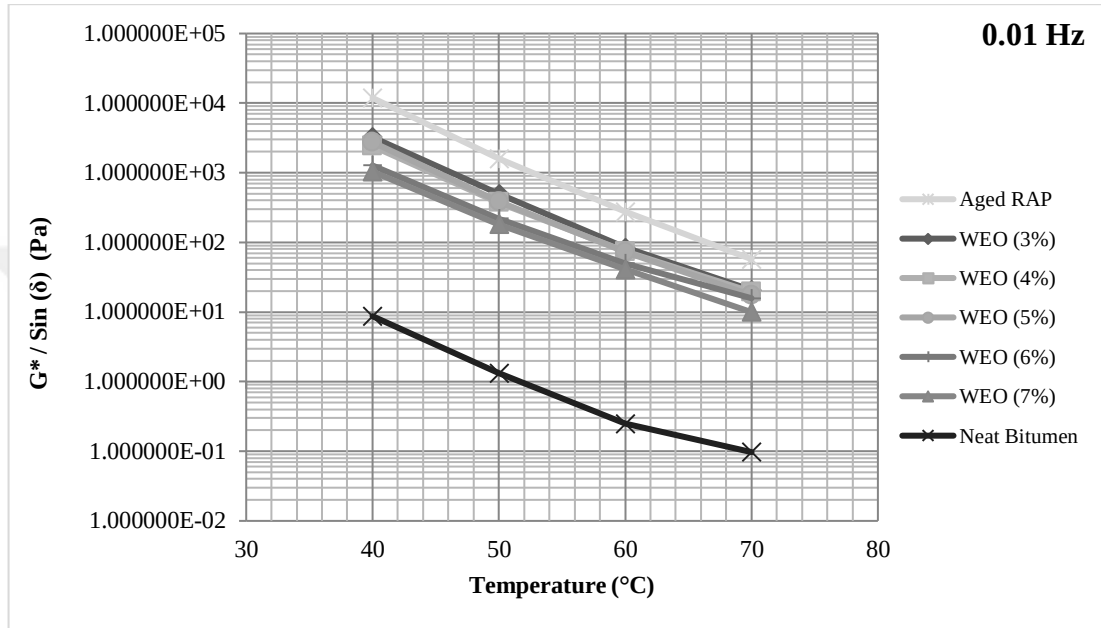


Figure 4.7 WEO rejuvenated samples rutting parameters at 0.01 Hz frequency

The asphalt rheology science suggests that the rutting parameter $G^*/\sin(\delta)$ of a bitumen sample should be maximum to ensure a high rut resistance under heavy traffic loads. Since $G^*/\sin(\delta)$ of aged bitumen is higher than the $G^*/\sin(\delta)$ of fresh neat bitumen, it does not mean that aged bitumen is more rut resistant than fresh neat bitumen. This fact is attributed to the high phase angles and high complex modulus of aged bitumen due to the hardening phenomenon.

In this context, the main apprehension is that to check if we get a bitumen sample like neat bitumen in terms of rutting performance or not. The results shown on Figure 4.7, shows that the rutting parameters of WEO rejuvenated samples decrease by adding more content of rejuvenating agents. This is because of that the bitumen softens in referenced temperatures by adding more rejuvenating agent. As seen on the figure, generally the rutting parameter decreases by increase of the temperature. Although the

WEO rejuvenating agent softens the aged RAP bitumen, it does not ensure to achieve a fresh neat bitumen in terms of $G^*/\sin(\delta)$.

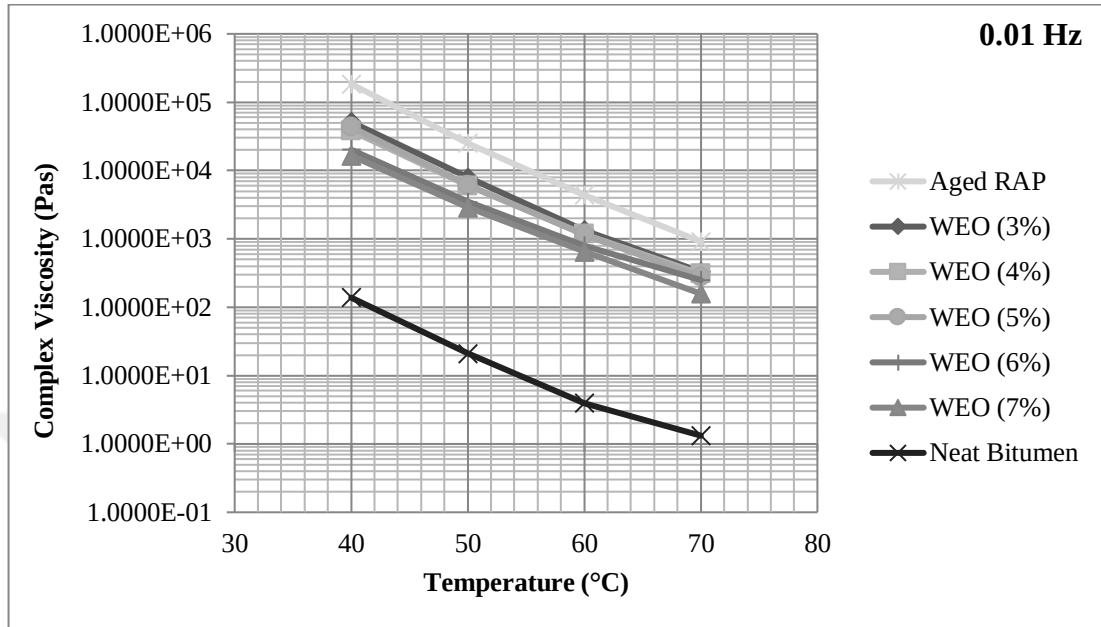


Figure 4.8 WEO rejuvenated samples complex viscosities at 0.01 Hz frequency

Like rutting parameters, as shown in Figure 4.8, the same story is true for complex viscosities. In this way, it can be concluded that the complex viscosity of aged RAP bitumen is rather high compared to neat bitumen. The results at 0.01 frequency level shows that the use of WEO decreases the complex viscosity of aged RAP bitumen at this frequency level but still not giving us a fresh neat bitumen.

Not surprisingly, the complex viscosity of all bitumen samples decreases by increasing of temperature. Almost the complex viscosities of all samples represent a linear drop with a very similar slope in logarithmic scale (exponential drop in normal scale) by increase of temperature level.

The phase angle (δ) ranges from 0° for completely elastic materials to 90° for completely viscous materials. For viscoelastic materials like bitumen, the loading time and the temperature has a great effect on phase angle value. For faster loading periods like 0.01 Hz in higher temperature levels, this value is very sensitive to measurements.

Figure 4.9 simply shows this fact. At 40°C it is obvious that the neat bitumen is more viscous than aged RAP bitumen. The use of WEO rejuvenating agent results in more viscous bitumen sample. In slower loading periods, the difference becomes more distinguishable even in higher temperatures. We will see this fact within the following phases at higher frequencies.

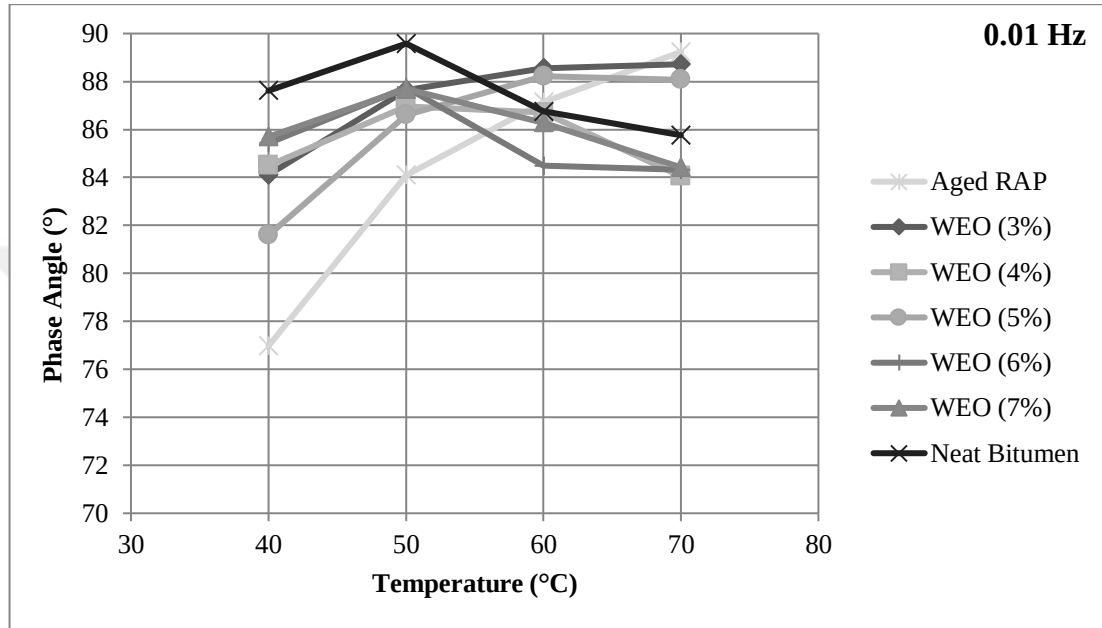


Figure 4.9 WEO rejuvenated samples phase angles at 0.01 Hz frequency

Figures 4.10 to 4.12, respectively, demonstrate the test results for rutting parameters, complex viscosities and phase angles of WEO rejuvenated bitumen samples at 0.1 frequency level.

The rutting parameters for 0.1 Hz of WEO rejuvenated bitumen samples, non-rejuvenated bitumen samples and neat bitumen at different temperatures are presented in Figure 4.10. The softening effect of WEO is cognizable in all frequency levels at any temperature.

Also, the results for complex viscosities presented in Figure 4.11 show the softening effect of WEO in terms of complex viscosity. The neat bitumen characteristic was not achieved by adding WEO rejuvenating agent but still we see that the hardened bitumen characteristics can improve by adding WEO as a rejuvenating agent.

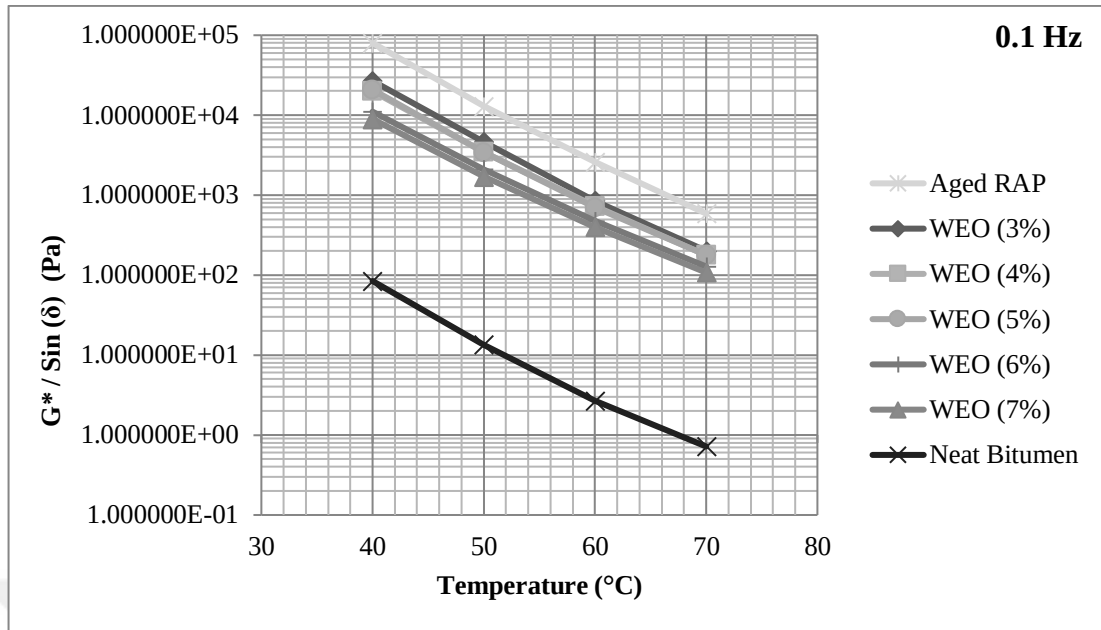


Figure 4.10 WEO rejuvenated samples rutting parameters at 0.1 Hz frequency

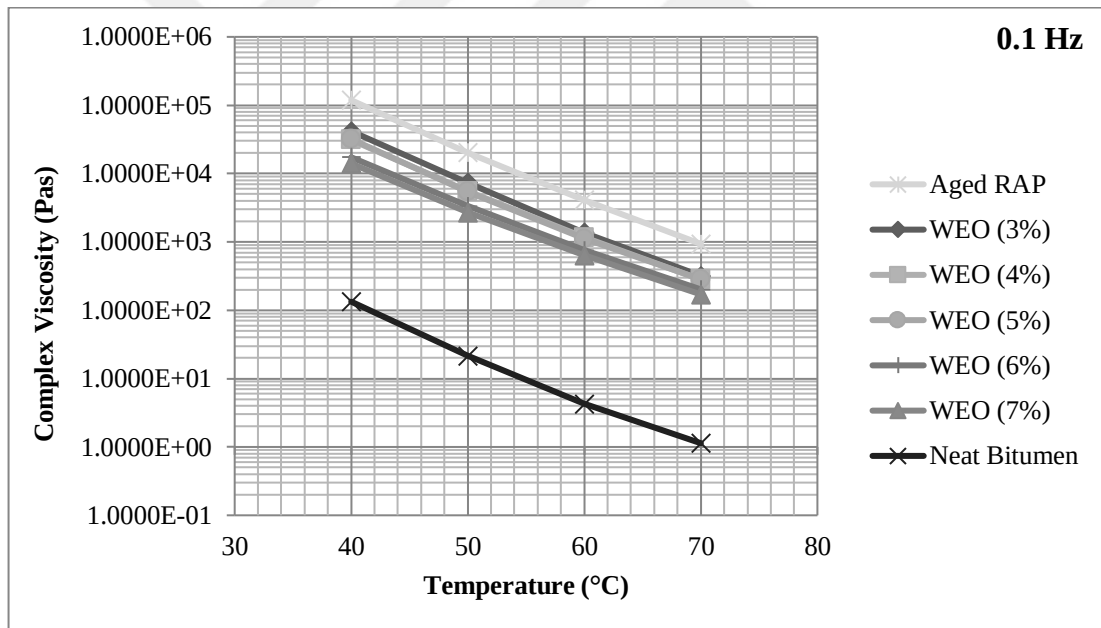


Figure 4.11 WEO rejuvenated samples complex viscosities at 0.1 Hz frequency

The phase angle results for 0.1 Hz at different temperatures are shown in Figure 4.12. In higher frequency levels, the differences between the samples are more obvious since the material visco-elastic characteristics become more measurable at higher frequencies. The same is true also for lower temperatures as shown in Figure 4.12. What we can conclude from this figure is that the aged bitumen which has been

hardened during the time, regains visco-elasticity through modification with WEO rejuvenating agent.

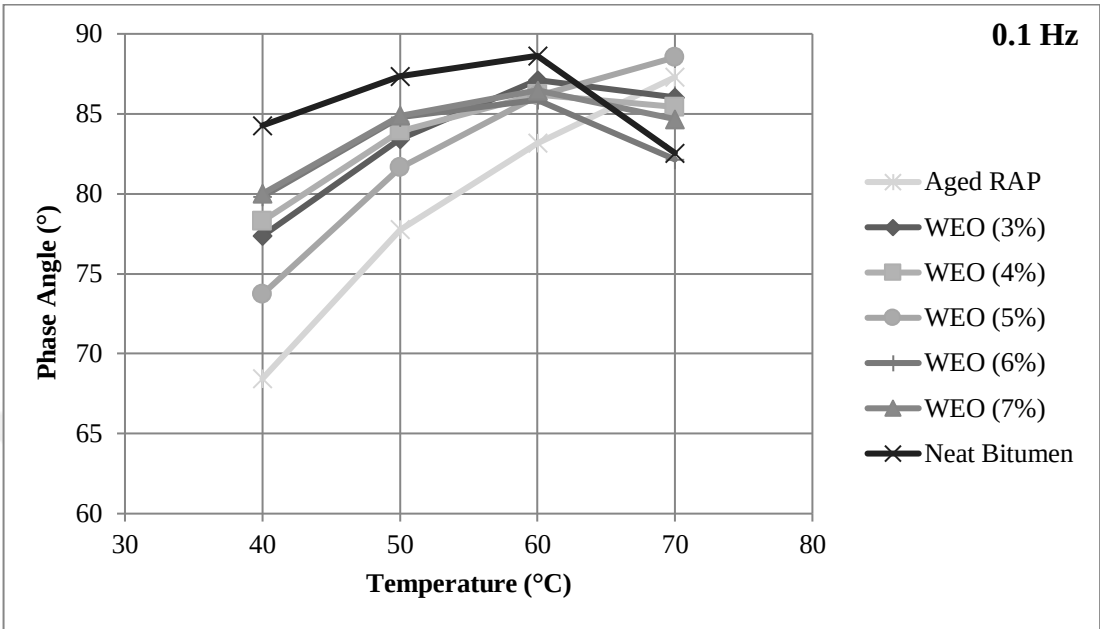


Figure 4.12 WEO rejuvenated samples phase angles at 0.1 Hz frequency

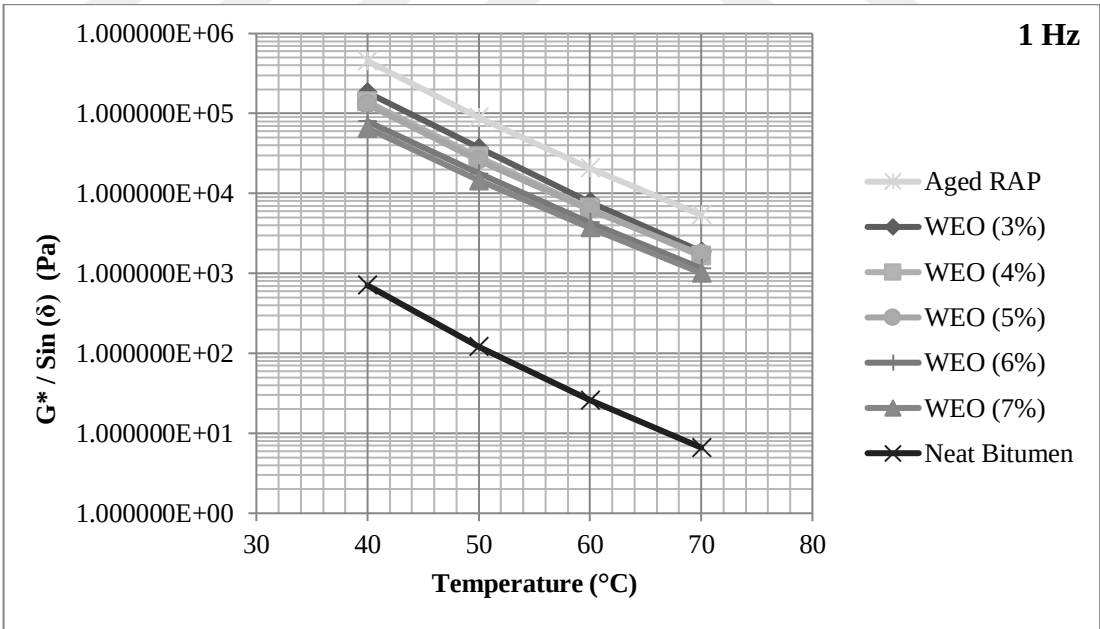


Figure 4.13 WEO rejuvenated samples rutting parameters at 1 Hz frequency

The same regime seen at other frequencies can be recognized for 1 Hz as can be seen in Figure 4.13 that demonstrates the WEO rejuvenated and non-rejuvenated bitumen samples rutting parameters.

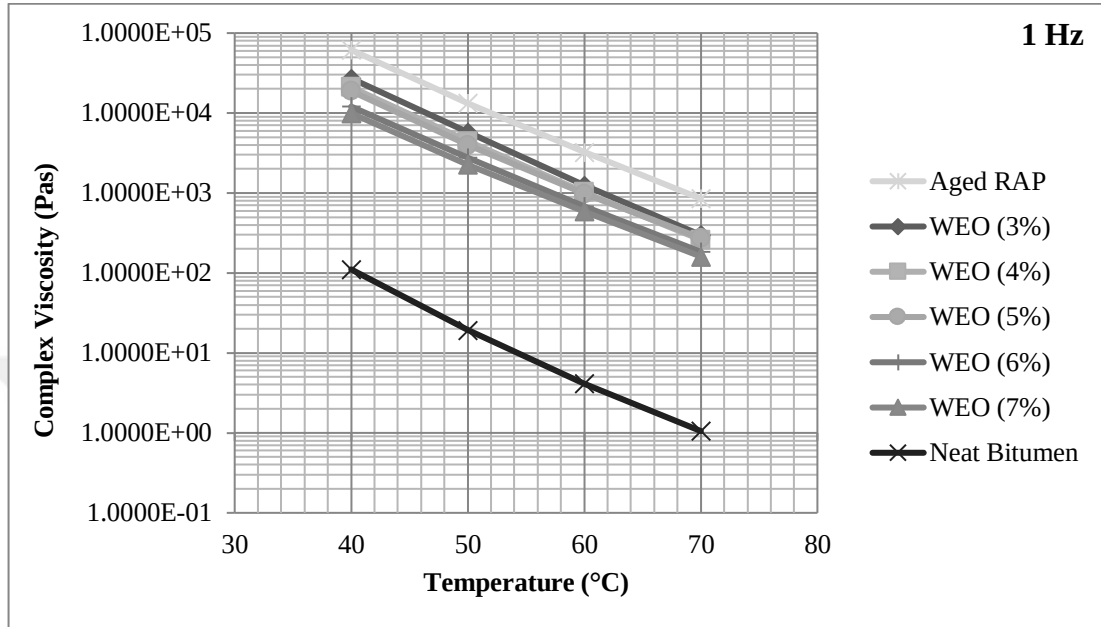


Figure 4.14 WEO rejuvenated samples complex viscosities at 1 Hz frequency

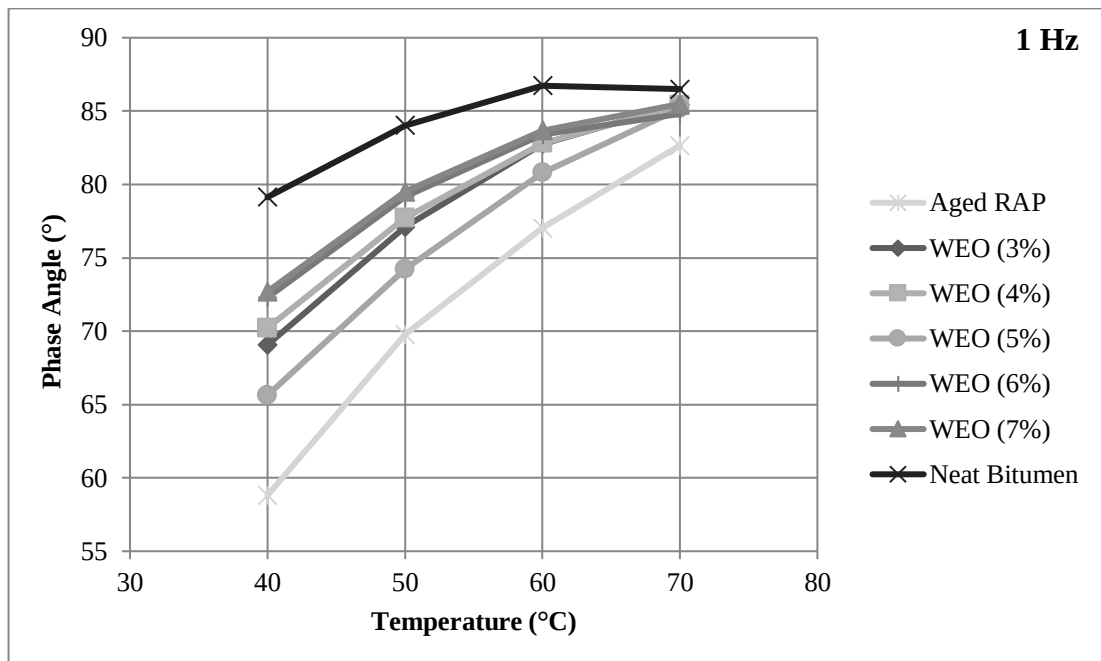


Figure 4.15 WEO rejuvenated samples phase angles at 1 Hz frequency

The complex viscosities of WEO rejuvenated and non-rejuvenated bitumen samples for 1 Hz at various temperatures are presented in Figure 4.14. The same behavior at previous frequency levels are also true for the samples at this frequency level.

As can be seen in Figure 4.15, the differences between phase angles become more recognizable at higher frequencies and lower temperatures. The aged bitumen visco-elastic characteristics improve by adding WEO rejuvenating agent.

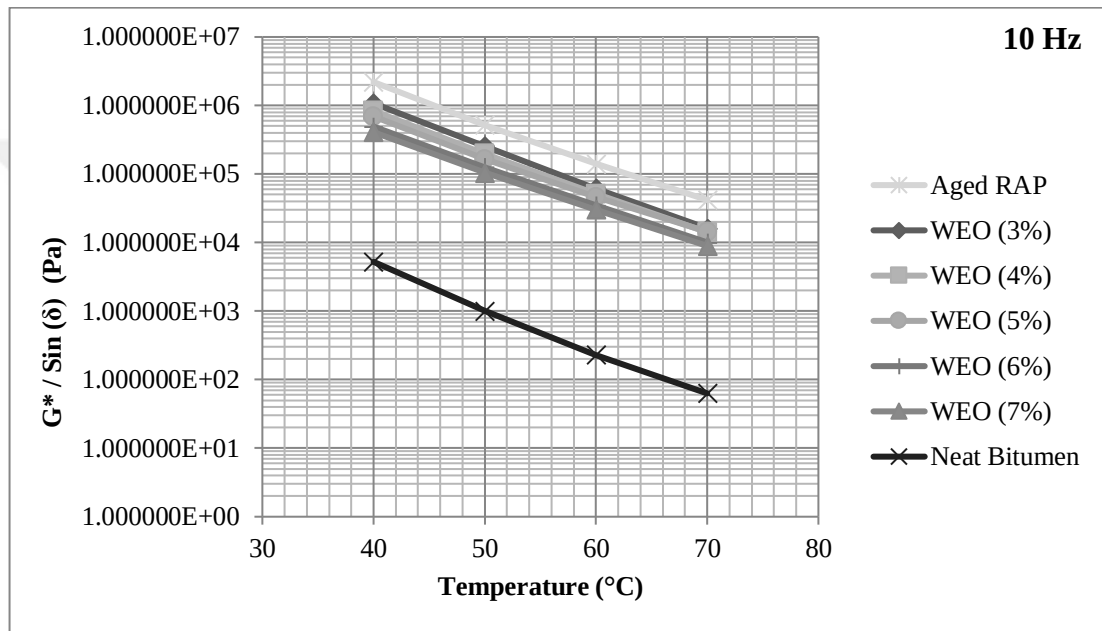


Figure 4.16 WEO rejuvenated samples rutting parameters at 10 Hz frequency

Figure 4.16 demonstrates the rutting parameters for WEO rejuvenated and non-rejuvenated bitumen samples at 10 Hz. Frequency level and different temperatures. As can be seen at this temperature level, the hardened aged RAP bitumen softens by adding WEO rejuvenating agent. Typically, the rutting parameters decrease by increasing of the temperature.

The complex viscosities are for WEO rejuvenated and non-rejuvenated bitumen samples at 10 Hz and different temperatures are presented in Figure 4.17. The same behavior experienced by adding WEO at other frequency levels is seen at this specific frequency level.

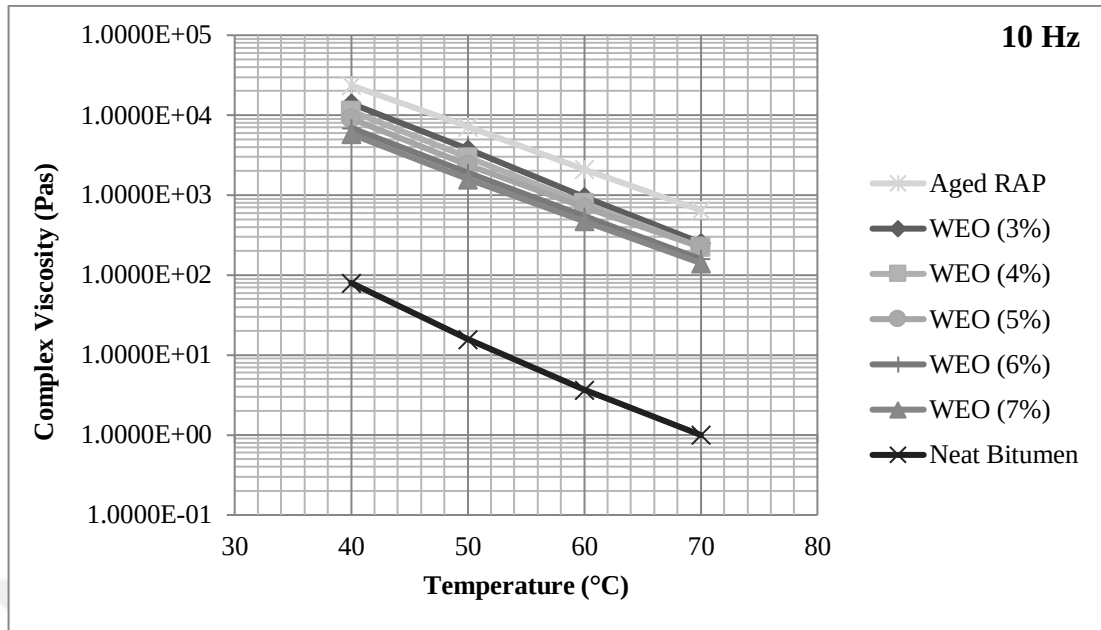


Figure 4.17 WEO rejuvenated samples complex viscosities at 10 Hz frequency

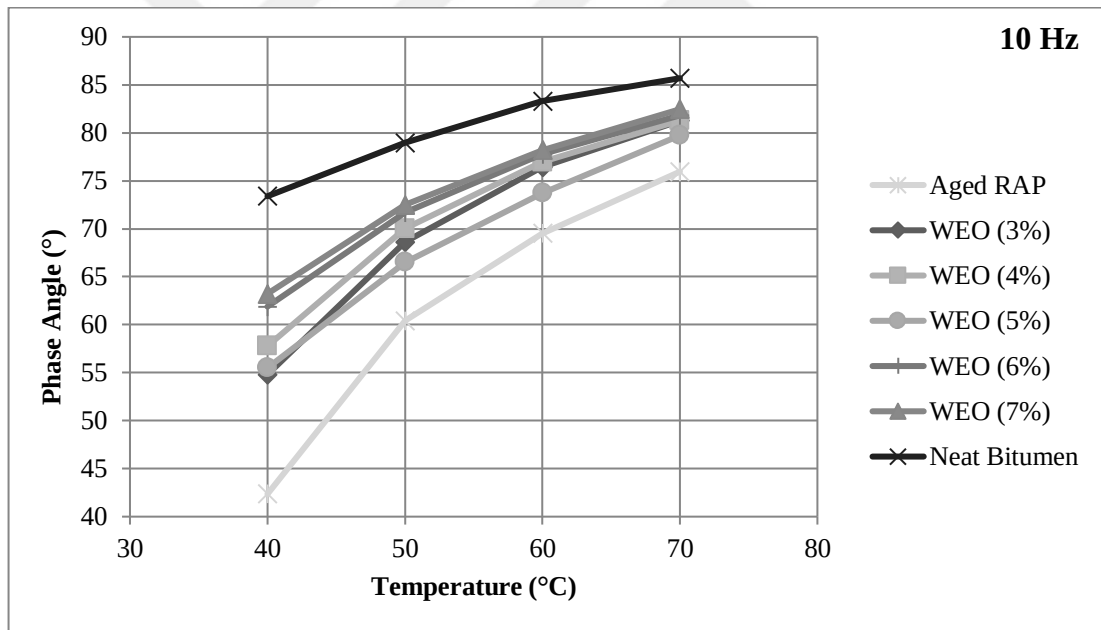


Figure 4.18 WEO rejuvenated samples phase angles at 10 Hz frequency

For highest frequency level (10 Hz) tested in the scope of DSR testing, the viscous and elastic behavior differences between WEO rejuvenated and non-rejuvenated bitumen samples are more obvious as shown in Figure 4.18. WEO rejuvenating agent improves the visco-elastic characteristics of aged RAP bitumen.

The following figures (Figure 4.19-4.30) are the presentation of the same results in another way. This time, the results have been plotted at a specific temperature and different frequency levels on X axis. In these figures the same results have been plotted to evaluate the effect of frequency level on rheological behaviors of WEO rejuvenated and non-rejuvenated bitumen samples.

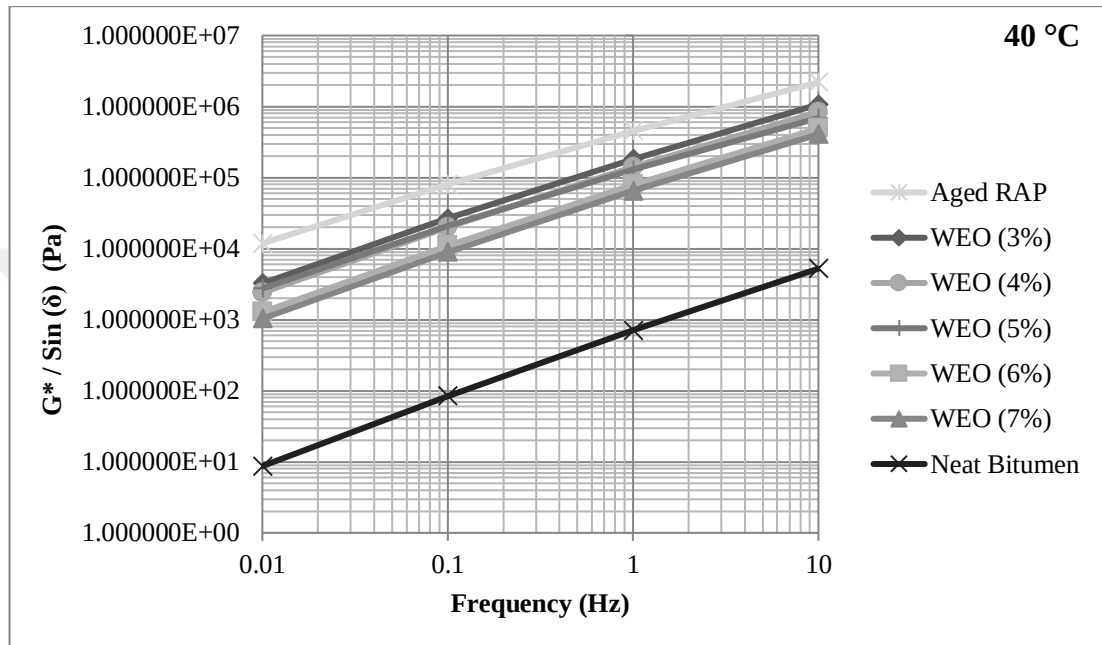


Figure 4.19 WEO rejuvenated samples rutting parameters at 40°C

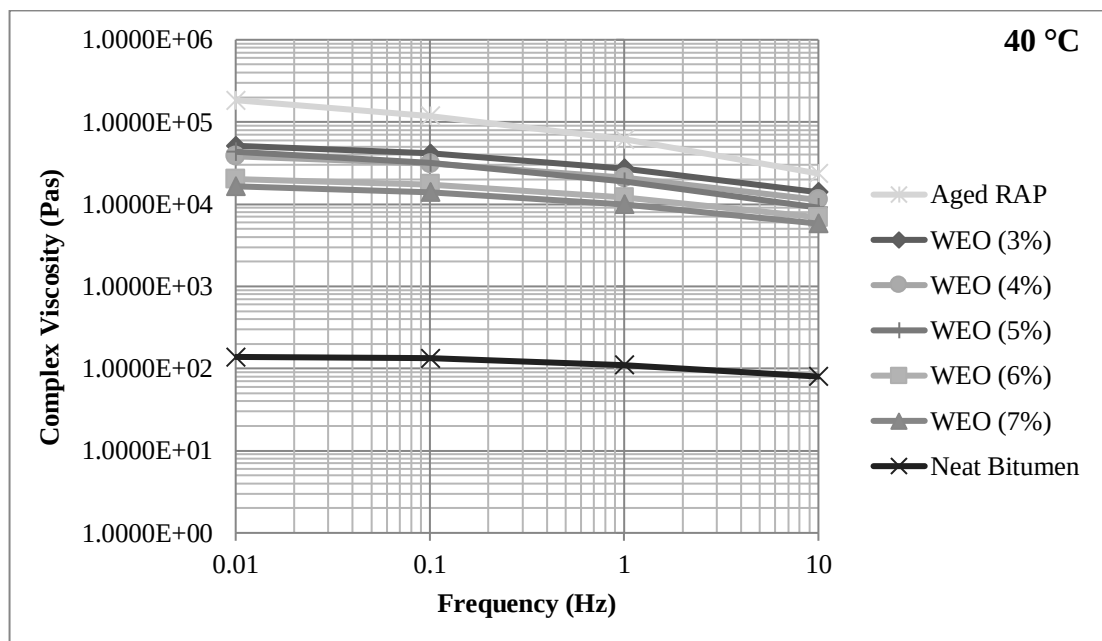


Figure 4.20 WEO rejuvenated samples complex viscosities at 40°C

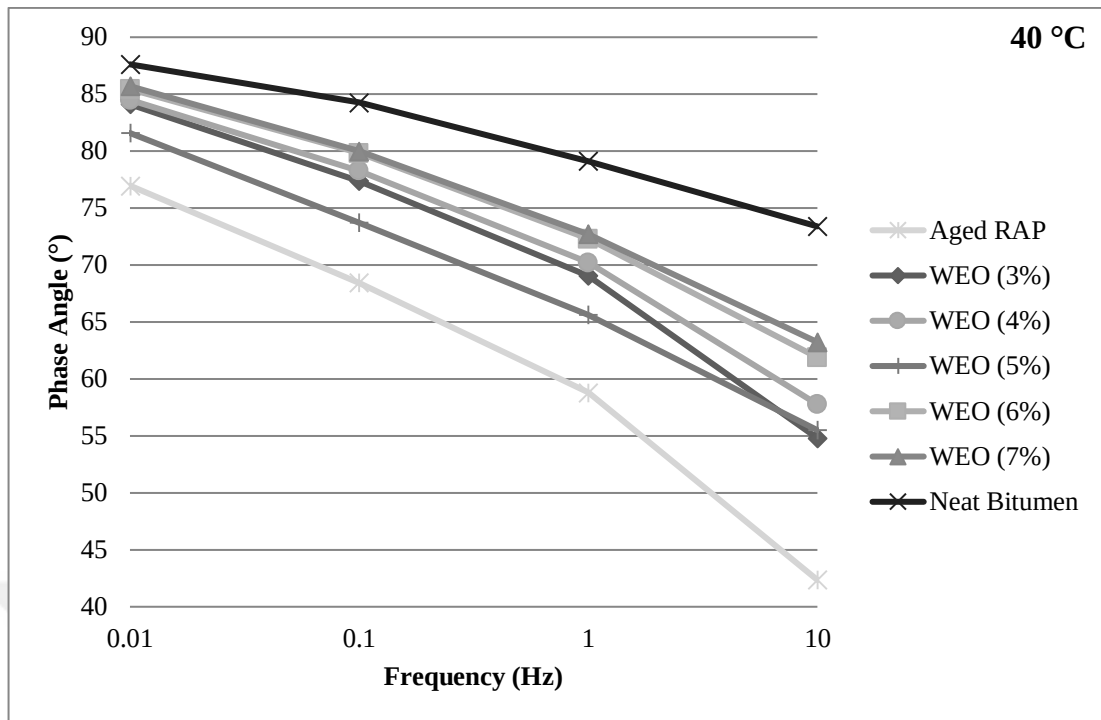


Figure 4.21 WEO rejuvenated samples phase angles at 40°C

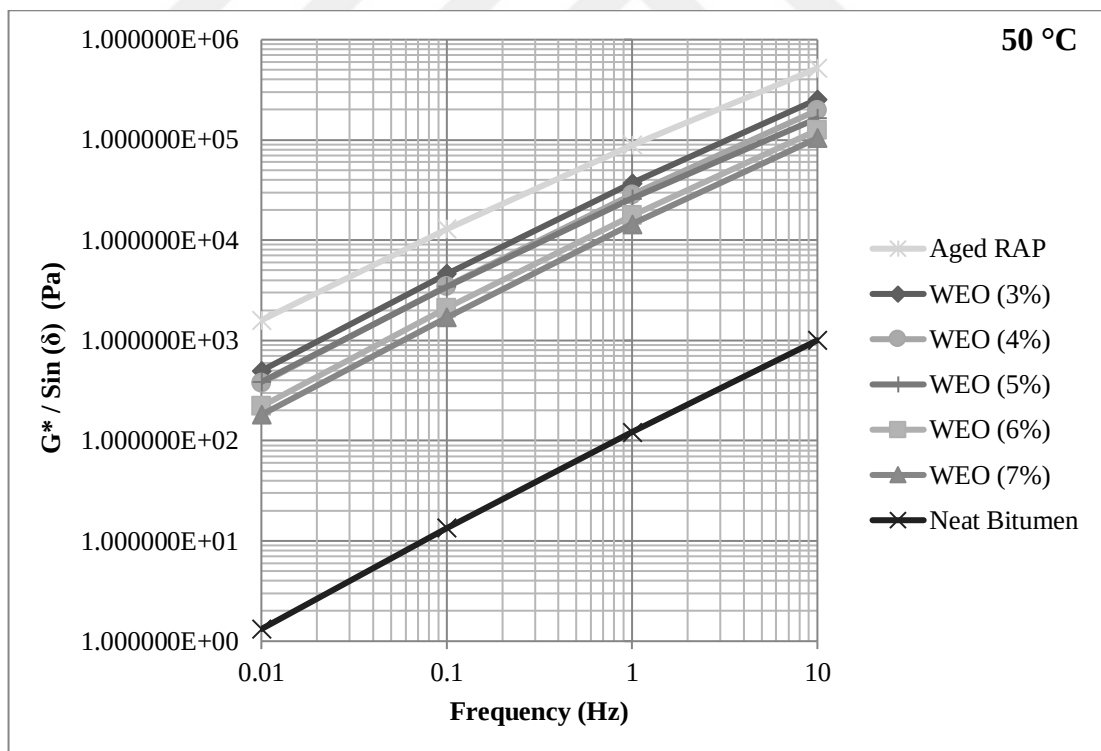


Figure 4.22 WEO rejuvenated samples rutting parameters at 50°C

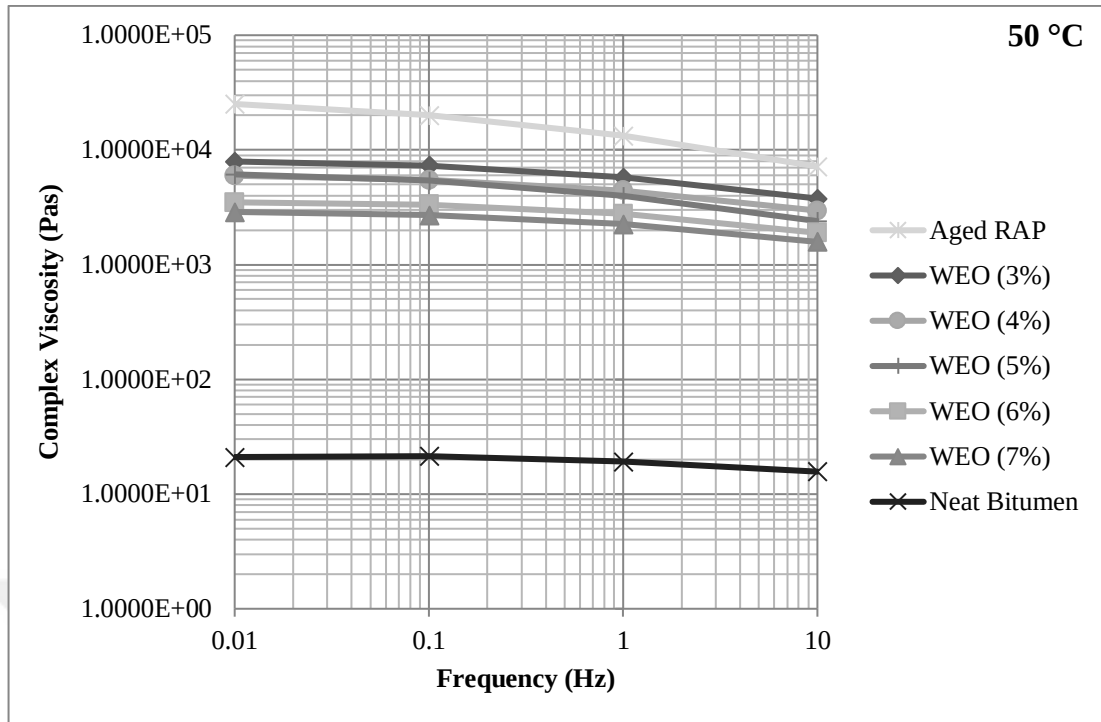


Figure 4.23 WEO rejuvenated samples complex viscosities at 50°C

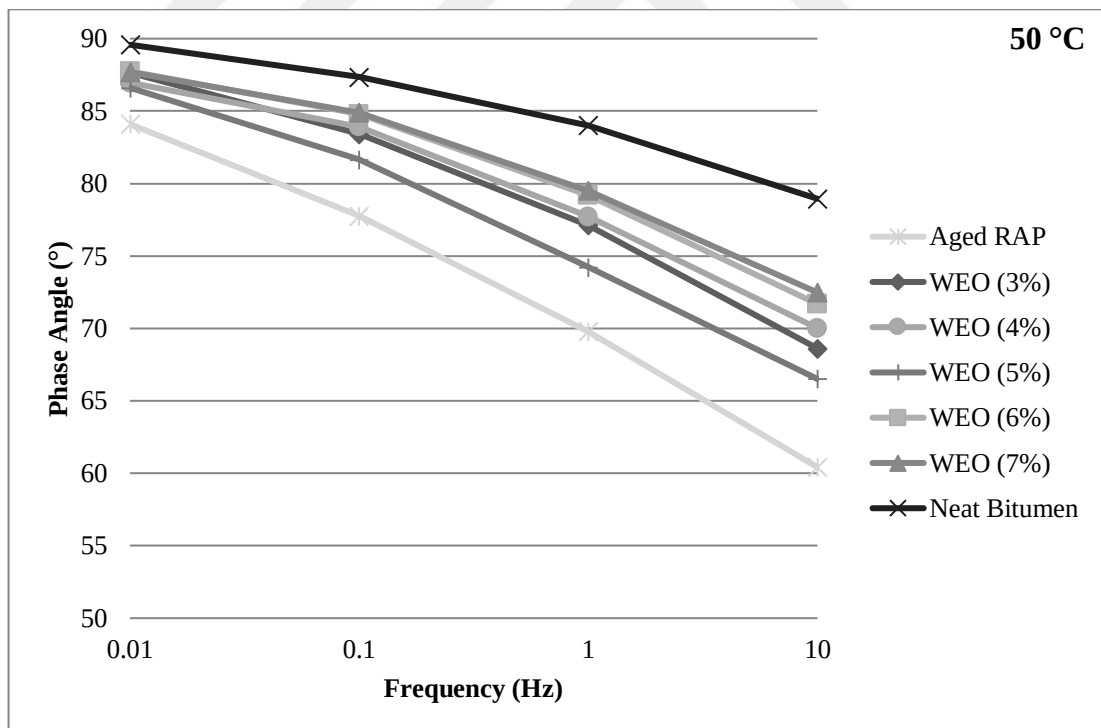


Figure 4.24 WEO rejuvenated samples phase angles at 50°C

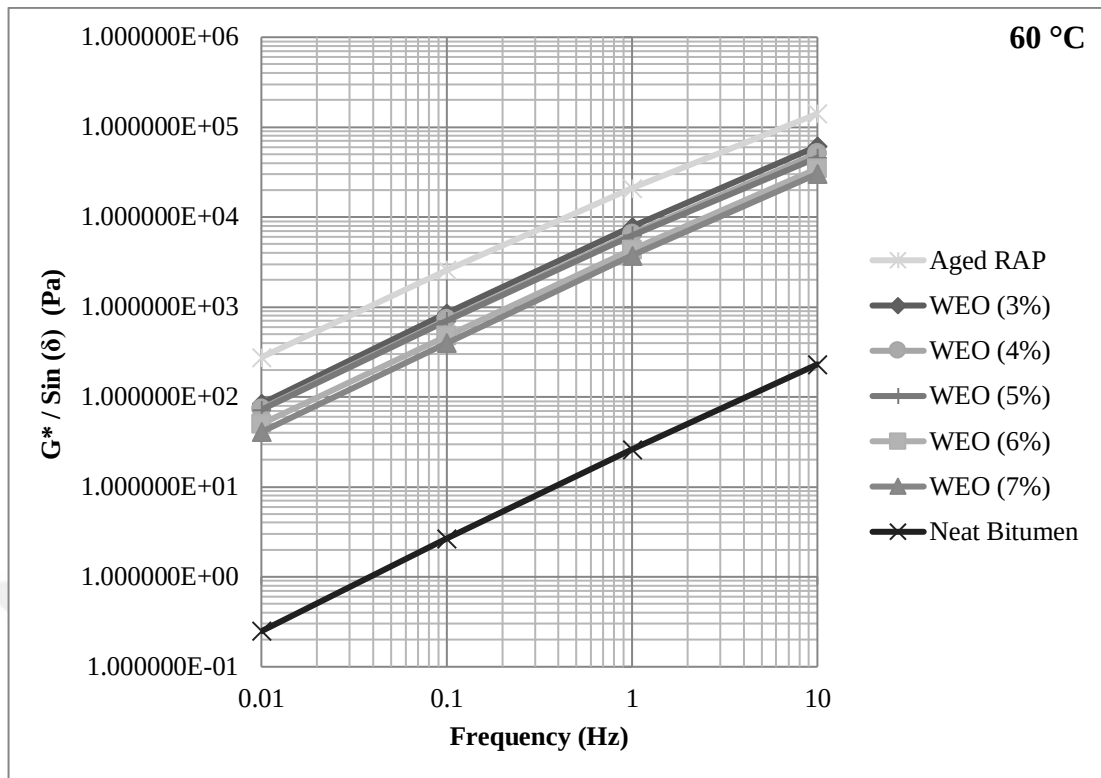


Figure 4.25 WEO rejuvenated samples rutting parameters at 60°C

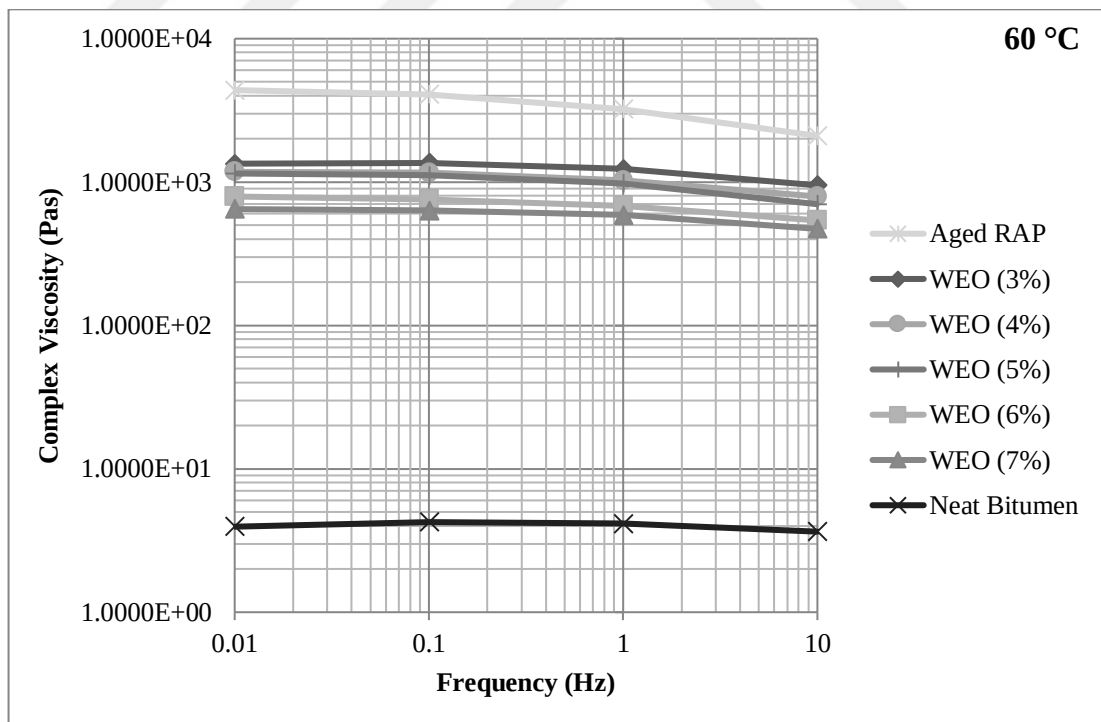


Figure 4.26 WEO rejuvenated samples complex viscosities at 60°C

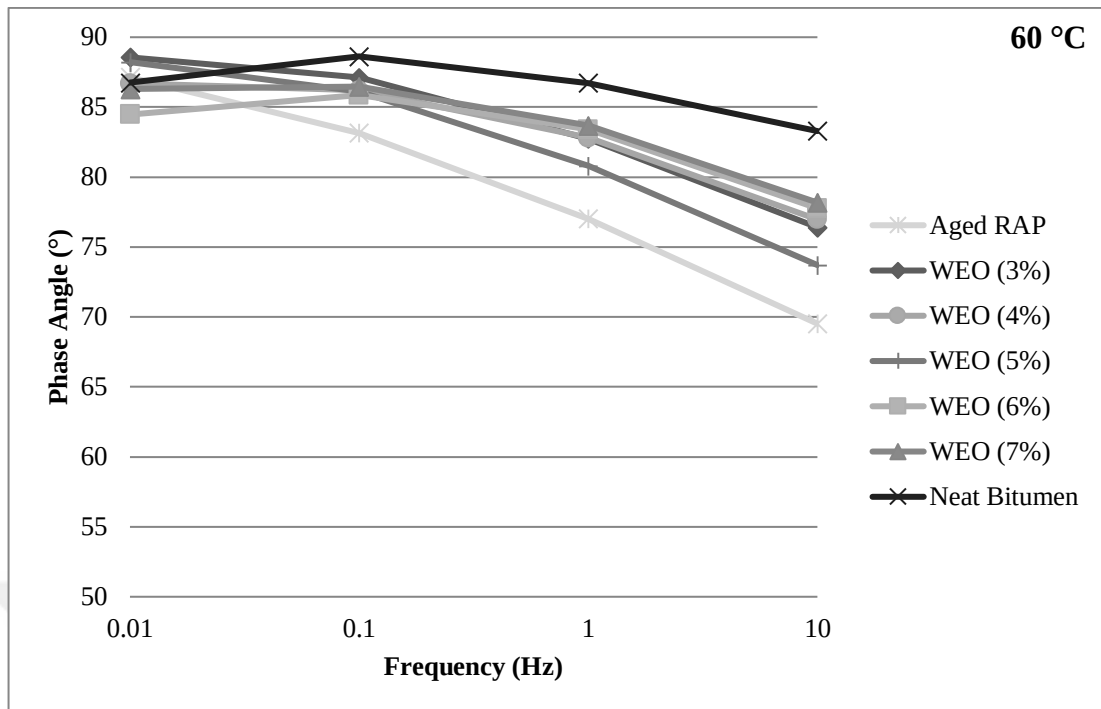


Figure 4.27 WEO rejuvenated samples phase angles at 60°C

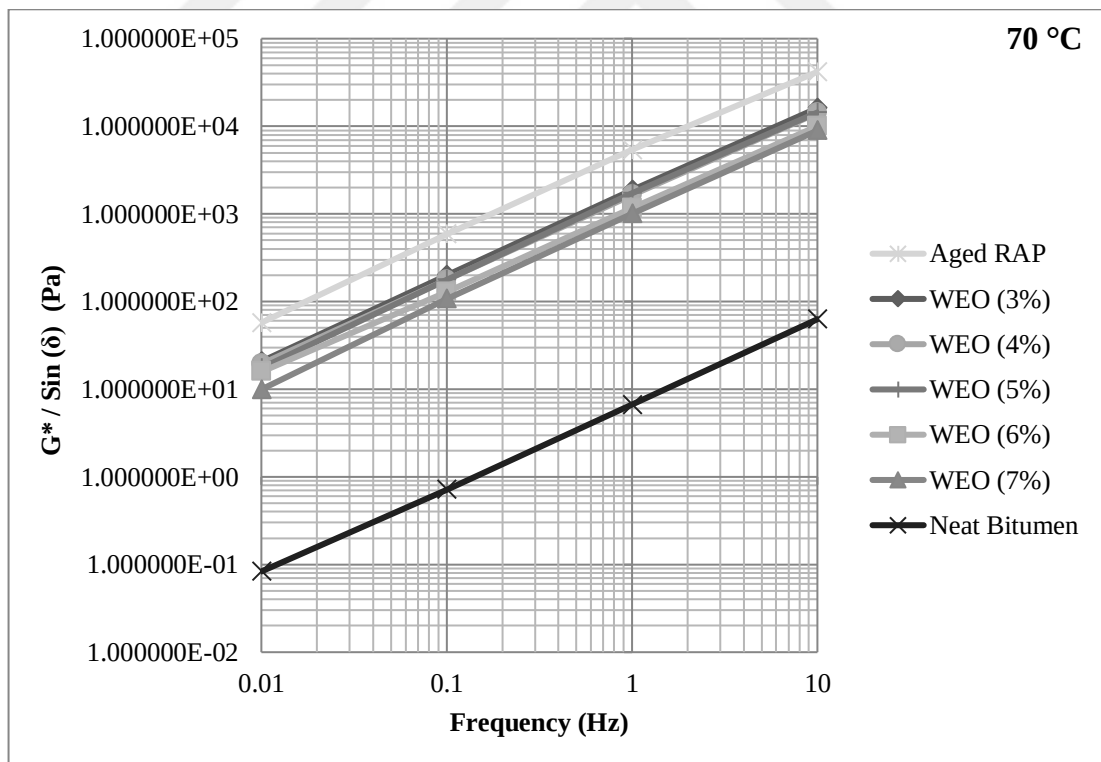


Figure 4.28 WEO rejuvenated samples rutting parameters at 70°C

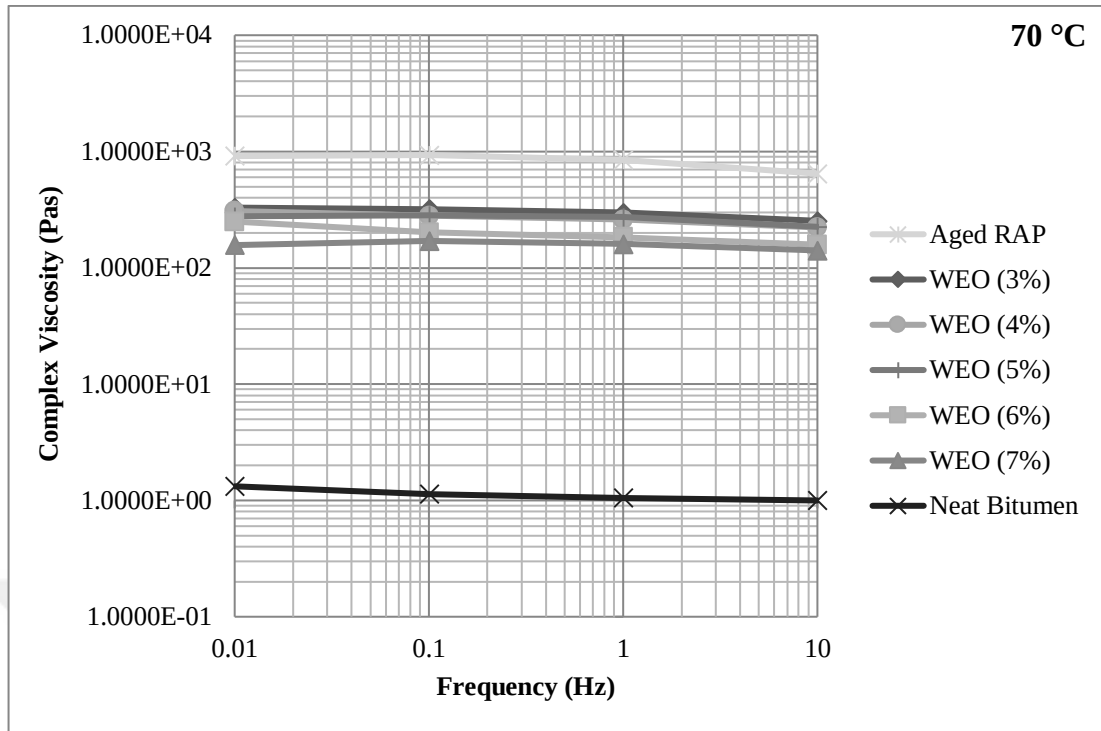


Figure 4.29 WEO rejuvenated samples complex viscosities at 70°C

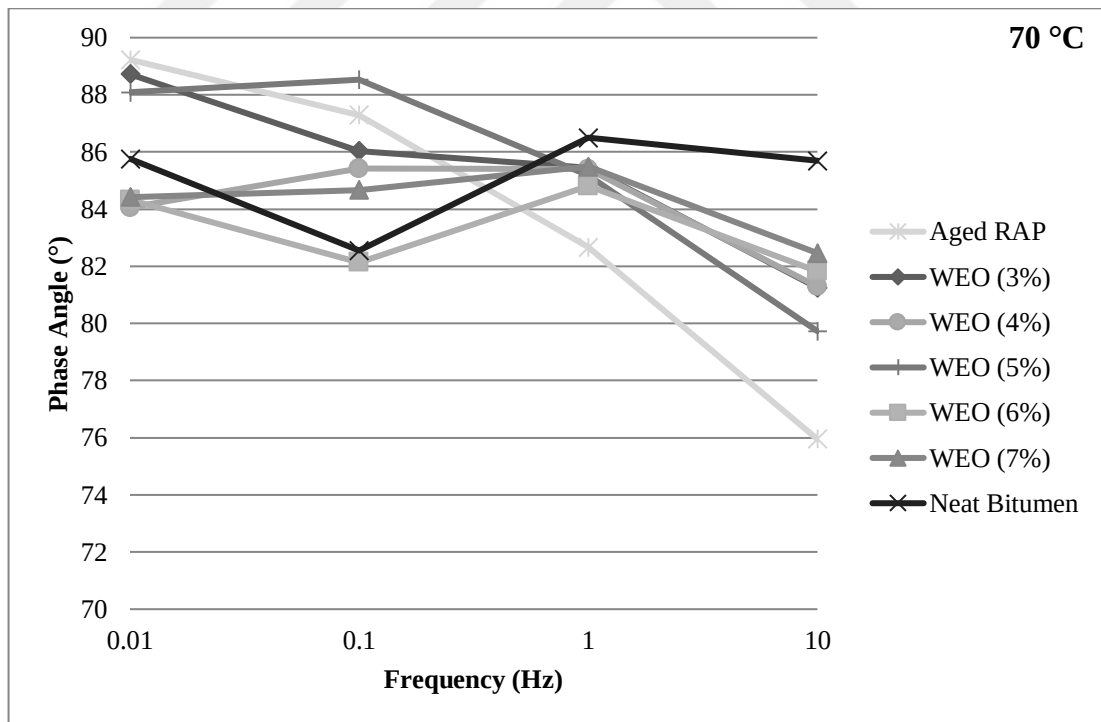


Figure 4.30 WEO rejuvenated samples phase angles at 70°C

4.1.2.2 Dynamic Shear Rheometer Results of WVO

The tested additive contents of WVO within the scope of this study were 3 to 7 percent of rejuvenating agent by weight of RAP aged bitumen like WEO rejuvenating agent. The extracted aged RAP bitumen was rejuvenated by adding the selected range of WVO.

The rheological behavior of all WVO rejuvenated bitumen samples were evaluated using DSR the same way experienced for WEO agent. Like WEO, all samples were tested at temperatures ranging from 40°C, 50°C, 60°C and 70°C in various loading frequencies using frequency sweep.

The results for all WVO rejuvenated samples are given in the Appendix for 0.01 Hz, 0.1 Hz, 1 Hz and 10 Hz. Like the previous tables for WEO, these tables also include the results for neat bitumen and aged RAP bitumen to provide comparisons with neat bitumen and to check the effects of WVO rejuvenating agent on aged RAP bitumen. $G^*/\sin(\delta)$ parameters were calculated for all samples at different temperatures and frequencies within the tables.

All test results are plotted for each frequency level and each temperature to investigate the rheological characteristics of WVO rejuvenated bitumen samples in different frequencies and different temperatures.

Figures 4.31 to 4.33, respectively, exhibit the plotted test results for rutting parameters, complex viscosities and phase angles of WVO rejuvenated bitumen samples at 0.01 Hz frequency level. Like figures for WEO, all results have been plotted in logarithmic scale on Y axis to help better demonstration of the results.

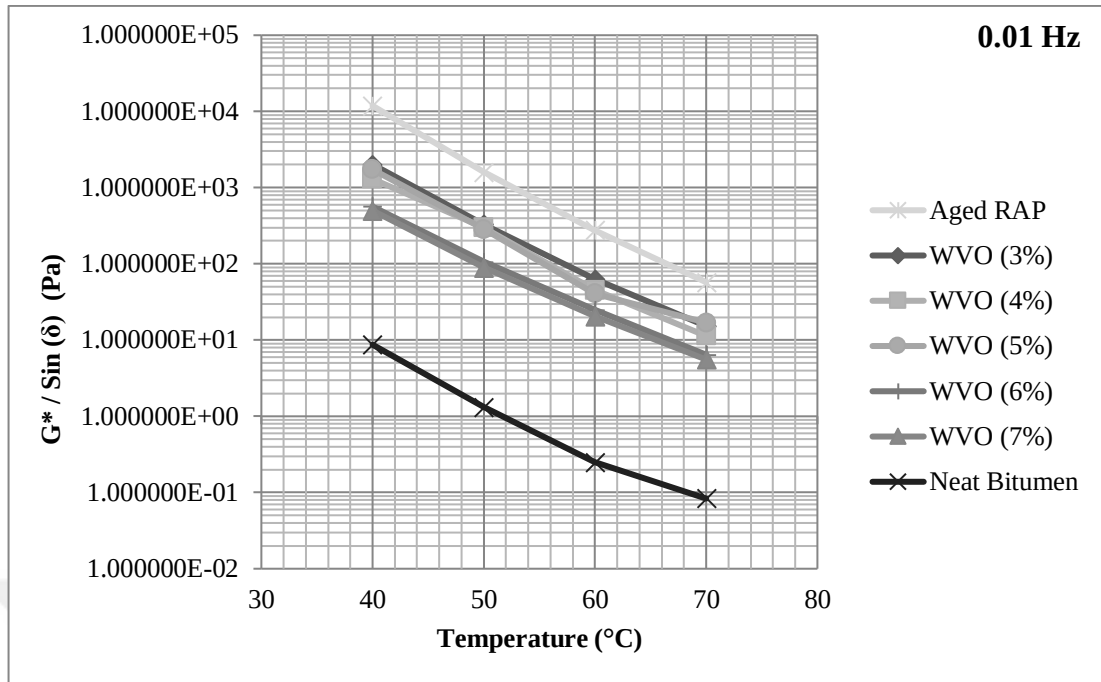


Figure 4.31 WVO rejuvenated samples rutting parameters at 0.01 Hz frequency

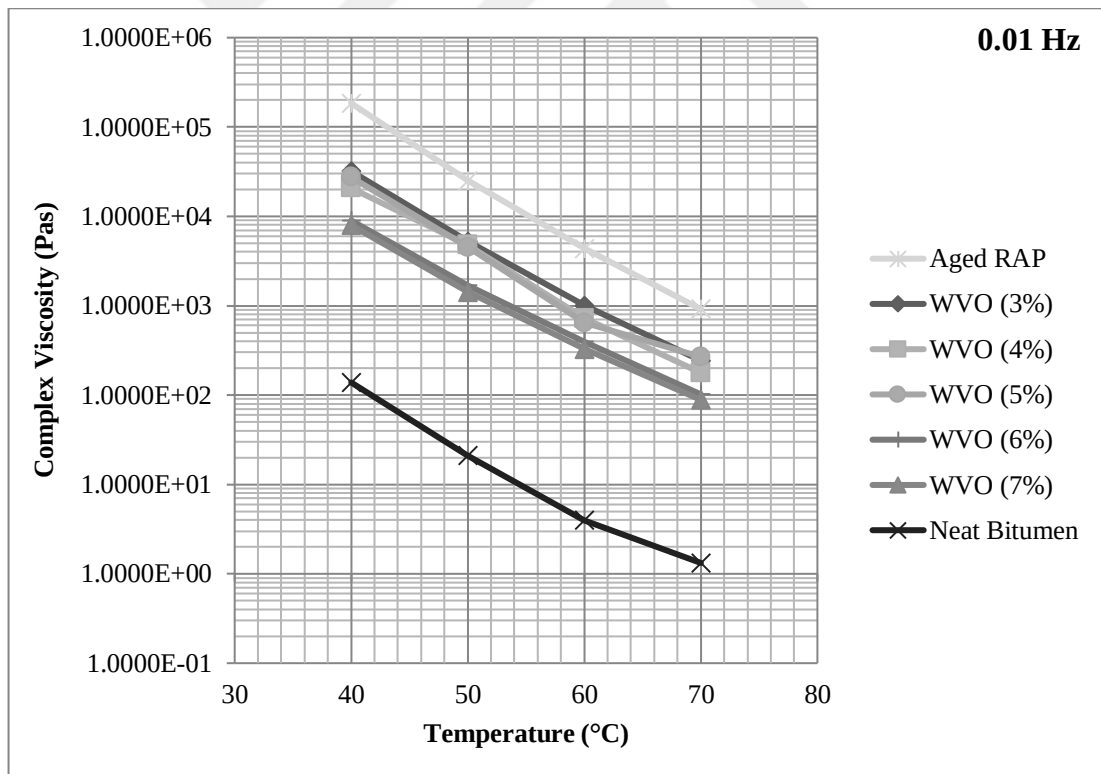


Figure 4.32 WVO rejuvenated samples complex viscosities at 0.01 Hz frequency

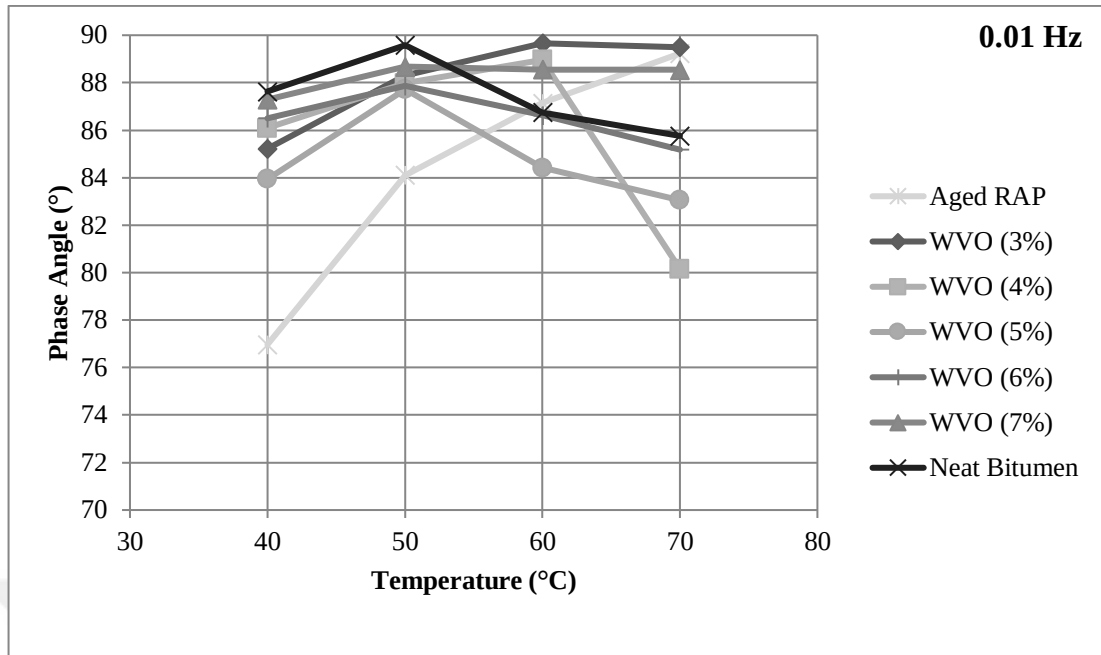


Figure 4.33 WVO rejuvenated samples phase angles at 0.01 Hz frequency

Figures 4.34 to 4.36, respectively, present the plotted test results for rutting parameters, complex viscosities and phase angles of WVO rejuvenated bitumen samples at 0.1 Hz frequency level.

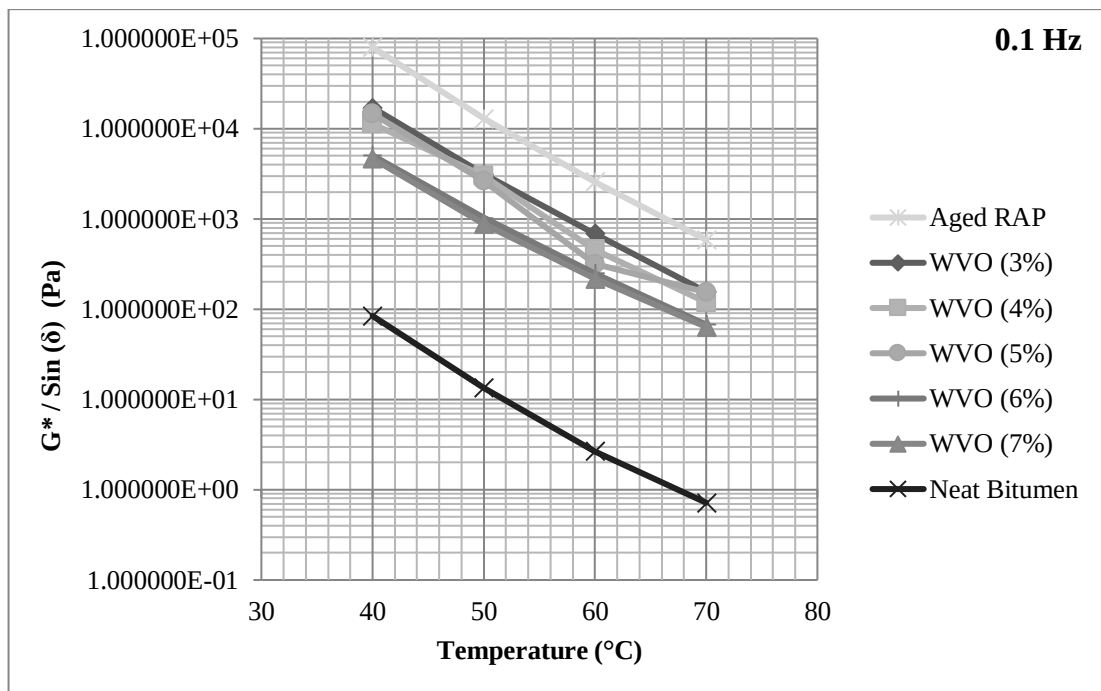


Figure 4.34 WVO rejuvenated samples rutting parameters at 0.1 Hz frequency

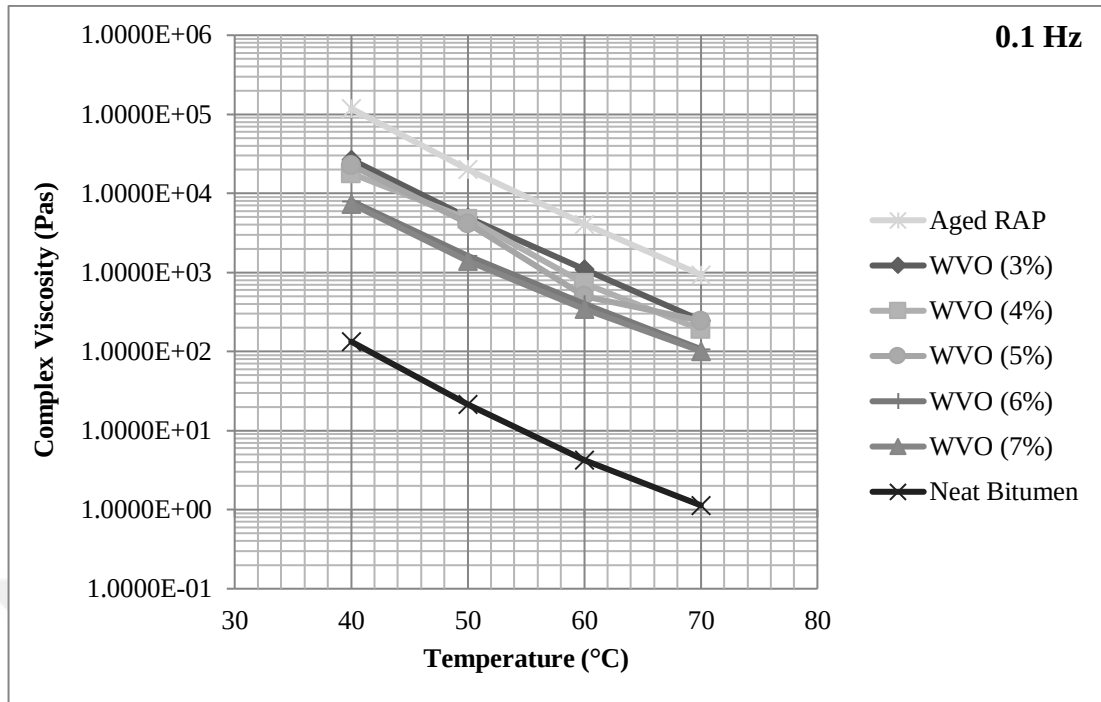


Figure 4.35 WVO rejuvenated samples complex viscosities at 0.1 Hz frequency

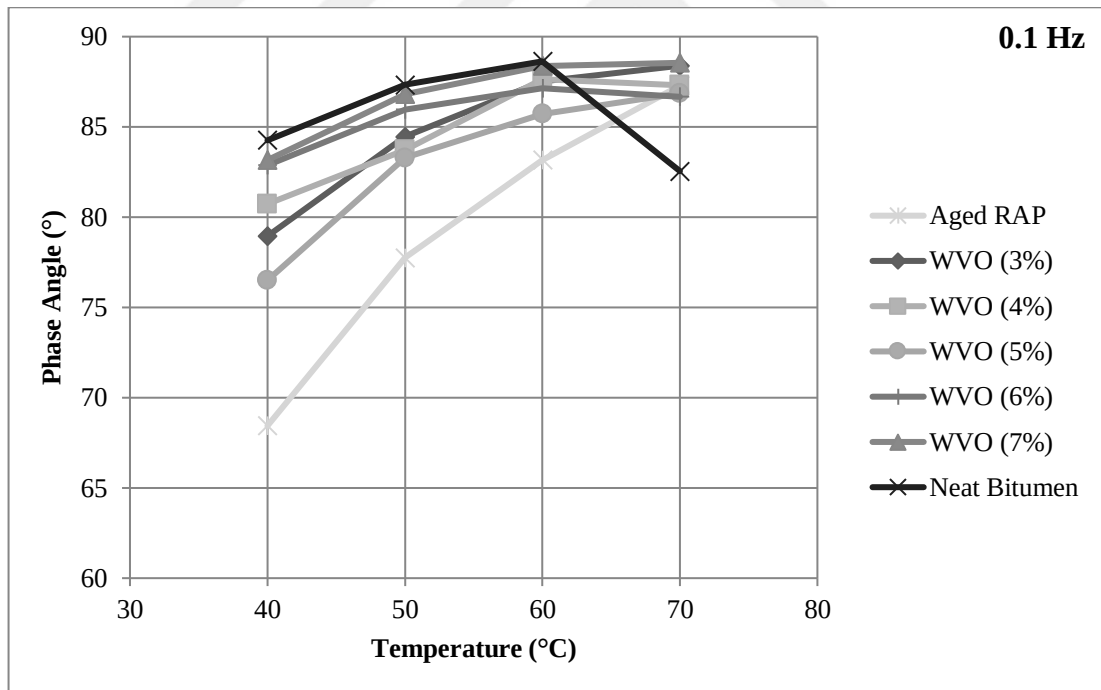


Figure 4.36 WVO rejuvenated samples phase angles at 0.1 Hz frequency

Figures 4.37 to 4.39, respectively, present the plotted test results for rutting parameters, complex viscosities and phase angles of WVO rejuvenated bitumen samples at 1 Hz frequency level.

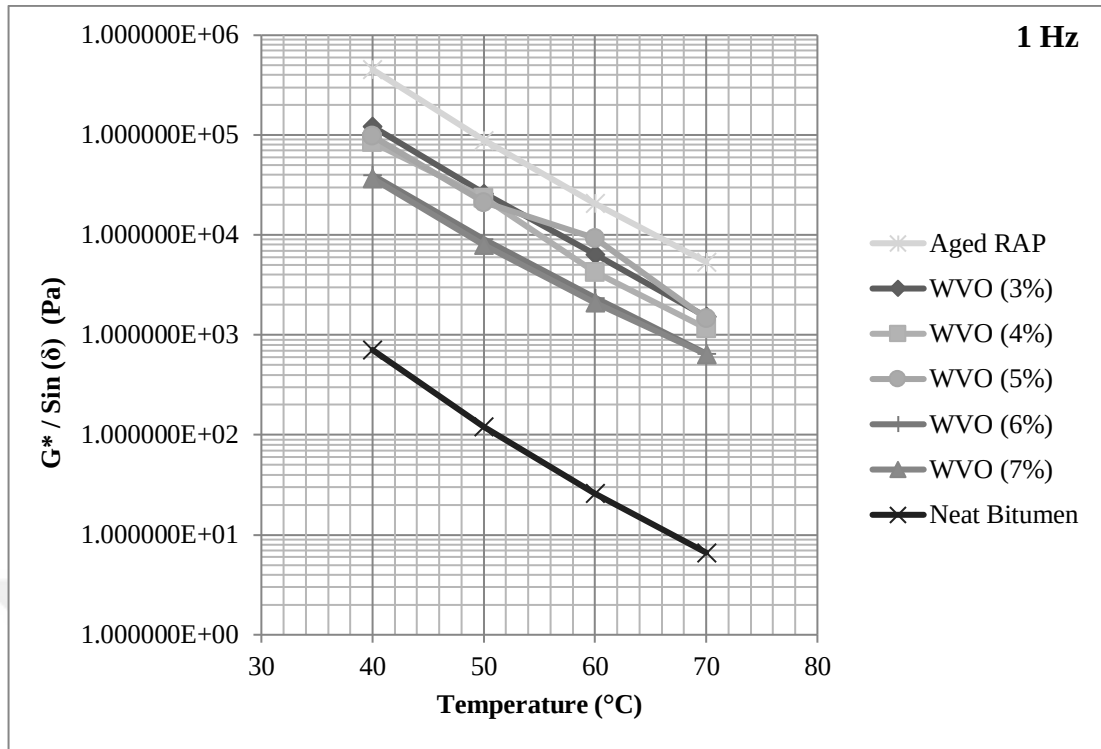


Figure 4.37 WVO rejuvenated samples rutting parameters at 1 Hz frequency

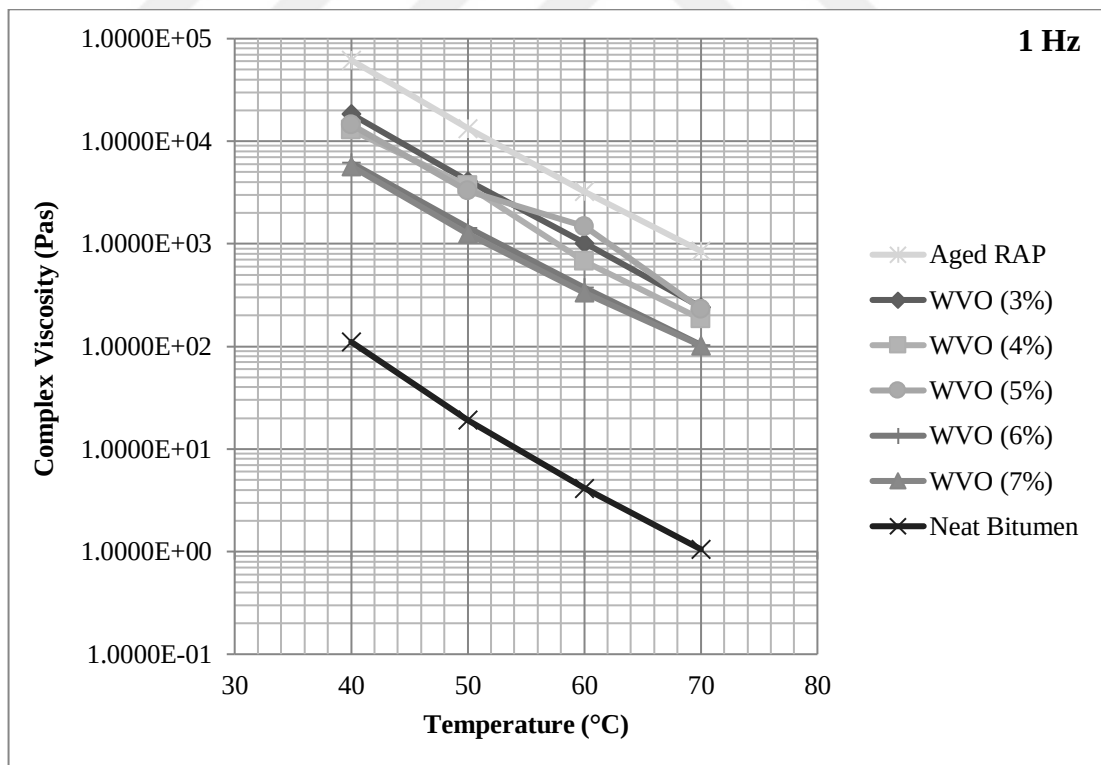


Figure 4.38 WVO rejuvenated samples complex viscosities at 1 Hz frequency

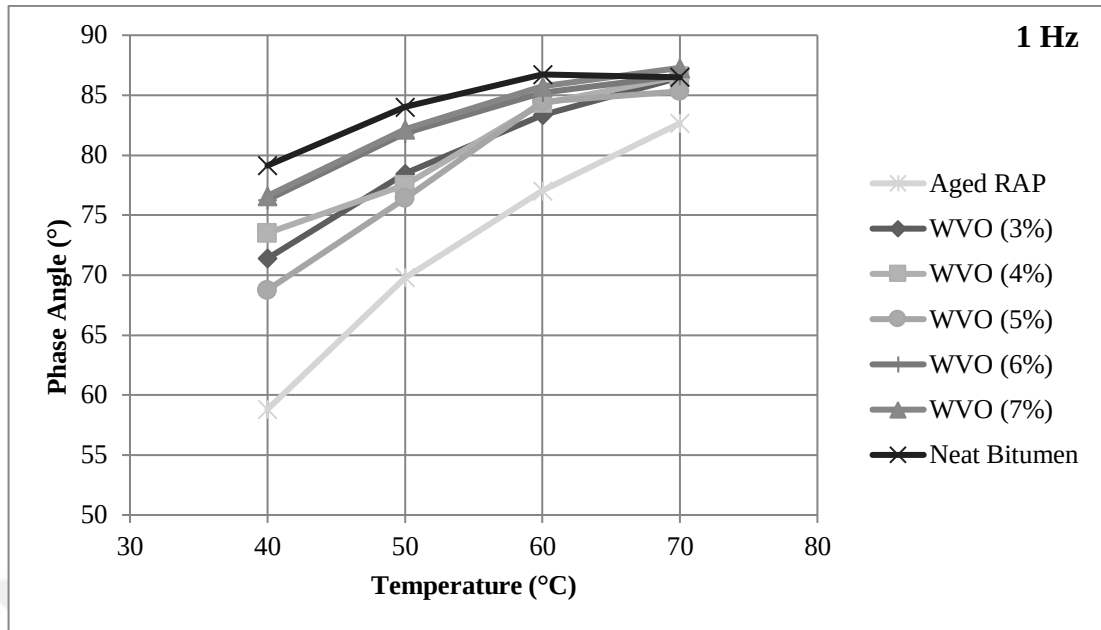


Figure 4.39 WVO rejuvenated samples phase angles at 1 Hz frequency

Figures 4.40 to 4.42, respectively, present the plotted test results for rutting parameters, complex viscosities and phase angles of WVO rejuvenated bitumen samples at 10 Hz frequency level.

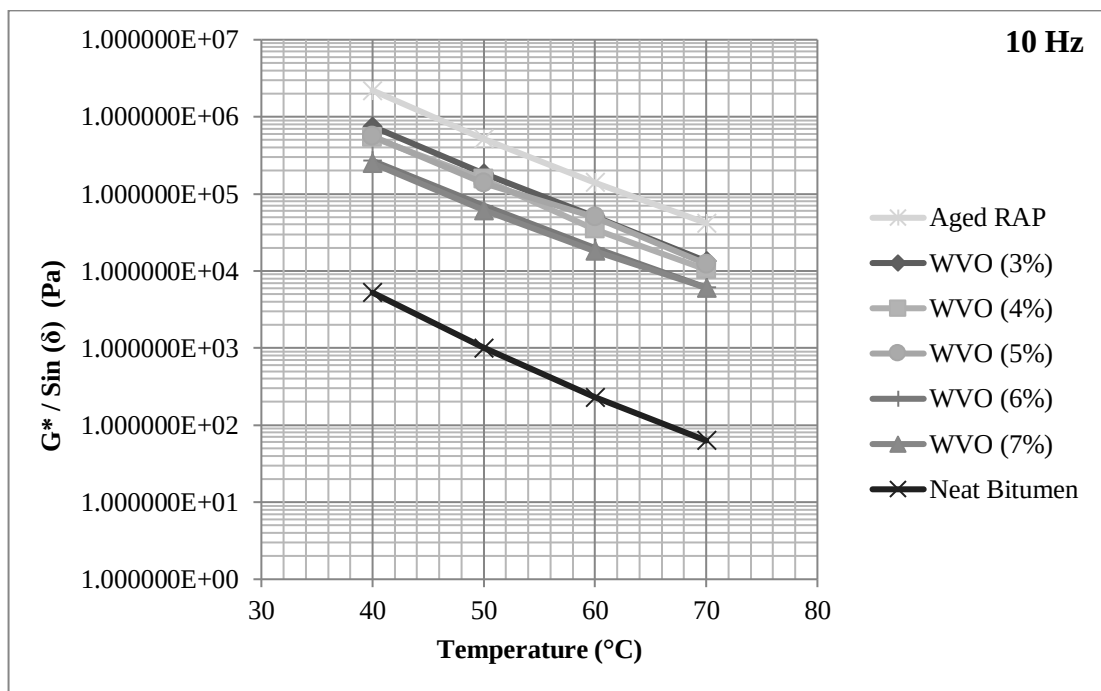


Figure 4.40 WVO rejuvenated samples rutting parameters at 10 Hz frequency

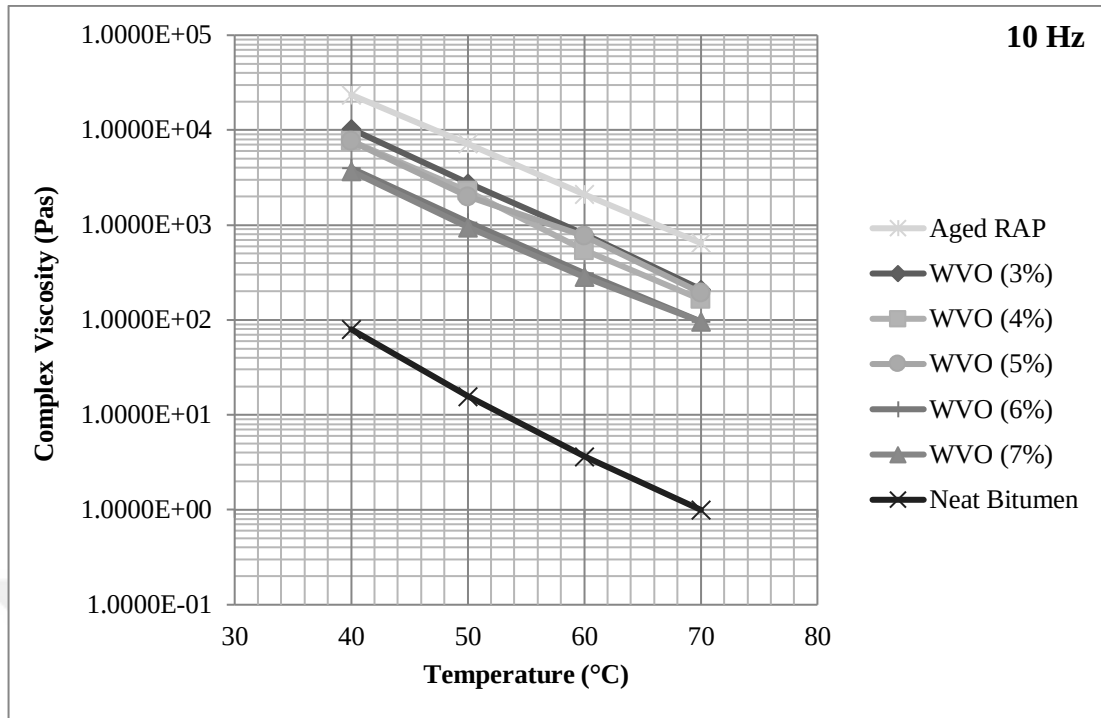


Figure 4.41 WVO rejuvenated samples complex viscosities at 10 Hz frequency

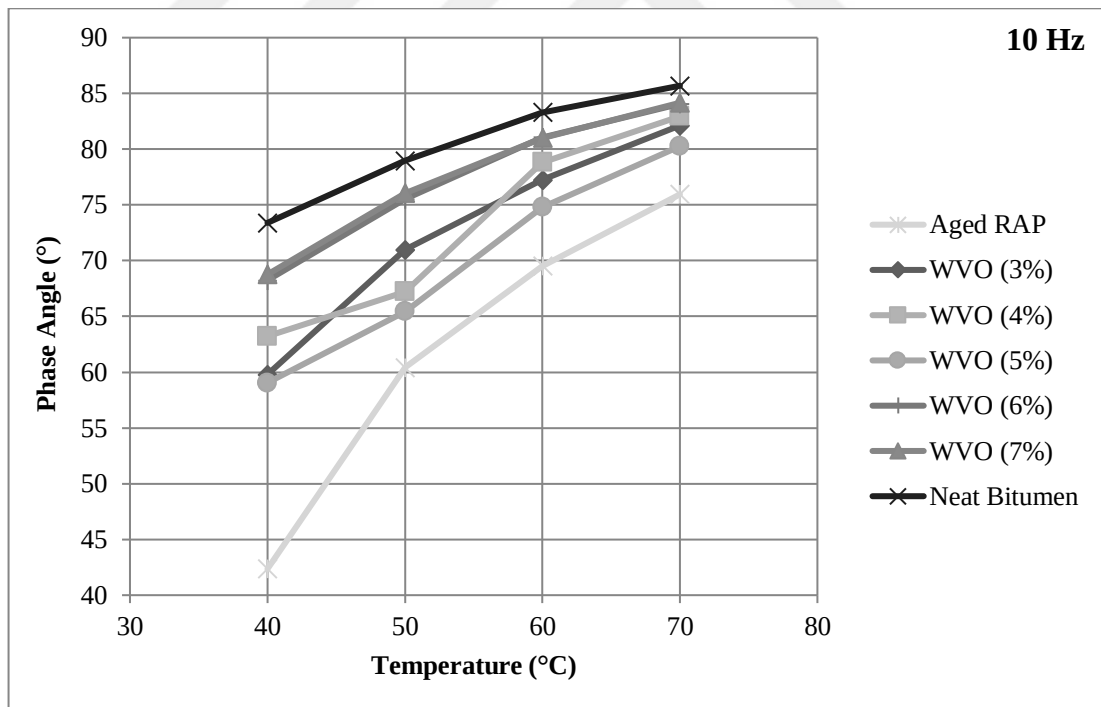


Figure 4.42 WVO rejuvenated samples phase angles at 10 Hz frequency

As can be seen in Figures 4.31 to 4.42, WVO rejuvenating agent has a very similar effect like WEO on aged RAP bitumen. WVO agent can soften the hardened aged RAP

bitumen and regain visco-elasticity to the bitumen. The typical behavior of bitumen samples for rutting parameters, complex viscosities and phase angles described for WEO rejuvenated bitumen samples, were observed for WVO rejuvenated bitumen samples. The comparisons between the rejuvenating agents are made within the section 4.2.1.4 (Rheological Comparison of Rejuvenating Agents). In this section we will see that the WEO and WVO agents act very similar in terms of rejuvenation.

Figures 4.43 to 4.54 show the demonstration of the same results at different temperatures (each figure at a specific temperature) aiming to conceive the outcome of increase in frequency level at X axis. In this way we can see the results for different frequency levels at each figure. As can be seen in Figures 4.43 to 4.54, the rutting parameters increase by increase in frequency level at all temperatures. The complex viscosities drop very slightly in logarithmic scale for each 10 times rank of frequency. As shown on all figures, the phase angles decrease as the bitumen normally behaves more elastic than viscous at lower temperature and higher frequency levels.

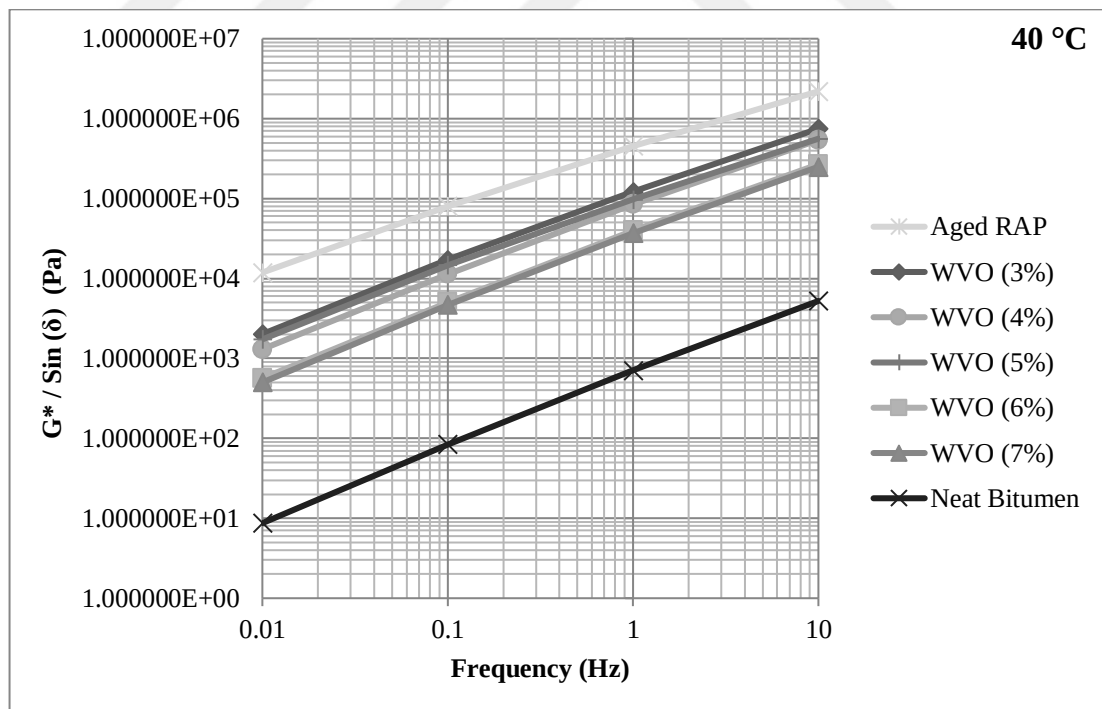


Figure 4.43 WVO rejuvenated samples rutting parameters at 40°C

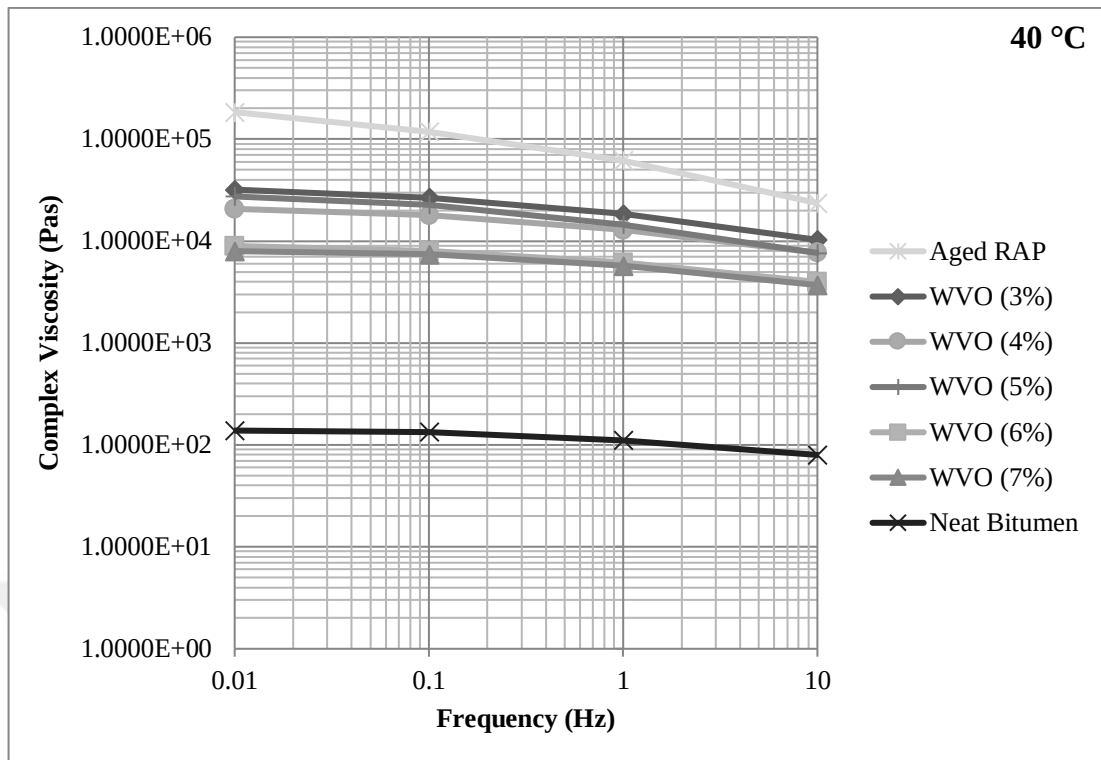


Figure 4.44 WVO rejuvenated samples complex viscosities at 40°C

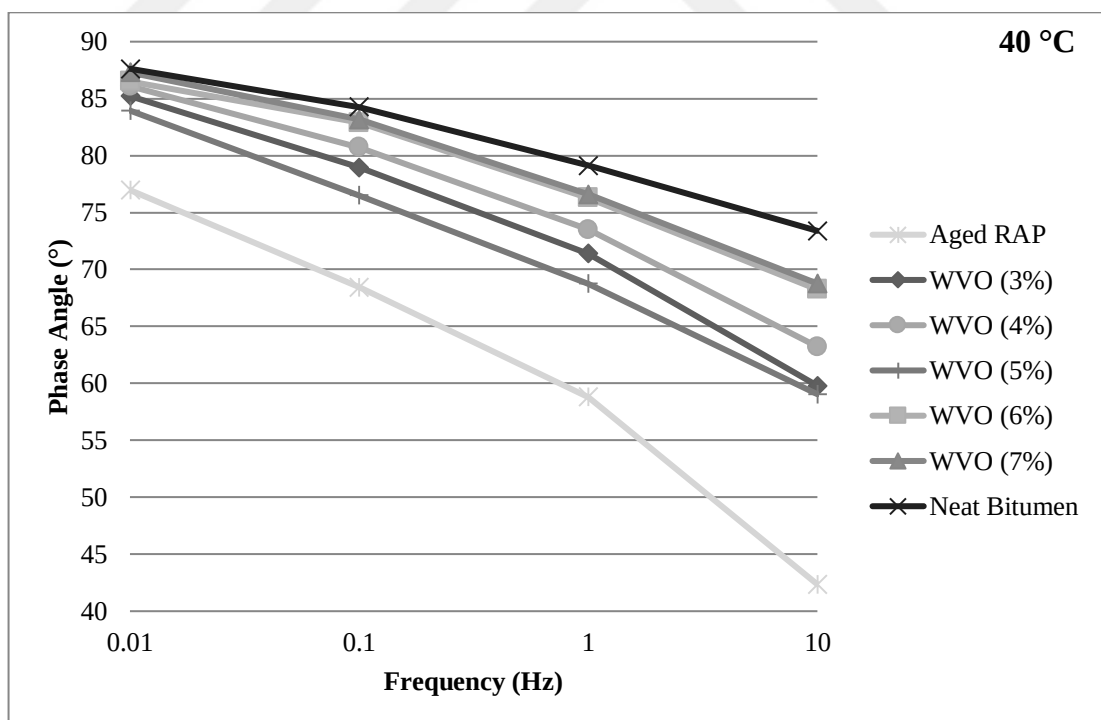


Figure 4.45 WVO rejuvenated samples phase angles at 40°C

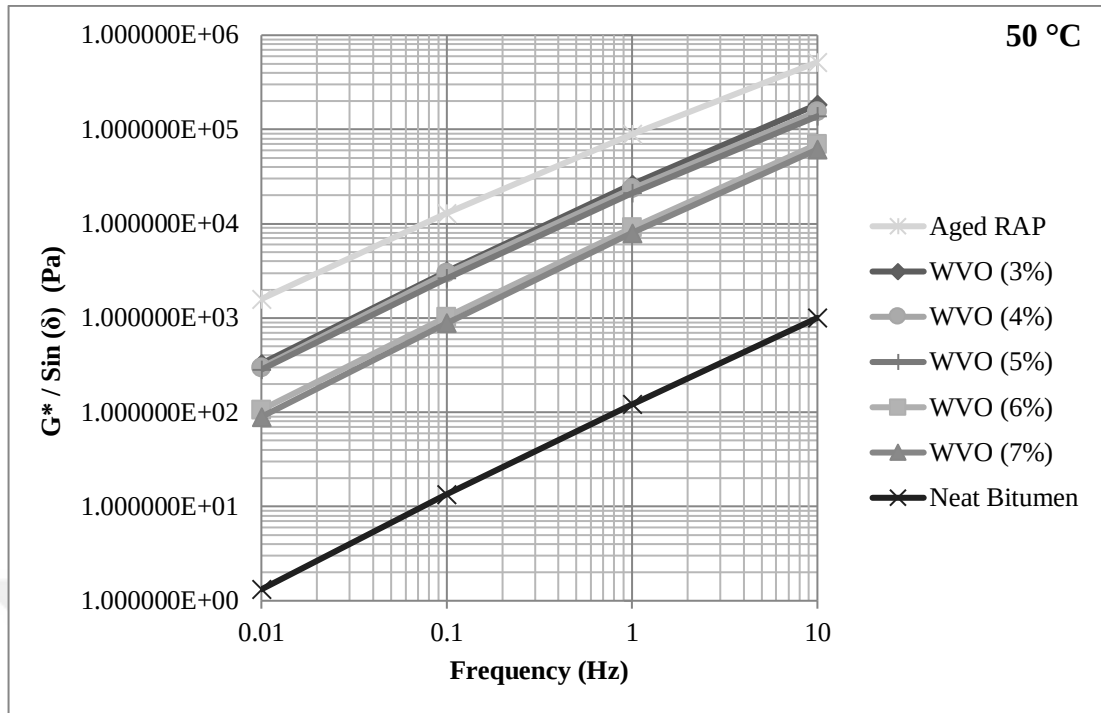


Figure 4.46 WVO rejuvenated samples rutting parameters at 50°C

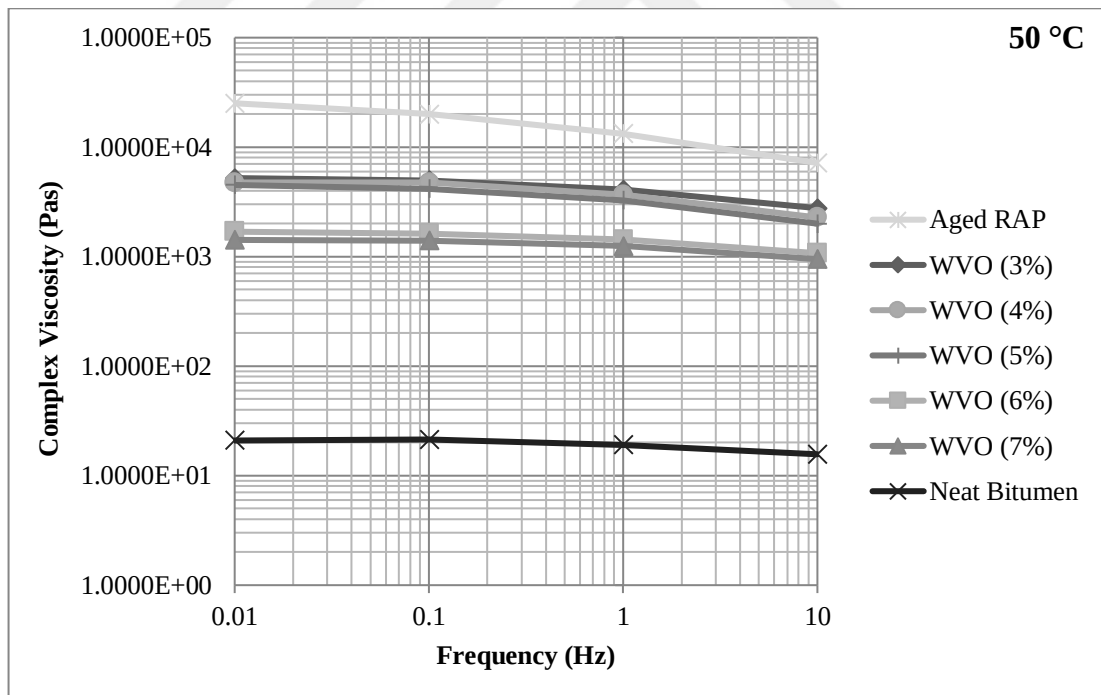


Figure 4.47 WVO rejuvenated samples complex viscosities at 50°C

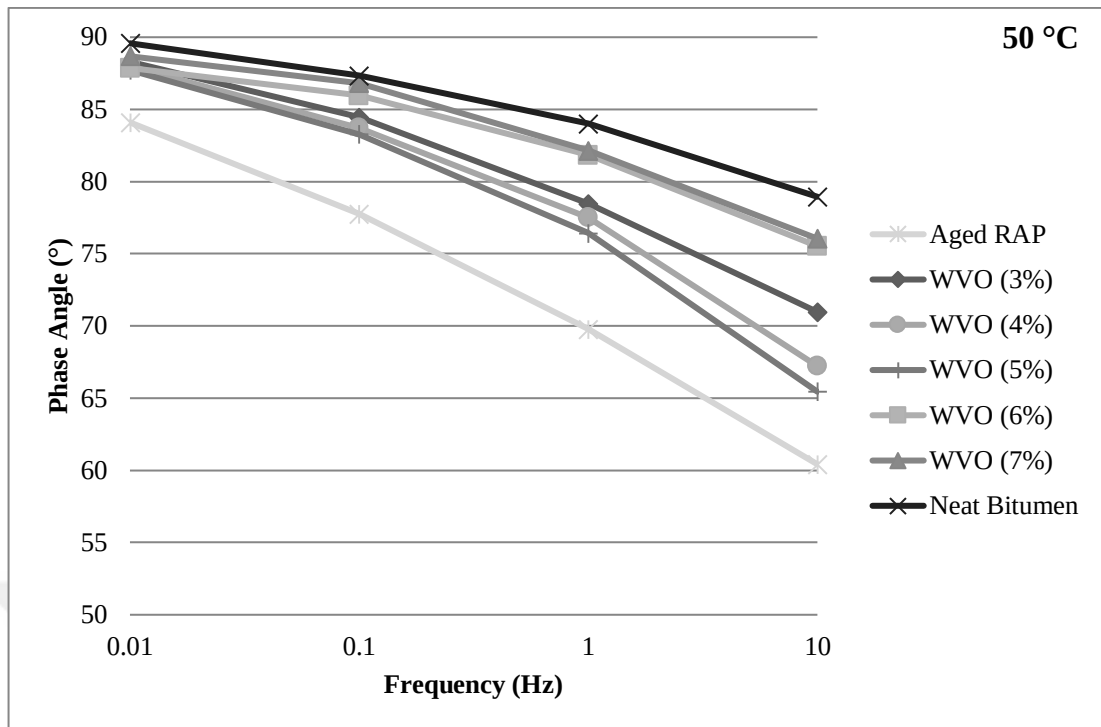


Figure 4.48 WVO rejuvenated samples phase angles at 50°C

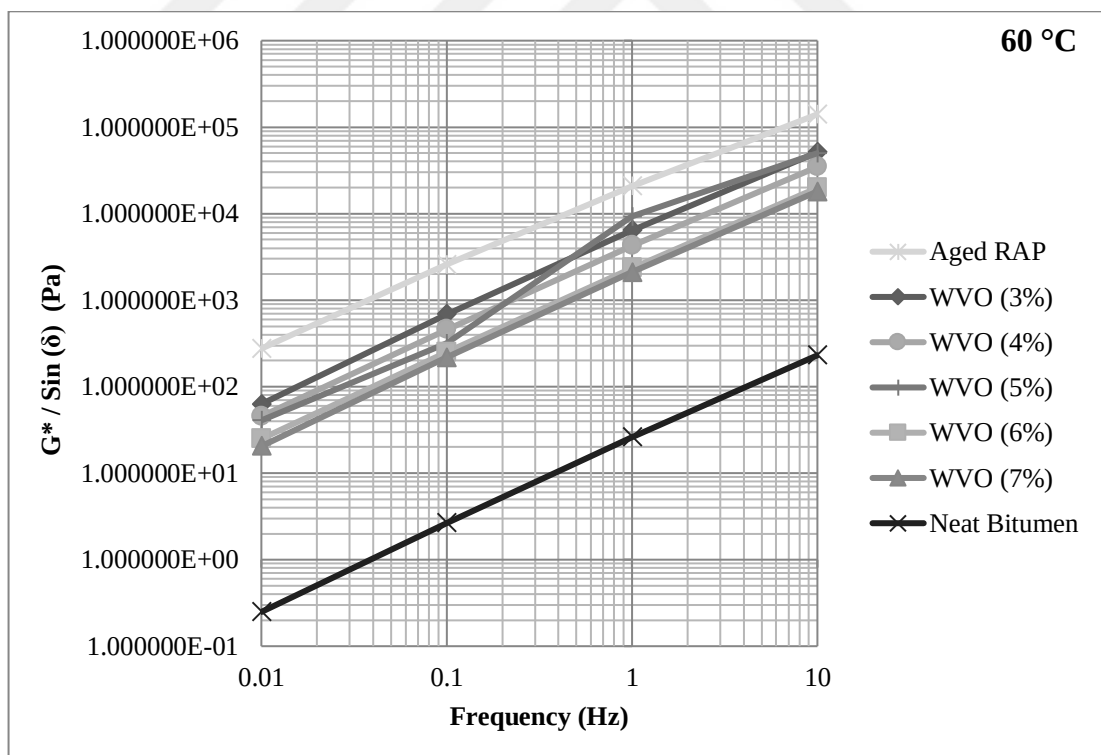


Figure 4.49 WVO rejuvenated samples rutting parameters at 60°C

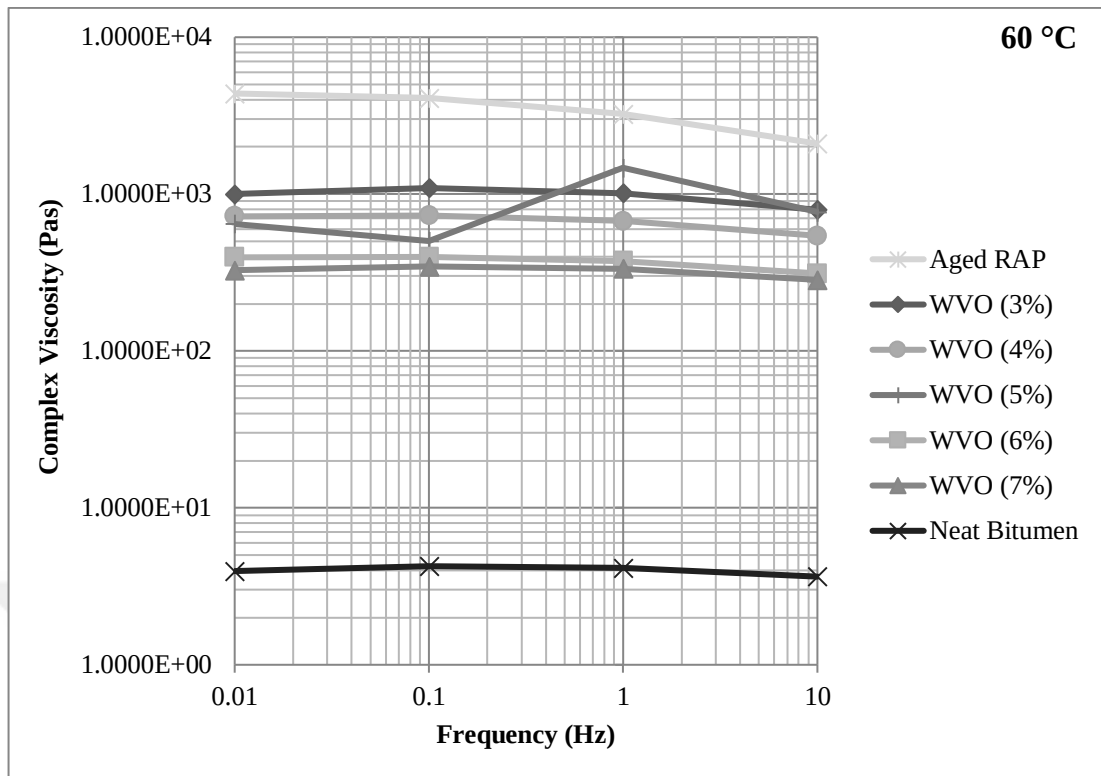


Figure 4.50 WVO rejuvenated samples complex viscosities at 60°C

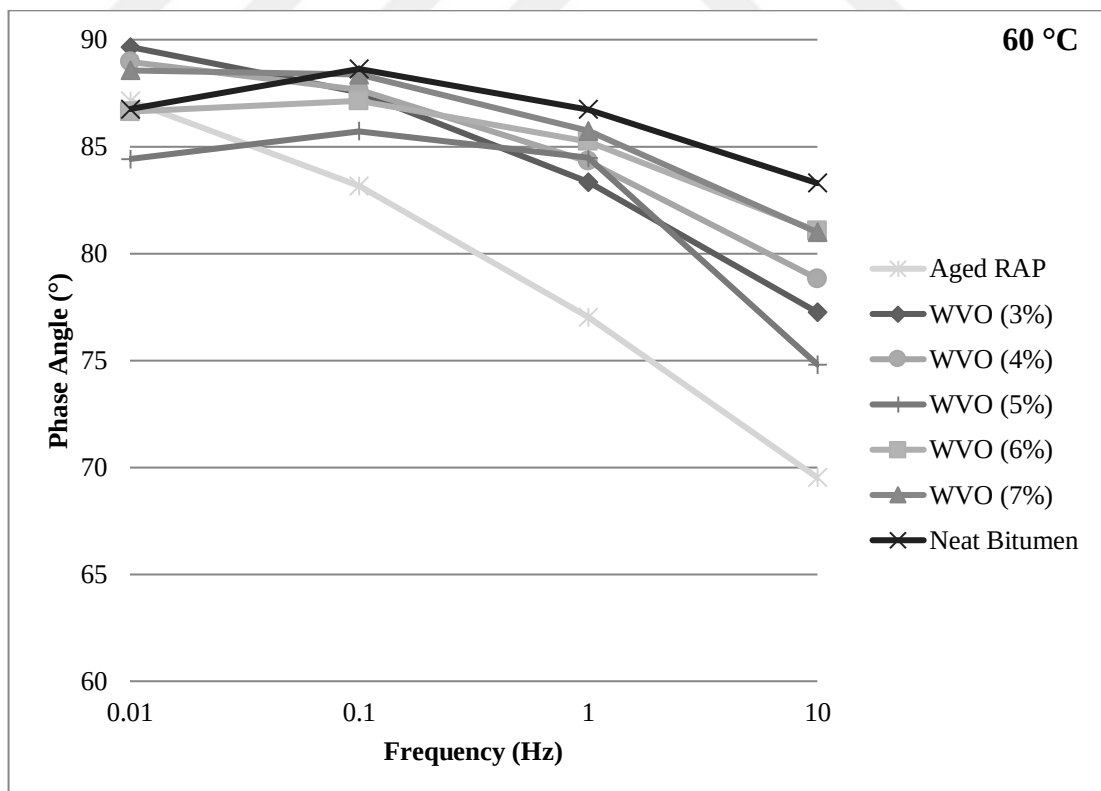


Figure 4.51 WVO rejuvenated samples phase angles at 60°C

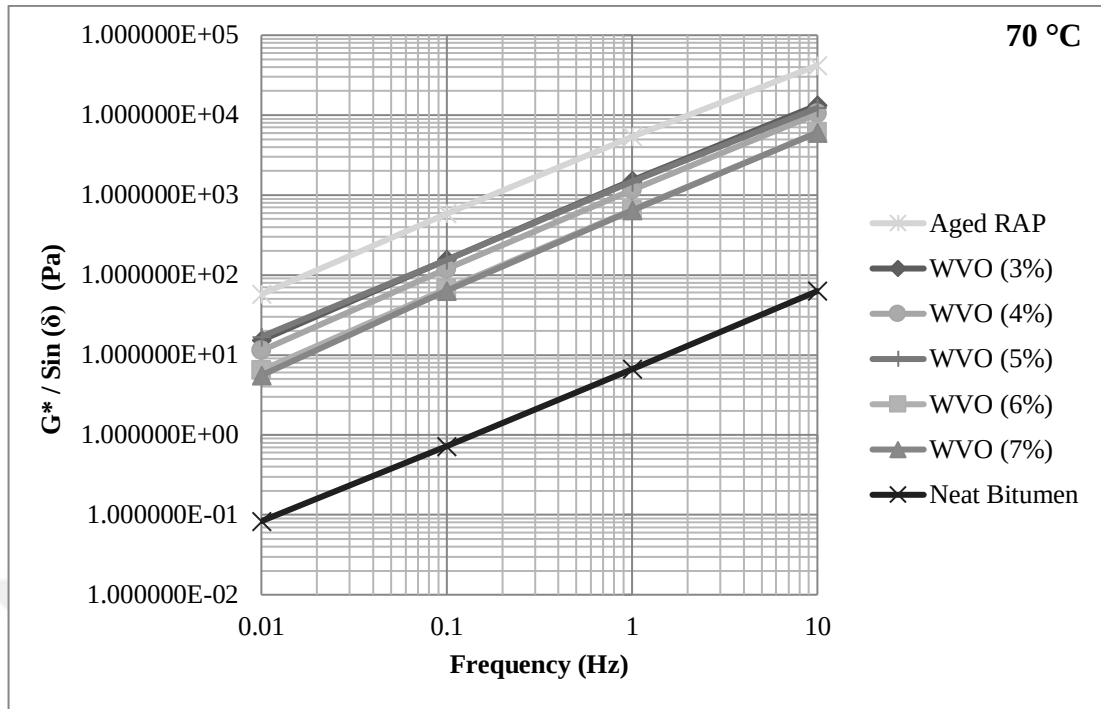


Figure 4.52 WVO rejuvenated samples rutting parameters at 70°C

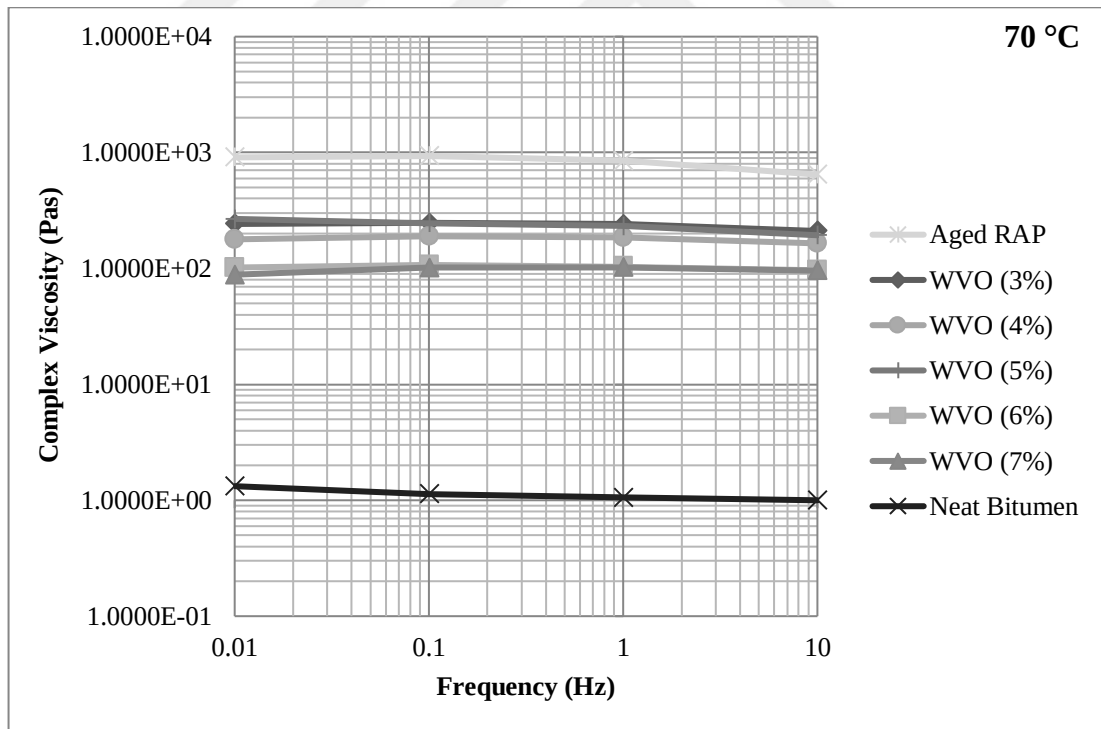


Figure 4.53 WVO rejuvenated samples complex viscosities at 70°C

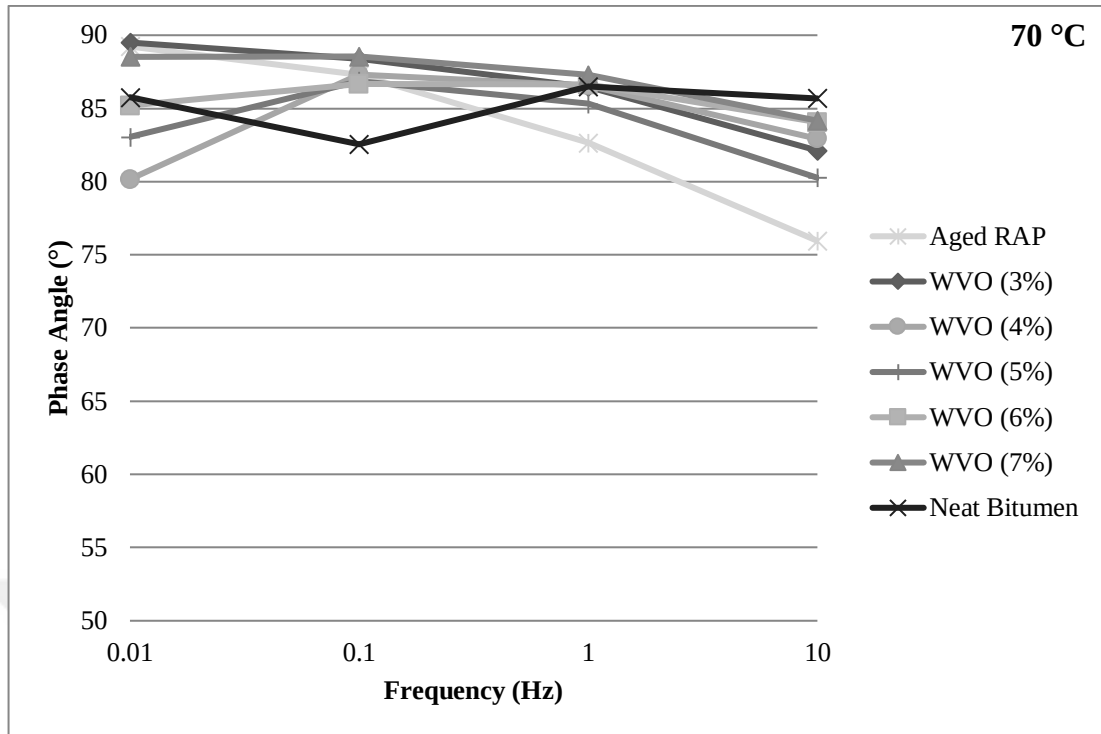


Figure 4.54 WVO rejuvenated samples phase angles at 70°C

4.1.2.3 Dynamic Shear Rheometer Results of CRA

The results for commercial agent are presented within this section. The manufacturing company suggested 7.5% of CRA for 50-70 penetration grading bitumen for optimum results. After testing the recommended content in terms of penetration, the contents selected for DSR testing were determined as 5.5%, 6.5%, 7.5%, 8.5% and 9.5% for CRA.

CRA as a commercial product was found a very effective product in terms of rejuvenation. By employing this agent, almost most visco-elastic characteristics of aged RAP bitumen were regained. This agent can soften the hardened aged RAP bitumen as to neat bitumen levels and the complex viscosity and phase angles values improved up to the neat bitumen values levels.

The Appendix consists of the DSR testing results recorded for all CRA rejuvenated and non-rejuvenated bitumen samples (including aged RAP bitumen and neat bitumen).

Figures 4.55 to 4.66 present the results for rutting parameters, complex viscosities and phase angles plotted for each frequency level at different temperatures. The results show that the CRA has an impressive effect on rheological properties of aged RAP bitumen. Typically, the rutting parameters decrease by increasing the temperature for every frequency level. The complex viscosities also show a very similar behavior to rutting parameters in terms of increasing the temperature for every frequency level.

Phage angles at lower frequency levels and higher temperatures are not recognizable as described before, since at higher frequencies and lower temperature levels, the phase angles are more distinguishable and the effect of CRA on visco-elastic characteristics of aged RAP bitumen becomes obvious. The CRA improves the rheological characteristics of aged RAP bitumen and treats visco-elastic behavior of aged RAP bitumen by helping it representing behavior like neat bitumen.

The differences between neat bitumen results and CRA rejuvenated results are obvious for the commercial product in comparison to waste oils. The comparisons between the bitumen samples rejuvenated using optimum rejuvenating agent contents is presented in 4.2.1.4 (Rheological Comparison of Rejuvenating Agents).

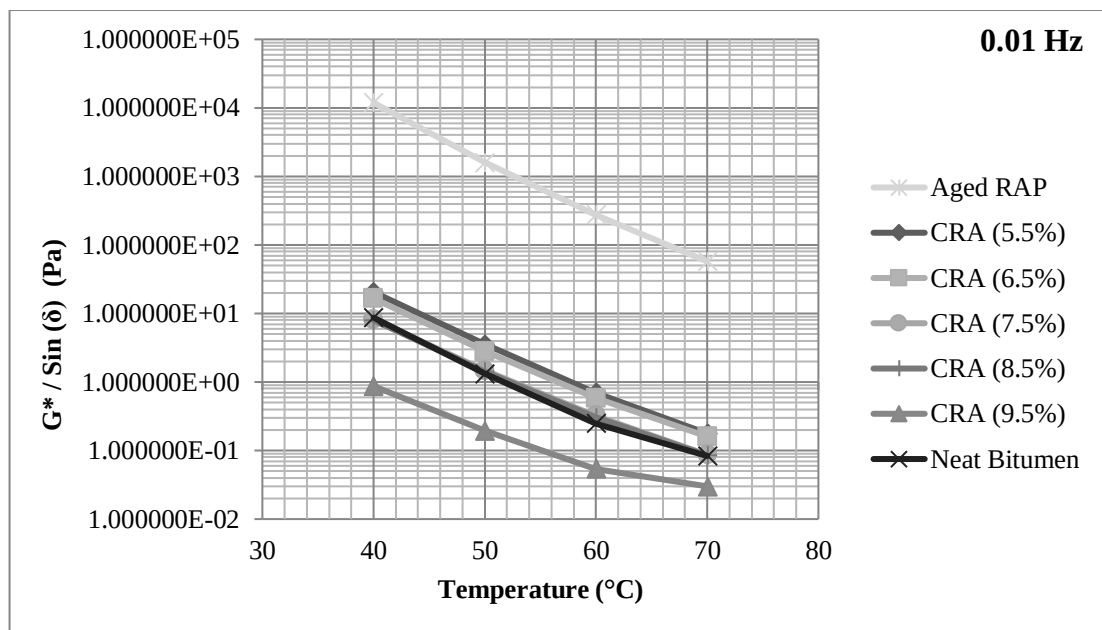


Figure 4.55 CRA rejuvenated samples rutting parameters at 0.01 Hz frequency

As can be seen, the increases in rejuvenating agent content helps aged RAP bitumen become more closer to neat bitumen in terms of all rheological properties. Since the increase of rejuvenating agent may have negative effects on the conventional properties of rejuvenated bitumen samples, 7.5% seem to be adequate for rejuvenation of 50-70 penetration grade aged RAP bitumen.

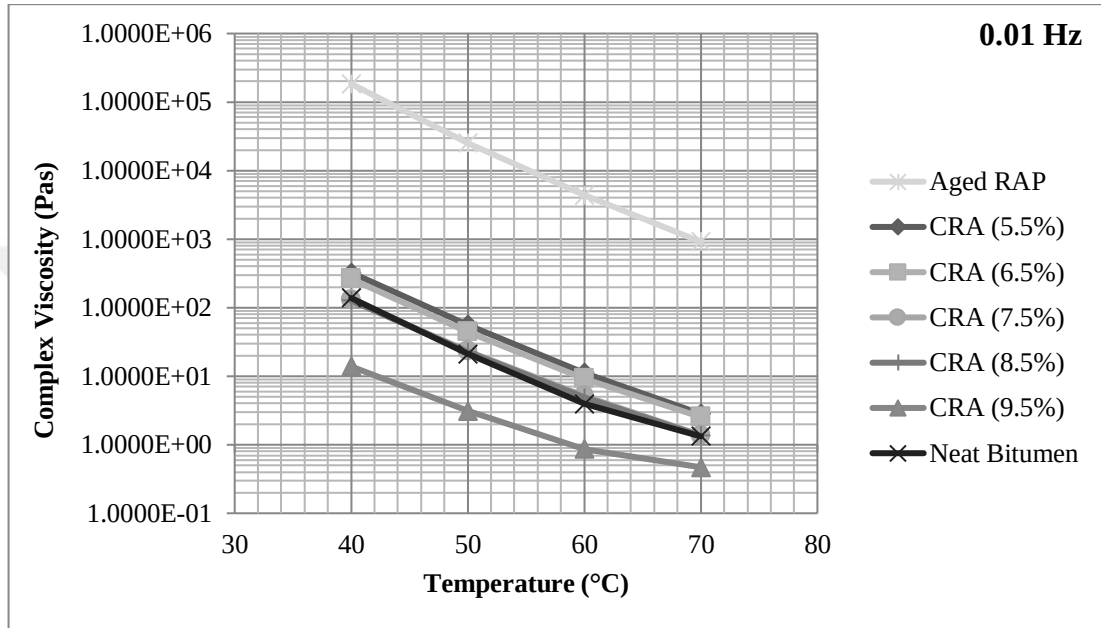


Figure 4.56 CRA rejuvenated samples complex viscosities at 0.01 Hz frequency

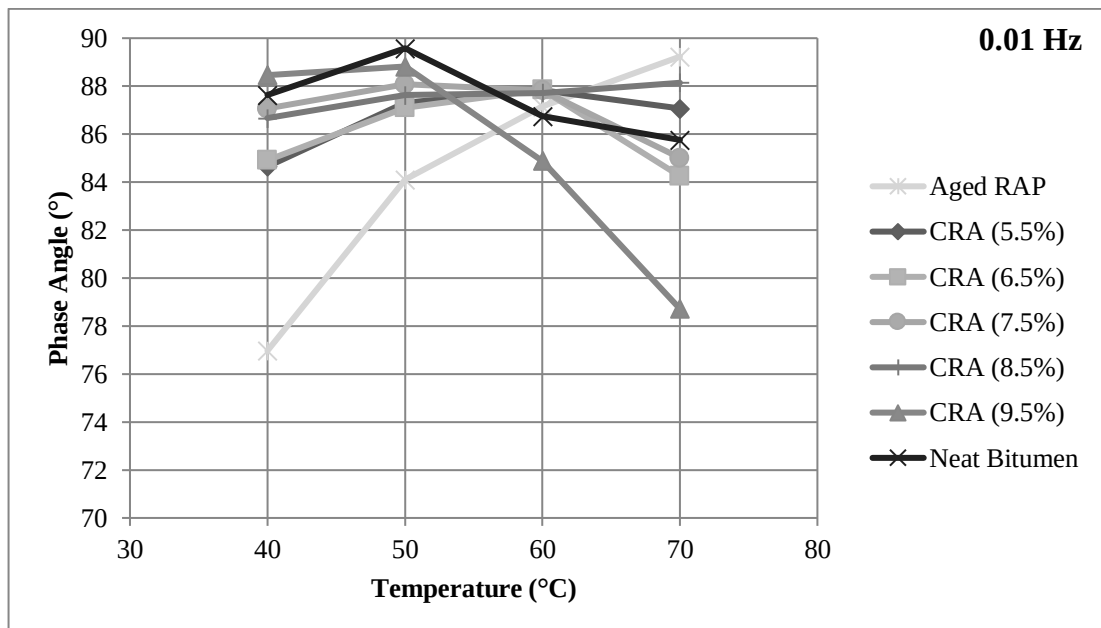


Figure 4.57 CRA rejuvenated samples phase angles at 0.01 Hz frequency

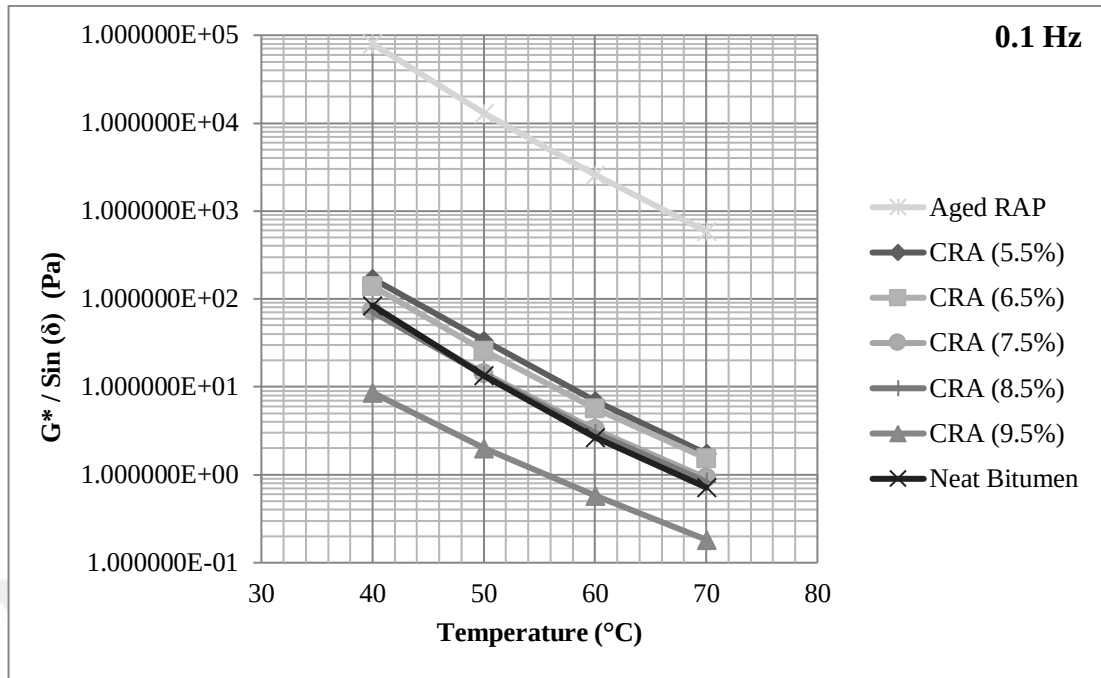


Figure 4.58 CRA rejuvenated samples rutting parameters at 0.1 Hz frequency

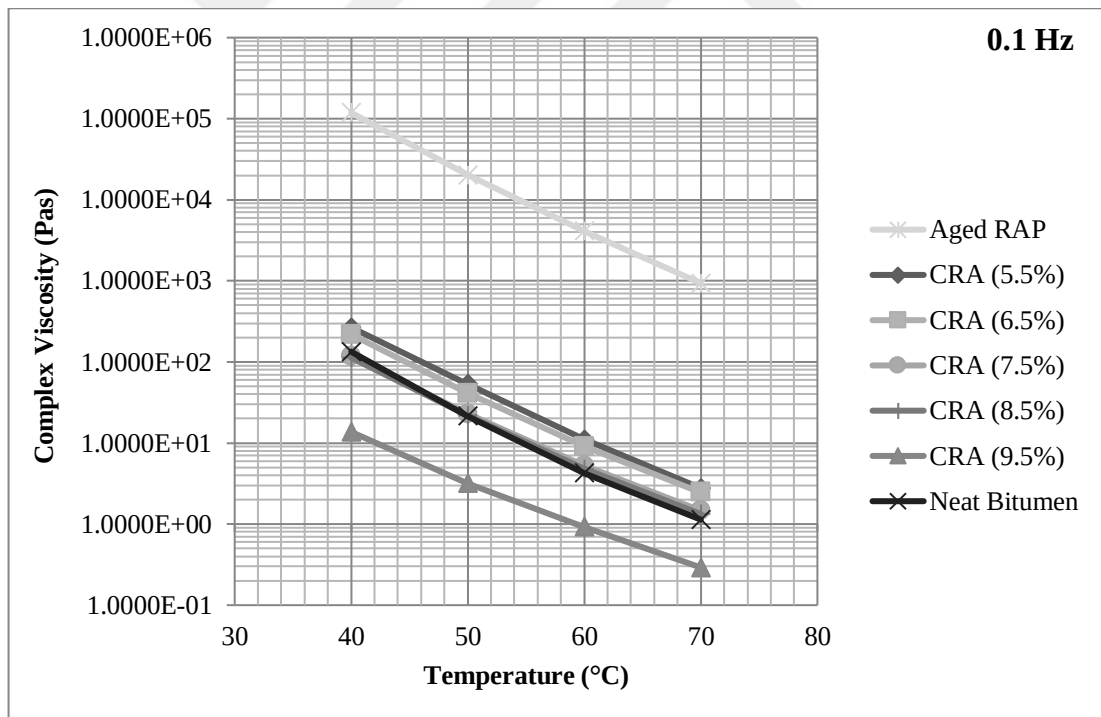


Figure 4.59 CRA rejuvenated samples complex viscosities at 0.1 Hz frequency

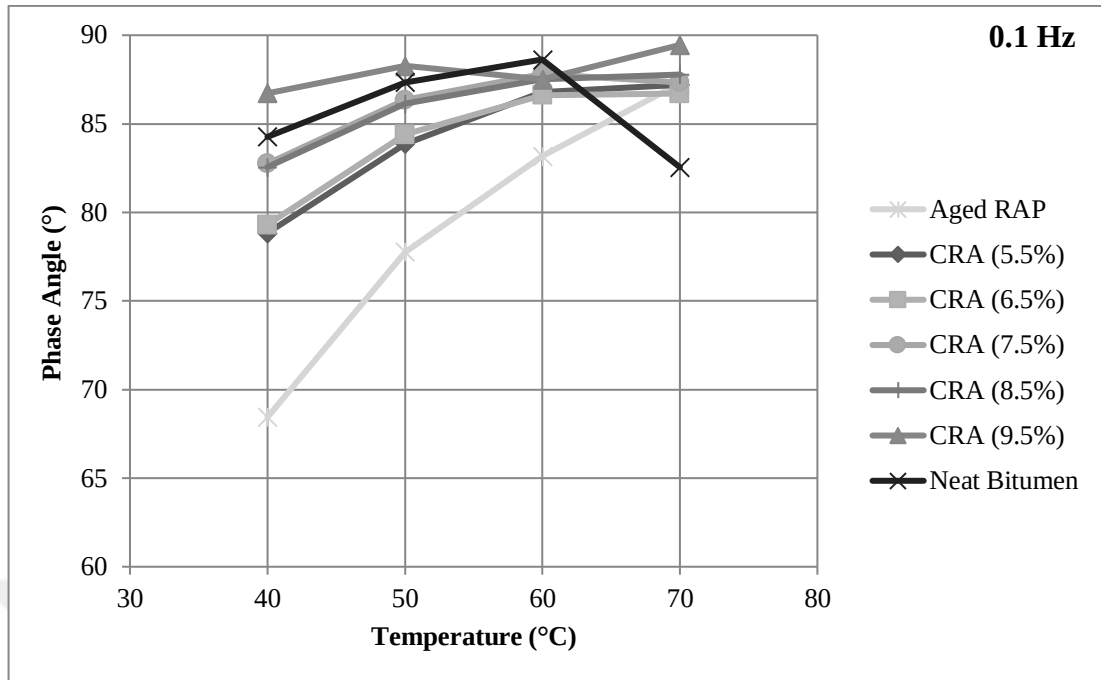


Figure 4.60 CRA rejuvenated samples phase angles at 0.1 Hz frequency

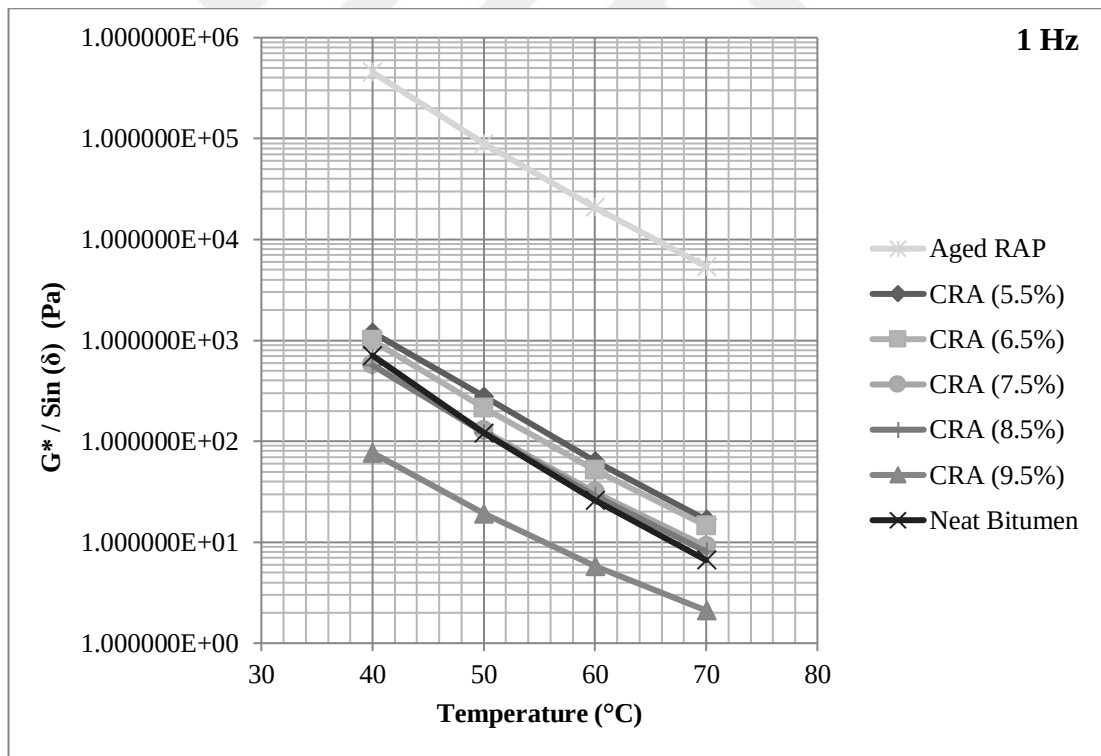


Figure 4.61 CRA rejuvenated samples rutting parameters at 1 Hz frequency

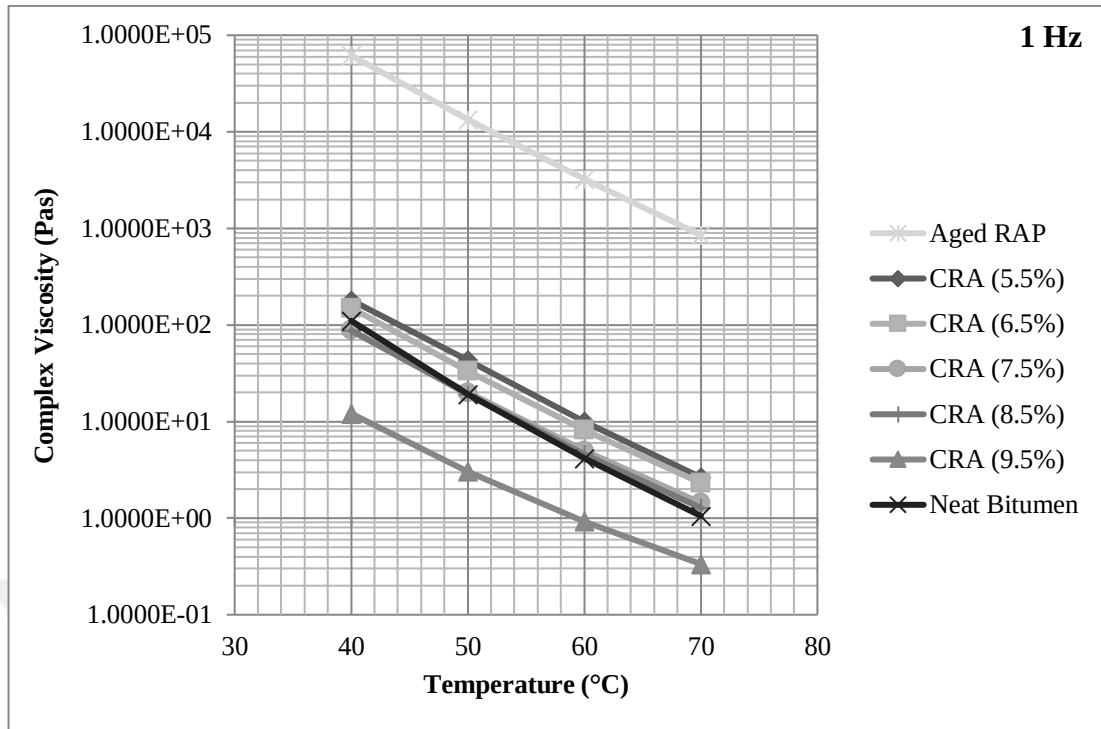


Figure 4.62 CRA rejuvenated samples complex viscosities at 1 Hz frequency

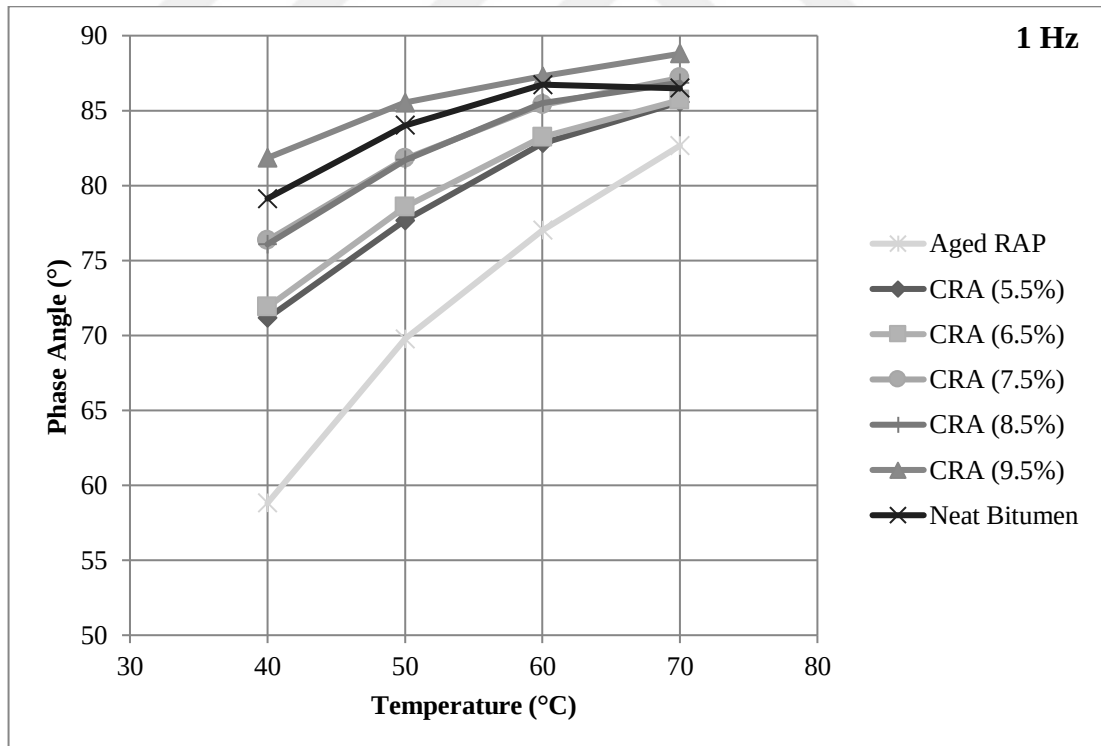


Figure 4.63 CRA rejuvenated samples phase angles at 1 Hz frequency

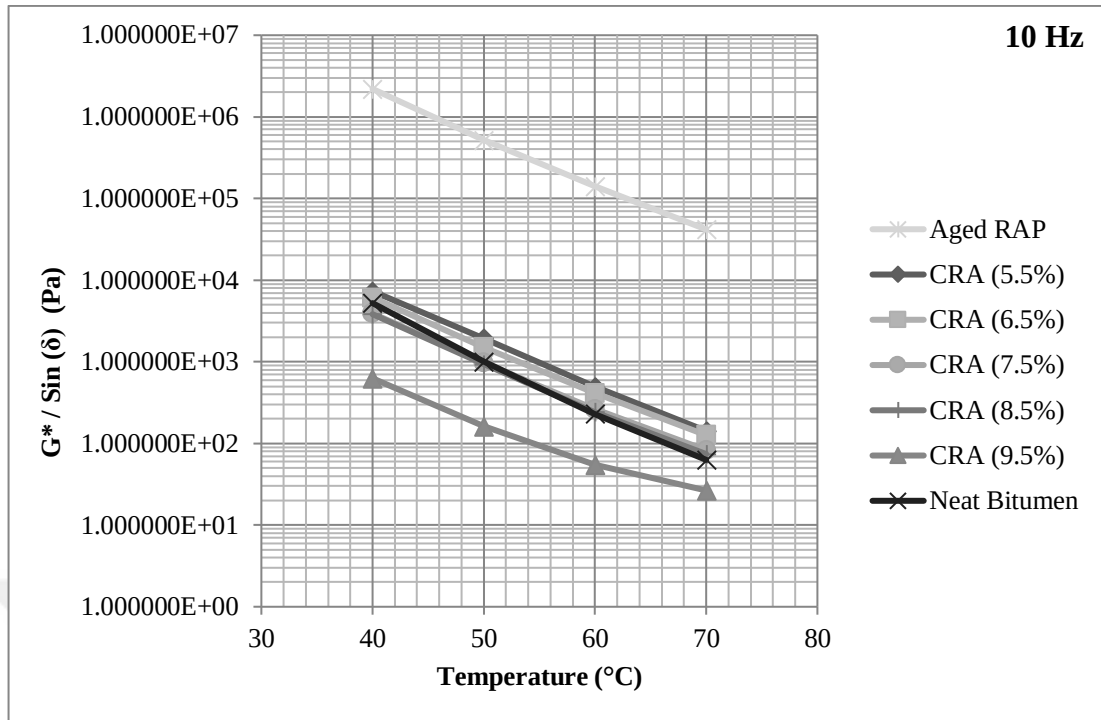


Figure 4.64 CRA rejuvenated samples rutting parameters at 10 Hz frequency

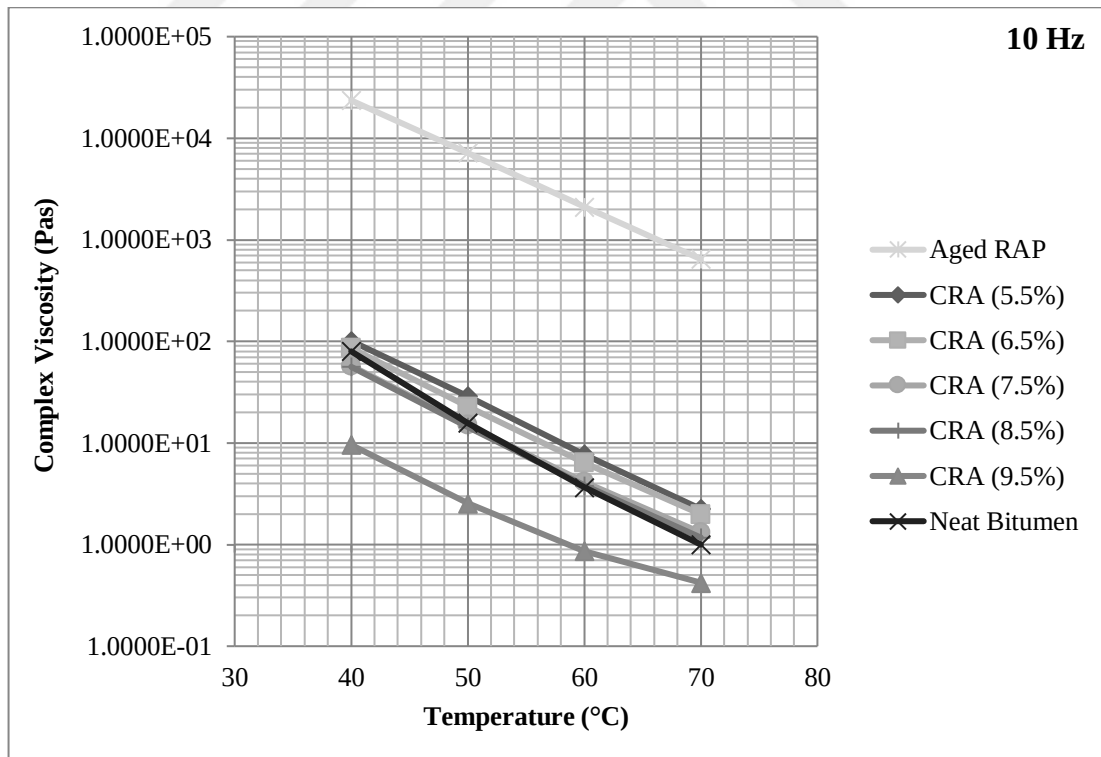


Figure 4.65 CRA rejuvenated samples complex viscosities at 10 Hz frequency

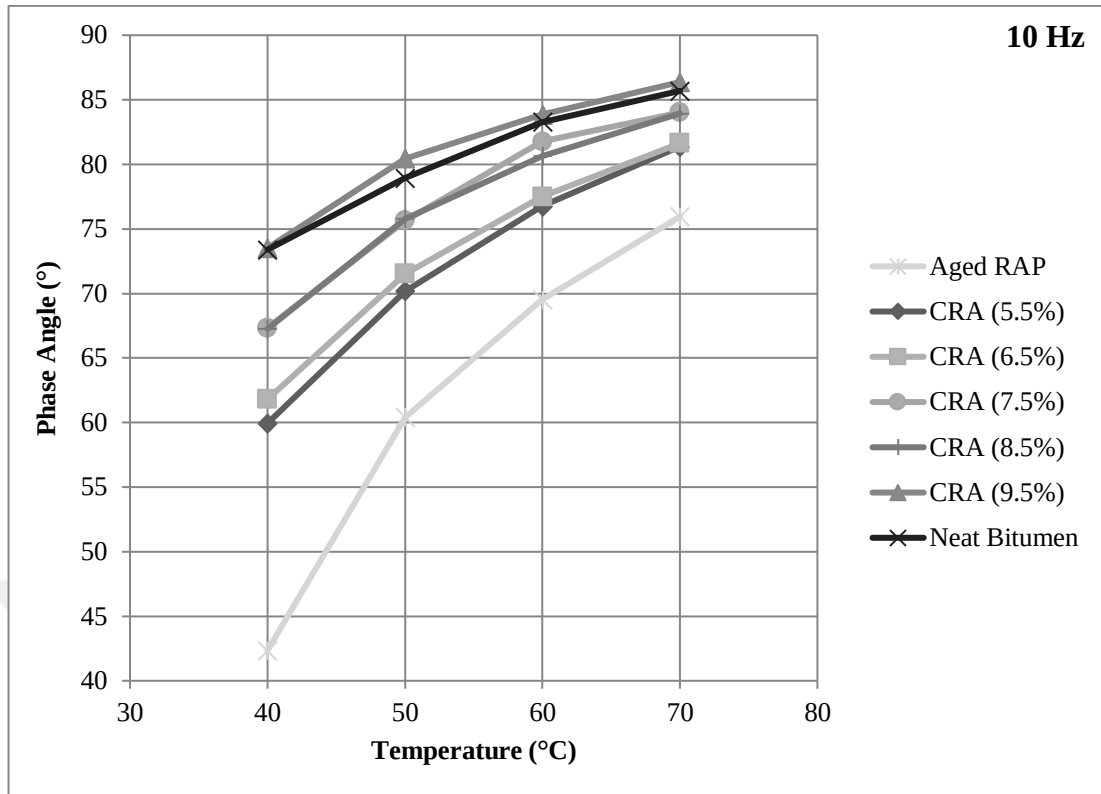


Figure 4.66 CRA rejuvenated samples phase angles at 10 Hz frequency

Another demonstration of the results for CRA rejuvenated and non-rejuvenated bitumen samples have been represented in Figures 4.67 to 4.78. In these figures, the effect of loading period in terms of frequency have been highlighted. The same regime demonstrates for waste oils have been observed for commercial rejuvenator in terms of rutting parameters, complex viscosities and phase angles. The difference is that the commercial product has been found more effective than waste oils in helping aged RAP bitumen regaining its pre-aging rheological characteristics.

At lower temperatures and higher frequency levels, the phase angles have more characteristic behavior and the effect of rejuvenation phenomenon is more revealed. As aforementioned, the effect of CRA on aged RAP bitumen on recovery of its pre-aging behavior is more recognizable at higher content uses of rejuvenator since 7.5% seems to be optimum both in terms of conventional and rheological properties.

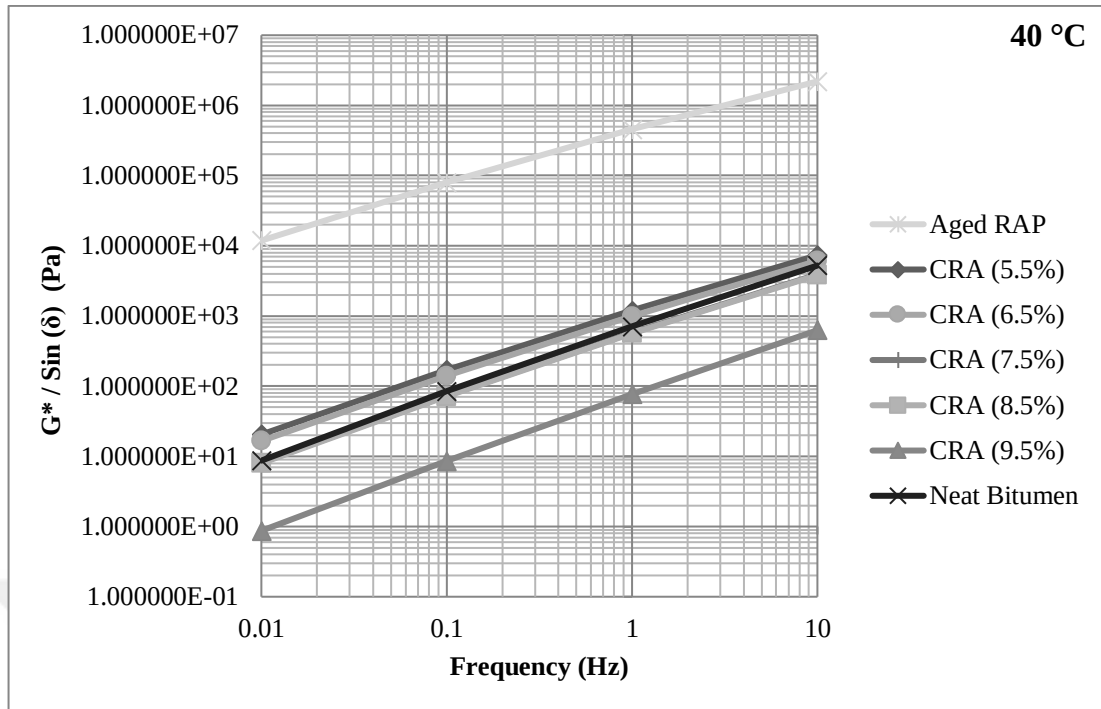


Figure 4.67 CRA rejuvenated samples rutting parameters at 40°C

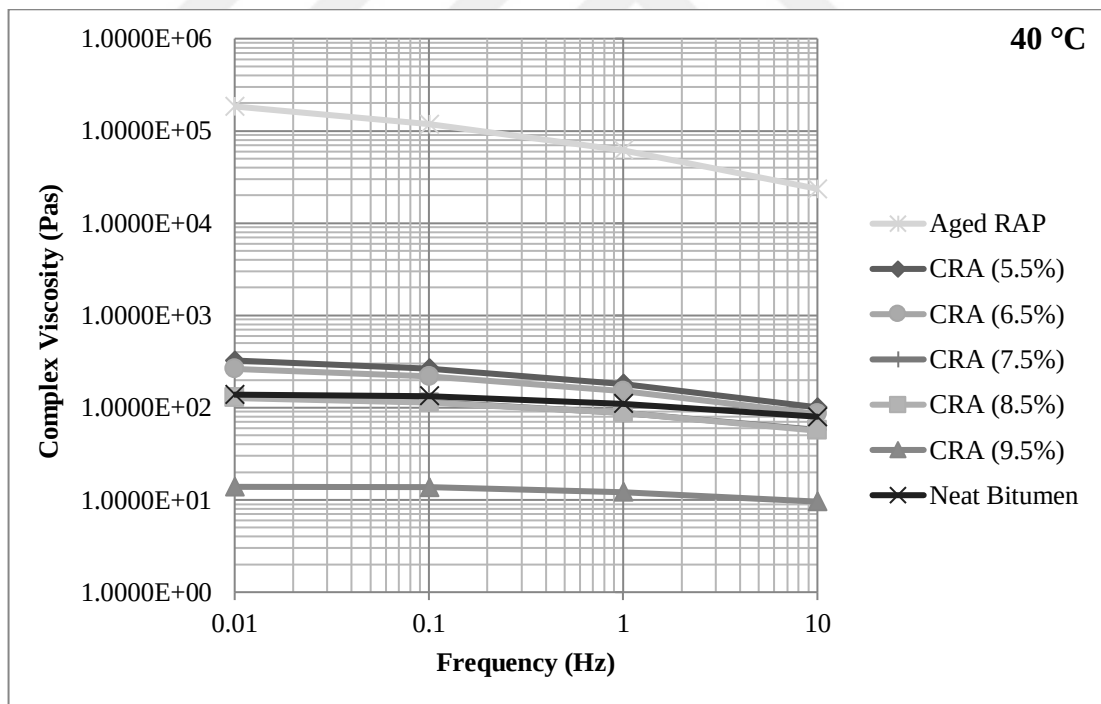


Figure 4.68 CRA rejuvenated samples complex viscosities at 40°C

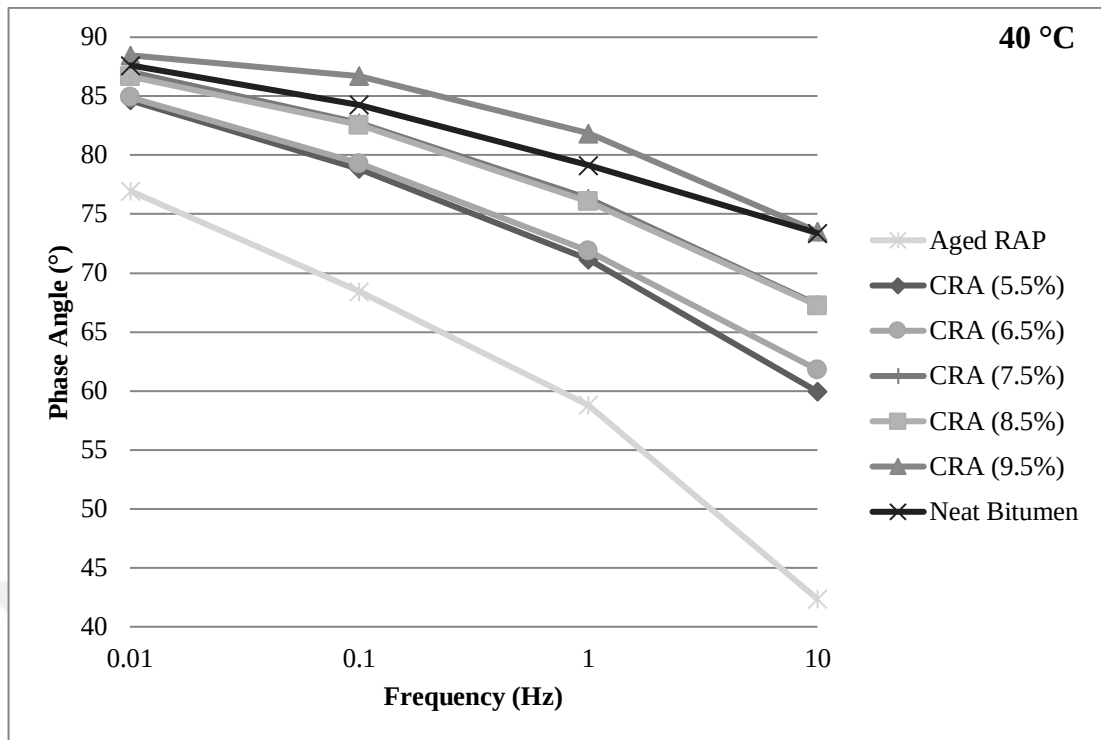


Figure 4.69 CRA rejuvenated samples phase angles at 40°C

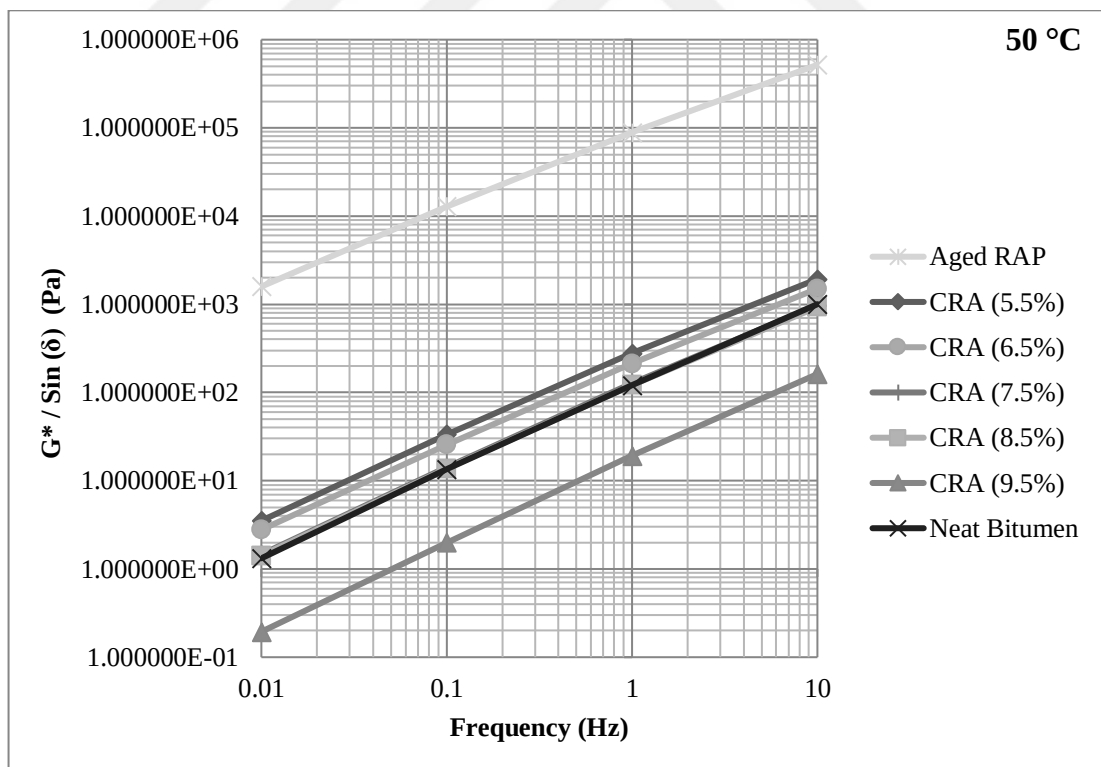


Figure 4.70 CRA rejuvenated samples rutting parameters at 50°C

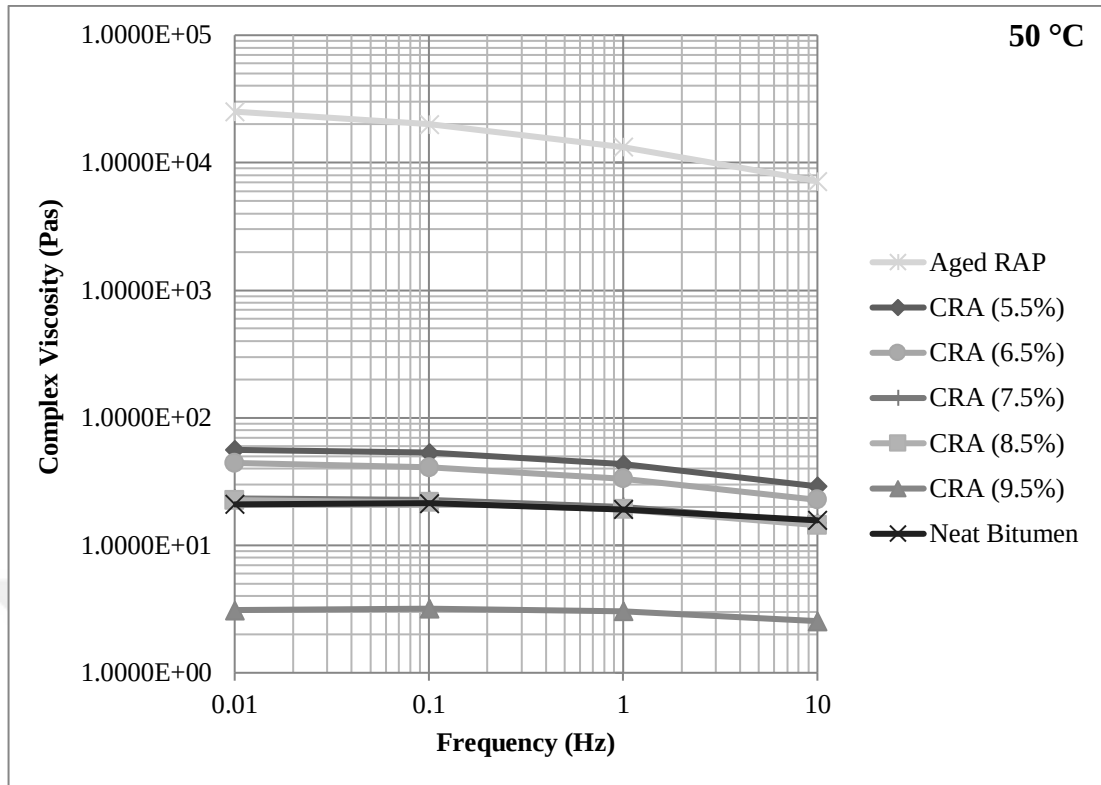


Figure 4.71 CRA rejuvenated samples complex viscosities at 50°C

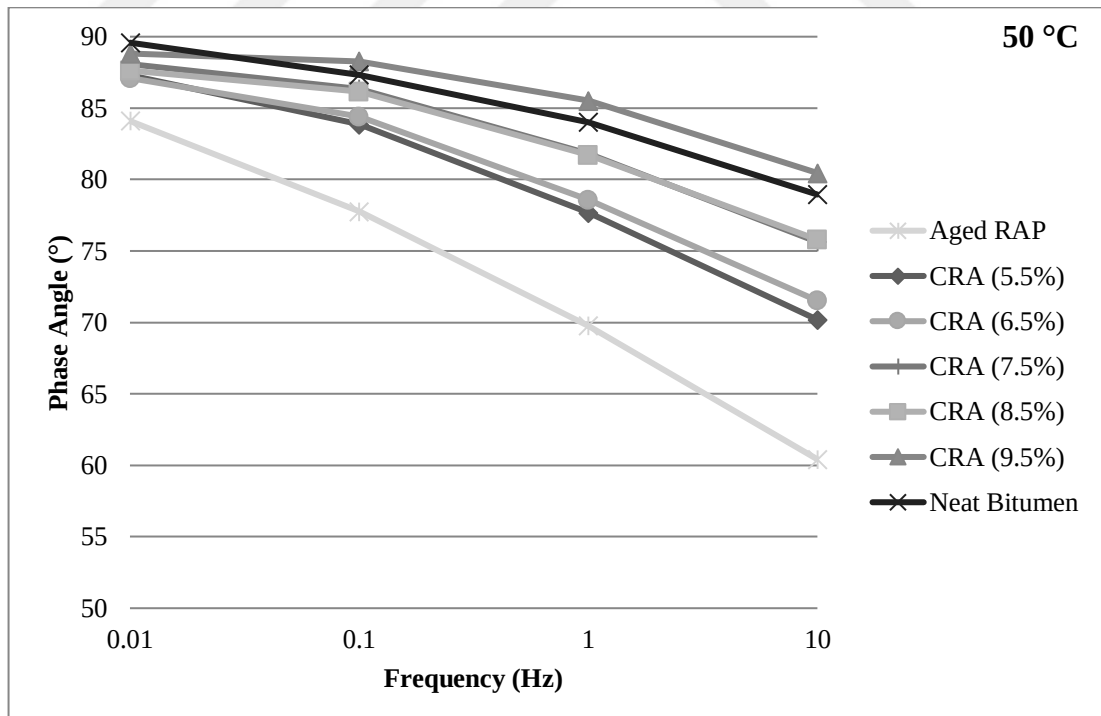


Figure 4.72 CRA rejuvenated samples phase angles at 50°C

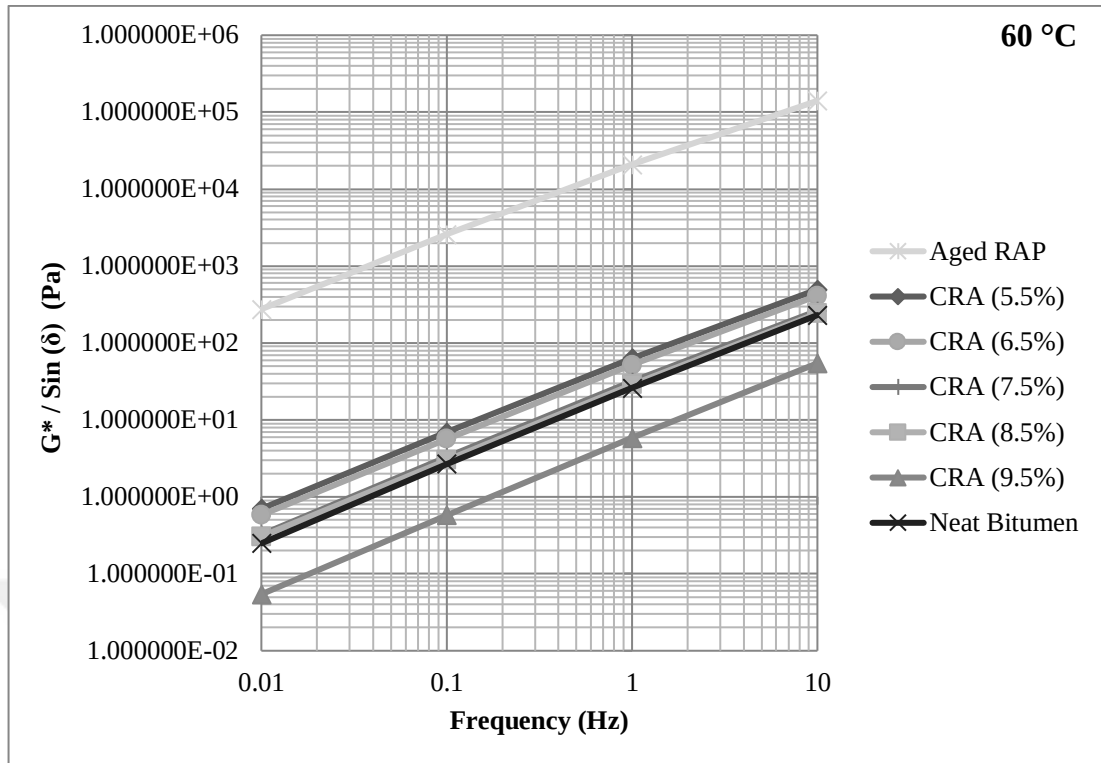


Figure 4.73 CRA rejuvenated samples rutting parameters at 60°C

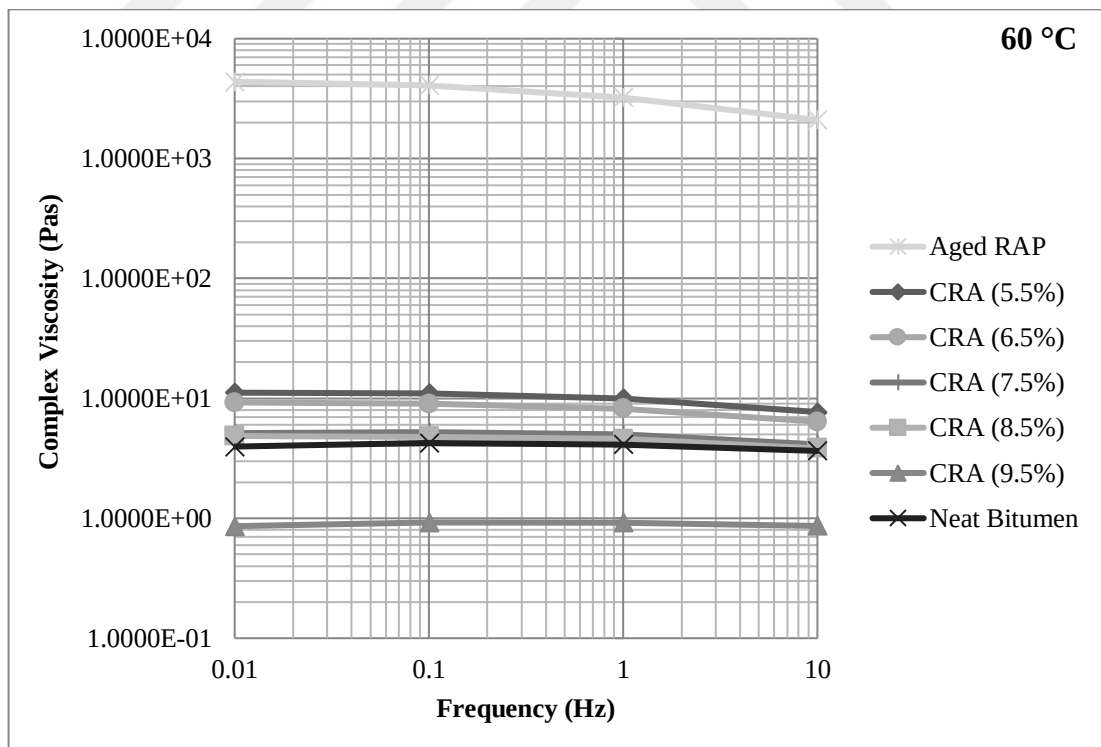


Figure 4.74 CRA rejuvenated samples complex viscosities at 60°C

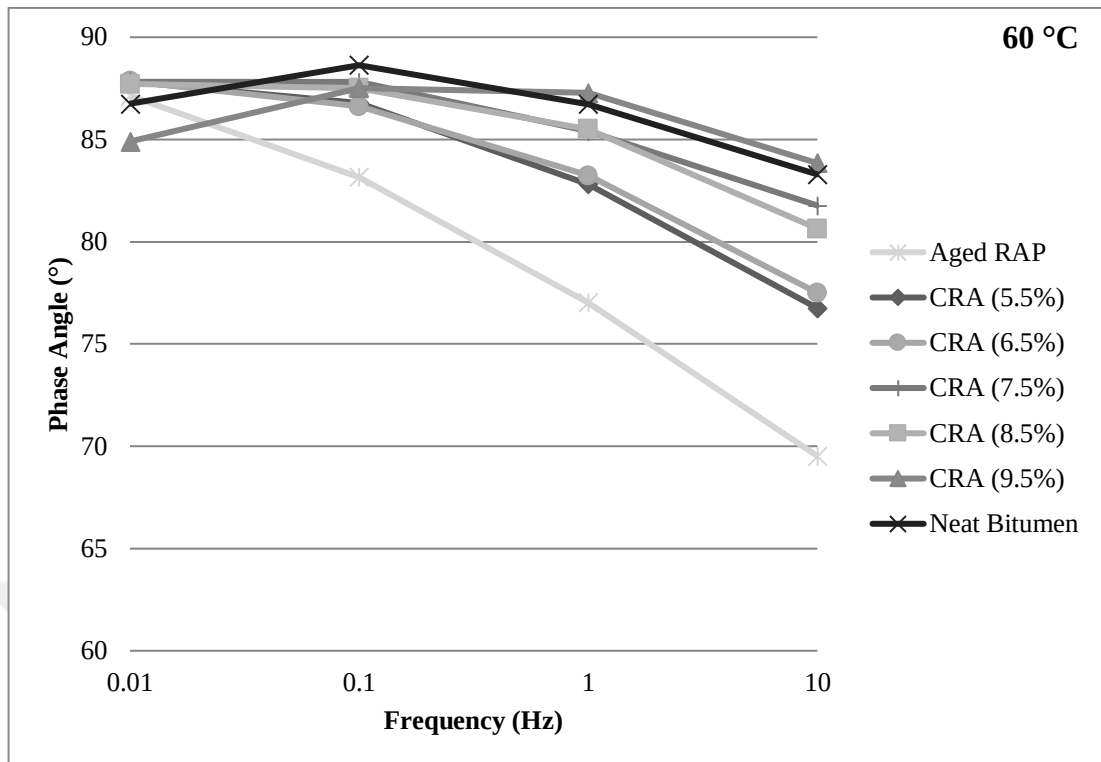


Figure 4.75 CRA rejuvenated samples phase angles at 60°C

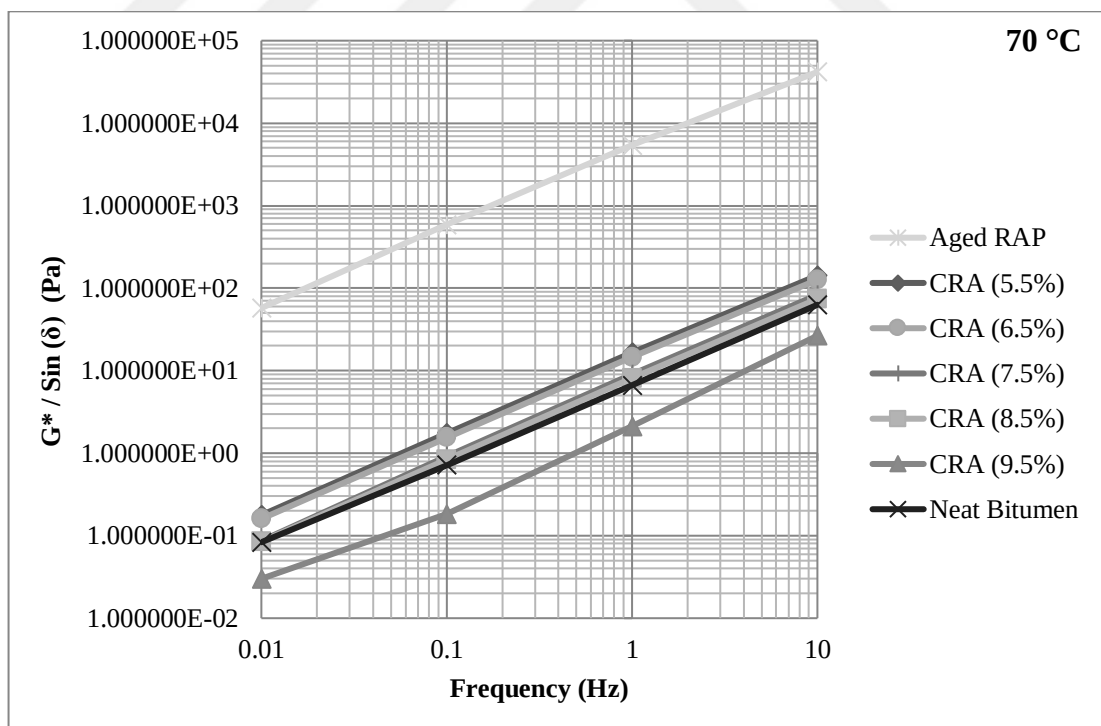


Figure 4.76 CRA rejuvenated samples rutting parameters at 70°C

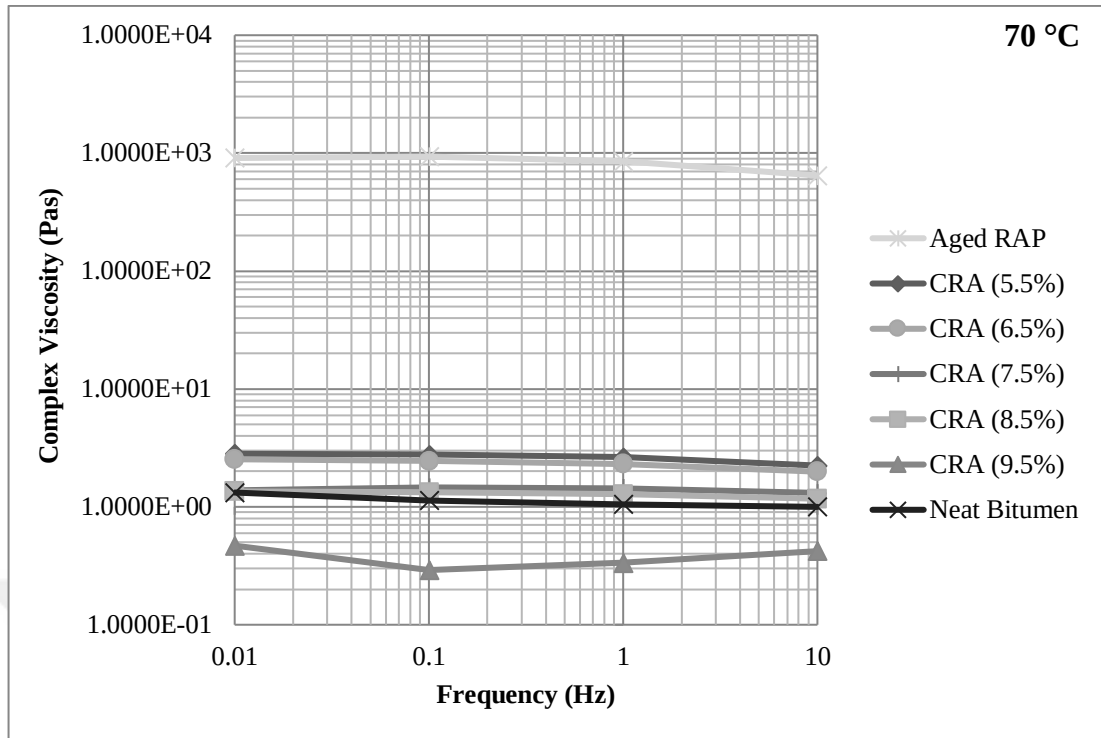


Figure 4.77 CRA rejuvenated samples complex viscosities at 70°C

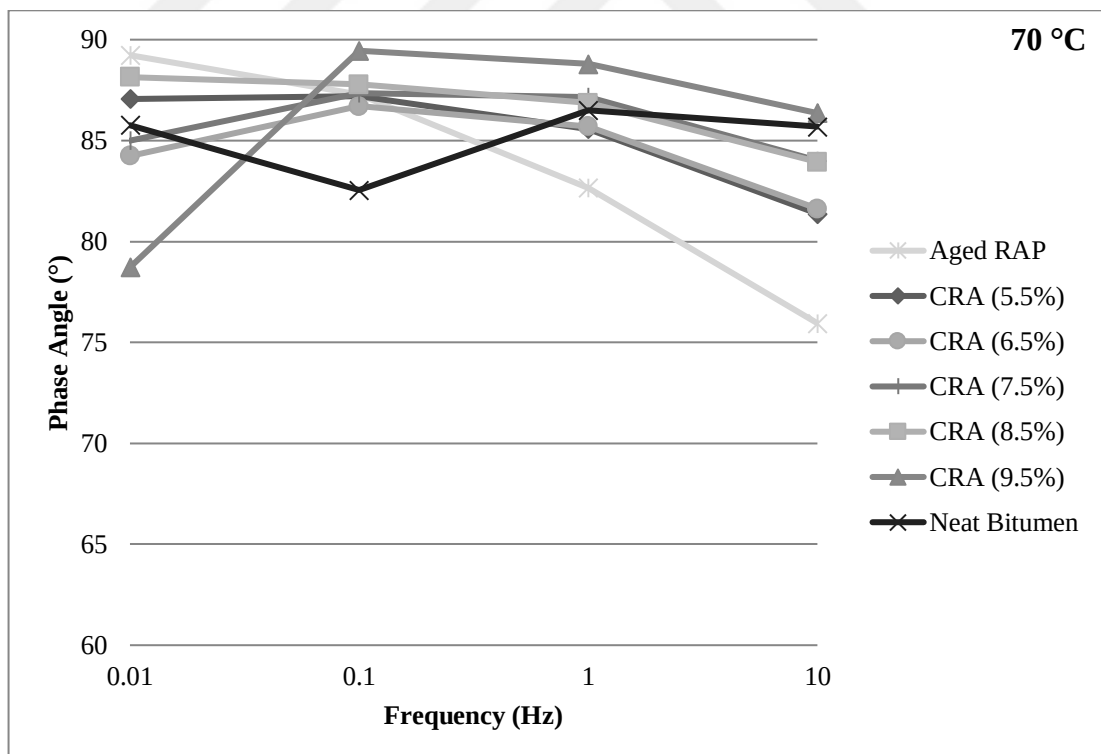


Figure 4.78 CRA rejuvenated samples phase angles at 70°C

4.1.2.4 Rheological Comparison of Rejuvenating Agents

The results for all bitumen samples which were rejuvenated using optimum contents of rejuvenating agents have been presented in this section. Like previous sections, the results for aged RAP bitumen and neat bitumen samples have also been presented to make comparisons between all rejuvenated samples using commercial product and waste oils and non-rejuvenated samples. The Appendix is consisted of all optimally rejuvenated and non-rejuvenated samples. All results were plotted on figures with temperatures on X axis and frequency levels on X axis separately to see the effect of temperature and loading times clearly.

Figures 4.79 to 4.81, respectively, show the plotted test results for rutting parameters, complex viscosities and phase angles of all optimally rejuvenated bitumen samples at 0.01 Hz frequency level. The results for rutting parameters and the complex viscosities have been plotted in logarithmic scale on Y axis to help better demonstration of the results.

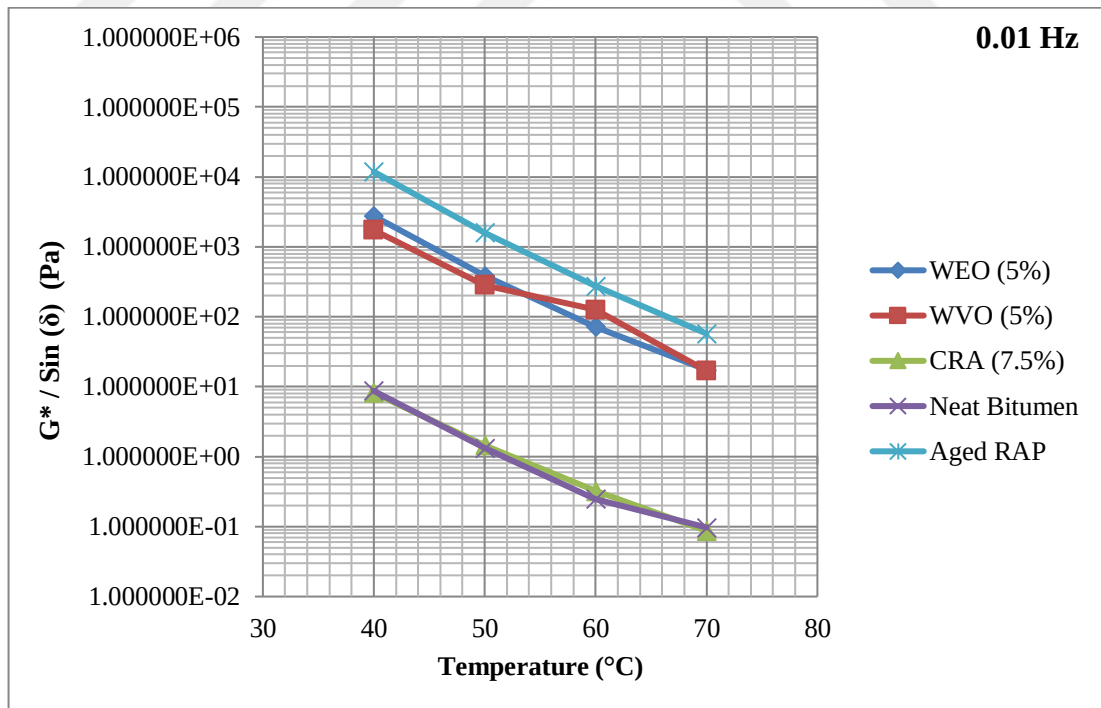


Figure 4.79 Optimally rejuvenated samples rutting parameters at 0.01 Hz frequency

As mentioned before, the rutting parameter $G^*/\sin(\delta)$ of a bitumen sample should be maximum to ensure adequate rut resistance. Although $G^*/\sin(\delta)$ of aged bitumen is typically higher than the $G^*/\sin(\delta)$ of fresh neat bitumen, we cannot say the aged bitumen is more rut resistant than fresh neat bitumen since it can be more susceptible to fatigue cracking and may be more brittle under heavy traffic loads especially in cold weather conditions.

Although the rheological parameter which should be checked for fatigue cracking at lower temperatures is $G^* \cdot \sin(\delta)$ and not the rutting parameter $G^*/\sin(\delta)$, the important matter here is that to check if the rejuvenated bitumen sample has been recovered or not. The point is to compare the results with the results of neat bitumen to check if the tested sample has demonstrated closer behavior to neat bitumen or not. In terms of rutting parameters,

Figures 4.79 for 0.01 Hz, 4.82 for 0.1 Hz, 4.85 for 1 Hz and 4.88 for 10 Hz can be checked. As seen on all these figures, almost at all frequencies and temperatures, CRA rejuvenating agent demonstrated better performance than waste oils.

Although WEO and WVO could not give out the results near neat bitumen, but both waste oils were capable of rejuvenating aged RAP bitumen in terms of all rheological characteristics.

Both waste oils rejuvenated samples demonstrated very similar behavior with regard to rutting, complex viscosity and phase angle. WVO rejuvenated samples in most cases demonstrated better performance than WEO rejuvenated samples in terms of rheological properties.

Typically, the rutting parameters of all bitumen samples decreased by increasing of temperature and increased by increasing of frequency.

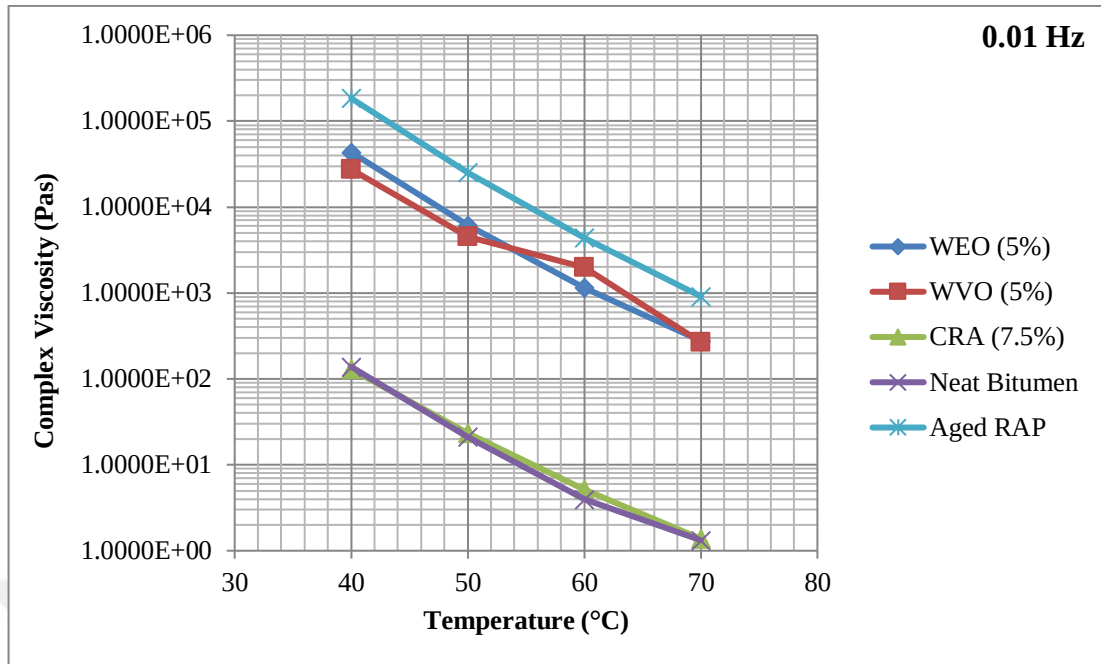


Figure 4.80 Optimally rejuvenated samples complex viscosities at 0.01 Hz frequency

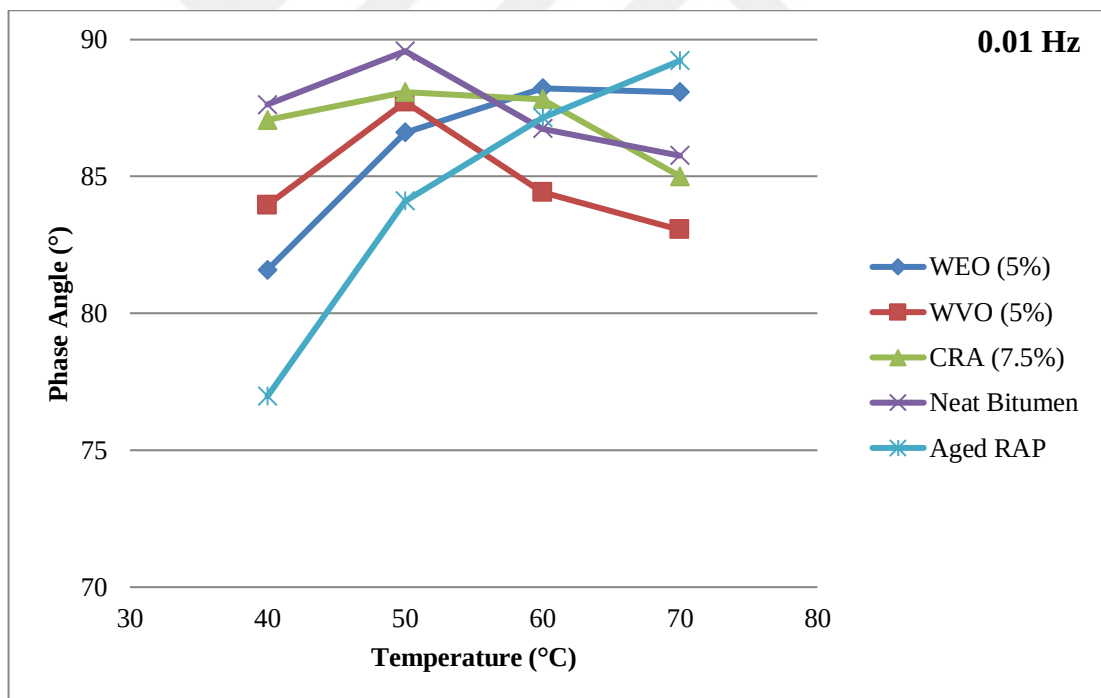


Figure 4.81 Optimally rejuvenated samples phase angles at 0.01 Hz frequency

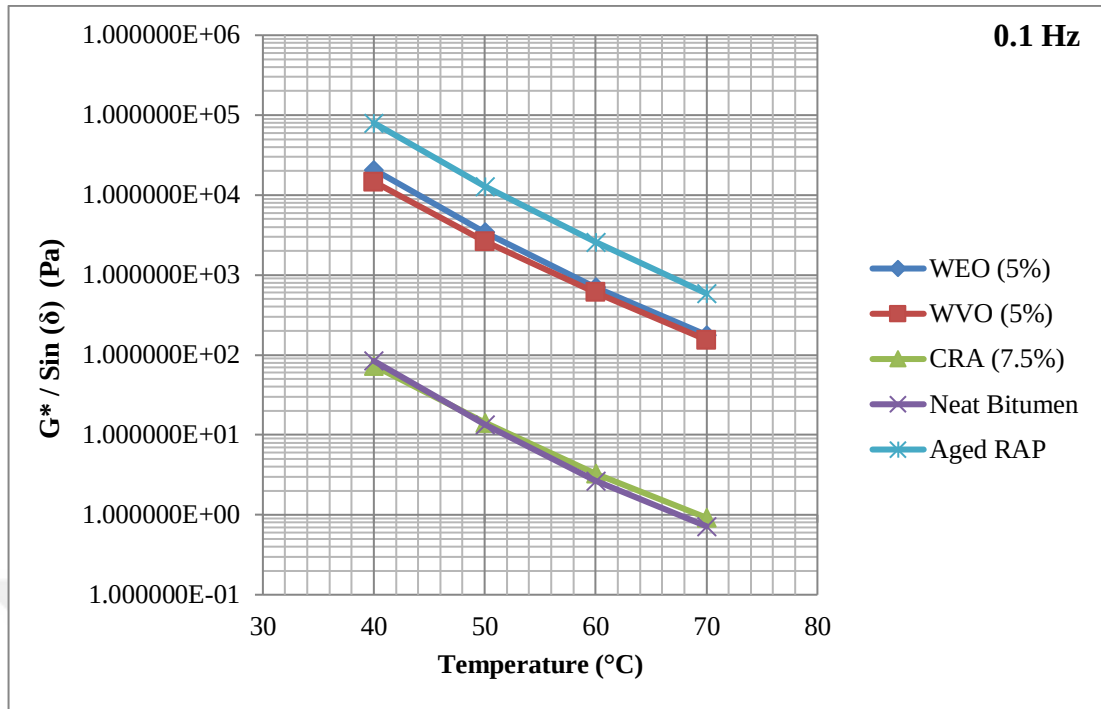


Figure 4.82 Optimally rejuvenated samples rutting parameters at 0.1 Hz frequency

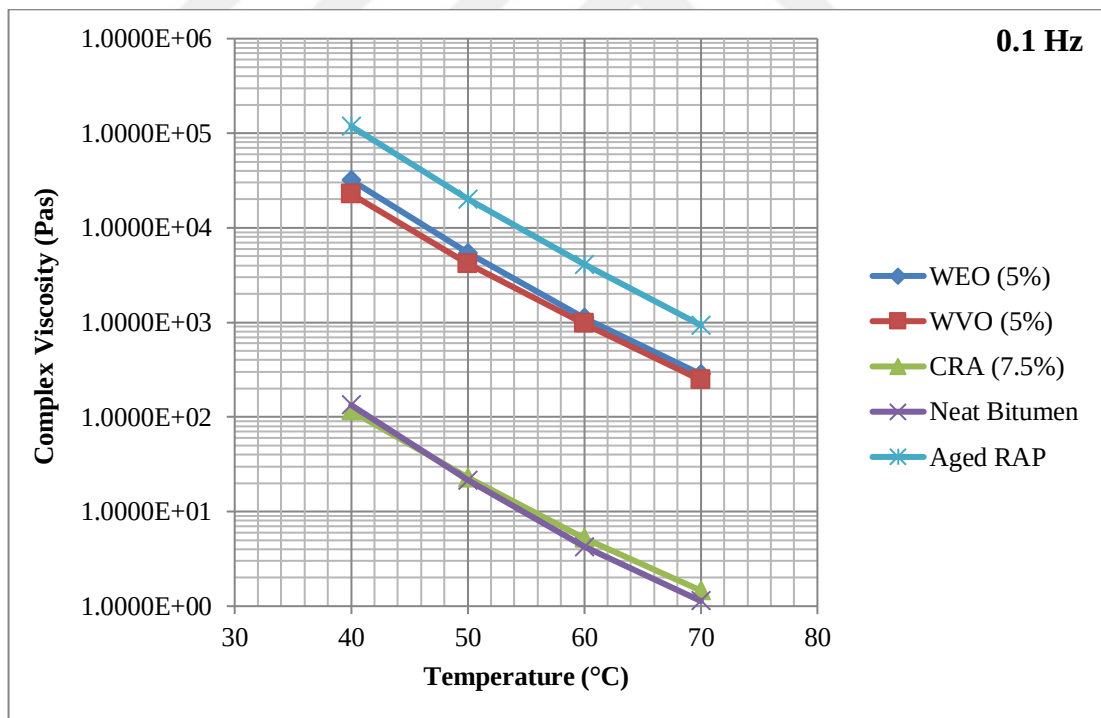


Figure 4.83 Optimally rejuvenated samples complex viscosities at 0.1 Hz frequency

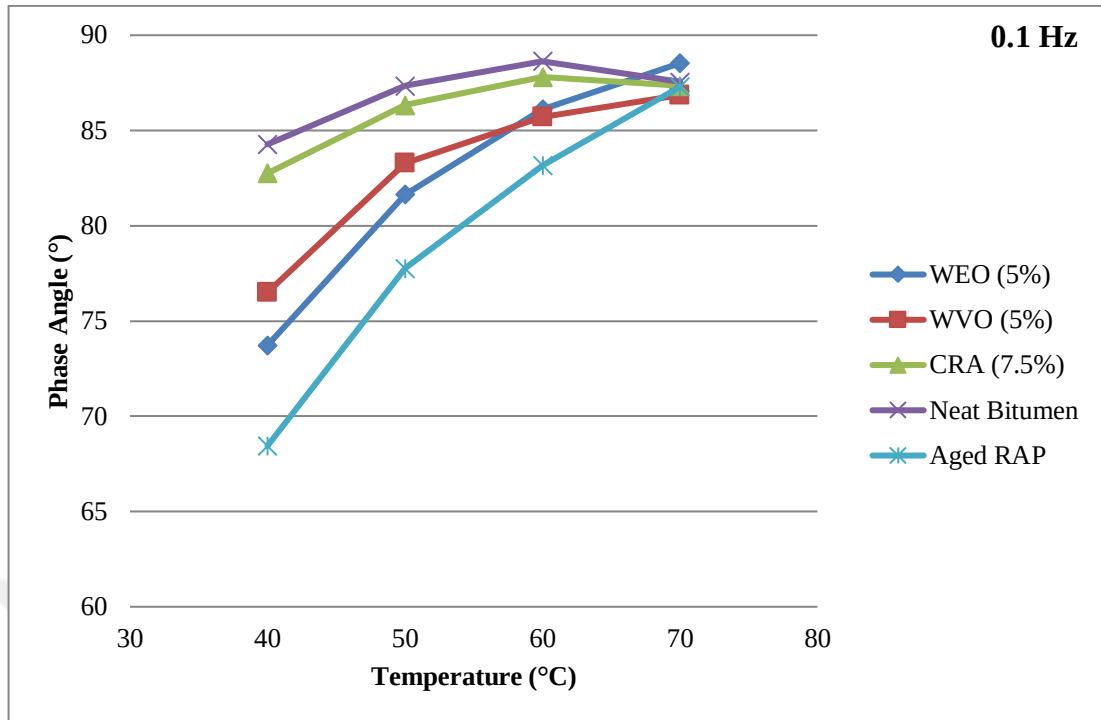


Figure 4.84 Optimally rejuvenated samples phase angles at 0.1 Hz frequency

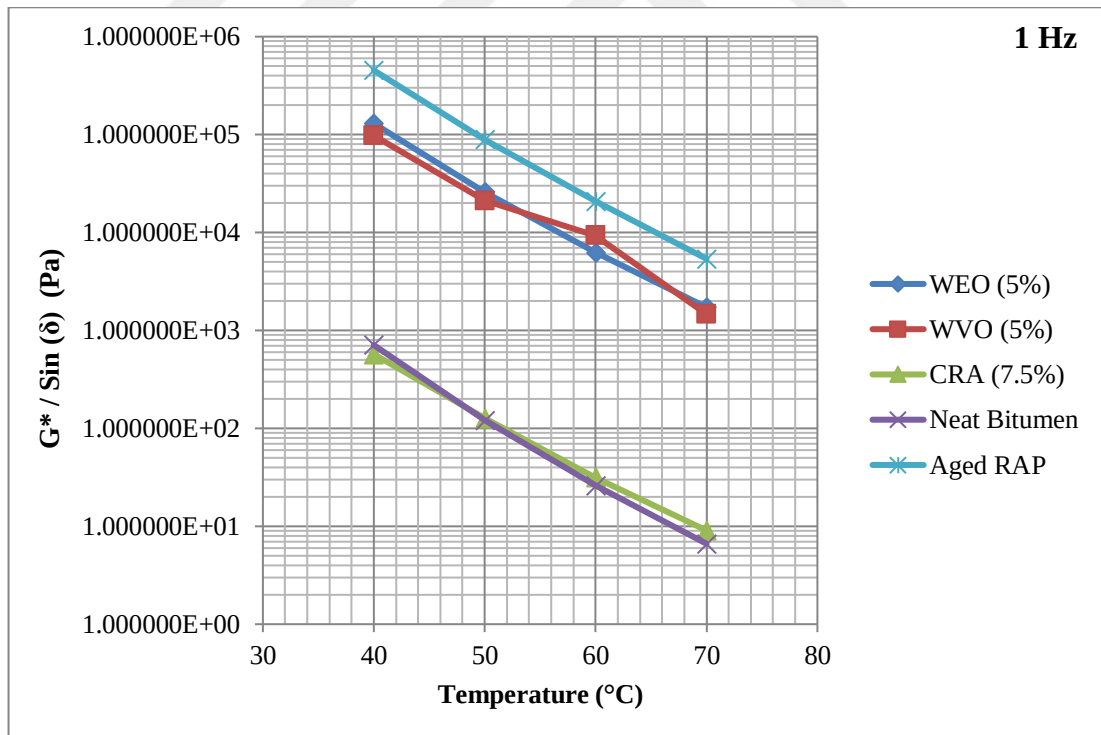


Figure 4.85 Optimally rejuvenated samples rutting parameters at 1 Hz frequency

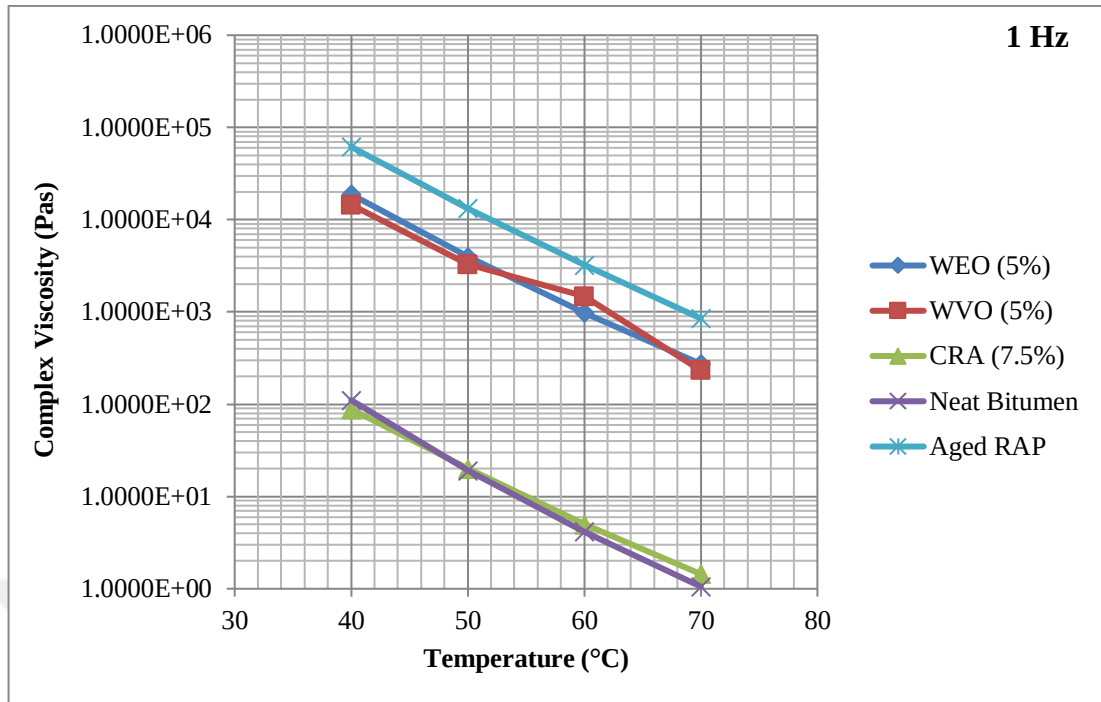


Figure 4.86 Optimally rejuvenated samples complex viscosities at 1 Hz frequency

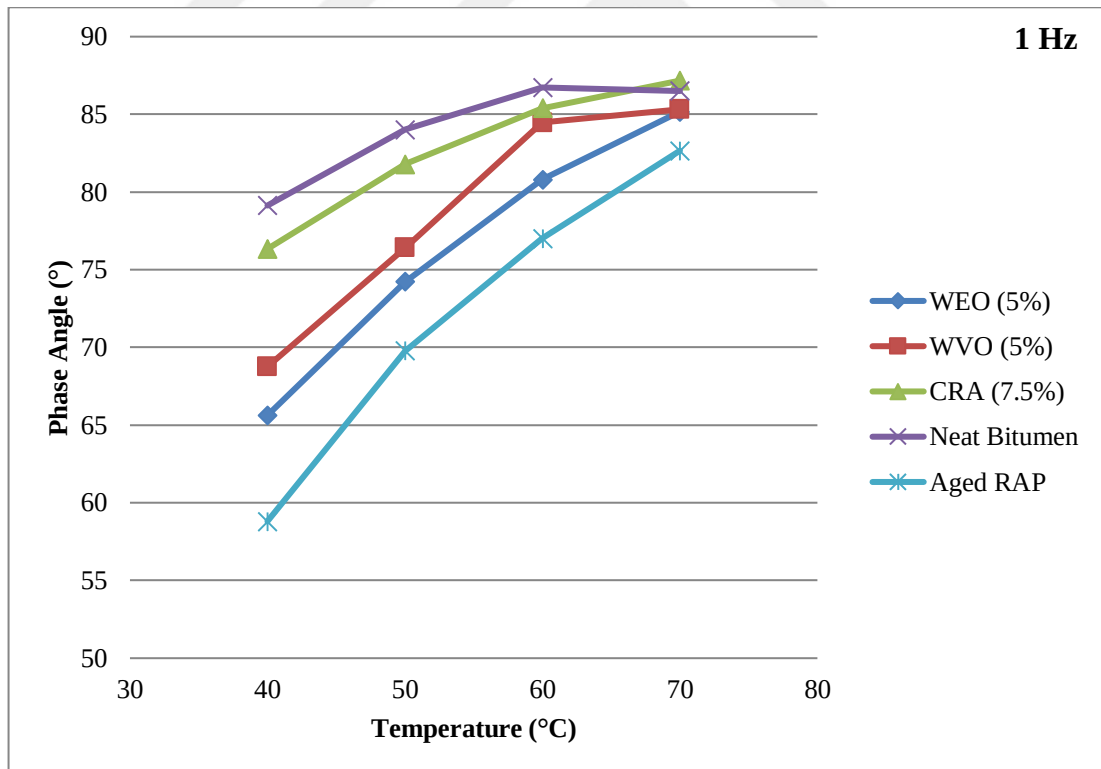


Figure 4.87 Optimally rejuvenated samples phase angles at 1 Hz frequency

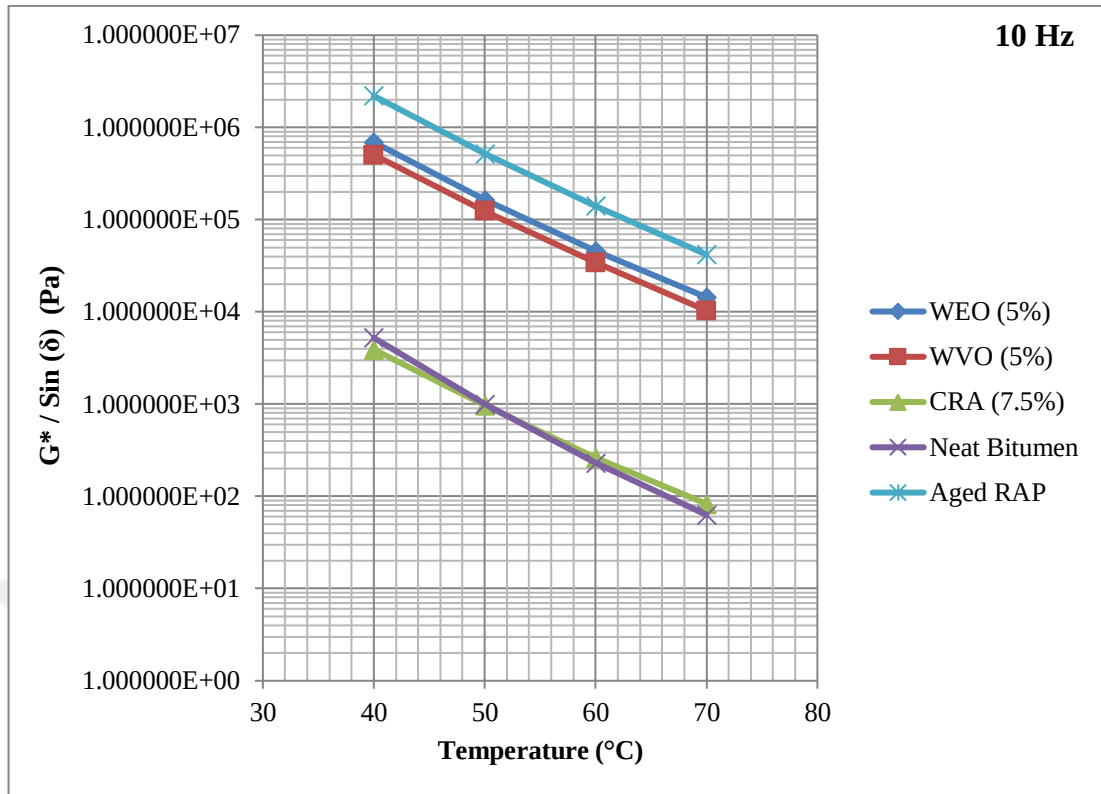


Figure 4.88 Optimally rejuvenated samples rutting parameters at 10 Hz frequency

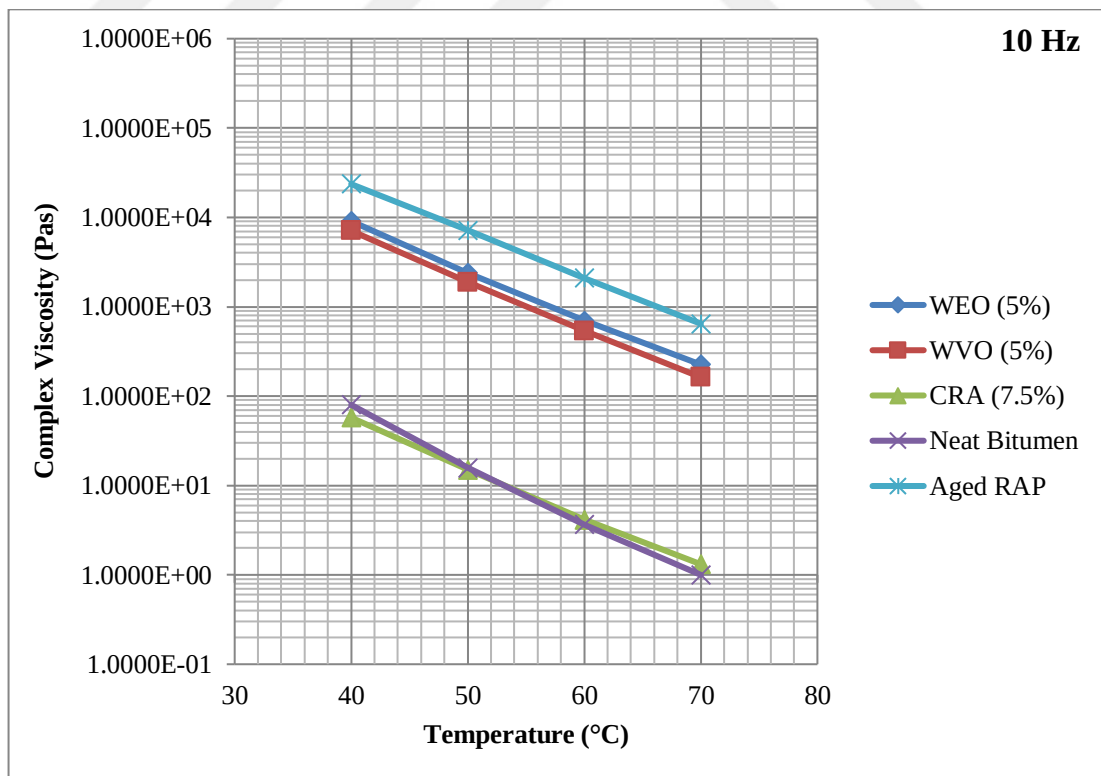


Figure 4.89 Optimally rejuvenated samples complex viscosities at 10 Hz frequency

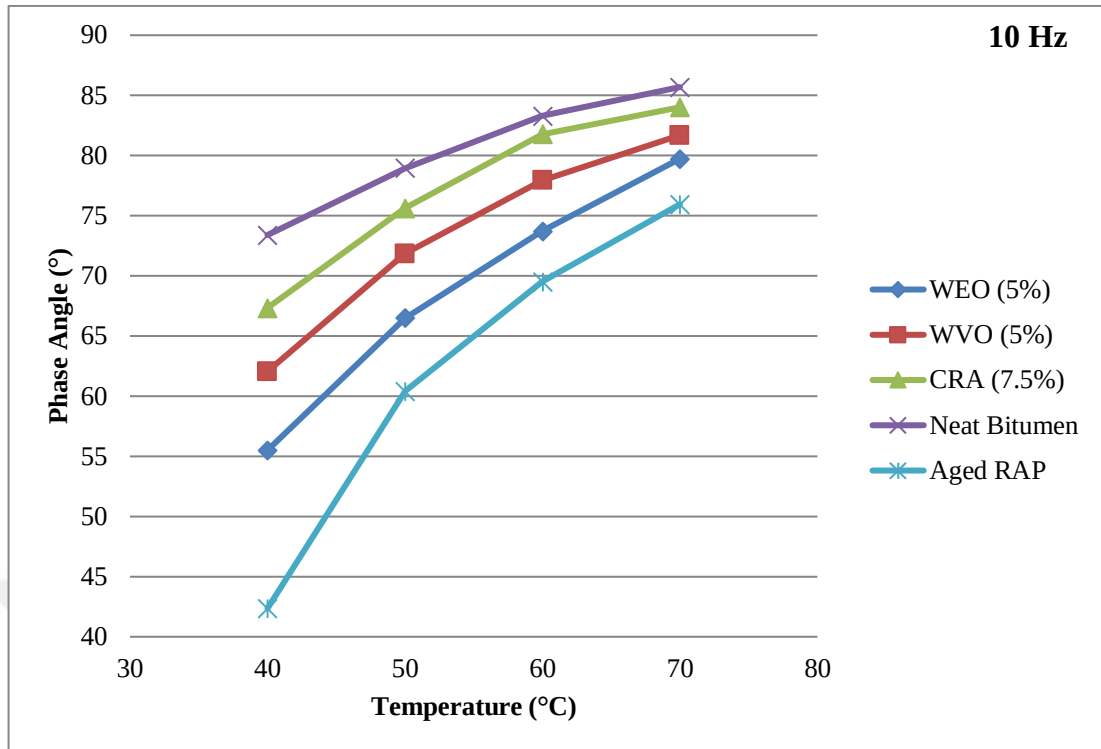


Figure 4.90 Optimally rejuvenated samples phase angles at 10 Hz frequency

Figures 4.91 to 4.102 show the other way plotting of the results for better demonstration of the effect of frequency level on rutting parameters, complex viscosities and phase angle values at each temperature. Normally, the rutting parameters increase linearly in logarithmic scale (exponentially in normal scale) by increasing of the frequency at all temperatures.

The complex viscosities, when plotted on logarithmic scale, decrease very slightly by increasing of the frequency level. Phase angles, obviously at lower temperatures, decrease by increasing the frequency level. This means that all bitumen samples behave more elastic rather than viscous at lower temperatures and higher frequencies.

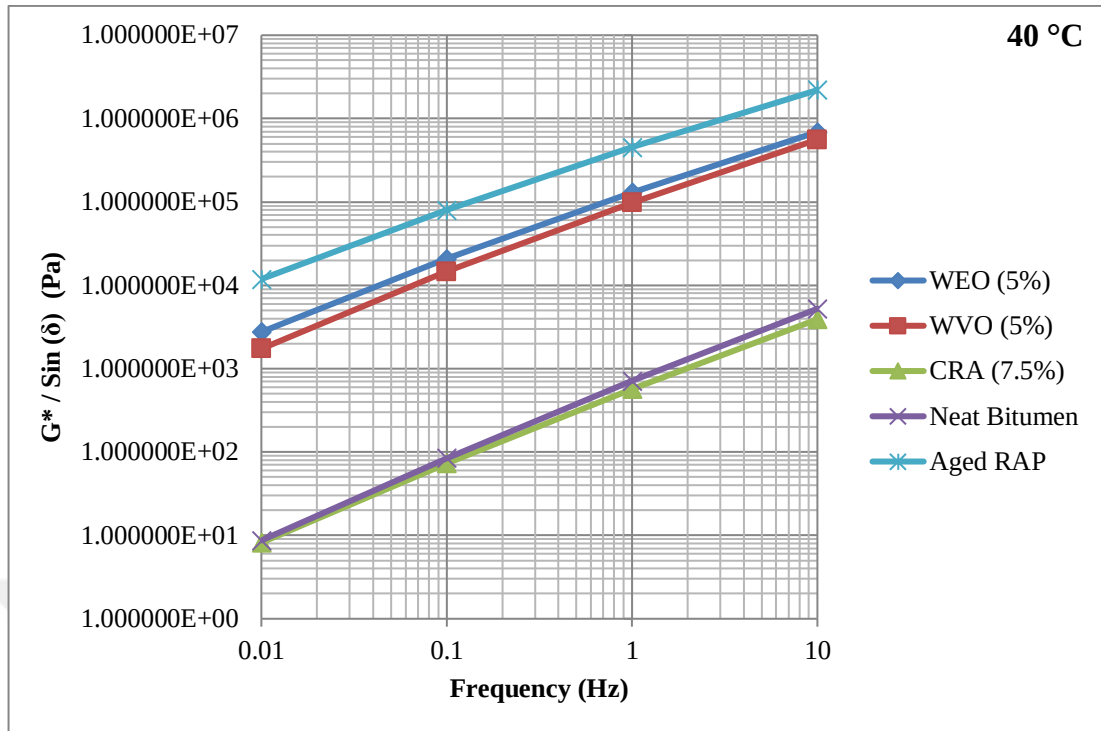


Figure 4.91 Optimally rejuvenated samples rutting parameters at 40°C

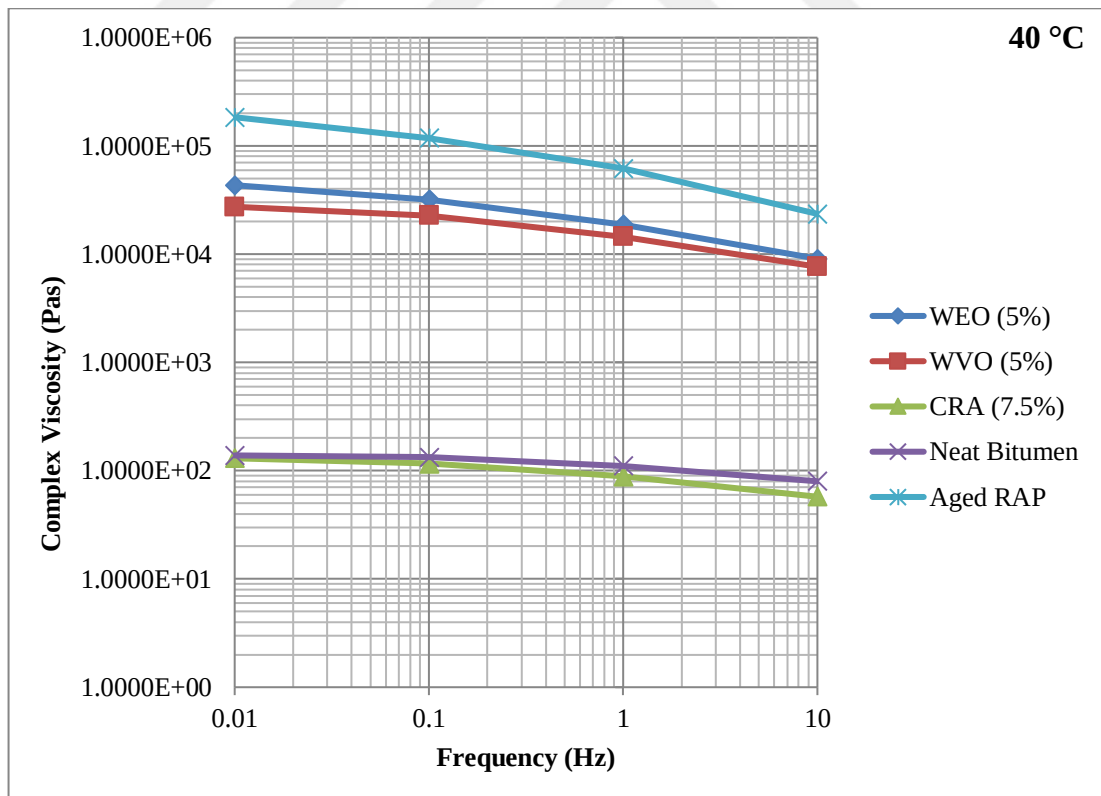


Figure 4.92 Optimally rejuvenated samples complex viscosities at 40°C

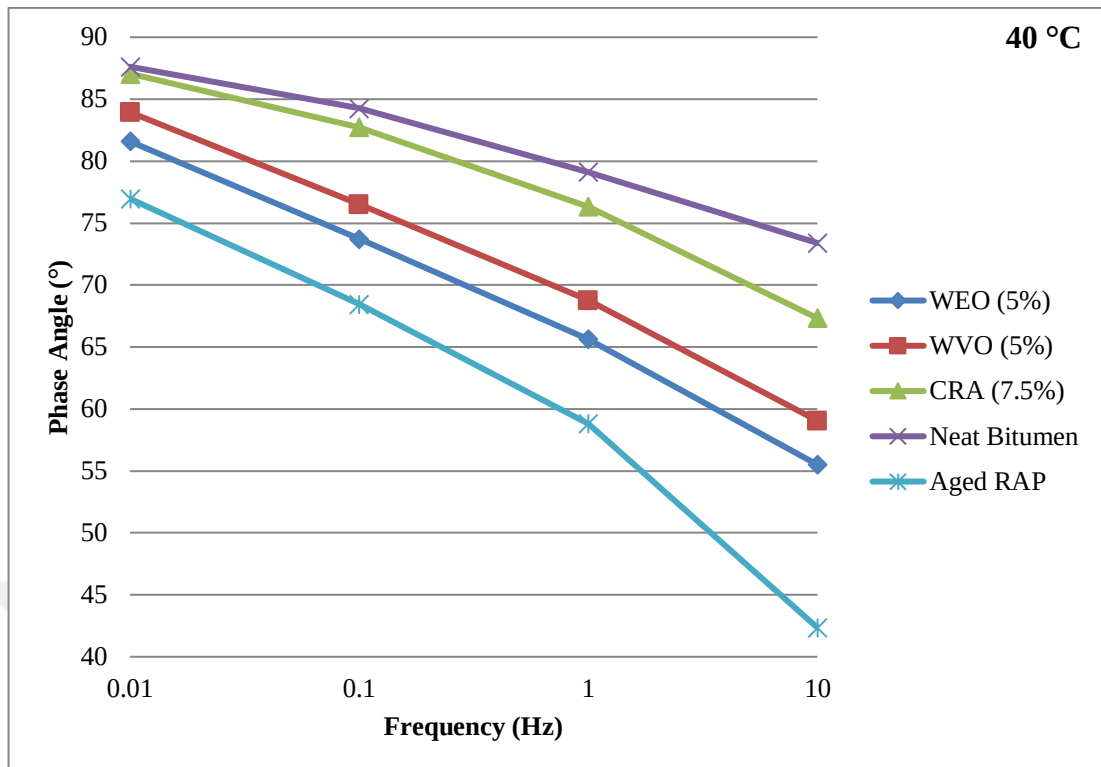


Figure 4.93 Optimally rejuvenated samples phase angles at 40°C

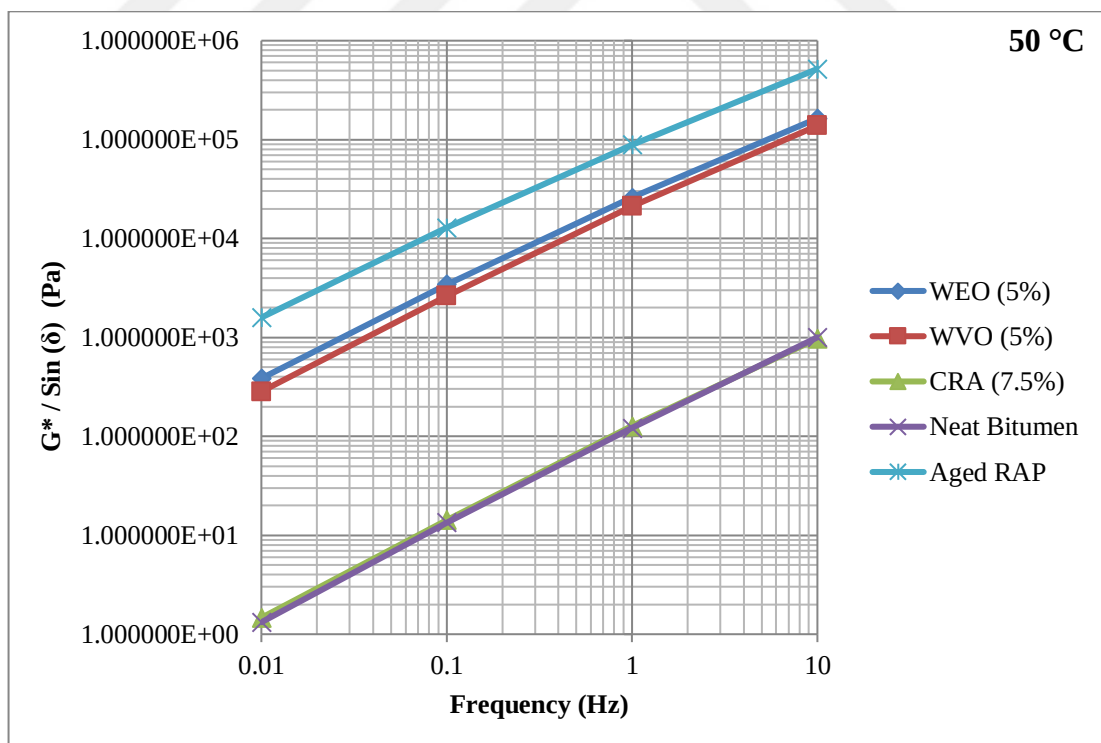


Figure 4.94 Optimally rejuvenated samples rutting parameters at 50°C

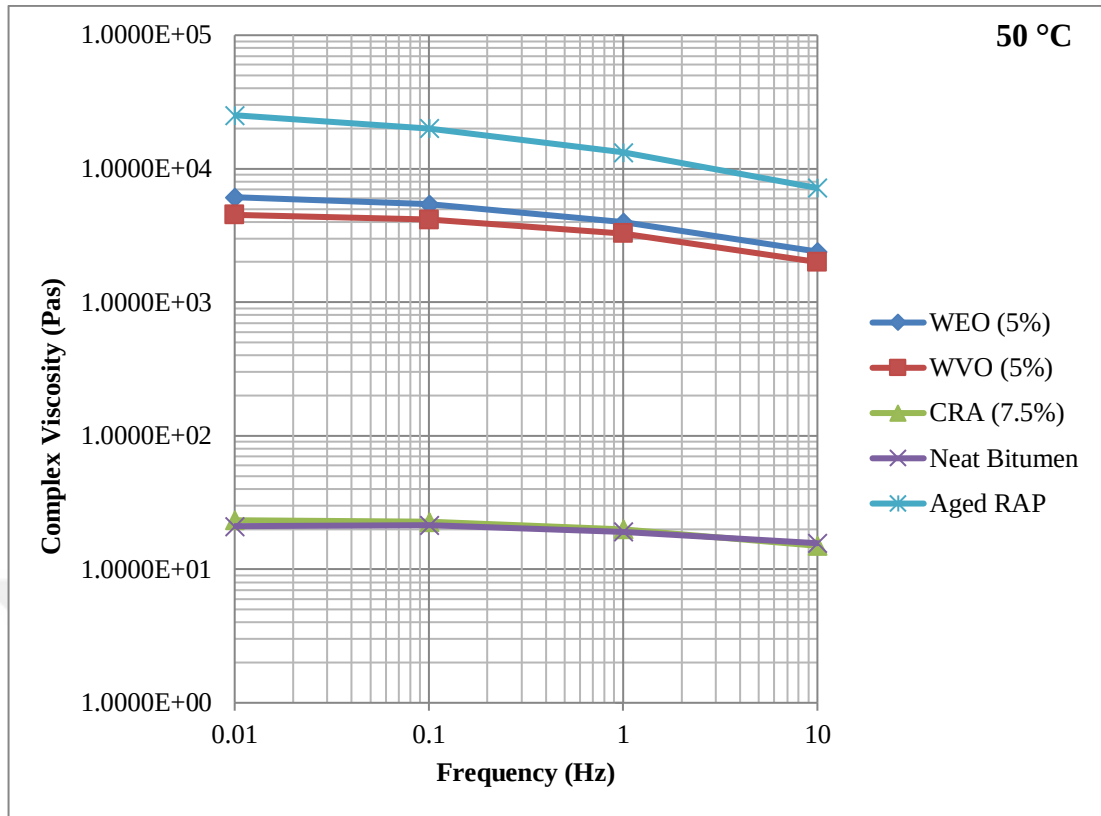


Figure 4.95 Optimally rejuvenated samples complex viscosities at 50°C

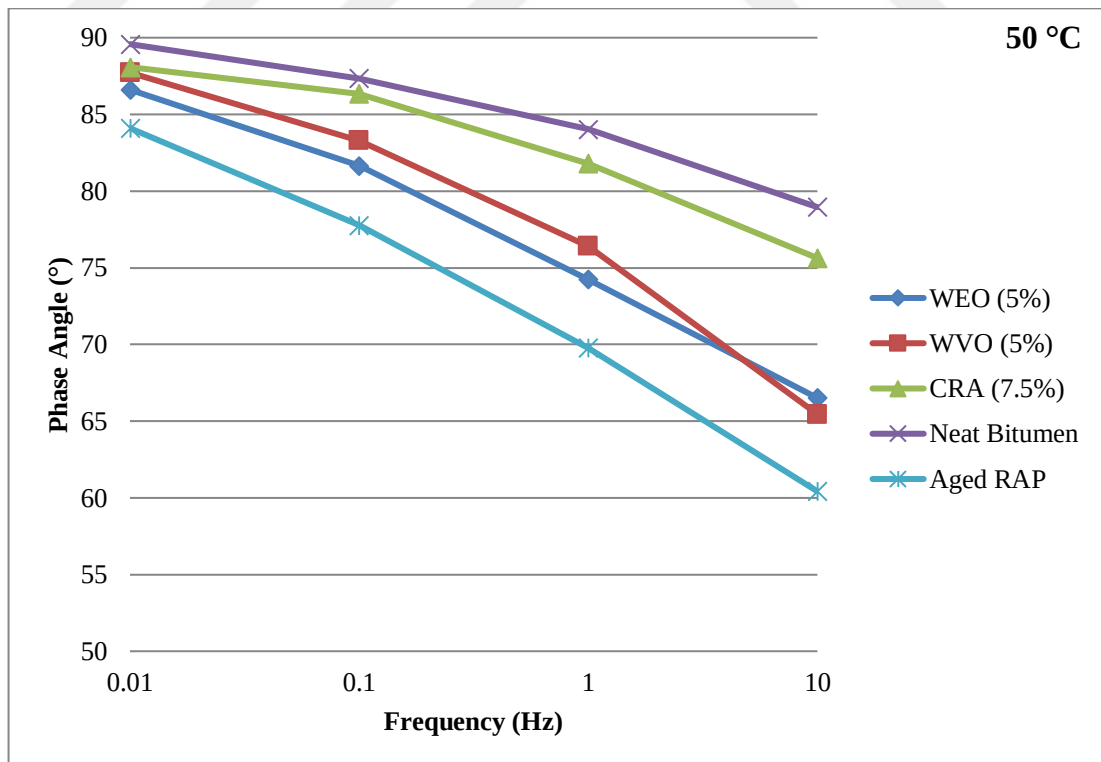


Figure 4.96 Optimally rejuvenated samples phase angles at 50°C

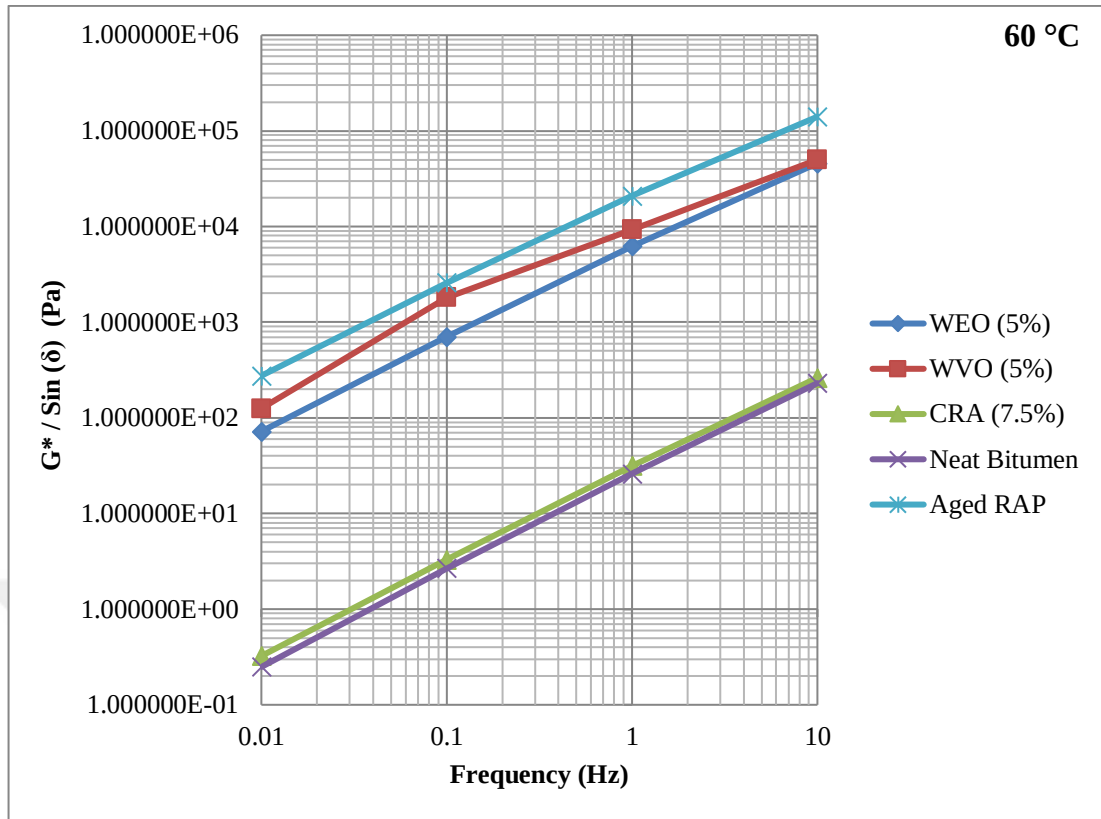


Figure 4.97 Optimally rejuvenated samples rutting parameters at 60°C

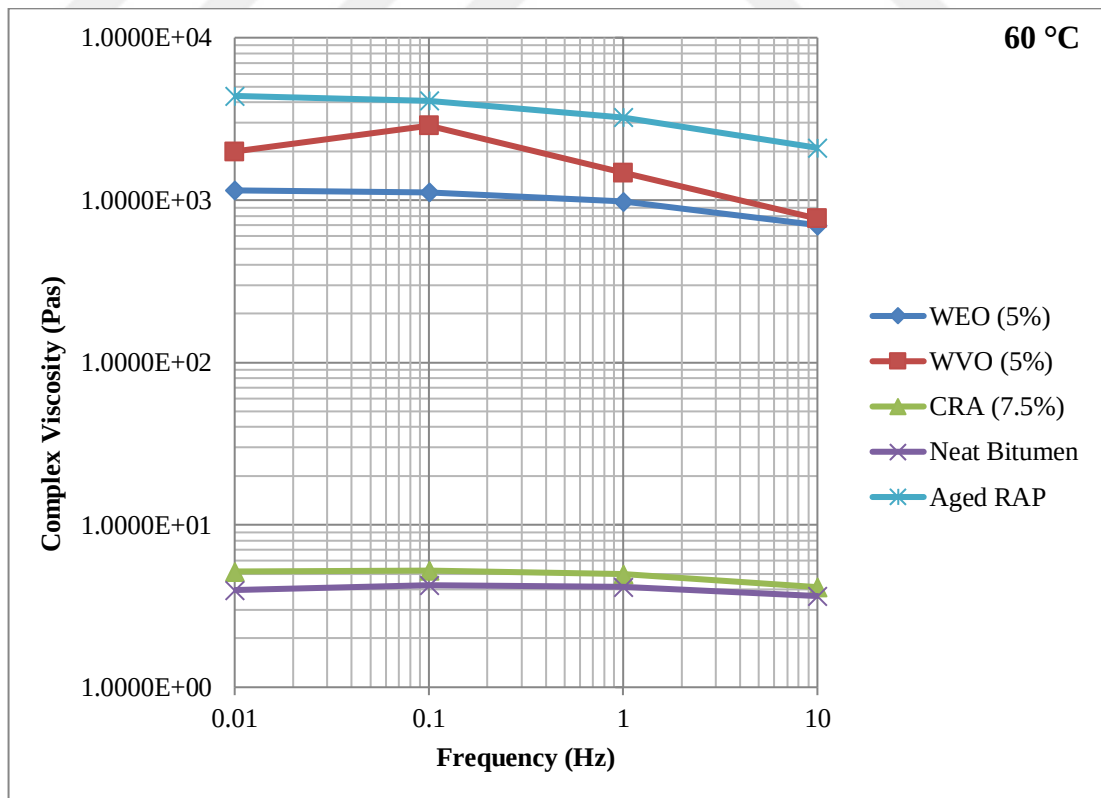


Figure 4.98 Optimally rejuvenated samples complex viscosities at 60°C

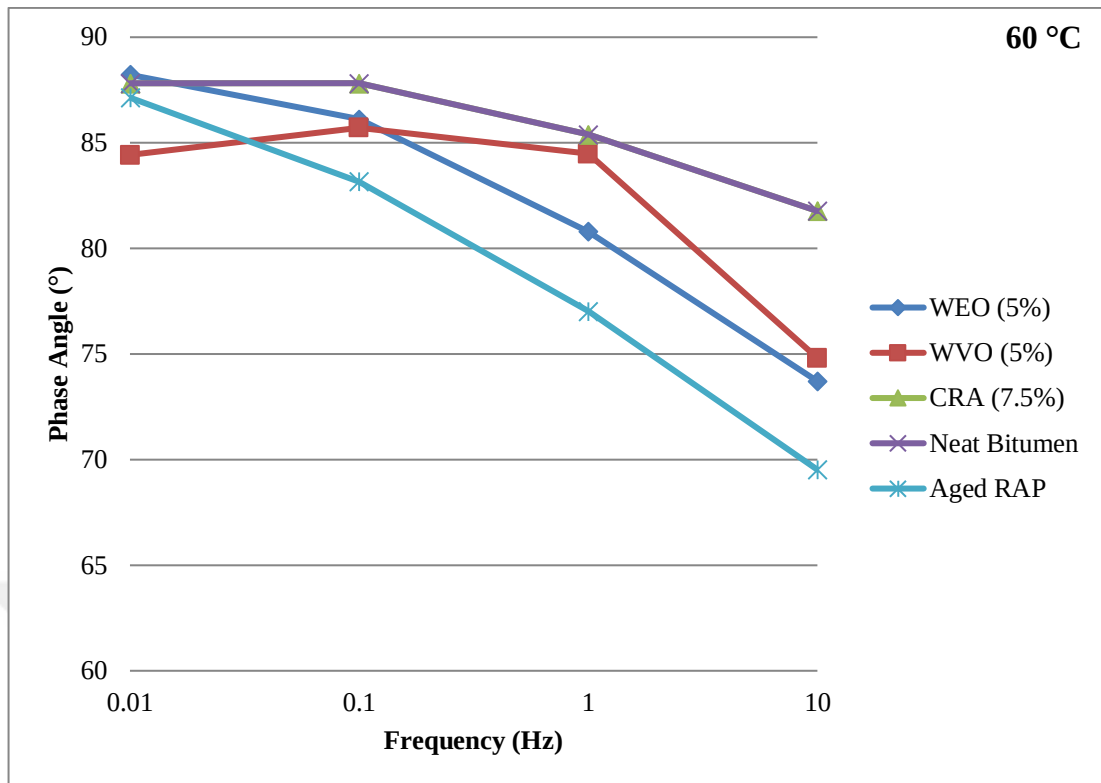


Figure 4.99 Optimally rejuvenated samples phase angles at 60°C

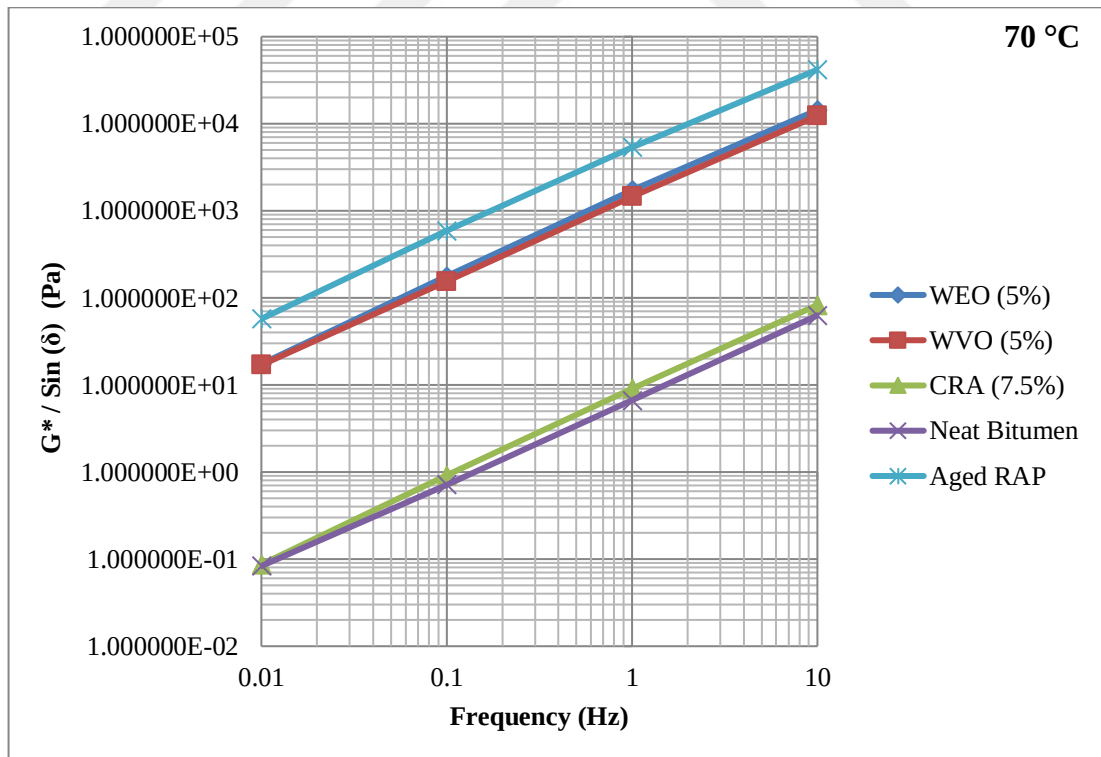


Figure 4.100 Optimally rejuvenated samples rutting parameters at 70°C

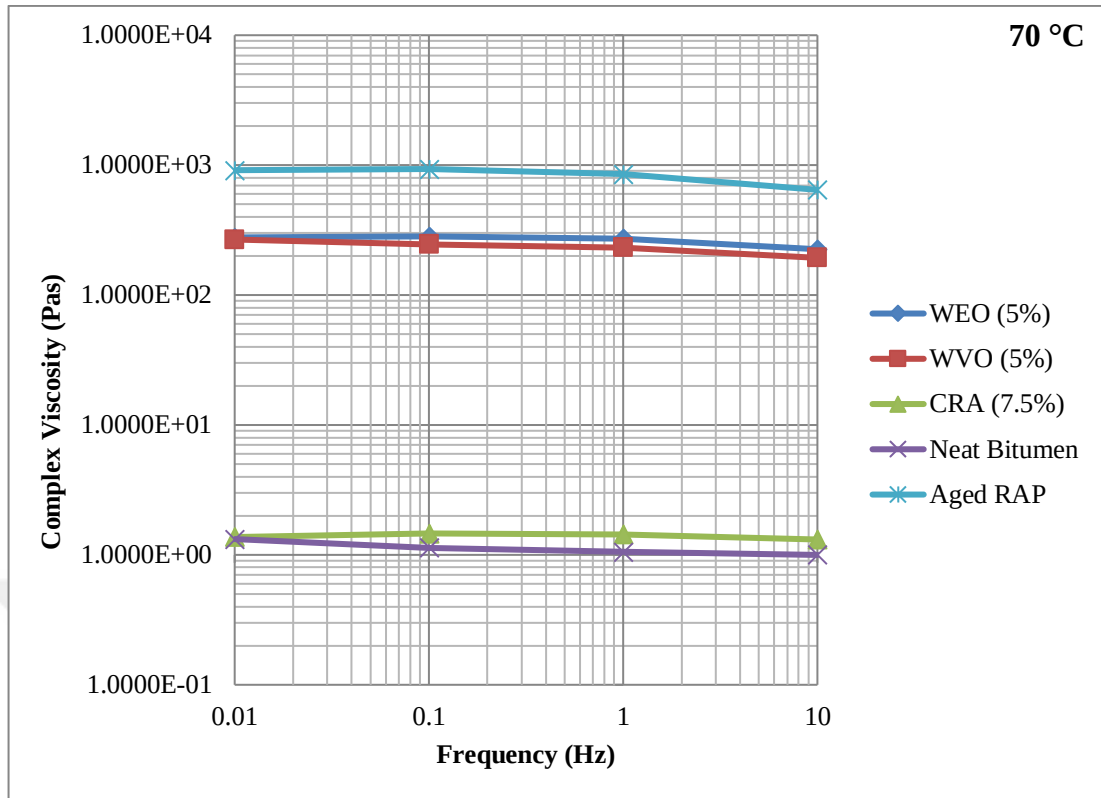


Figure 4.101 Optimally rejuvenated samples complex viscosities at 70°C

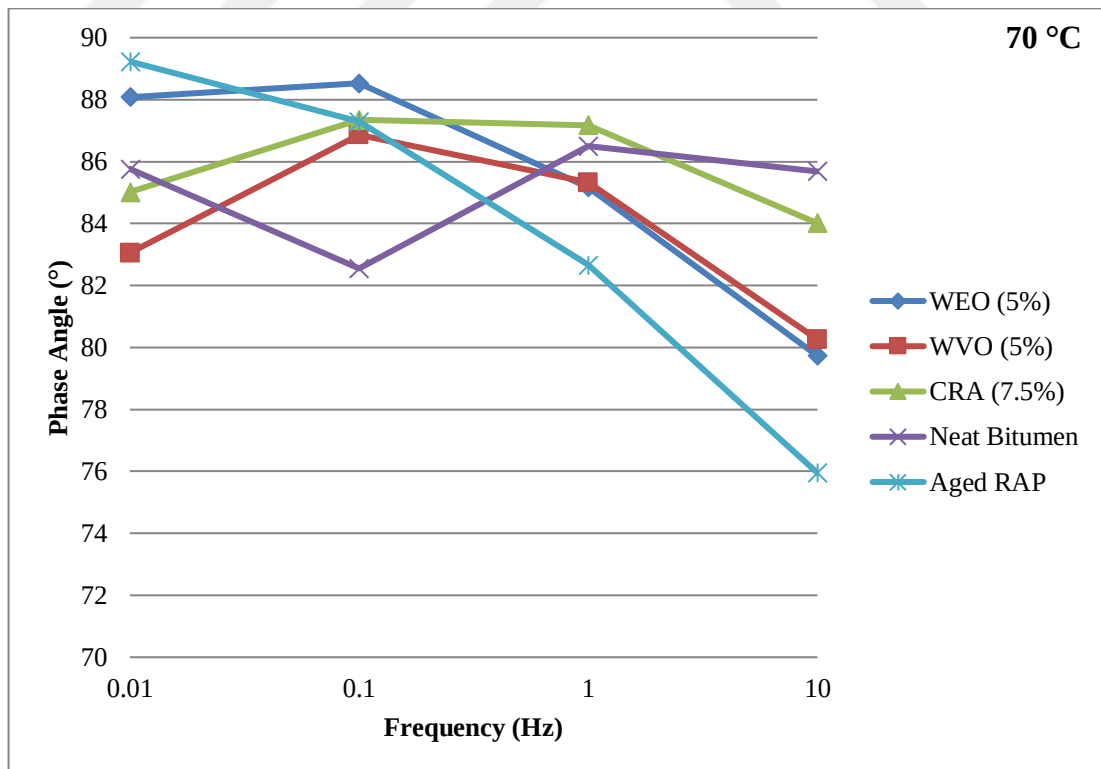


Figure 4.102 Optimally rejuvenated samples phase angles at 70°C

4.1.2.5 After RTFO Rheological Characteristics of Optimally Rejuvenated Bitumen Samples

One of the most concerned issues in bitumen rejuvenation is the capability of rejuvenated bitumen samples for preserving its characteristics especially during short term aging which corresponds the construction phases mainly including the manufacturing, hauling and application operations. As discussed, most rejuvenators are consisted of oily base materials. It is important to ensure that these oils remain within the binder or mixture and not evaporate during the construction phase. At this point, the evaporation point of the used oil is essential. The boiling point of WEO, WVO and CRA are ≥ 280 , ≥ 250 and ≥ 350 respectively. So, we can expect that the CRA rejuvenated samples be more resistant to short term aging compared to waste oils rejuvenated samples. On the other hand, we know that the volatilization is not the only factor affecting the aging phenomenon since other factors like oxidation, polymerization, thixotropy, syneresis and separations are essential.

The short-term aging as mentioned before, is simulated by RTFO aging process. Since we know that the rejuvenation should give out a long-lasting asphalt product, we can assume that the rejuvenated samples should be treated like a new-born fresh bitumen. So, not only in terms of investigations, but also in terms of regulations, all rejuvenated samples must meet the qualification criteria for after RTFO testing.

The Appendix contains the tables that summarize after RTFO test results for optimally rejuvenated bitumen samples. Test results show that the CRA represented better after RTFO performance than WEO and WVO agents since waste oils rejuvenated bitumen samples could meet the criteria for both conventional and Superpave specifications.

Figures 4.103 to 4.105, Figures 4.106 to 4.108, Figures 4.109 to 4.111 and Figures 4.112 to 4.114, respectively, demonstrate after RTFO test results at 0.01 Hz, 0.1 Hz, 1 Hz and 10 Hz for rutting parameters, complex viscosities and phase angles. The results

for rutting parameters and the complex viscosities have been plotted in logarithmic scale on Y axis to help better demonstration of the results.

As can be observed in Figures 4.103 to 4.14, all rejuvenated bitumen samples become stiffer and their DSR characteristics closes to the characteristics of aged RAP bitumen. Although short term aging has negative effects on the rejuvenated bitumen samples, all samples remained better than aged RAP bitumen. But we know that the aged RAP bitumen to some how has experienced a long term natural aging process. It is important to investigate the long term properties of rejuvenated bitumen samples. Neat bitumen sample could stand softer than all rejuvenated bitumen samples after RTFO aging. The CRA rejuvenated bitumen sample expectedly performed better than waste oils. Among waste oils, WVO exhibited slightly better performance than WEO. This can be attributed to the high temperature resistance of WVO as a vegetable frying oil. Engine oil should also have high resistance to high engine temperature, yet we know that a used engine oil like WEO may lack some of its properties giving it the ability to withstand high temperatures.

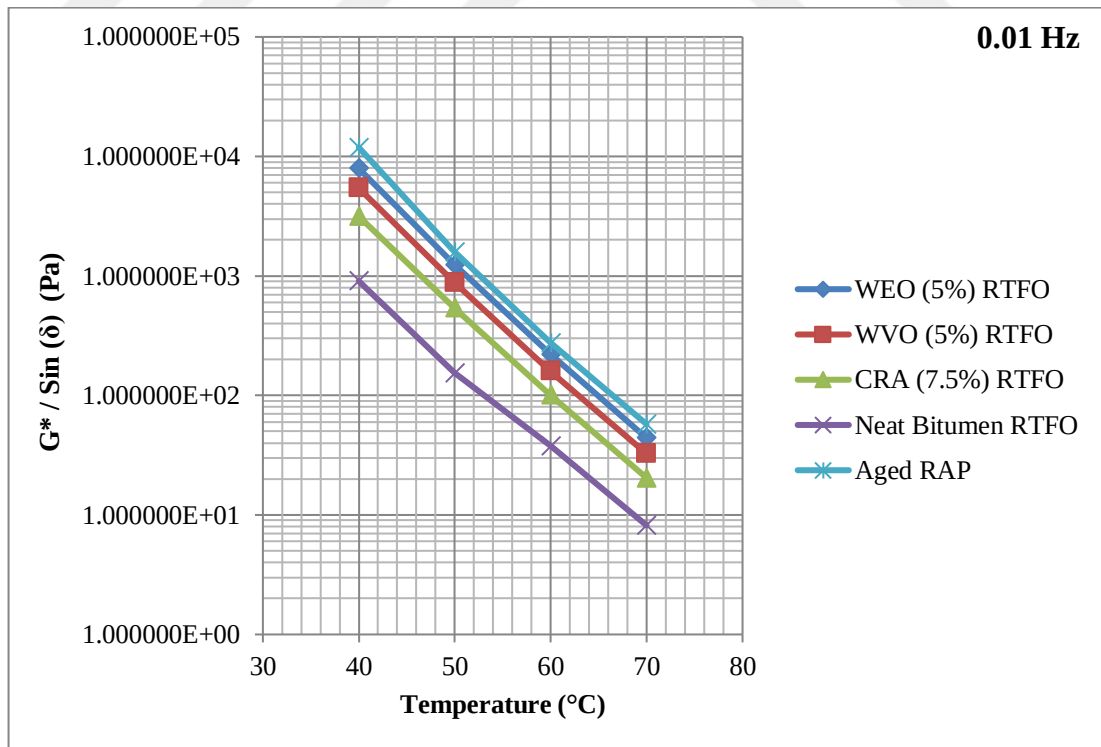


Figure 4.103 After RTFO optimum samples rutting parameters at 0.01 Hz frequency

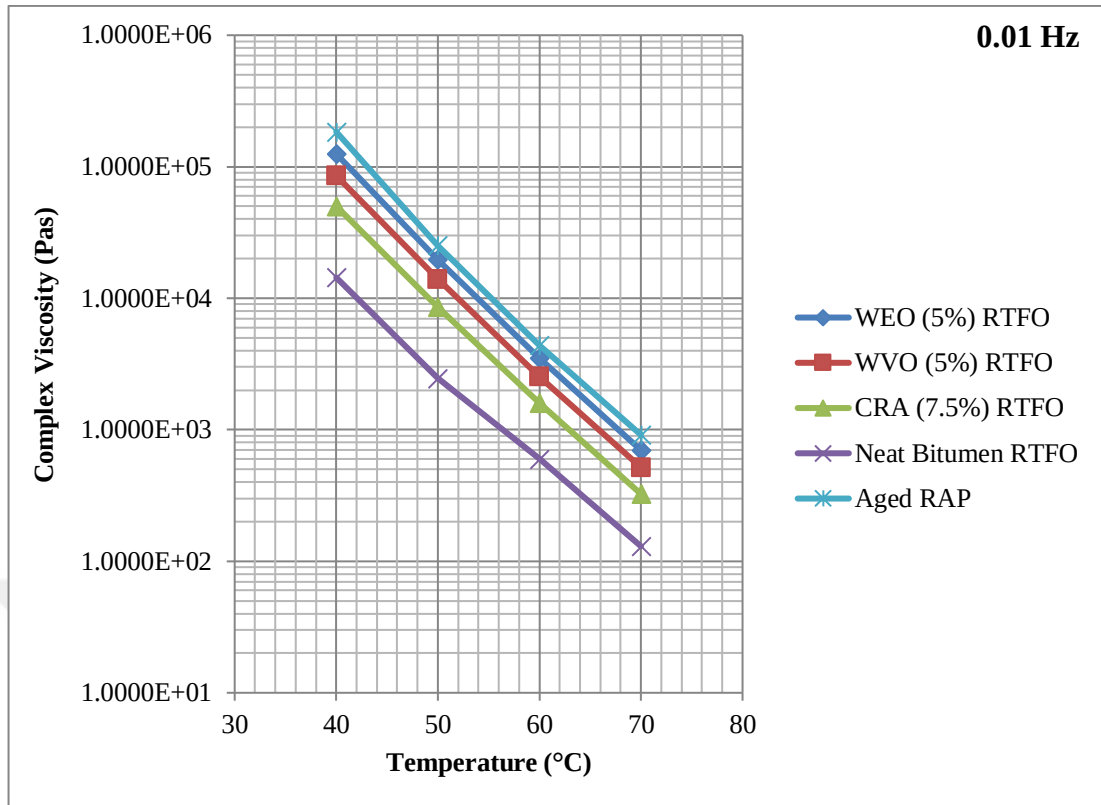


Figure 4.104 After RTFO optimum samples complex viscosities at 0.01 Hz

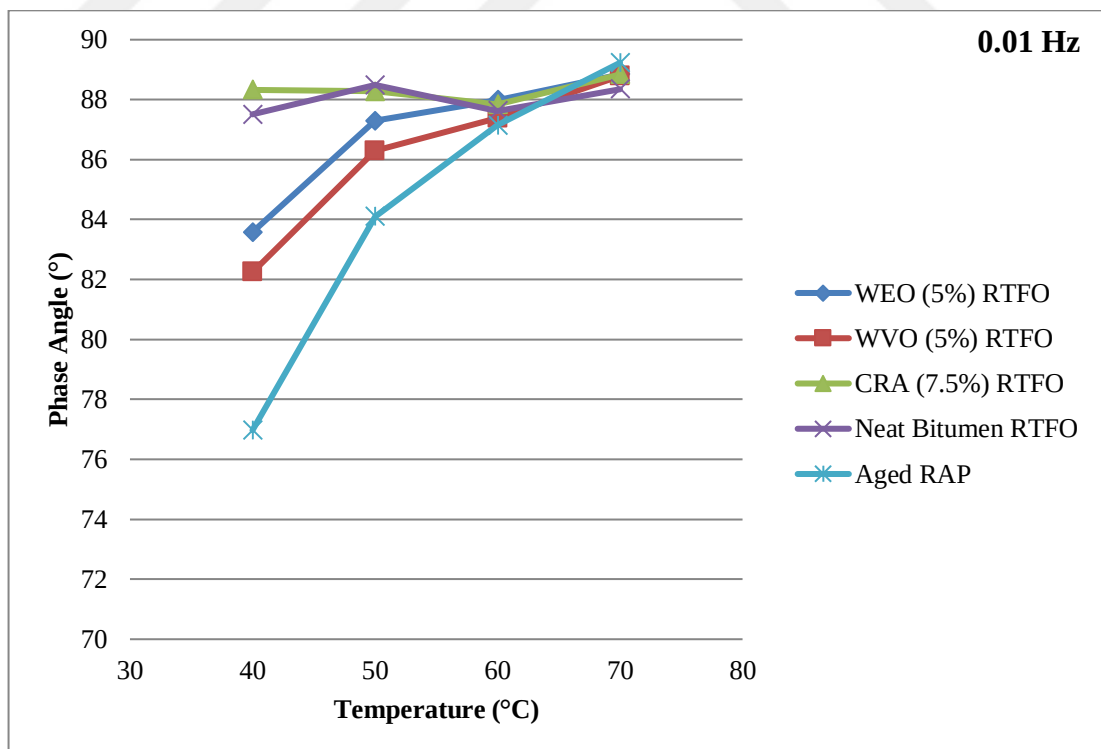


Figure 4.105 After RTFO optimum samples phase angles at 0.01 Hz frequency

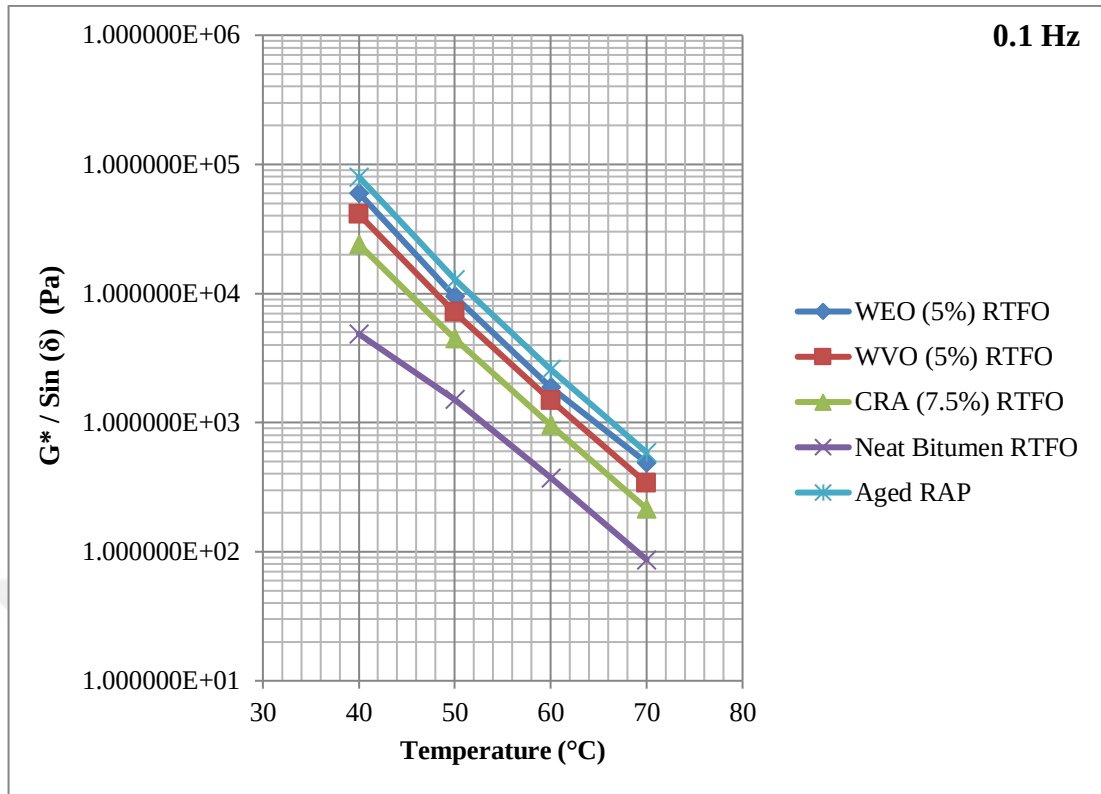


Figure 4.106 After RTFO optimum samples rutting parameters at 0.1 Hz frequency

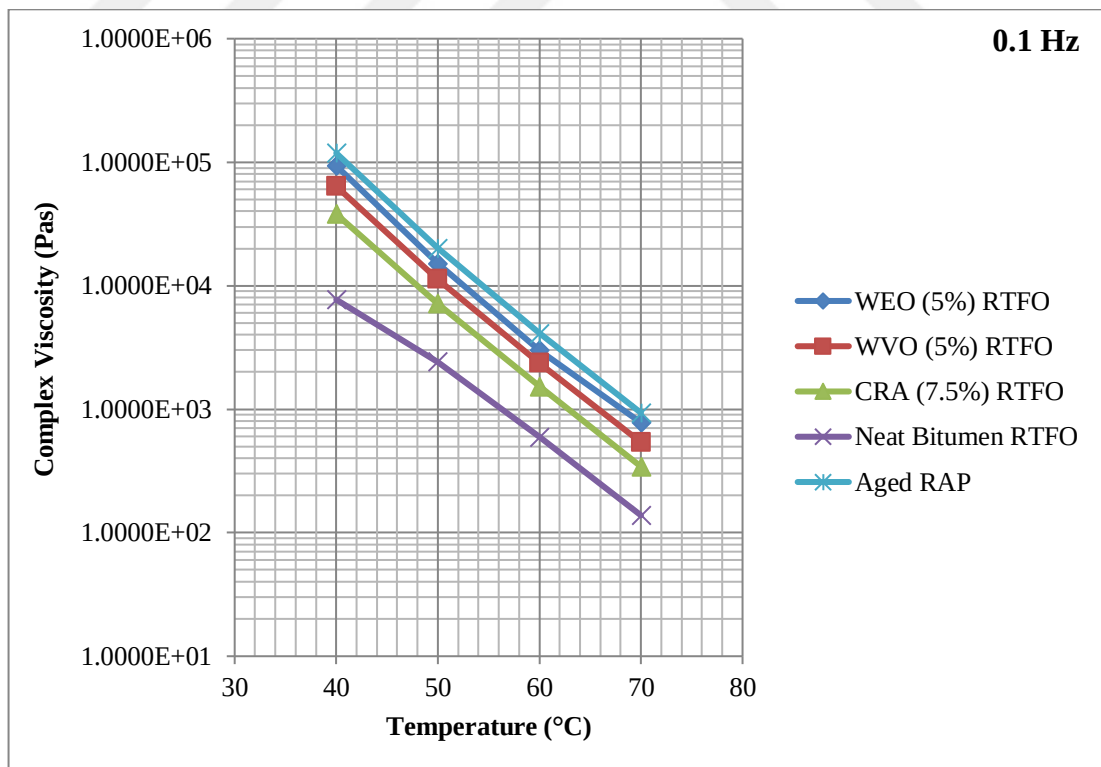


Figure 4.107 After RTFO optimum samples complex viscosities at 0.1 Hz frequency

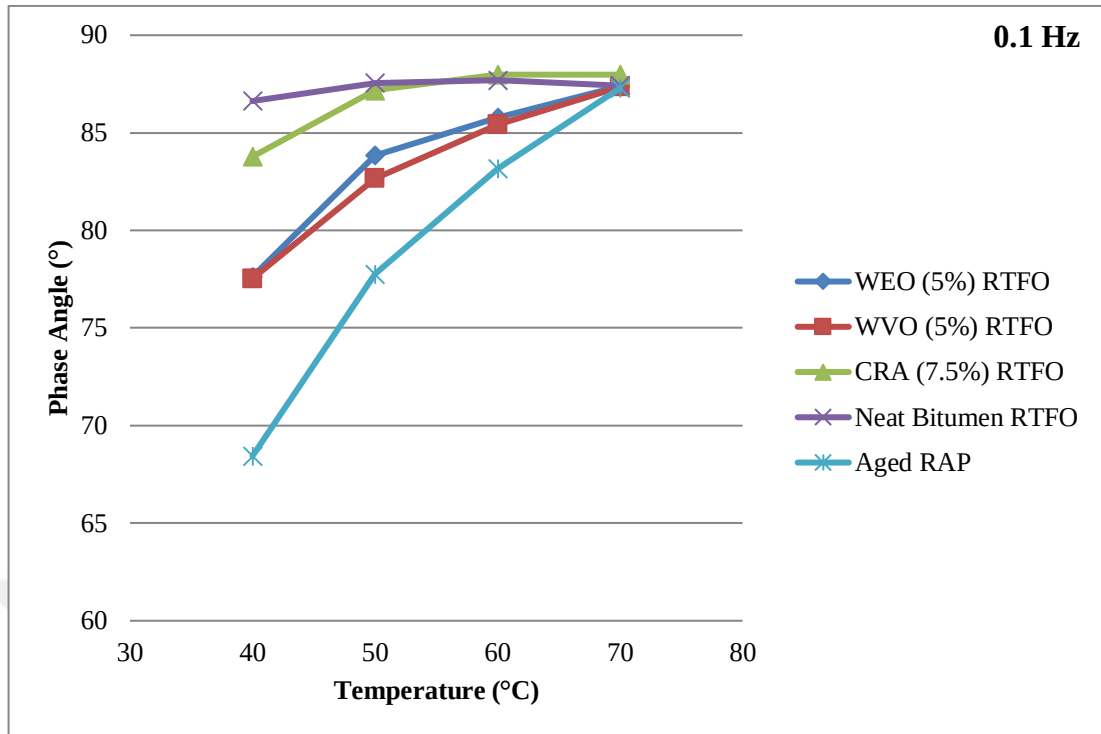


Figure 4.108 After RTFO optimum samples phase angles at 0.1 Hz frequency

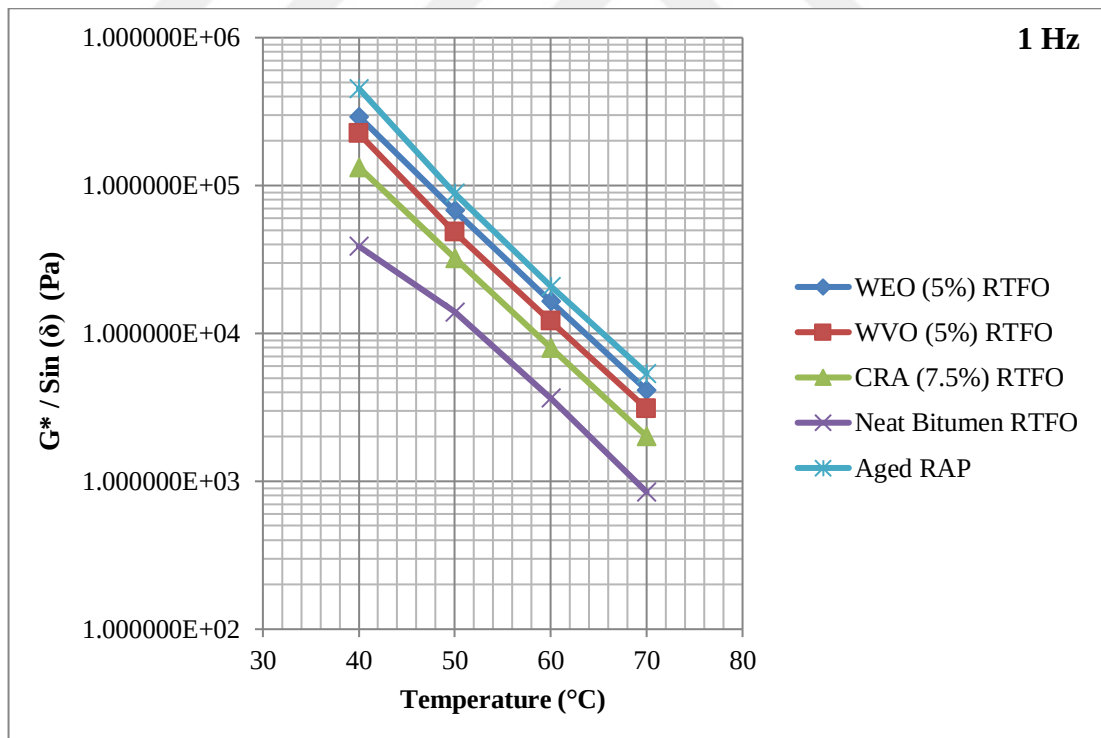


Figure 4.109 After RTFO optimum samples rutting parameters at 1 Hz frequency

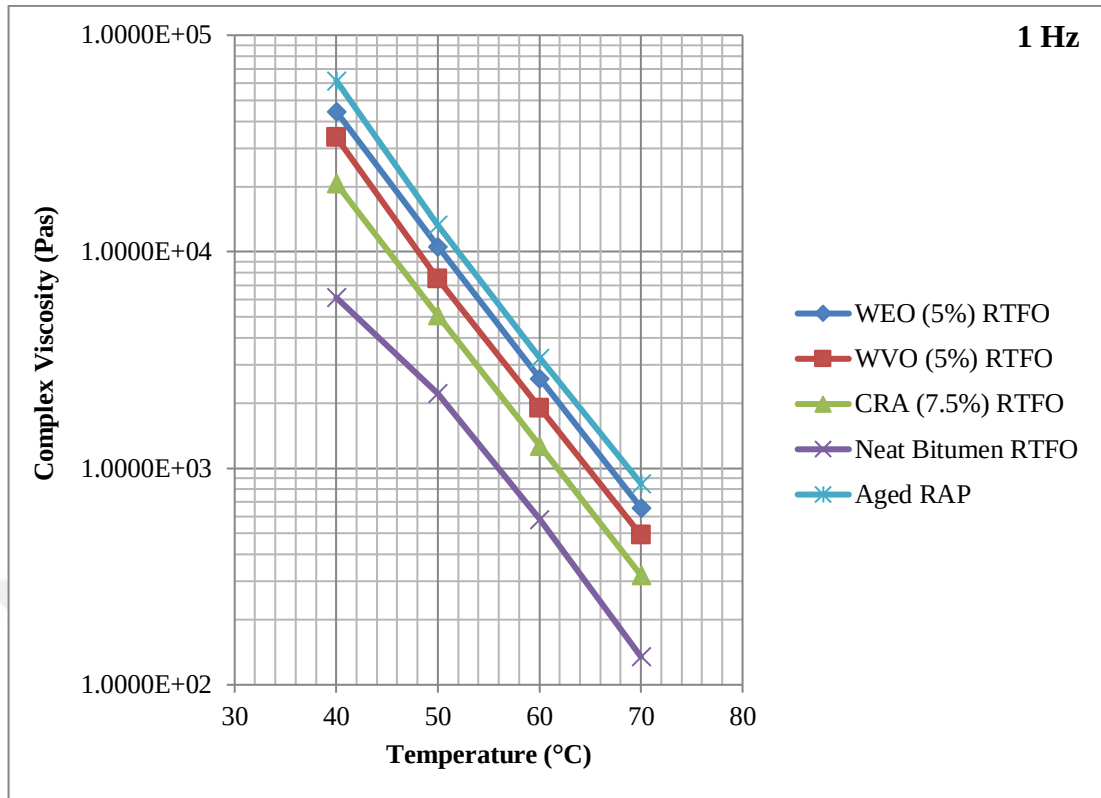


Figure 4.110 After RTFO optimum samples complex viscosities at 1 Hz frequency

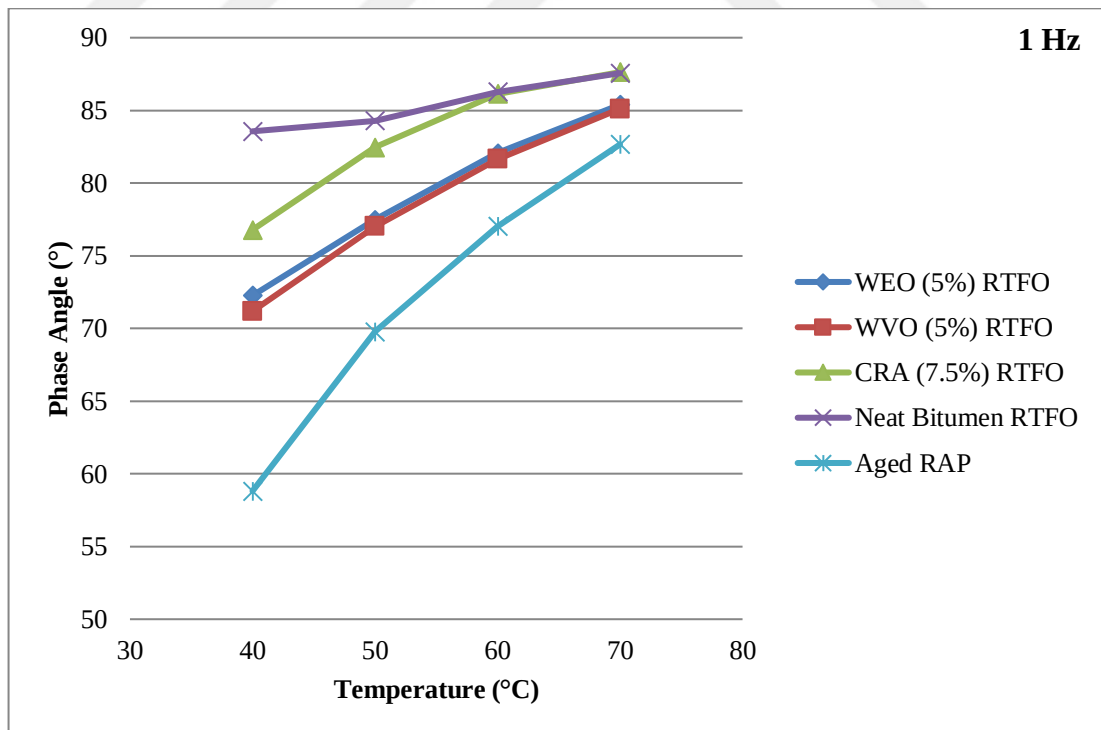


Figure 4.111 After RTFO optimum samples phase angles at 1 Hz frequency

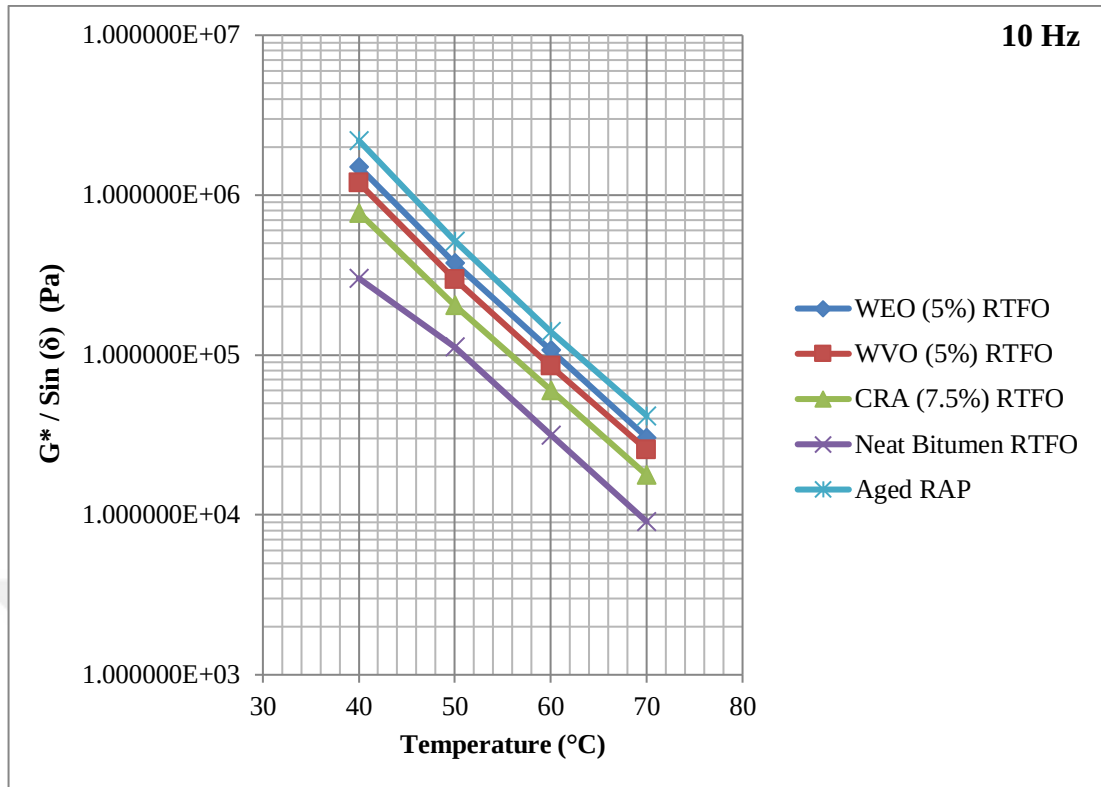


Figure 4.112 After RTFO optimum samples rutting parameters at 10 Hz frequency

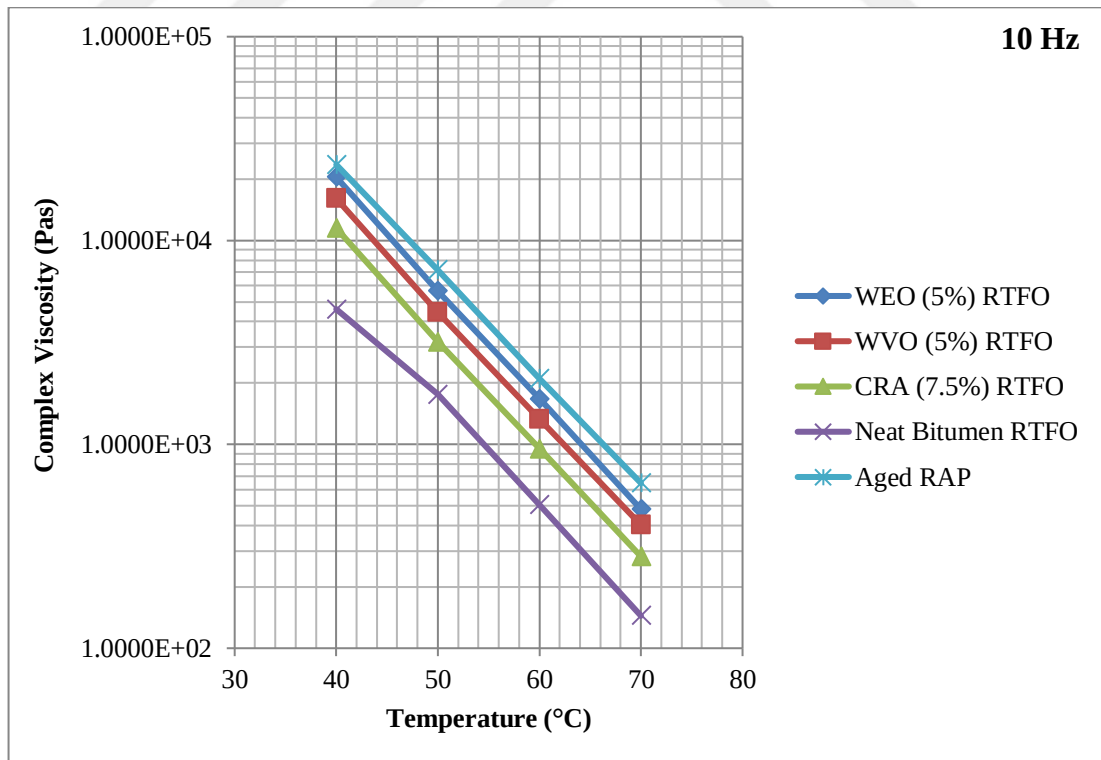


Figure 4.113 After RTFO optimum samples complex viscosities at 10 Hz frequency

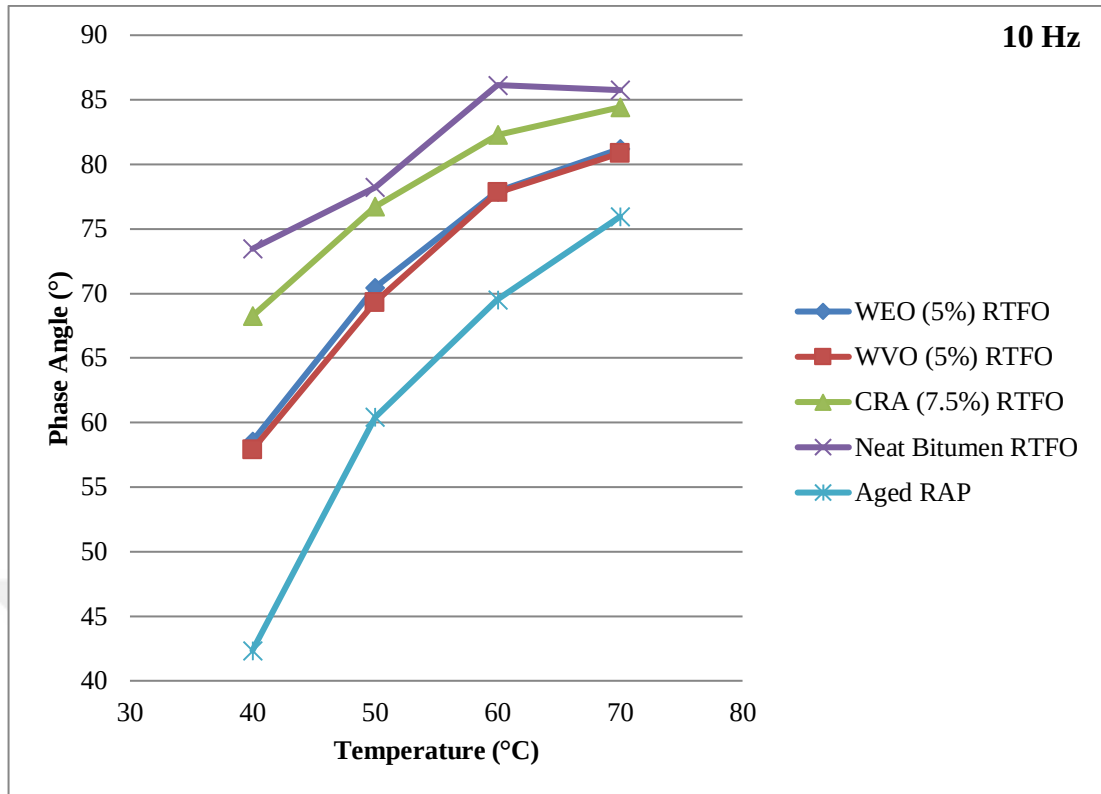


Figure 4.114 After RTFO optimum samples phase angles at 10 Hz frequency

Figures 4.105 to 4.117, Figures 4.118 to 4.120, Figures 4.121 to 4.123 and Figures 4.124 to 4.126, respectively, demonstrate after RTFO test results at 40 °C, 50 °C, 60 °C and 70 °C for rutting parameters, complex viscosities and phase angles. Like previous plots, the results for rutting parameters and the complex viscosities have been plotted in logarithmic scale on Y axis to help better demonstration of the results.

In terms of rutting parameters, all rejuvenated samples at each temperature exhibited an exponential (linear in logarithmic scale) increase by increasing of the frequency level after RFTO aging process. A slight drop in logarithmic scale can be observed for complex viscosities for all samples almost at every temperature by a tenfold increase in the frequency levels. As expected, phase angles were recorded low for lower temperatures and high frequencies.

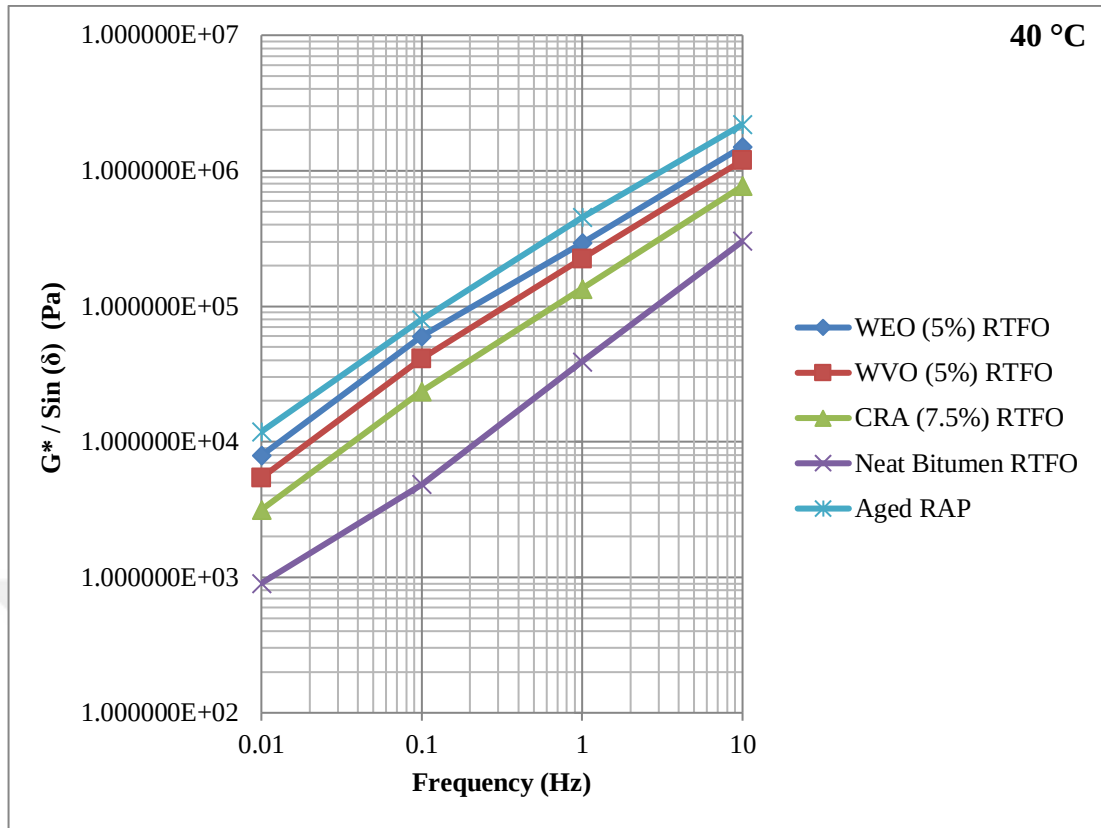


Figure 4.115 After RTFO optimum samples rutting parameters at 40°C

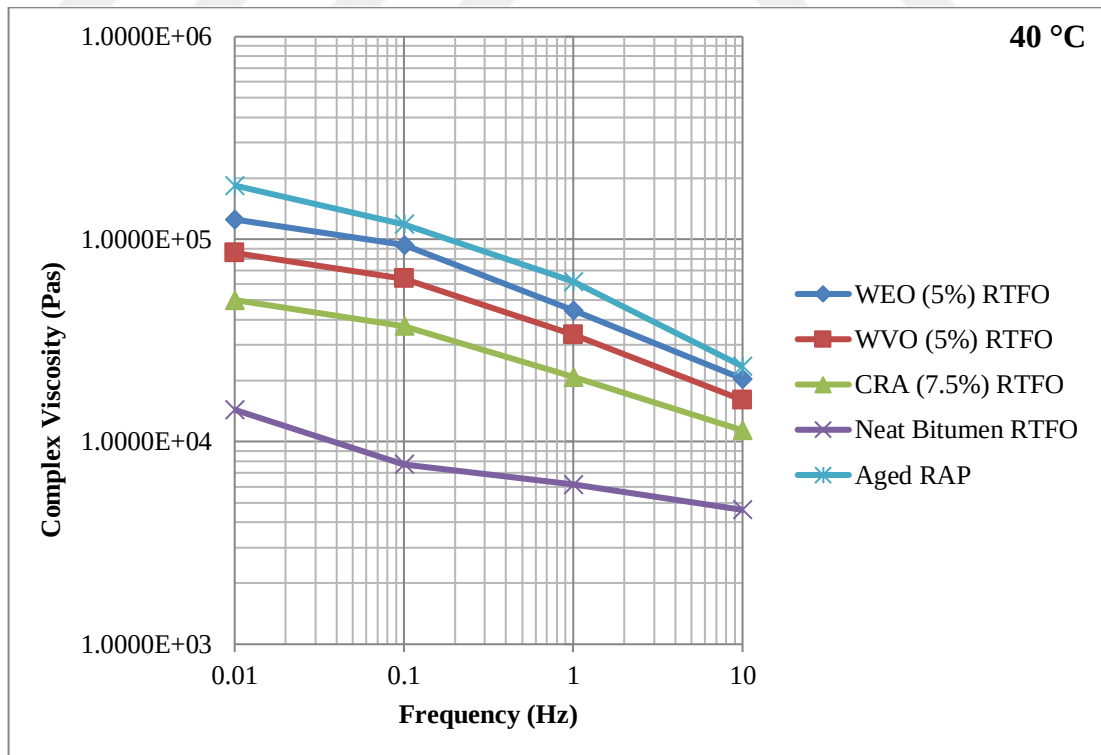


Figure 4.116 After RTFO optimum samples complex viscosities at 40°C

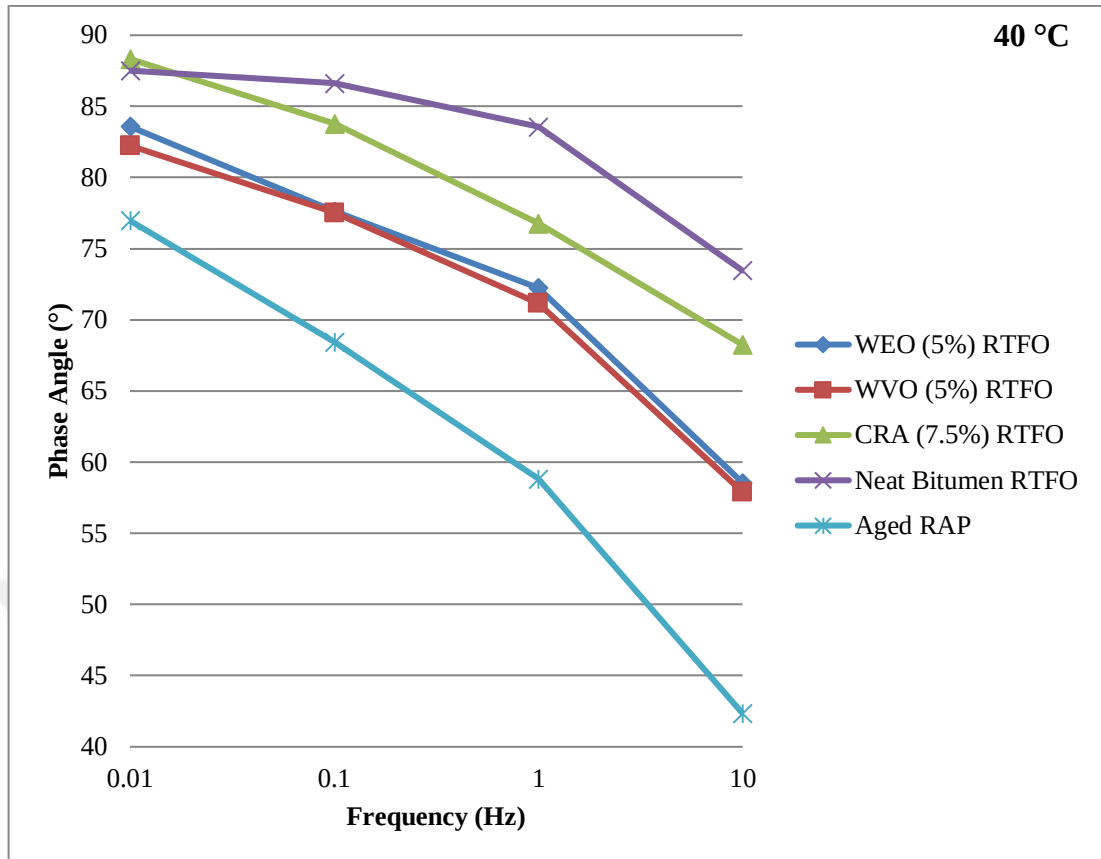


Figure 4.117 After RTFO optimum samples phase angles at 40°C

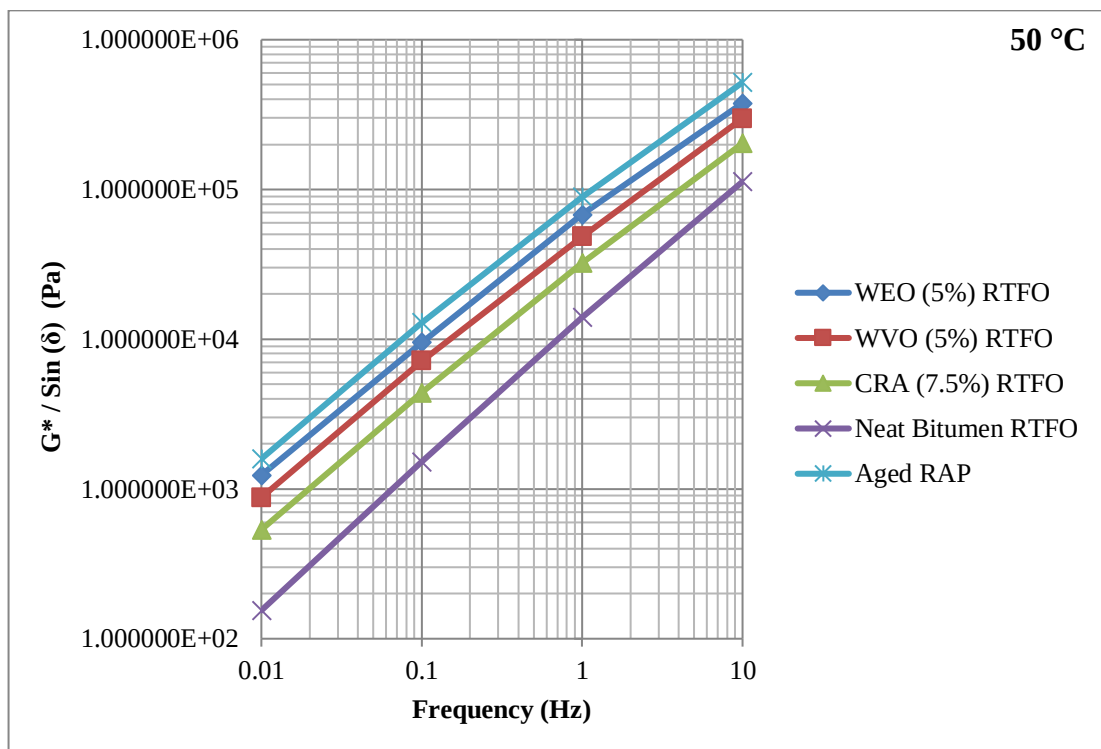


Figure 4.118 After RTFO optimum samples rutting parameters at 50°C

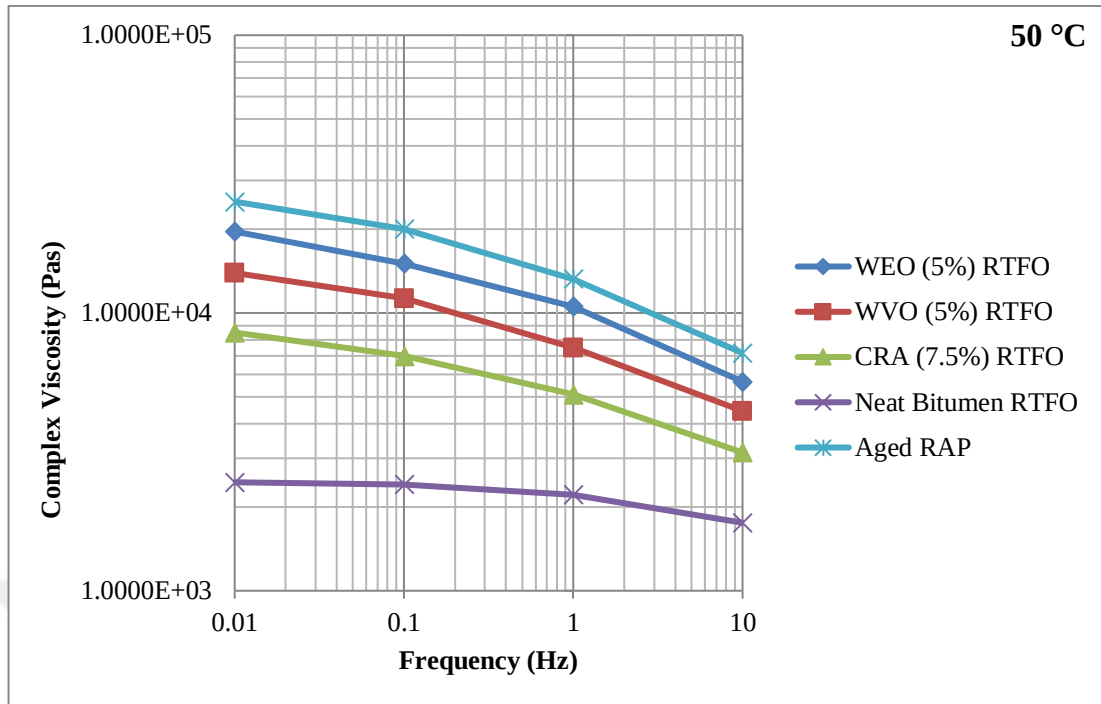


Figure 4.119 After RTFO optimum samples complex viscosities at 50°C

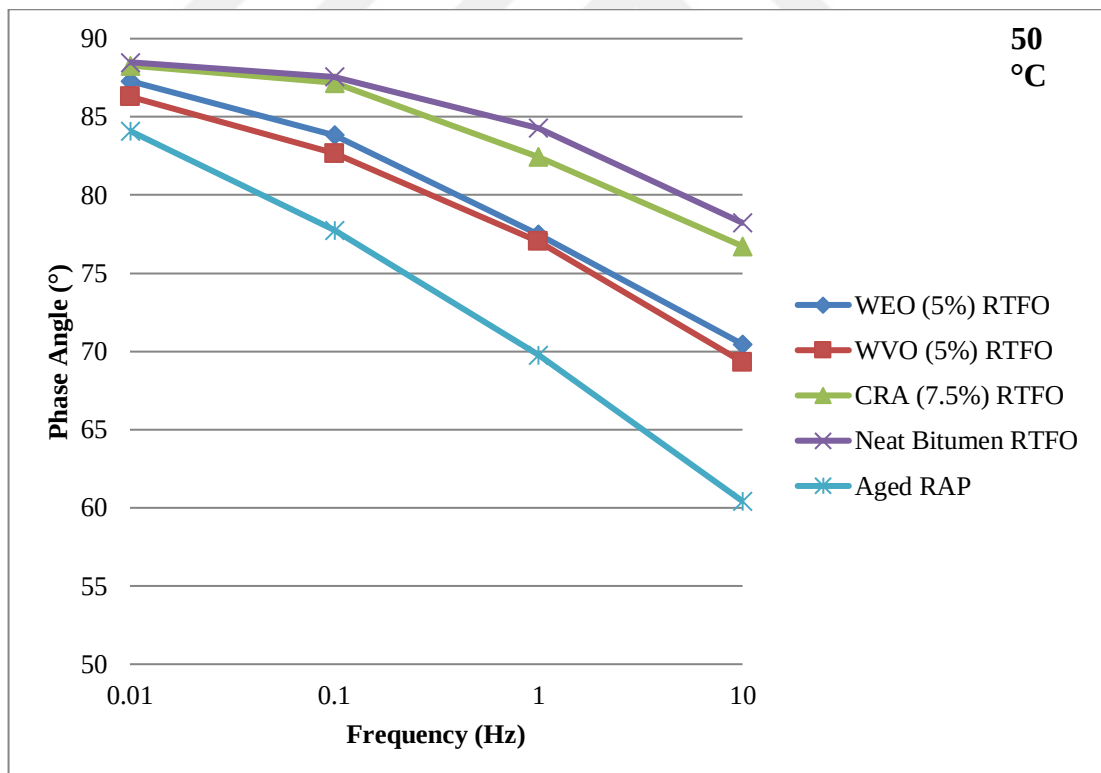


Figure 4.120 After RTFO optimum samples phase angles at 50°C

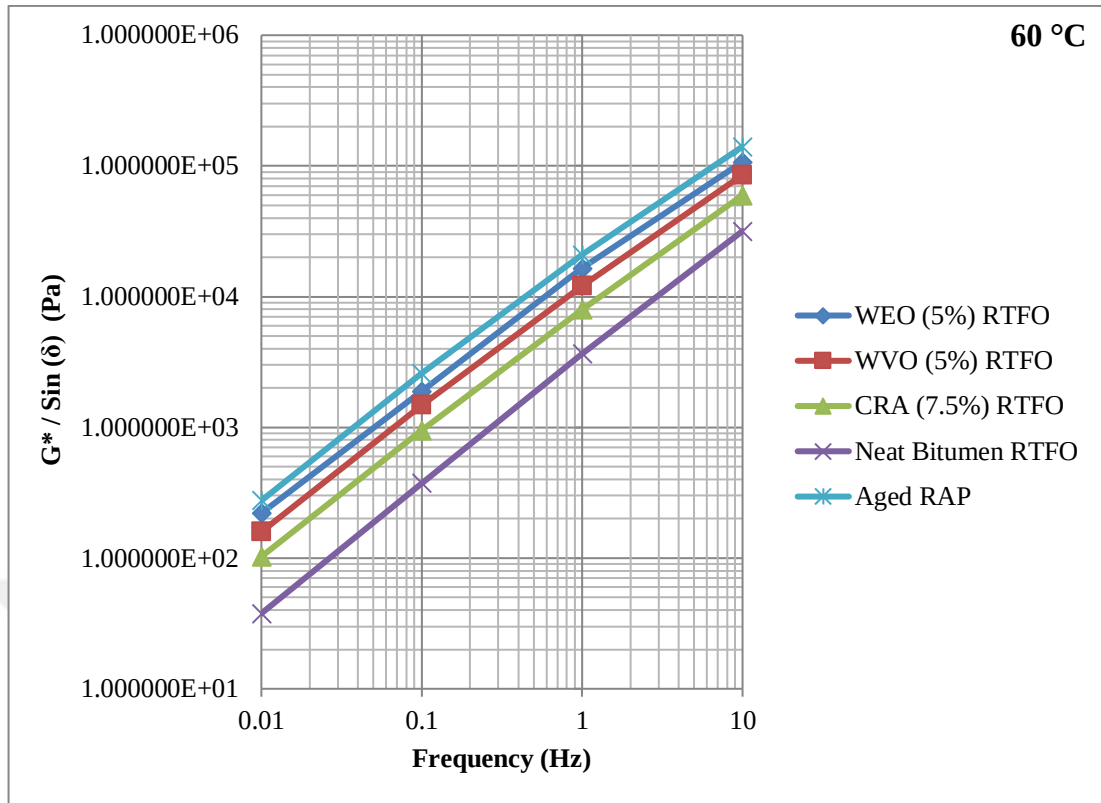


Figure 4.121 After RTFO optimum samples rutting parameters at 60°C

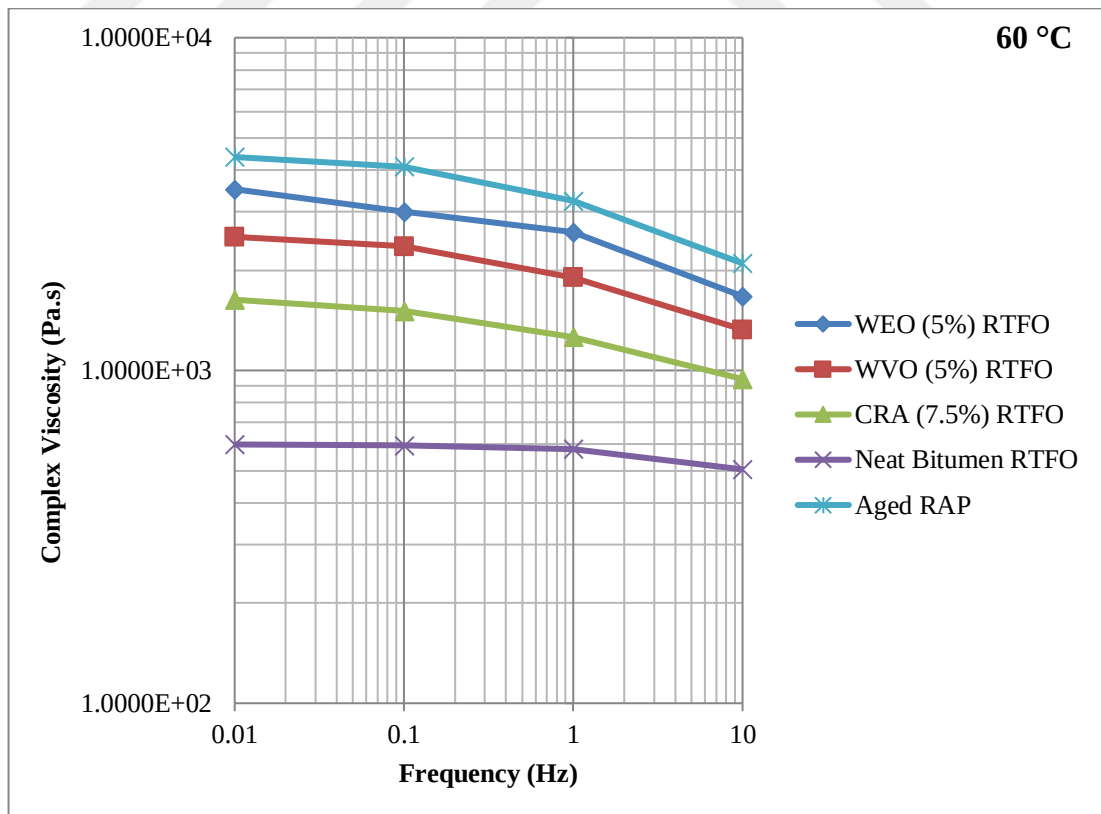


Figure 4.122 After RTFO optimum samples complex viscosities at 60°C

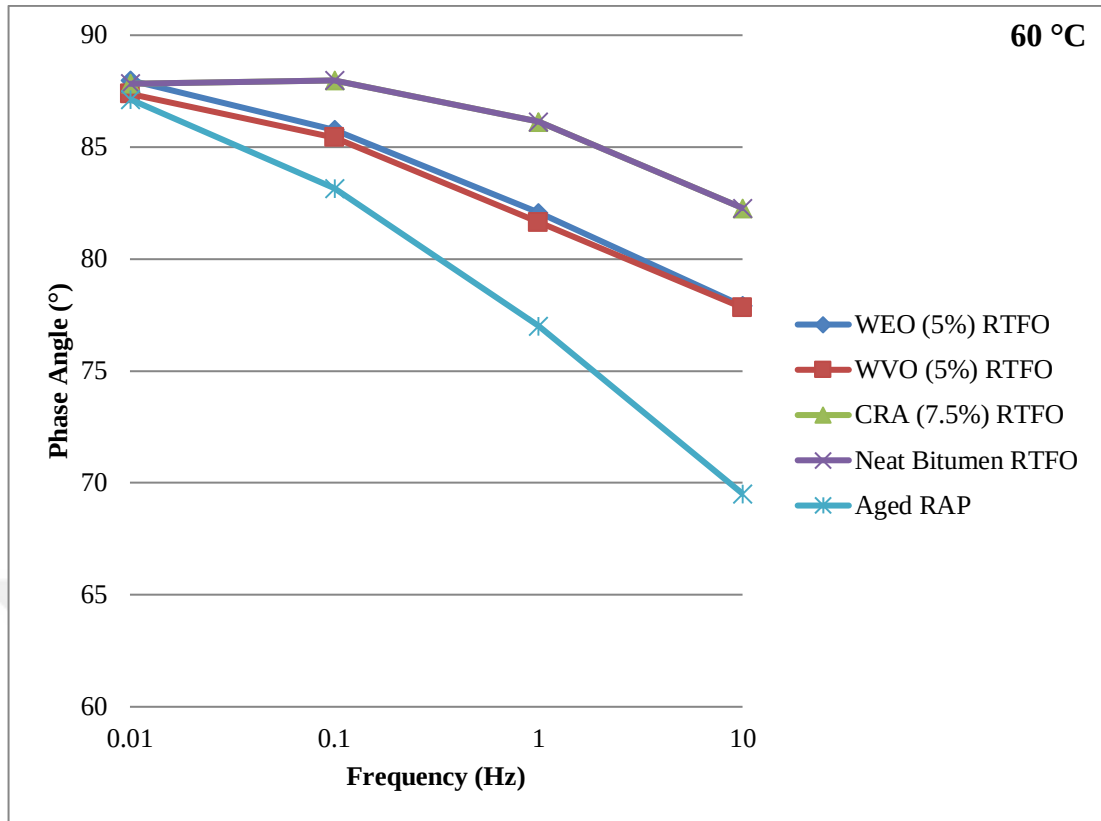


Figure 4.123 After RTFO optimum samples phase angles at 60°C

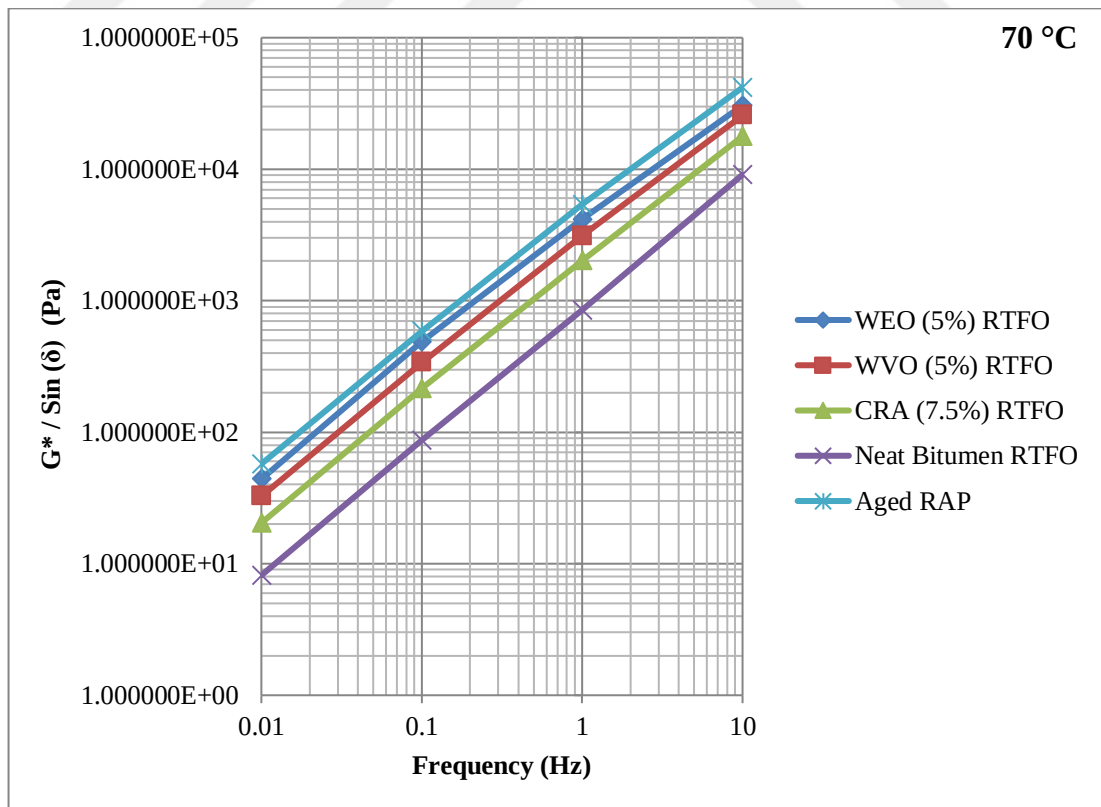


Figure 4.124 After RTFO optimum samples rutting parameters at 70°C

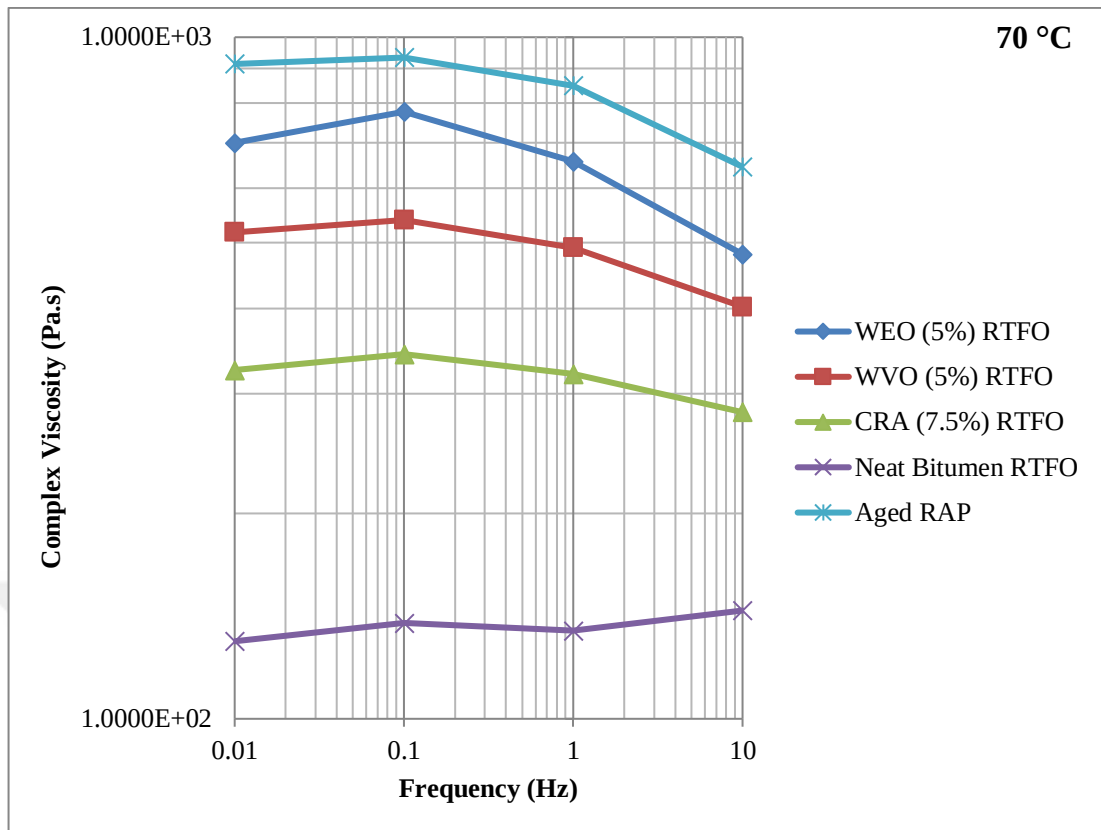


Figure 4.125 After RTFO optimum samples complex viscosities at 70°C

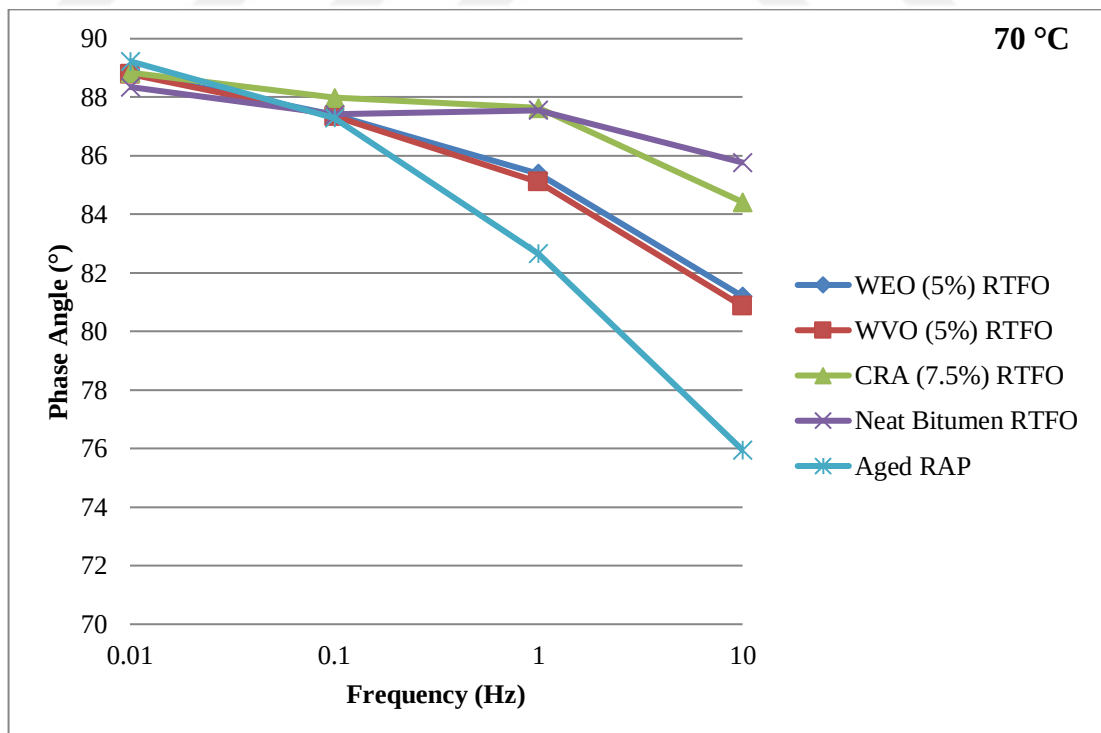


Figure 4.126 After RTFO optimum samples phase angles at 70°C

4.1.2.6 Performance Gradings of Optimally Rejuvenated Bitumen Samples

As discussed, the rejuvenated bitumen samples should be treated like new -bitumen samples which can fulfill every specifications limit. All optimally rejuvenated bitumen samples were subjected to PG test to determine new born samples performance gradings upper limits. Although penetration grading was the reference grading system which was considered within this study, performance gradings had also to be confirmed for the optimally rejuvenated bitumen samples according to AASHTO T315.

The catalogue upper limit of neat bitumen was confirmed as 64 °C. In case of rejuvenated samples, we know that the lower PG limit is most concerned than the upper limit since the upper limit must be checked for the rejuvenated samples to avoid high temperatures distresses.

Normally the upper limit of an aged bitumen can be recorded higher than a neat bitumen yet having trouble with after RTFO limits. The upper limit of the extracted RAP bitumen was recorded as 76 °C. The upper limits of rejuvenated bitumen samples were recorded as 76 °C, 70 °C and 64 °C respectively for WEO, WVO and CRA rejuvenated bitumen samples. Following RTFO limit checking, all samples' high PG limits were detected as 64 °C. The results showed conformity with the conventional test results. The PG results presented within Table 4.5. The Appendix also include the detailed results of PG inspections.

Figures 4.127 to 4.131, respectively, show the rutting parameters, phase angles, complex modulus, elastic modulus and viscous modulus of all bitumen samples. As can be seen in all these figures, the CRA rejuvenated sample behaved so similar to neat bitumen sample since waste oils rejuvenated samples demonstrated a performance between neat bitumen and aged RAP bitumen in terms of rheological characteristics at higher temperatures. WVO acted somewhat better than WEO as an asphalt rejuvenator.

Table 4.5 After RTFO $G^*/\sin(\delta)$ values @ 64°C

Neat Bitumen								
Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	$G^*/\sin(\delta)$
(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(kPa)
64	85.03	3.5278E+03	3.0563E+02	3.5146E+03	3.5278E+02	3.54E+02	1.00E-01	3.541136E+00

Extracted Aged RAP Bitumen + 5.4% WEO								
Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	$G^*/\sin(\delta)$
(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(kPa)
64	71.64	1.6756E+04	5.2781E+03	1.5904E+04	1.6756E+03	1.70E+03	1.01E-01	1.76552E+01

Extracted Aged RAP Bitumen + 5.1% WVO								
Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	$G^*/\sin(\delta)$
(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(kPa)
64	71.64	1.4724E+04	4.6377E+03	1.3974E+04	1.4724E+03	1.49E+03	1.01E-01	1.55133E+01

Extracted Aged RAP Bitumen + 7.5% CRA								
Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	$G^*/\sin(\delta)$
(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(kPa)
64	71.64	4.7690E+03	1.5022E+03	4.5262E+03	4.7690E+02	4.79E+02	1.00E-01	5.02474E+00

The rutting performance of all the samples decrease almost linearly in the logarithmic scale by increasing the temperature.

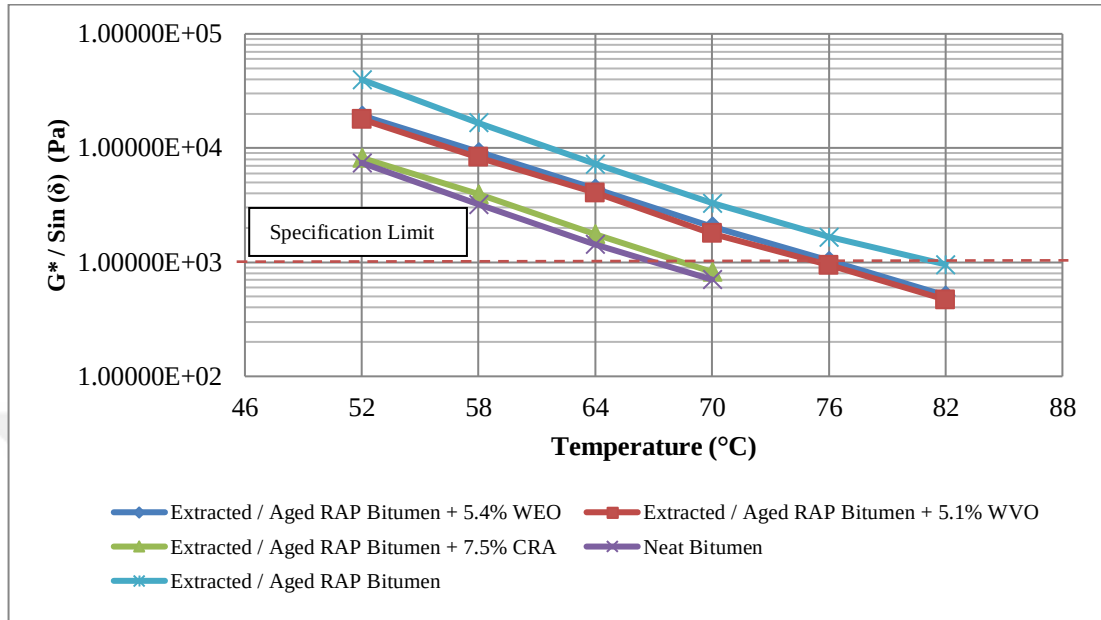


Figure 4.127 PG test rutting parameters corresponding to temperature variations

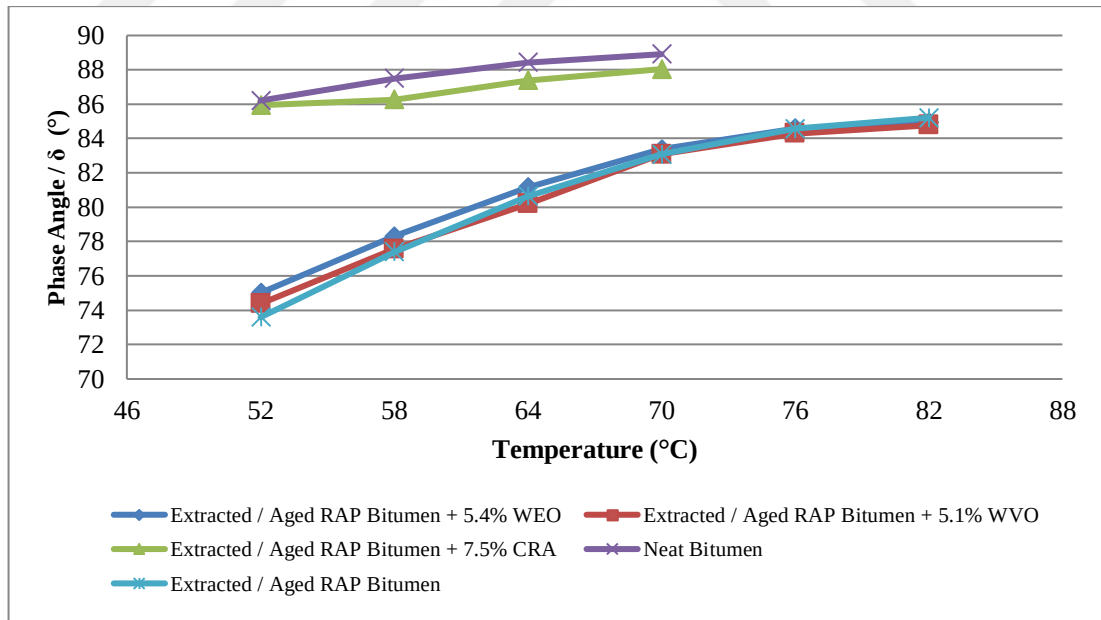


Figure 4.128 PG test phase angles corresponding to temperature variations

Figure 4.132 shows the rutting parameters values checked as per AASHTO T315 specification limits. $G^*/\sin(\delta)$ must be equal or greater than 2.2 kPa. As can be observed, all rejuvenated and neat bitumen samples exhibited greater values than the

specification limit after RTFO aging. CRA rejuvenated sample performed better than the waste oils in this context. All results are in high conformity with the previous DSR testing results. WVO's rutting parameter is slightly lower than WEO's value. It means that WVO rejuvenated sample remains still softer than WEO rejuvenated sample following the RTFO aging process.

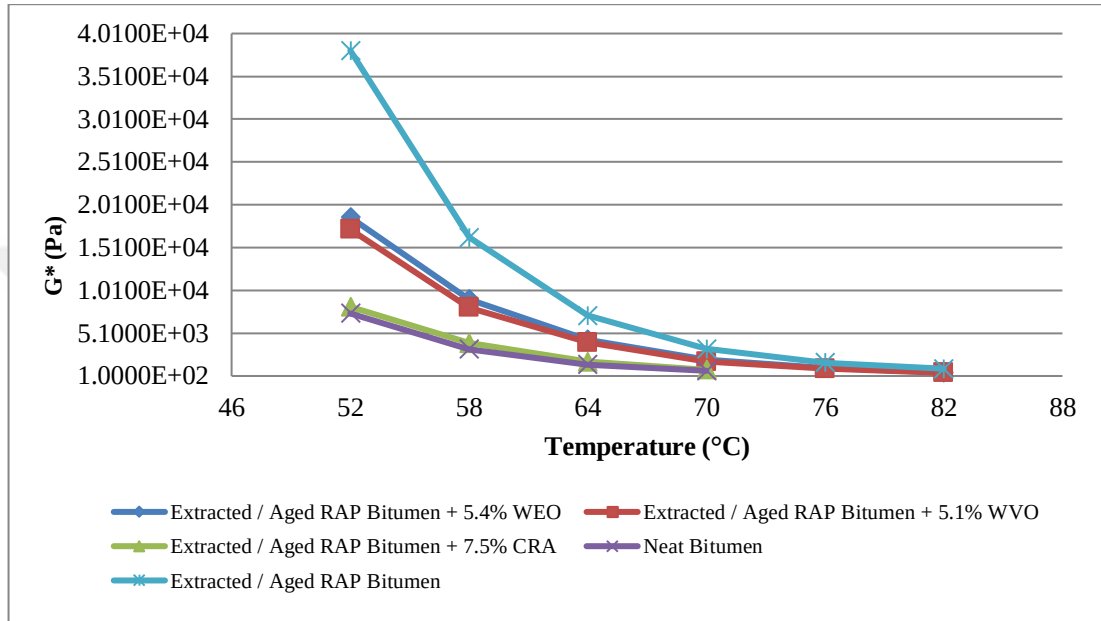


Figure 4.129 PG test complex moduli corresponding to temperature variations

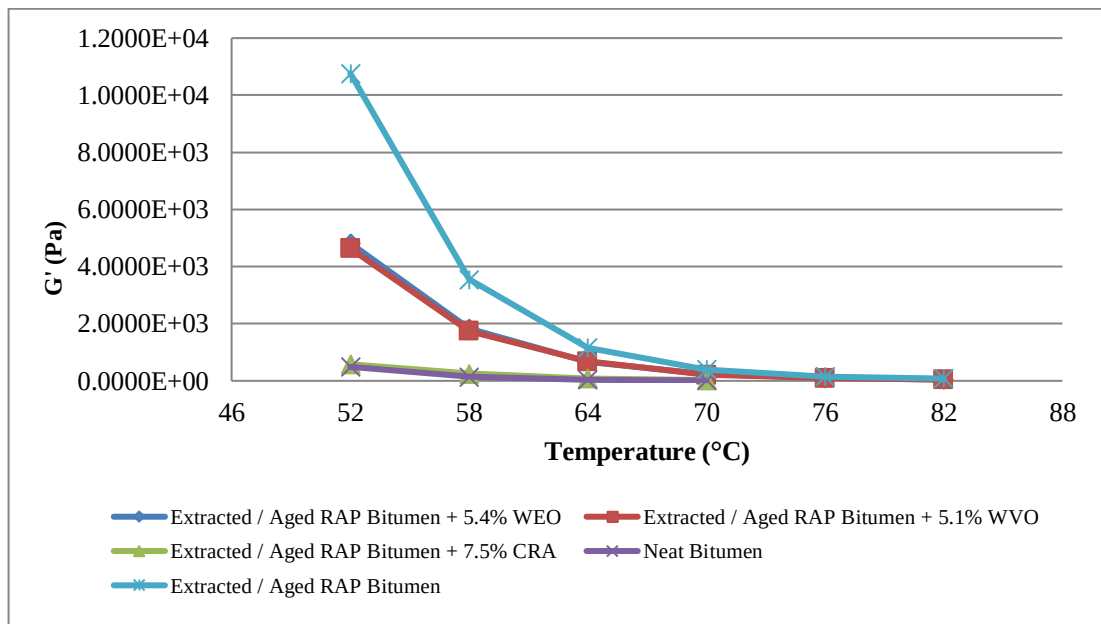


Figure 4.130 PG test elastic moduli corresponding to temperature variations

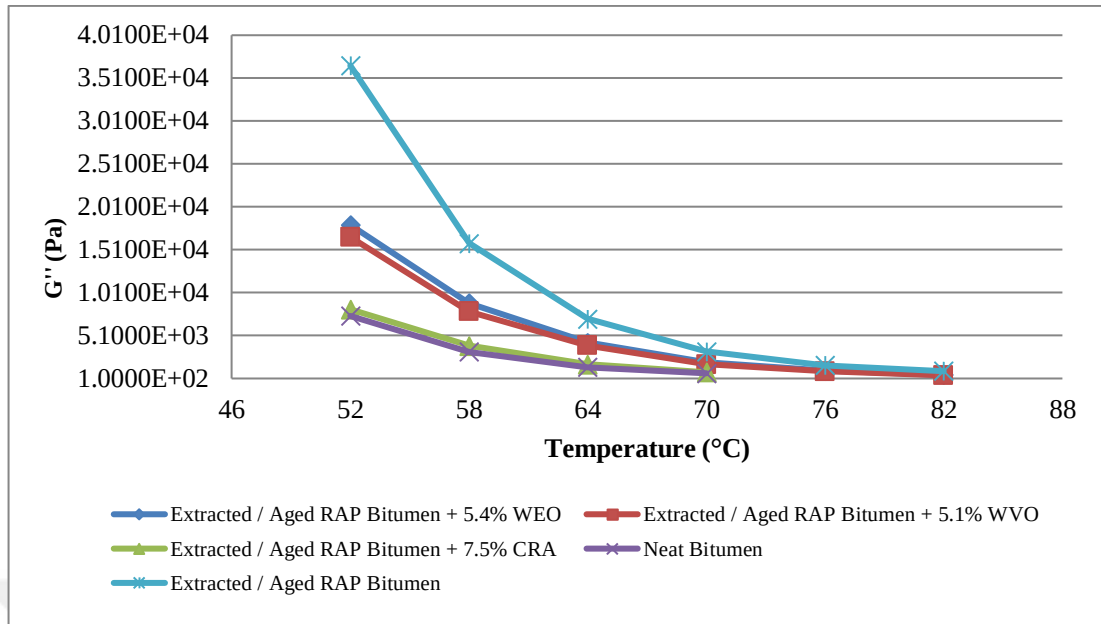


Figure 4.131 PG test viscous moduli corresponding to temperature variations

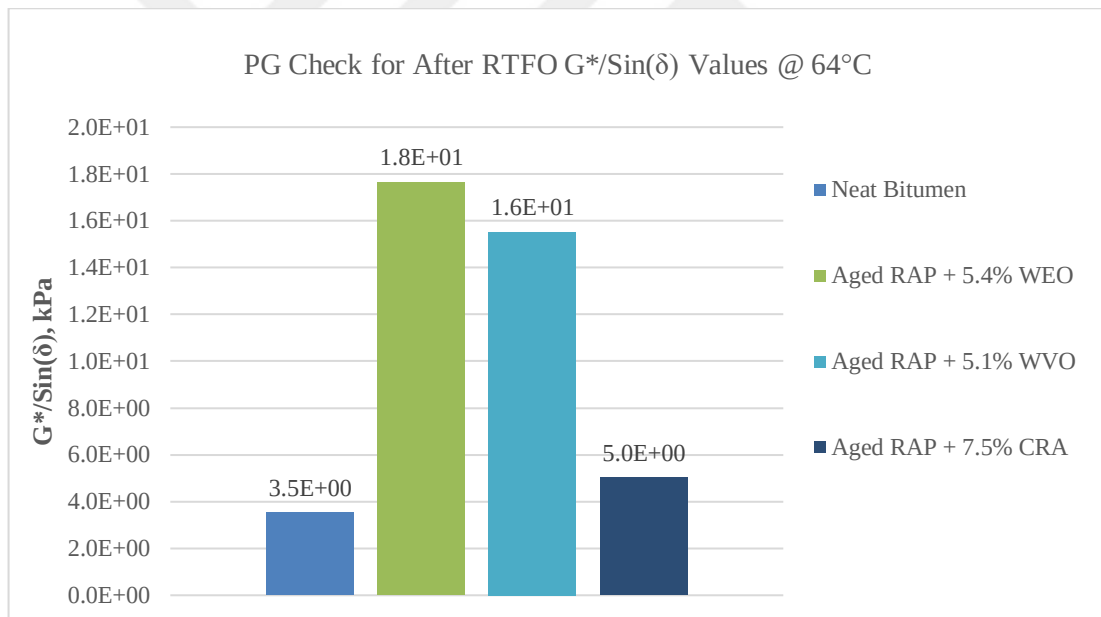


Figure 4.132 PG check for after RTFO testing

4.1.3 Morphological Test Results

Within the scope of this study, two different microscopy methods were used to differentiate the rejuvenated and non-rejuvenated bitumen samples. As discussed in Chapter 2 of this dissertation, there are many studies related to AFM and the aging of asphalt binder which prove that the morphology of bitumen changes through aging

process. As mentioned within the literature review, dark and light bars so-called bee structures which are visible on un-aged neat bitumen images disappear on aged bitumen images. Within the scope of this study, the AFM microscopy was employed to investigate these differences on aged RAP bitumen, neat bitumen and rejuvenated RAP bitumen samples.

In addition to AFM, SEM was used to investigate the differences of images drawn through electron microscopy between aged RAP bitumen, neat bitumen and rejuvenated RAP bitumen samples. As presented within the following sub-sections, AFM images were successful to differentiate the morphological properties of aged RAP bitumen, neat bitumen and rejuvenated RAP bitumen samples since SEM images and the methodology used in this dissertation did not help as the images were subject to sampling method and many other factors.

4.1.3.1 Atomic Force Microscopy Results

Figures 4.133 to 4.143 respectively show the AFM images of neat bitumen, Aged RAP bitumen, 3% WEO rejuvenated, 5% WEO rejuvenated, 7% WEO rejuvenated, 3% WVO rejuvenated, 5% WVO rejuvenated, 7% WVO rejuvenated, 5.5% CRA rejuvenated, 7.5% CRA rejuvenated and 9.5% CRA rejuvenated bitumen samples.

All figures include 3 different images Z-axis (a), Phase scan (b) and Amplitude (c) of the same sample's scan. All images are squares with dimensions set to 15 μm . Z-axis shows the actual topography of the sample since phase scan is to recognize varying stiffness or different adhesion properties which is not visible in the topographic Z-axis image. On the other hand, variations in the oscillation amplitude or phase provide the response signal for imaging. Therefore, in amplitude range, variations in the phase of oscillation helps to distinguish different types of materials on the sample's surface. In all three kind of images, bee structures can be seen on the surface of the samples. It can be concluded that the differences in images between aged and un-aged samples prove the existence of a material which is found at micro-surface

dimension. These bee-structures are from a different source and give the sample's surface in micro level a different topography.

Figure 4.133 shows the AFM images of neat bitumen samples. 1.5 μm to 2.5 μm lengths bee-structures can be seen in Z-axis image. Phase scan shows the differences in stiffness of material on the exact coordinates on which the bee-structures are located. The amplitude image shows that these bee structures are from another source different from other parts of the bitumen sample. Almost 40-50 numbers of bee-structures can be counted in 225 μm^2 of the surface of the neat bitumen sample. On the other hand, Figure 4.134 contains the AFM images of extracted aged RAP bitumen sample. As shown in Figure 4.134, except dark and light areas, no bee-structures can be found on the surface of the aged RAP bitumen sample due to the changes in phase polarity. The phase scan and the amplitude images of the same sample show almost a homogenous material with no polarity in terms of stiffness and material type.

The rejuvenated samples with three dosages of rejuvenating agents were also investigated by AFM imaging. Rejuvenated bitumen samples are obtained by adding rejuvenating agents into aged RAP bitumen through stirring in standardized method explained within the Chapter 3 to ensure a homogenous bitumen sample. In micro levels, we do not know what happens to rejuvenating agents in terms of micro bonds.

Figures 4.135 to 4.137, respectively, include the AFM images for 3% WEO, 5% WEO and 7% WEO rejuvenated bitumen samples. Compared to aged RAP bitumen AFM images, WEO rejuvenated bitumen samples differ in Z-axis topographic images, phase scan and amplitude images. In terms of topography, a very tiny (approximately 0.5 to 1 μm) bee-structures appeared in WEO rejuvenated bitumen samples. The disperse parts of WEO rejuvenated bitumen also differ in topography compared to aged RAP bitumen. Phase scan images show that the material stiffness changes dramatically especially for 5% and higher contents of WEO rejuvenating agent. The amplitude images also prove the existence of an external material (rejuvenating agent) inside of the rejuvenated bitumen sample. The new-born tiny bee-structures inside of the optimally WEO rejuvenated bitumen (5% rejuvenating agent) are approximately

half in numbers and one-third in size compared to neat bitumen sample. Although the exact AFM images of neat bitumen is not observed in case of rejuvenated bitumen samples, but similarities can be seen.

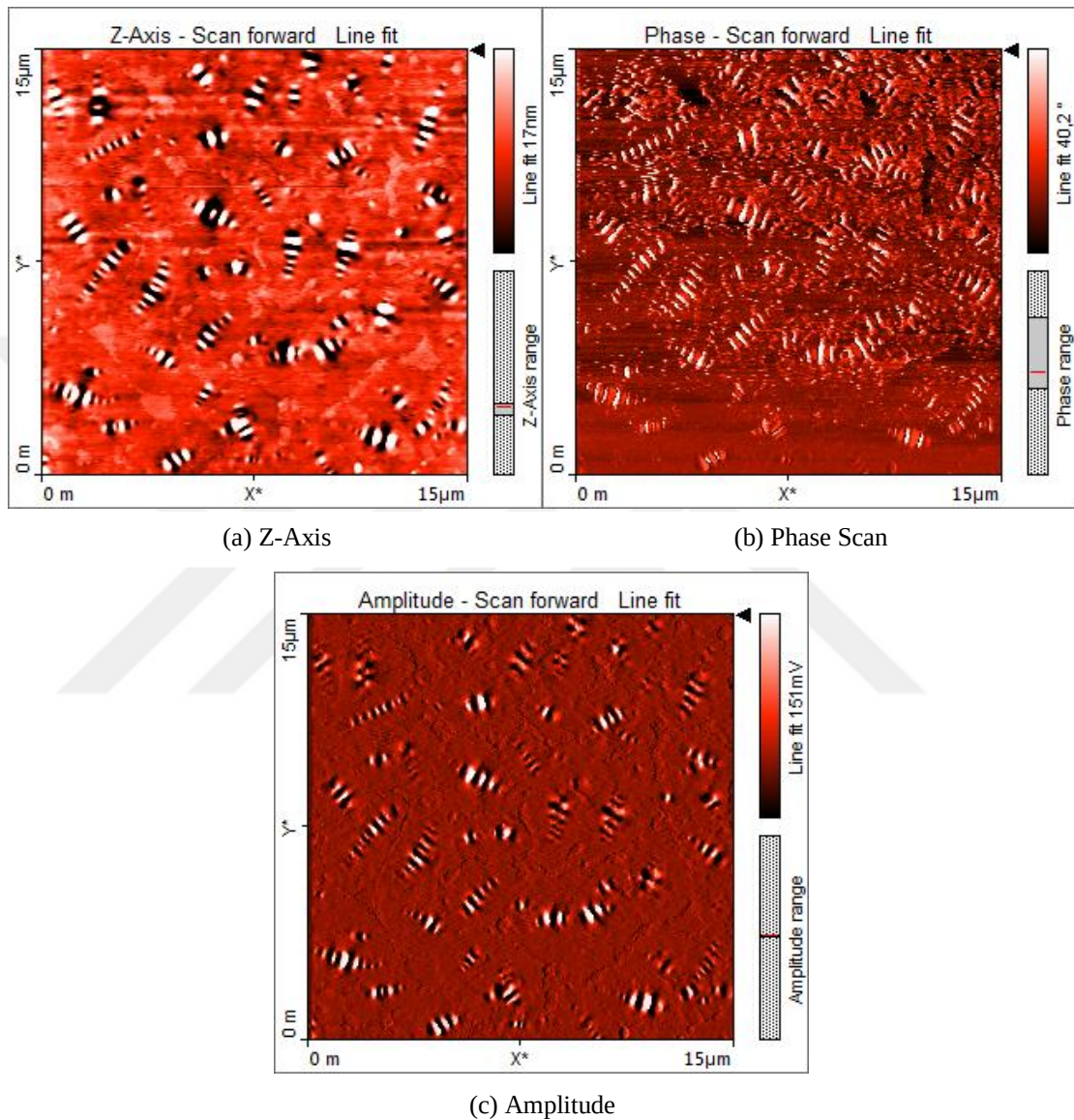
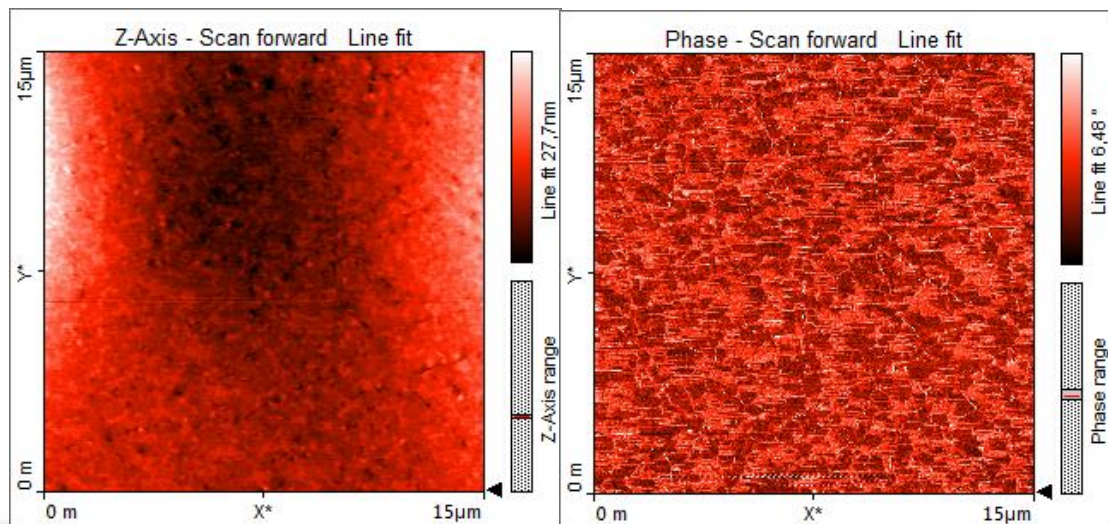


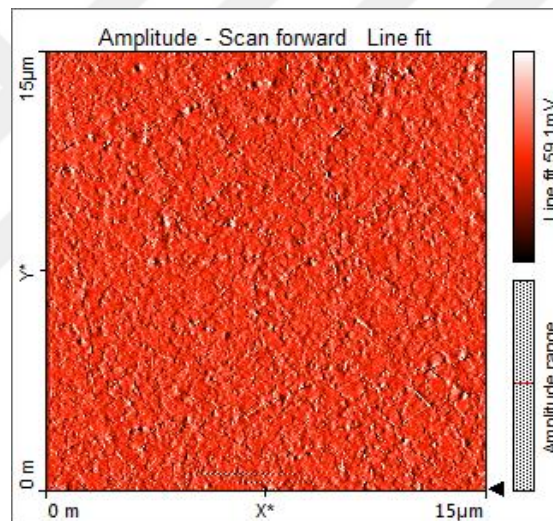
Figure 4.133 Atomic Force Microscopy Images of Neat Bitumen Sample

In the case of WVO rejuvenated samples, the AFM images seem to be very similar to WEO rejuvenated bitumen samples with some differences as shown in Figures 4.138 to 4.140. Compared to aged RAP bitumen AFM images, WVO rejuvenated bitumen samples Z-axis topographic images, phase scan and amplitude images show a huge change.



(a) Z-Axis

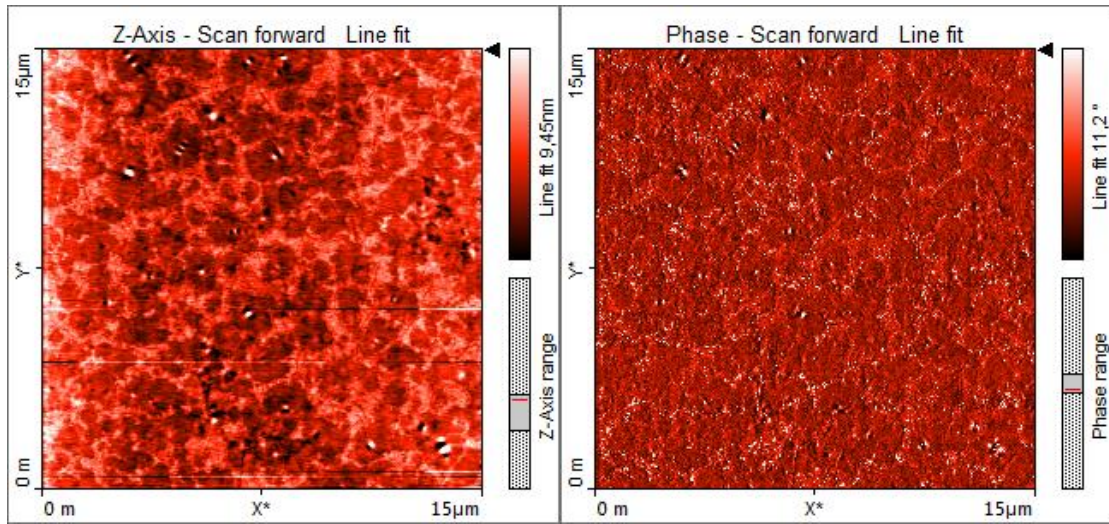
(b) Phase Scan



(c) Amplitude

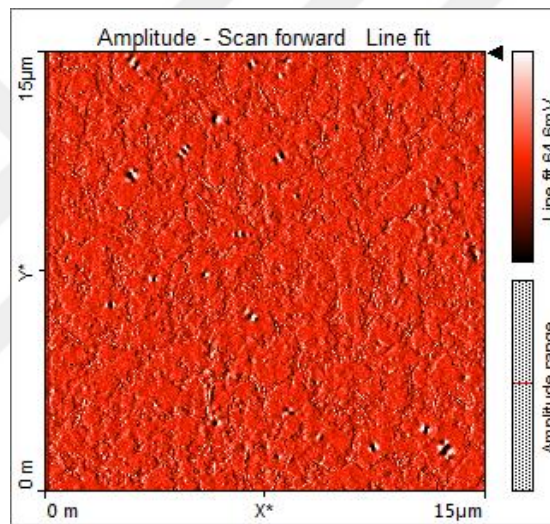
Figure 4.134 Atomic Force Microscopy Images of Aged RAP Bitumen Sample

Z-axis topographic images show tiny (approximately 0.5 to 1 μm) bee-structures in WVO rejuvenated samples very similar to WEO rejuvenated bitumen samples. The disperse parts of WVO rejuvenated bitumen but is slightly different from WEO rejuvenated disperse phase and significantly different in topography compared to aged RAP bitumen. Phase scan images show that the material stiffness significantly changes through adding WVO rejuvenating agent.



(a) Z-Axis

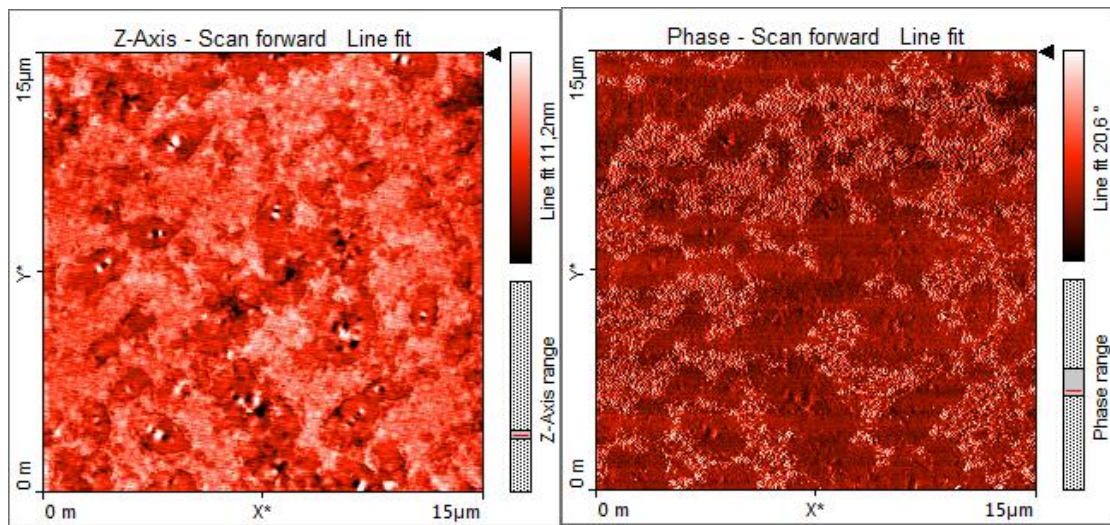
(b) Phase Scan



(c) Amplitude

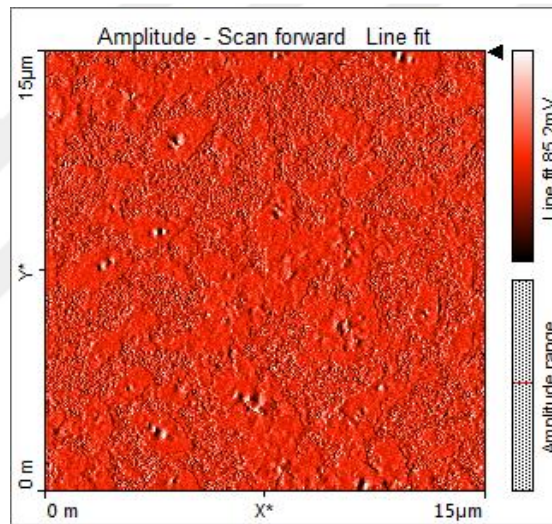
Figure 4.135 Atomic Force Microscopy Images of WEO (3%) Rejuvenated Bitumen Sample

The amplitude images show the presence of WVO rejuvenating agent inside the rejuvenated bitumen sample. The new-born tiny bee-structures inside of the 5% optimally WVO rejuvenated bitumen show similarity to WEO bitumen samples, means half in numbers and one-third in size compared to neat bitumen. But the disperse phase of the Z-axis is different from WEO rejuvenated bitumen samples. WVO bitumen samples AFM images show that the images of neat bitumen are not achieved through rejuvenation with WVO agent still similarities are detected.



(a) Z-Axis

(b) Phase Scan



(c) Amplitude

Figure 4.136 Atomic Force Microscopy Images of WEO (5%) Rejuvenated Bitumen Sample

In the case of WVO rejuvenated samples, the AFM images seem to be very similar to WEO rejuvenated bitumen samples with some differences as shown in Figures 4.138 to 4.140. Compared to aged RAP bitumen AFM images, WVO rejuvenated bitumen samples Z-axis topographic images, phase scan and amplitude images show a huge change. Z-axis topographic images show tiny (approximately 0.5 to 1 μm) bee-structures in WVO rejuvenated samples very similar to WEO rejuvenated bitumen samples.

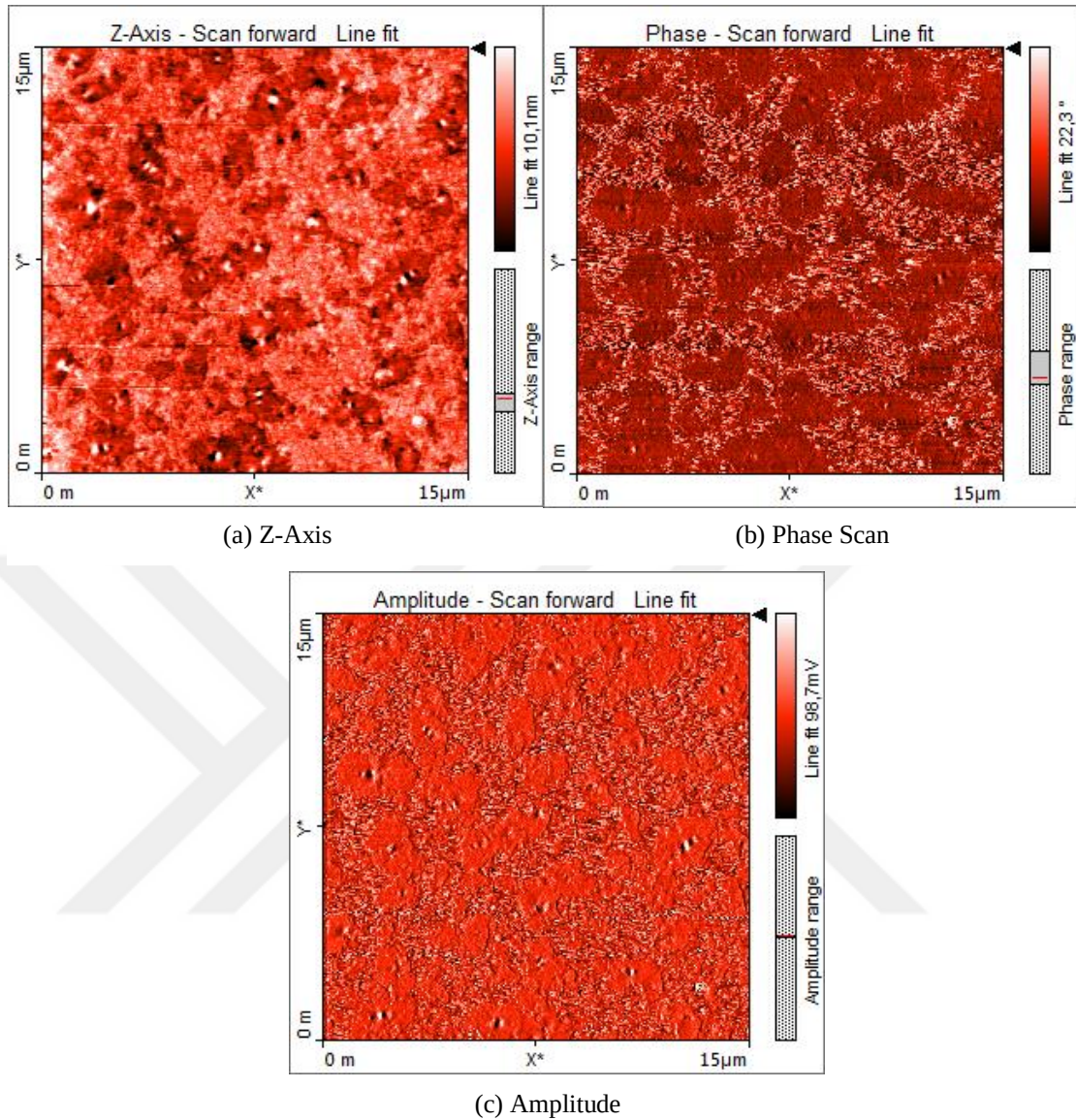


Figure 4.137 Atomic Force Microscopy Images of WEO (7%) Rejuvenated Bitumen Sample

The disperse parts of WVO rejuvenated bitumen but is slightly different from WEO rejuvenated disperse phase and significantly different in topography compared to aged RAP bitumen. Phase scan images show that the material stiffness significantly changes through adding WVO rejuvenating agent. The amplitude images show the presence of WVO rejuvenating agent inside the rejuvenated bitumen sample.

The new-born tiny bee-structures inside of the 5% optimally WVO rejuvenated bitumen show similarity to WEO bitumen samples, means half in numbers and one-third in size compared to neat bitumen. But the disperse phase of the Z-axis is different

from WEO rejuvenated bitumen samples. WVO bitumen samples AFM images show that the images of neat bitumen are not achieved through rejuvenation with WVO agent still similarities are detected.

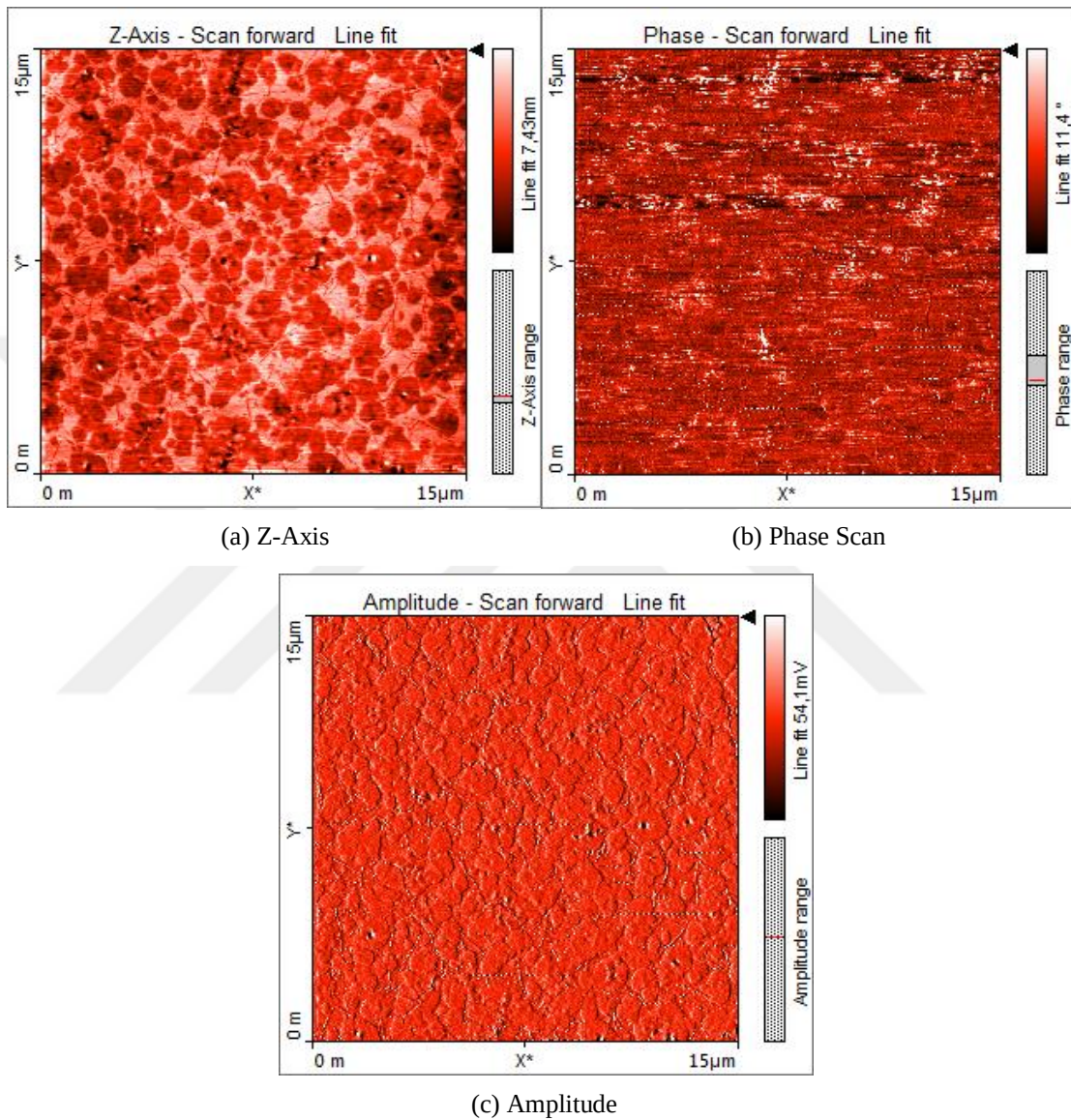


Figure 4.138 Atomic Force Microscopy Images of WVO (3%) Rejuvenated Bitumen Sample

The commercial rejuvenating agent AFM imaging showed slightly different results from waste oils and neat bitumen since it is obvious that significant changes in material micro topography, stiffness and homogeneity occurs through rejuvenation with CRA. Figures 4.141 to 4.143 respectively include Z-axis, phase scan and amplitude images for 5.5% CRA rejuvenated, 7.5% CRA rejuvenated and 9.5% CRA rejuvenated

bitumen samples. Like waste oils, tiny bee-structures alike showed up in CRA rejuvenated bitumen samples since the disperse phase of the topographic scan is significantly different from waste oils rejuvenated samples. It can be understood that the bitumen stiffness changes dramatically through rejuvenation with CRA. The consistency of rejuvenation dispersion inside of the rejuvenated bitumen samples seem to be better than WEO and WVO rejuvenated bitumen samples.

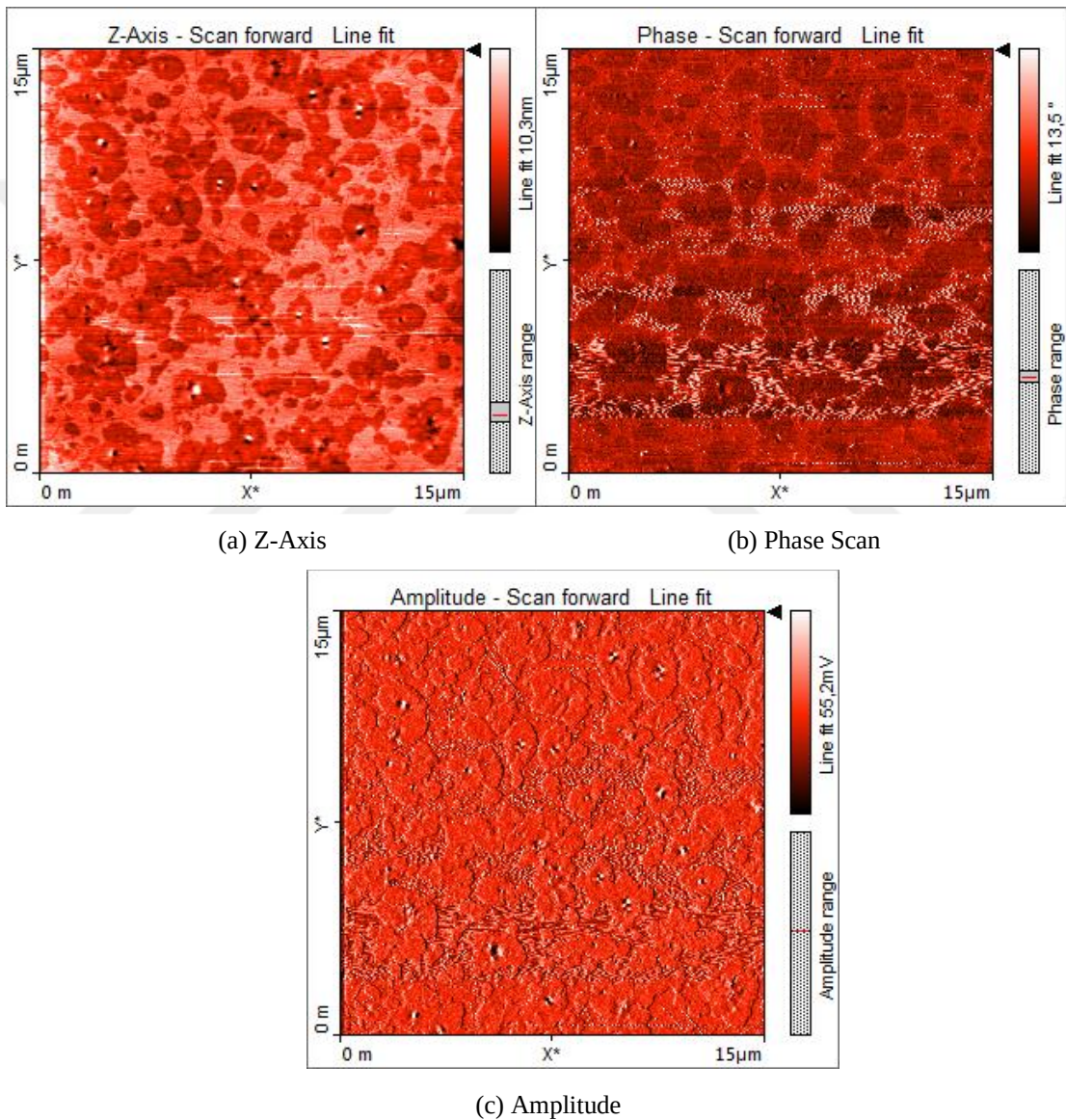
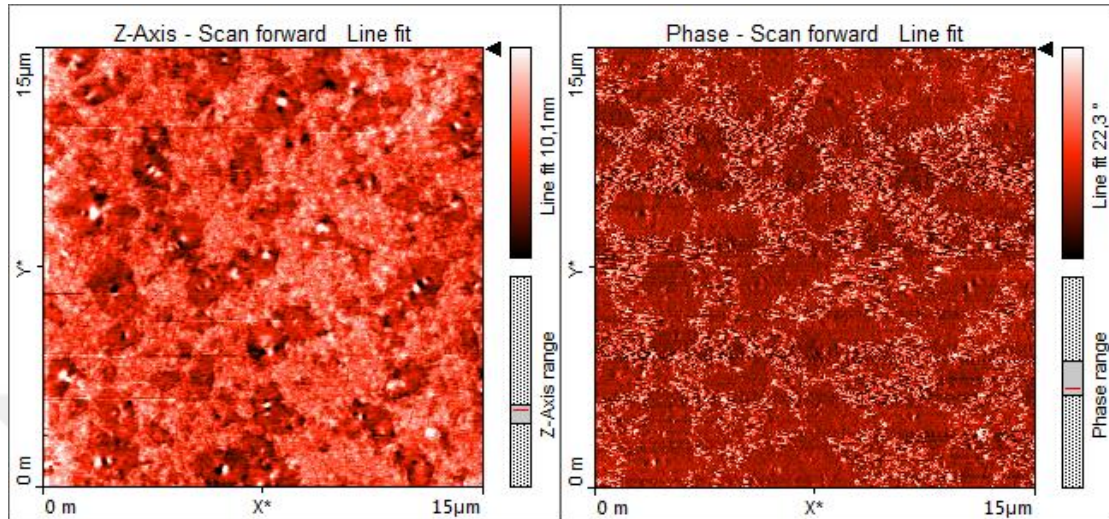


Figure 4.139 Atomic Force Microscopy Images of WVO (5%) Rejuvenated Bitumen Sample

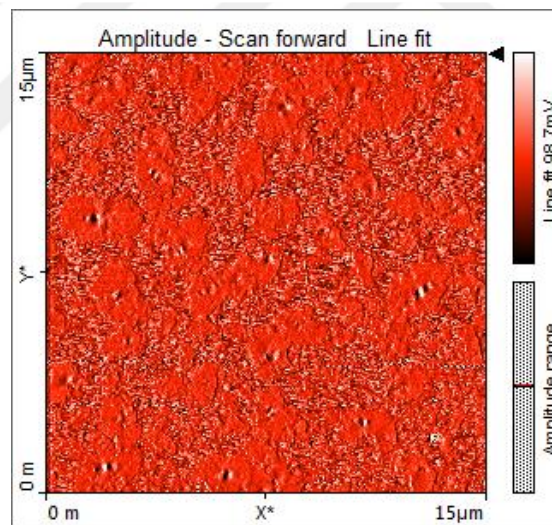
Coming to conclusion, all rejuvenating agents change the aged bitumen micro topography and stiffness significantly. Although the neat bitumen AFM images are

significantly different from rejuvenated bitumen samples but similarities in terms of stiffness and bee-structures can be observed compared to aged RAP bitumen.



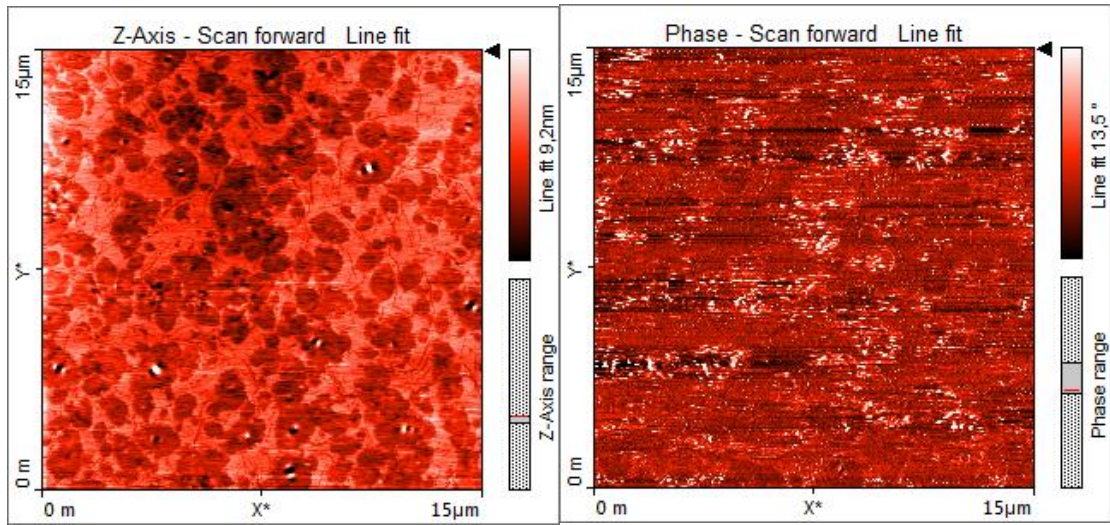
(a) Z-Axis

(b) Phase Scan



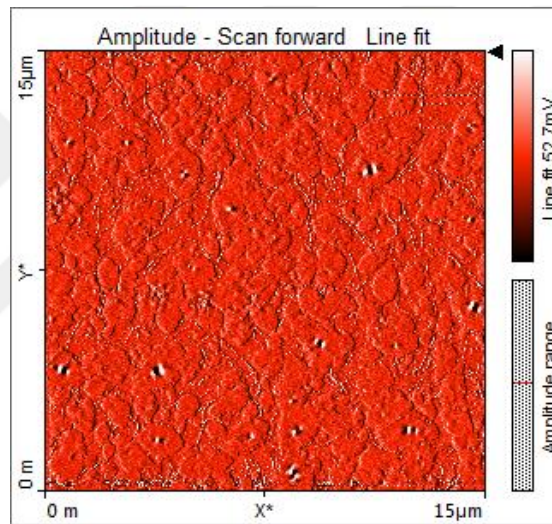
(c) Amplitude

Figure 4.140 Atomic Force Microscopy Images of WVO (7%) Rejuvenated Bitumen Sample



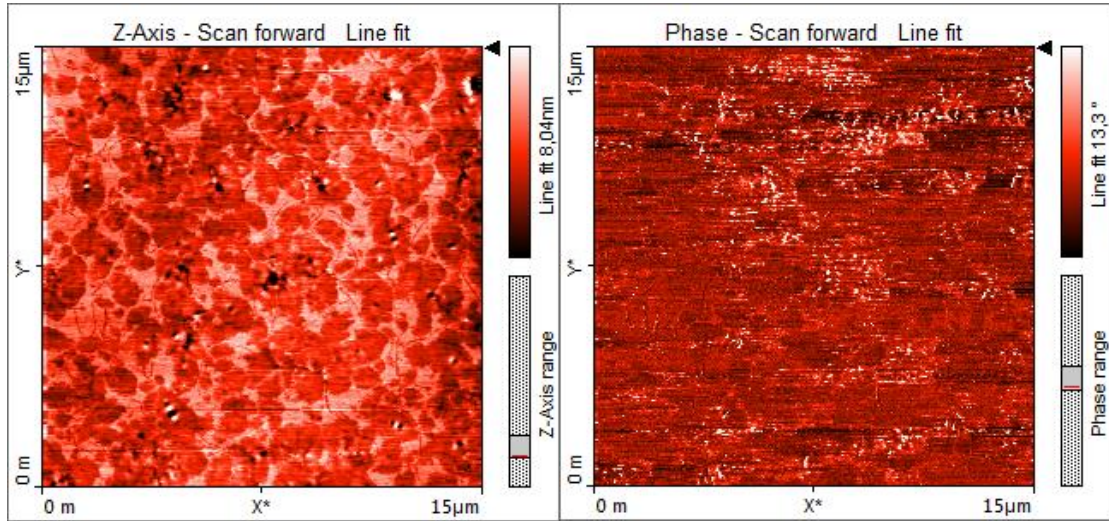
(a) Z-Axis

(b) Phase Scan



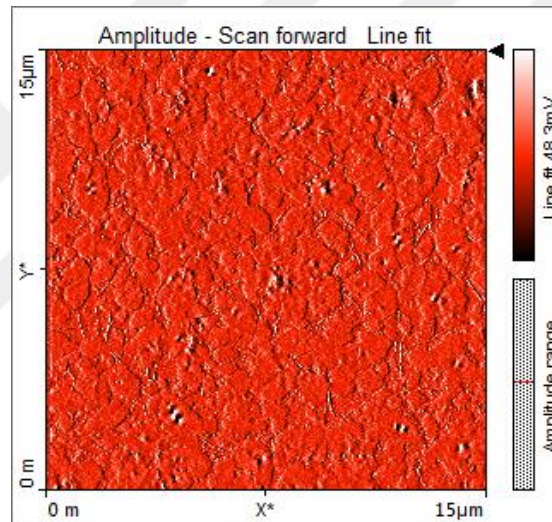
(c) Amplitude

Figure 4.141 Atomic Force Microscopy Images of CRA (5.5%) Rejuvenated Bitumen Sample



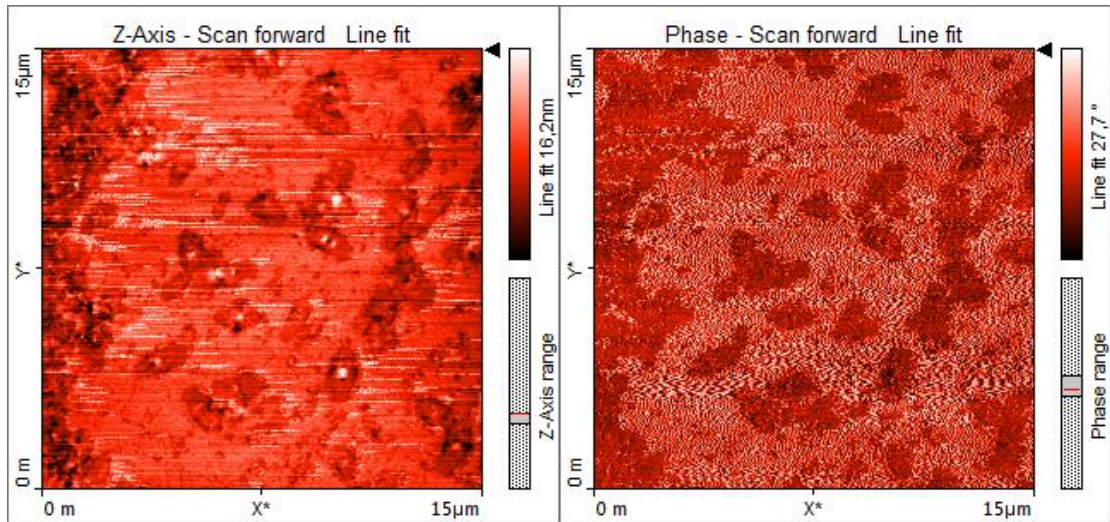
(a) Z-Axis

(b) Phase Scan



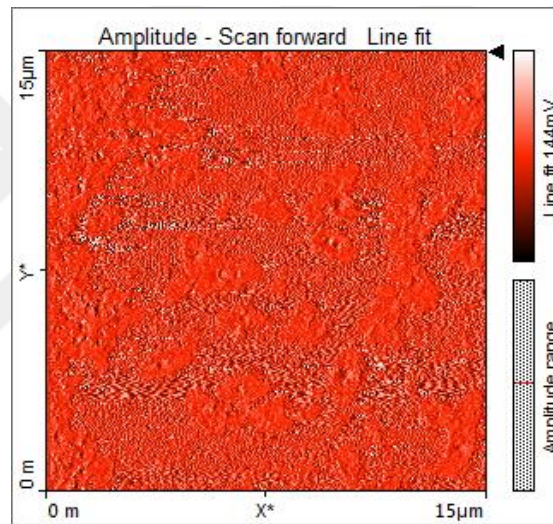
(c) Amplitude

Figure 4.142 Atomic Force Microscopy Images of CRA (7.5%) Rejuvenated Bitumen Sample



(a) Z-Axis

(b) Phase Scan



(c) Amplitude

Figure 4.143 Atomic Force Microscopy Images of CRA (9.5%) Rejuvenated Bitumen Sample

4.1.3.2 Scanning Electron Microscopy (SEM) Results

As aforementioned, Rejuvenated and non-rejuvenated bitumen samples were inspected by SEM microscopy. According to literature presented in Chapter 2, almost all SEM microscopic investigations of bitumen rejuvenation are related to capsuling method in which the rejuvenating agent is reserved inside micro capsules to be released when aged bitumen is cracked. Capsules used inside the bitumen sample can be observed by means of SEM microscopy since no interpretable results could be

obtained in case of rejuvenated bitumen samples. Figure 4.144 shows an example of bitumen sample image shot by SEM method.

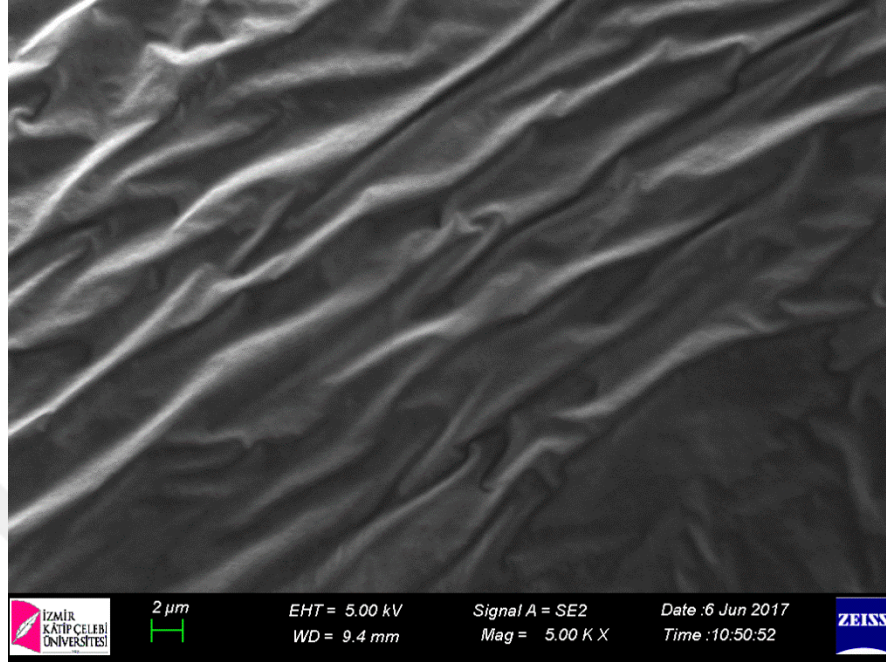


Figure 4.144 A bitumen sample SEM image example

4.1.4 Chemical Test Results

4.1.4.1 Fourier Transform Infrared Spectroscopy (FT-IR) Results

The rejuvenated and non-rejuvenated bitumen samples including aged RAP and neat bitumen as well as the rejuvenating agents used within the scope of this study were all studied in terms of spectroscopy analysis by means of FTIR. Figures 4.145 to 4.149 respectively include the FTIR spectral diagrams. All spectrums have been normalized and offset to positive absorbance levels to make reasonable comparisons between the spectrums. Figure 4.145 shows the spectral diagrams for all bitumen samples including neat, aged RAP bitumen, 5% WEO rejuvenated, &5 WVO rejuvenated and 7.5% CRA rejuvenated bitumen samples. As shown in Figure 4.145, for wavenumbers ranging from 400 to 4000 cm^{-1} , all bitumen samples spectral rise and falls track a similar trendline with differences in absorbance levels. For instance, carbonyl and sulfoxide peaks at the wavenumbers of 1700 and 1300 cm^{-1} as expected

according to literature. Since at these ranges of wavenumbers, for aged RAP bitumen the increase in content of polar components which constitute the higher molecular sizes corresponding asphaltenes results in the increase in absorbance level.

As can be seen in Figure 4.145, aged RAP bitumen recorded high absorbance than neat bitumen for almost all wavenumbers ranging from 400 to 4000 cm^{-1} . In the case of aged RAP bitumen associated with aging, which is rather oxidized compared to neat bitumen, the areas linked to the carbonyl functional group which characterize the bitumen oxidation, the increase in carbonyl concentration due to aging can be observed. The sulfoxide group is normally found more than carbonyl, in bitumen samples with high contents of sulfur. Additionally, the groups associated with the oxidation (S-O and C-O) rated high absorbance peaks in aged RAP bitumen compared to neat bitumen. As can be seen from all spectrums, the absorbance levels have been decreased through rejuvenation process. The drops in absorbance level in CRA rejuvenated bitumen is more effective than WEO and WVO rejuvenated bitumen samples. Each rejuvenated bitumen sample also recorded high peaks in specific wavenumbers associated with the rejuvenating agent used within it. Figures 4.146 to 4.149 respectively show the plotted spectrums for WEO, WVO and CRA rejuvenated bitumen samples including each rejuvenating agent spectrum in the diagrams. For instance, as can be seen in Figure 4.147, the FTIR spectrum of the WVO rejuvenated bitumen samples present the absorption peaks at wavenumbers from 1150 to 1700 to cm^{-1} , which are attributed to the ester carbonyl functional group found in WVO stretching. These peaks can also be found in the FTIR spectra of WVO rejuvenating agent. The same peaks can be observed in Figure 4.148 for CRA rejuvenating agent which has resulted in peaks in absorbance level of CRA rejuvenated bitumen sample in the same wavenumbers.

Figure 4.149 show the spectrums for all rejuvenating agents in one diagram. As shown in the Figure 4.149, CRA and WVO rejuvenators showed more similarities to each other than WEO which is linked to vegetable source of these two organic rejuvenating agents.

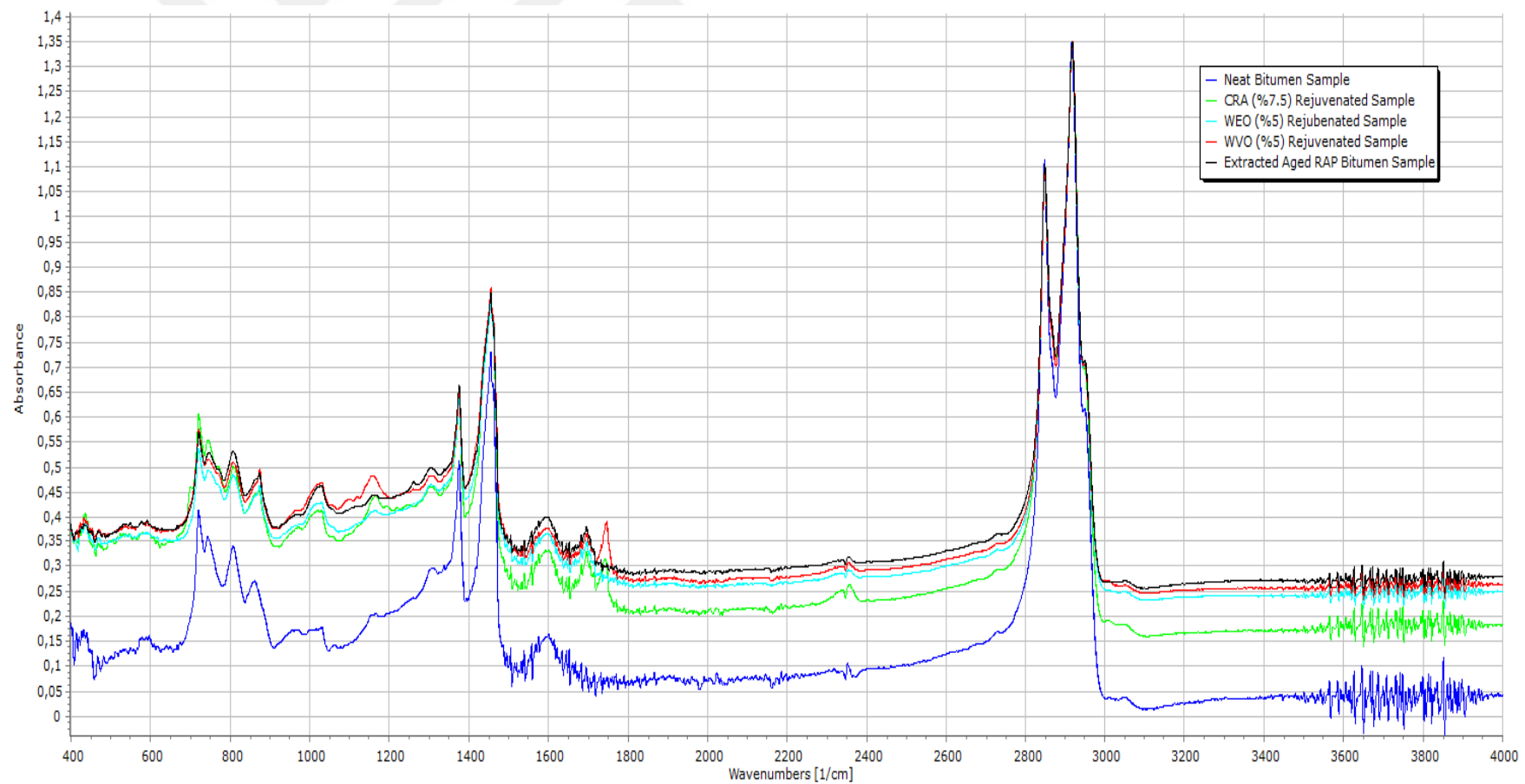


Figure 4.145 Neat, aged RAP and all optimally rejuvenated bitumen samples FTIR spectral diagrams

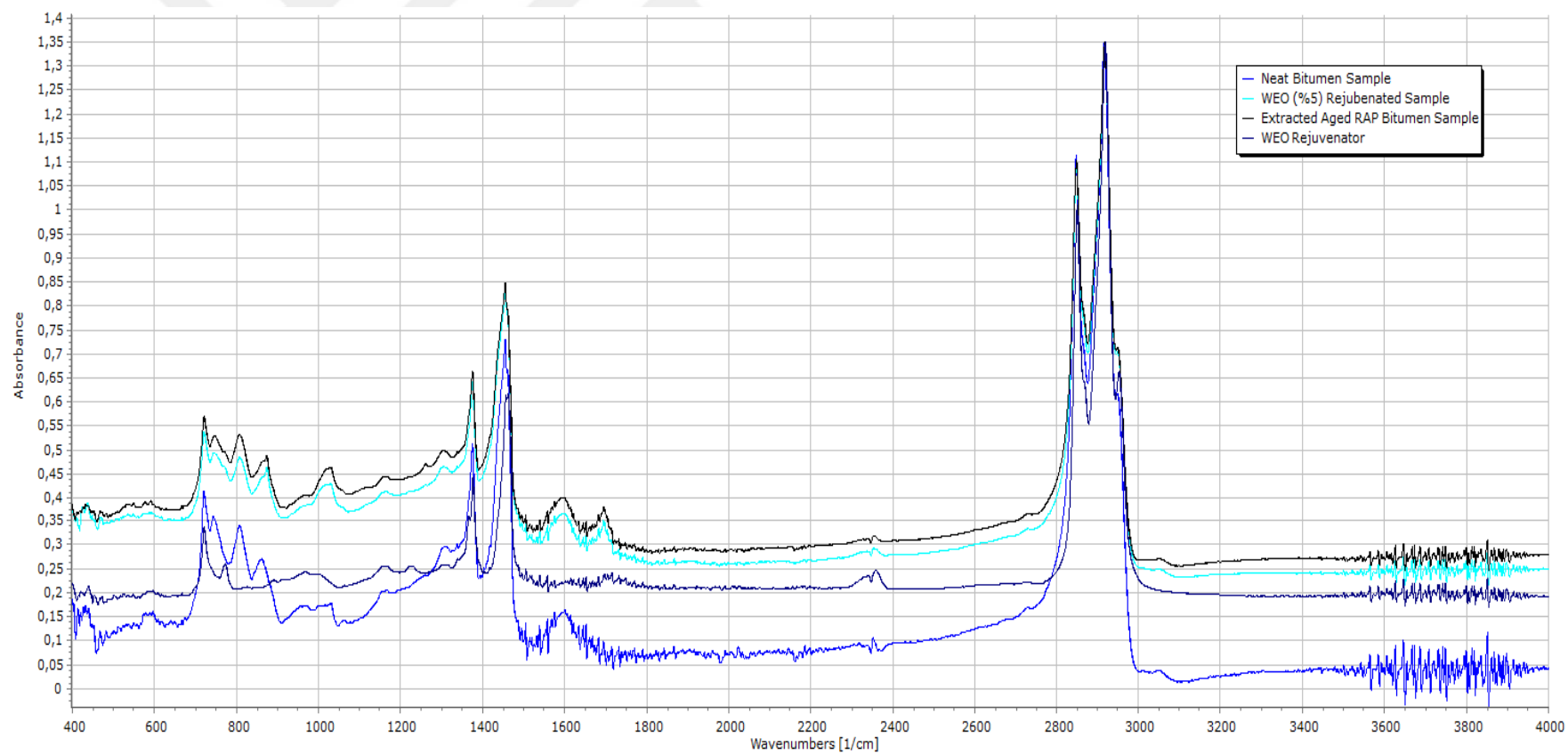


Figure 4.146 Neat, aged RAP and optimum WEO rejuvenated bitumen samples FTIR spectral diagrams

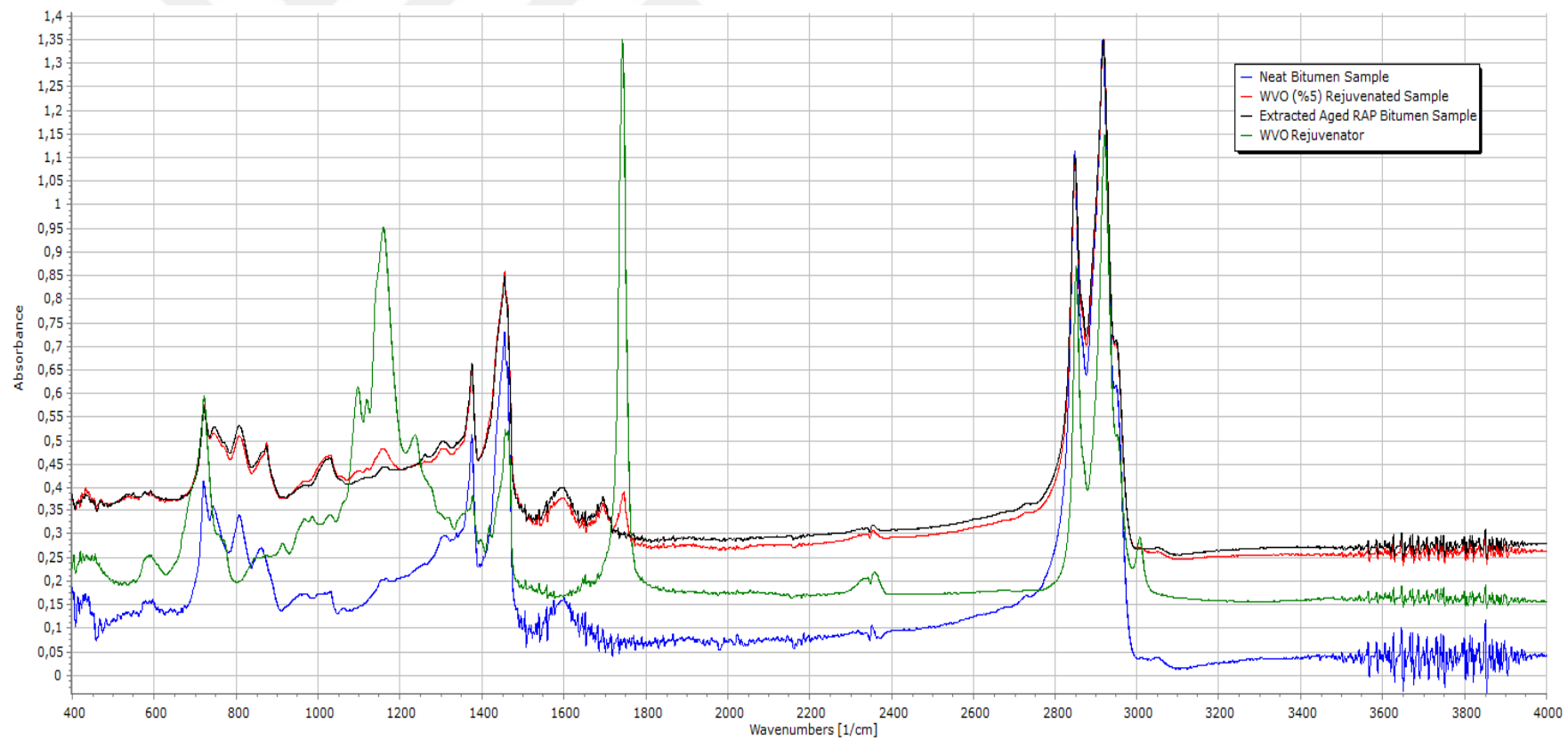


Figure 4.147 Neat, aged RAP and optimum WVO rejuvenated bitumen samples FTIR spectral diagrams

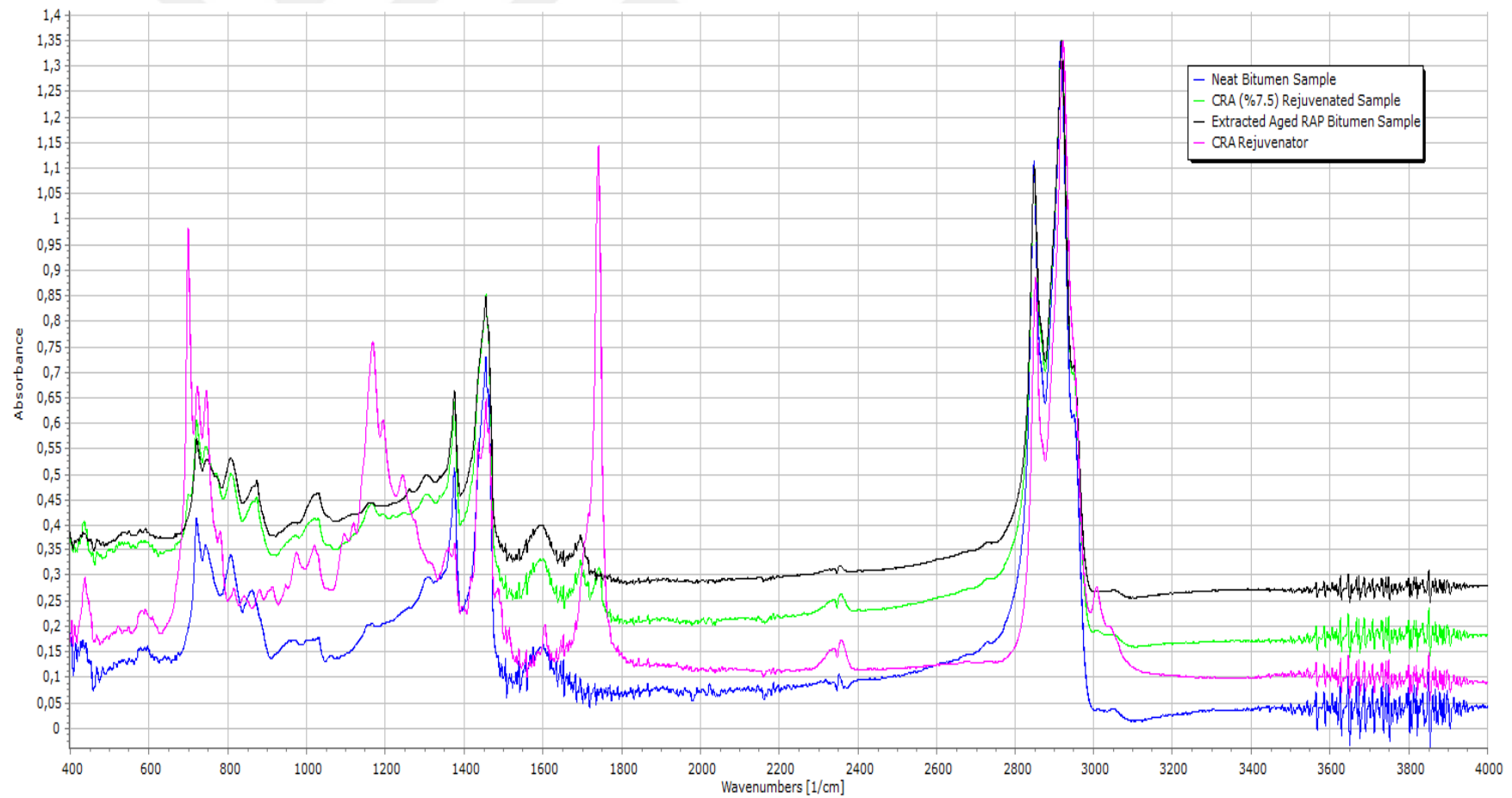


Figure 4.148 Neat, aged RAP and optimum CRA rejuvenated bitumen samples FTIR spectral diagrams

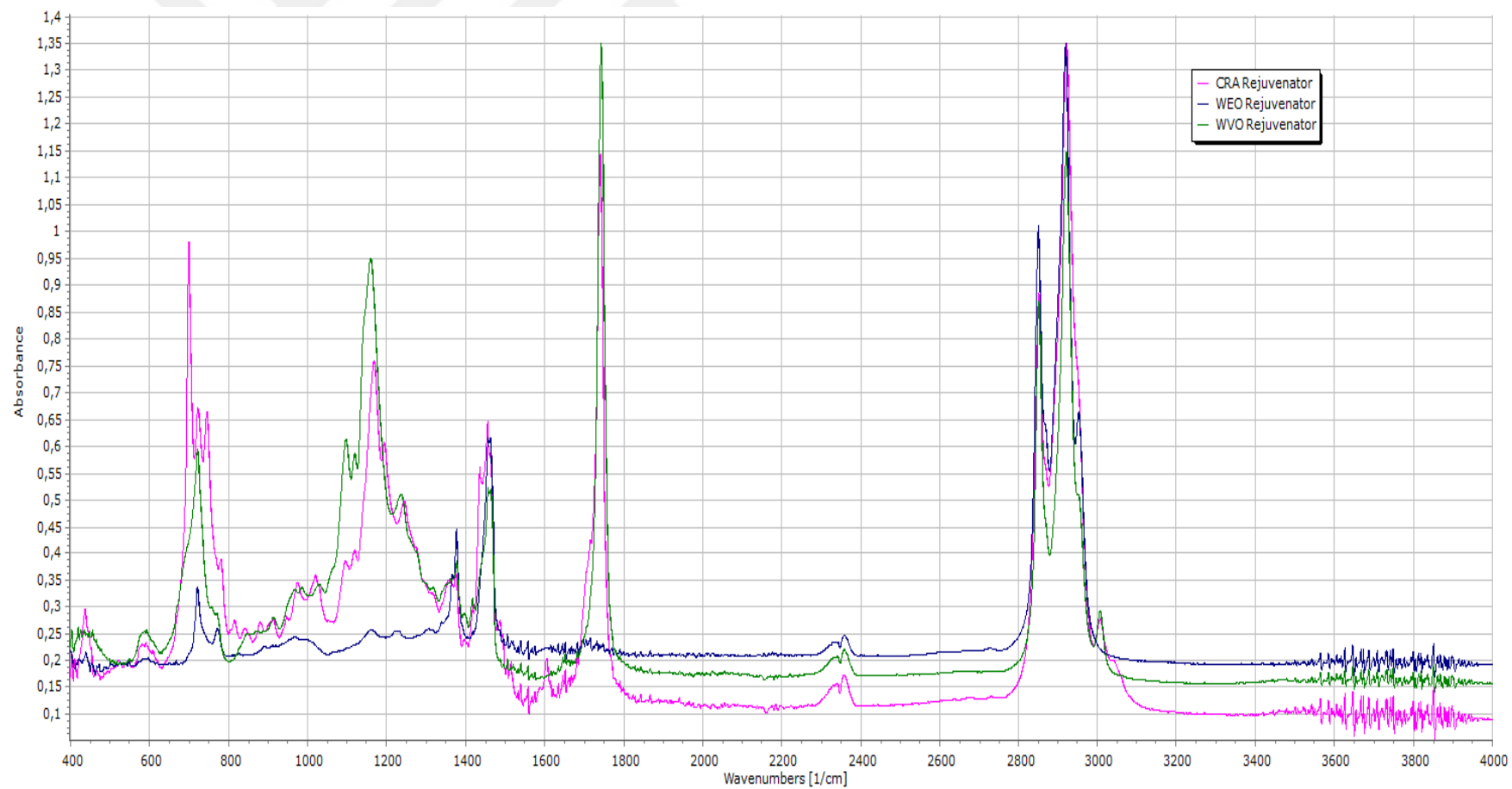


Figure 4.149 FTIR spectral diagrams of WEO, WVO and CRA rejuvenators

4.2 Mixture Test Results

Within the scope of this study, the effect of three different kinds of rejuvenators (WEO, WVO and CRA) was evaluated on aged bitumen. As we know, rejuvenators finally aim to increase the applicable amount of RAP in bituminous mixtures. Within the scope of this section, rejuvenated asphalt specimens were produced using a laboratory Marshall compactor according to Marshall mix design method. Marshall stability and flow test were employed to determine the mechanical properties. The volumetric analysis was the determinant criteria in selection of highest amount of RAP within the mixtures.

All the control and rejuvenated samples were tested and checked as per Turkish Specification limits. Subsequently, the specimens containing highest RAP contents were prepared and tested for the Indirect Tensile Strength (ITS). Finally, the ITS values were taken into consideration in calculation of aging indices of all rejuvenated specimens with highest RAP contents. The results were employed to make comparisons based on specimen's stiffness.

Two main steps were followed in design of asphalt specimens with different amount of RAP; at the first step, the material properties were determined and at the second step, the content of RAP and virgin materials (aggregates and binder) were defined. Appropriate ratios of RAP to virgin materials in target mixtures were defined considering the overall properties of RAP and virgin materials.

4.2.1 Marshall Test Results

Volumetric analysis together with Marshall stability and flow values were the determinant criteria in selection of maximum possible RAP content for all mixtures involving non-rejuvenated and rejuvenated RAP. Results for air void contents, stabilities and flow rates are respectively presented in Figure 4.150 to Figure 4.152 for all rejuvenated and non-rejuvenated control specimens.

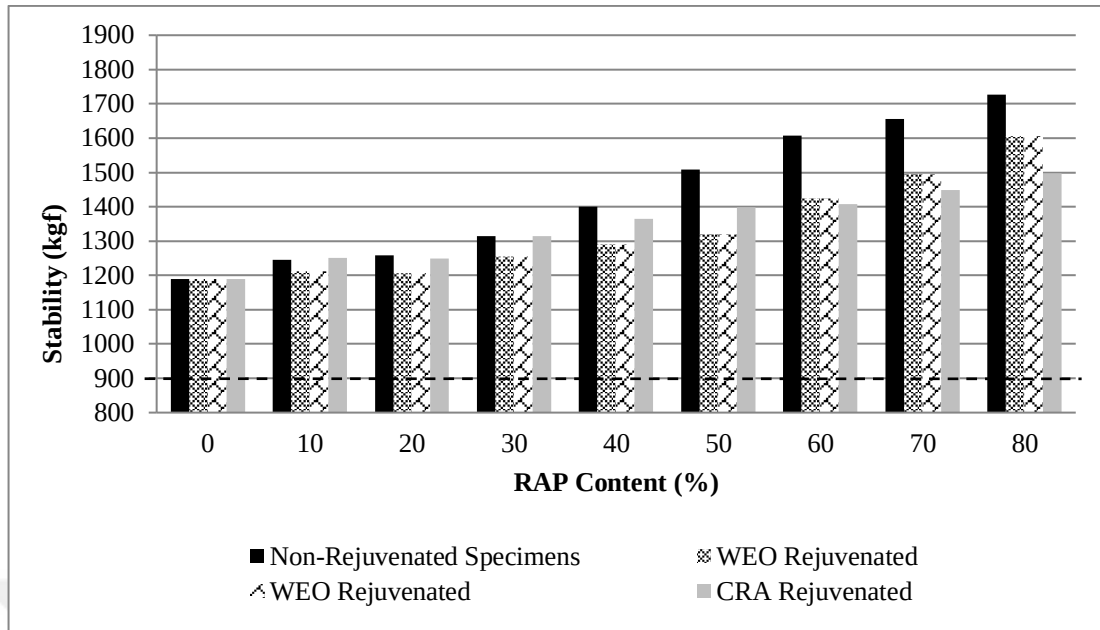


Figure 4.150 Marshall stability values corresponding RAP contents

When Figure 4.150 is analyzed, it is seen that all stability values are over specification limit (900 kgf) for all rejuvenated and non-rejuvenated specimens. This result is expected since the bitumen within RAP material is aged binder and thus the mixtures containing RAP are stiffer than virgin bituminous mixtures hence the stabilities were recorded higher for specimens including RAP material. In fact, the most concerned issue for RAP involving mixtures is the matter of durability rather than stability. Therefore, volumetric characteristics and flow rates (somehow, as an indicator of flexibility) are more determinative for maximum possible RAP content than stability values.

As can be seen in the Figure 4.151, all mixtures containing rejuvenated RAP could meet volumetric criteria in terms of air voids since mixtures containing non-rejuvenated RAP fail to satisfy desired air voids content for mixtures containing more than 40% of non-rejuvenated RAP. The reason that volumetric characteristic of rejuvenated RAP containing mixtures remained within the desired contents is attributed to lower viscosity values of rejuvenated RAP bitumen and hence improved workability.

At standard mixing and compaction temperatures, it is more convenient to process rejuvenated RAP mixtures than non-rejuvenated RAP mixtures. CRA rejuvenated specimens performed better than waste oils with WVO slightly better than WEO in terms of softening the stiff RAP materials. This is in parallel with the results of conventional and rheological investigations. This can be attributed to higher resistance of CRA to short term aging (mixing & compaction phase) at high temperatures.

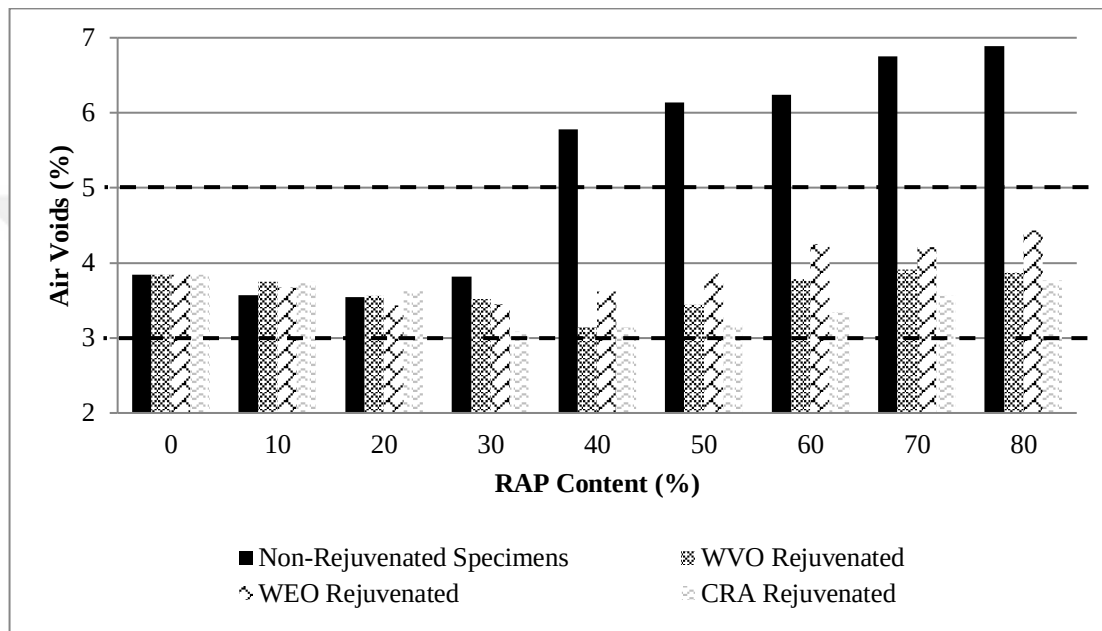


Figure 4.151 Air voids contents corresponding RAP contents

When evaluating flow rates as shown in Figure 4.152, it is understood that this criterion is determinative for both non-rejuvenated and rejuvenated RAP involving mixtures. It is seen that, mixtures containing over 20% of non-rejuvenated RAP almost fail to meet the flow rate criteria since rejuvenated mixtures exhibited better flow rates.

Flow rates however, have been decisive in determination of maximum possible amount of rejuvenated RAP within type I wearing course as per Turkish specifications. In this sense, 70% of rejuvenated (WEO modified) RAP can be employed within type-I wearing course since on the other hand, 80% of WVO rejuvenated RAP can be implemented and even more than 80% of CRA rejuvenated RAP can be used.

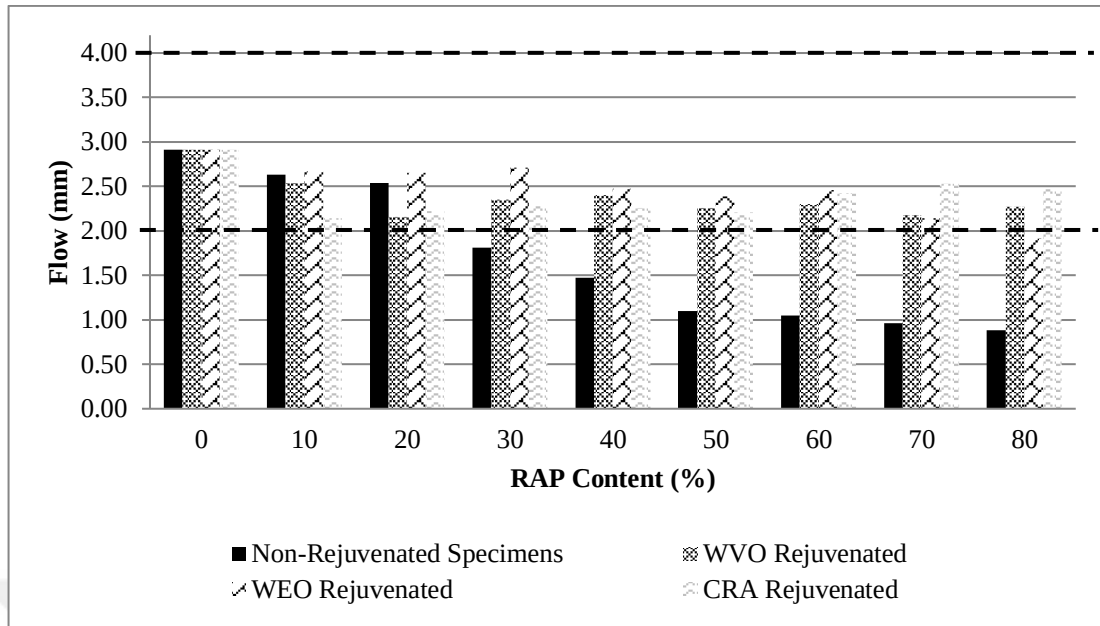


Figure 4.152 Marshall flow rates corresponding RAP contents

It is obvious that the amount of maximum RAP which can be implemented without failing to meet all Turkish criteria increases substantially by rejuvenation process. Evidently, 50%, 60% and 70% more RAP can be employed by using WEO, WVO and CRA respectively.

4.2.2 Indirect Tensile Strength and Stiffness Indices

ITS results provide an insight about the mixtures stiffness. As it is seen in Figure 4.153, the mixtures involving rejuvenated RAP recorded lower ITS than non-rejuvenated mixtures containing RAP materials. The use of rejuvenators can soften the bituminous mixtures and give the mixtures a better workability and eventually better flexibility.

Aging indices based on ITS values were calculated as 1.39 for non-rejuvenated mixture, 1.16 for WEO rejuvenated mixture, 1.15 for WVO rejuvenated mixture and 1.1 for CRA rejuvenated mixture. The closer the aging index gets to 1, the less the mixture is stiffer. It can be concluded that the use of CRA, WEO and WVO can soften the mixtures containing RAP. Taking the aging indices into consideration, CRA

softens the mixture to high extent better than waste oils with WVO performing slightly better than WEO in terms of softening.

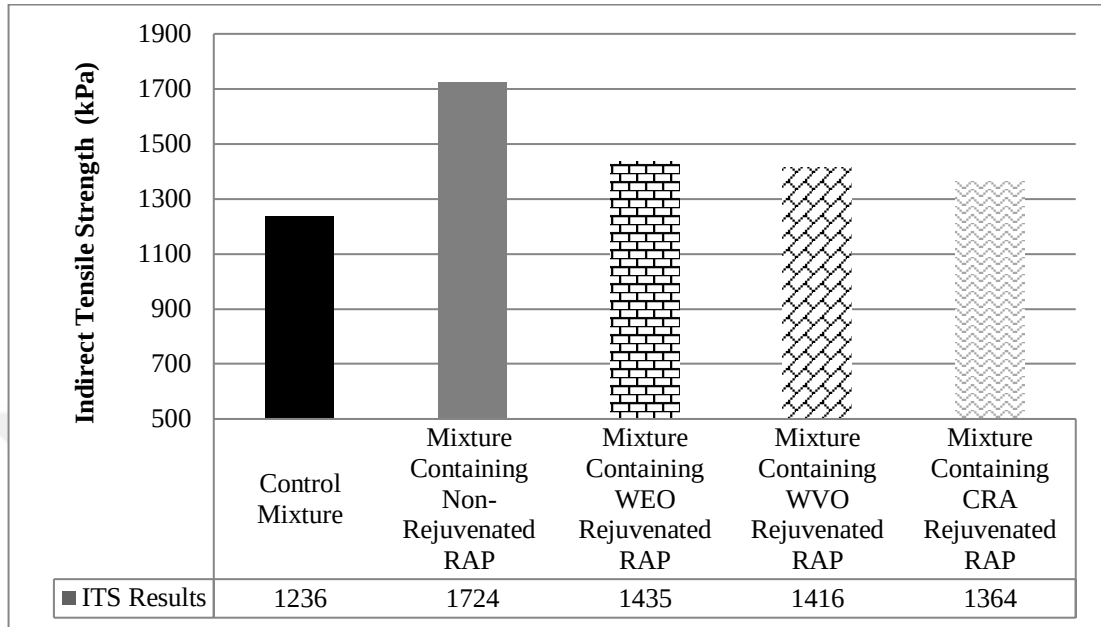


Figure 4.153 Indirect tensile test results

CHAPTER FIVE

COST ANALYSIS

In this chapter, the cost analysis and simple feasibility studies for implementing of studied rejuvenating agents have been presented. The aim of this study is to determine the financial benefits to be derived from RAP according to the criteria of the Turkish Highways Technical Specification. According to the specifications, in Turkey, only 25% RAP is allowed to be used with bituminous base courses. Cost analyzes for producing 1 ton of bituminous base course in an asphalt plant with 1500 tons daily manufacturing capacity has been compared to manufacturing cost of 1 ton of base course including 25% of rejuvenated RAP. In this regard, the manufacturing costs for three types of rejuvenated RAP materials have been calculated. In the calculations, the cost of transportation of manufactured Asphalt Concrete (AC) to the application area has not been included in the calculation. For now, the cost of RAP material only includes the transportation costs since the RAP itself belongs to the state roads authorities or the municipalities and no price is available for the RAP. Many other indirect benefits such as disposal benefits of RAP have not been taken into account.

5.1 Manufacturing Costs

5.1.1 Manufacturing Costs for 1 ton of Neat Base Course AC

In this section, the cost analysis for manufacturing 1 ton of neat bituminous base course in an asphalt plant with 1500 tons of manufacturing capacity is presented. In this regard, the following items must be considered, and some assumptions must be made followingly. All prices are in US\$ and the calculations are for the second quarter of 2018.

Main Items:

- Daily Operational Cost for An Asphalt Plant with 1500 tons capacity
 - Personnel Wage
 - Personnel Food etc.
 - Energy Costs

- Maintenance Costs
- Material Costs for Manufacturing 1 ton of Base Course AC
 - Aggregates (Including Transportation Costs)
 - Bitumen (Including Transportation Costs)
 - 5% Optimum Bitumen Content is assumed.

Assumptions Including Approximate Market Based Prices:

- Distances:
 - Aggregate Quarry – Plant 5 km.
 - Refinery – Plant 100 km.
- Prices:
 - 1 ton of Aggregate in Quarry: 3\$
 - 1 ton of Neat Bitumen in Refinery: 400\$
 - Plant Daily Personnel Wages: 350\$
 - Plant Daily Personnel Food: 100\$
 - Plant Daily Energy Cost: 500\$
 - Plant Daily Maintenance Cost: 50\$
 - 1 km Transportation Cost for 1 ton of Solid Materials: 1.5¢
 - 1 km Transportation Cost for 1 ton of Liquid Materials: 3¢

For 1500 tons daily manufacturing of neat base course AC:

Aggregates Cost (Including Transportation) + Bitumen Cost (Including Transportation) + Daily Personnel Cost + Daily Energy Cost + Daily Maintenance Cost

Aggregates Cost (Including Transportation) = 4,275.00\$ + 106.88\$ = 4,381.88\$

Bitumen Cost (Including Transportation) = 30,000.00\$ + 225.00\$ = 30,225.00\$

Daily Personnel Cost = 350.00\$ + 100.00\$ = 450.00\$

Daily Energy Cost = 500.00\$

Daily Maintenance Cost: 50.00\$

Therefore for 1 ton of neat base course AC,

$$\text{AC (neat)} = (4,381.88\$ + 30,225.00\$ + 450.00\$ + 500.00\$ + 50.00\$) / 1500 = 23.74\$/\text{ton}$$

5.1.2 Manufacturing Costs for 1 ton of Base Course AC including 25% Rejuvenated RAP

In this section, the cost analysis for manufacturing 1 ton of neat bituminous base course including 25% of rejuvenated RAP manufactured in a plant with 1500 tons of manufacturing capacity is presented. In this regard, the following items must be considered, and some assumptions must be made followingly. All prices are in US\$ and the calculations are for the second quarter of 2018.

Main Items:

- Daily Operational Cost for An Asphalt Plant with 1500 tons capacity
 - Personnel Wage
 - Personnel Food etc.
 - Energy Costs
 - Maintenance Costs
- Material Costs for Manufacturing 1 ton of Base Course AC including 25% RAP
 - Aggregates (Including Transportation Costs)
 - Bitumen (Including Transportation Costs)
 - WEO Rejuvenating Agent
 - WVO Rejuvenating Agent
 - CRA Rejuvenating Agent

Assumptions Including Approximate Market Based Prices:

- Distances:
 - RAP Stock – Plant 20 km.
 - Aggregate Quarry – Plant 5 km.
 - Refinery – Plant 100 km.
 - Rejuvenators Stock – Plant 100 km.

➤ Prices:

- 1 ton of RAP in Stock: free
- 1 ton of Aggregate in Quarry: 3\$
- 1 ton of Neat Bitumen in Refinery: 400\$
- 1 kg. of WEO Rejuvenating Agent: 95¢
- 1 kg. of WVO Rejuvenating Agent: 1.20\$
- 1 kg. of CRA Rejuvenating Agent: 2.85\$
- Plant Daily Personnel Wages: 350\$
- Plant Daily Personnel Food: 100\$
- Plant Daily Energy Costs: 500\$
- Plant Extra Daily Rejuvenation Energy Costs: 100\$
- Plant Daily Maintenance Cost: 50\$
- 1 km Transportation Cost for 1 ton of Solid Materials: 1.5¢
- 1 km Transportation Cost for 1 ton of Liquid Materials: 3¢

For 1500 tons daily manufacturing of neat base course AC including 25% of CRA Rejuvenated RAP:

Aggregates Cost (Including Transportation) + Bitumen Cost (Including Transportation) + RAP Cost (Including Transportation) + CRA Rejuvenating Agent Cost + Daily Personnel Cost + Daily Energy Cost (Including Rejuvenation) + Daily Maintenance Cost

Aggregates Cost (Including Transportation) = 3,206.25\$ + 80.16\$ = 3,286.41\$

Bitumen Cost (Including Transportation) = 22,500.00\$ + 168.75.00\$ = 22,668.75\$

RAP Cost (Including Transportation) = 0.00\$ + 112.50\$ = 112.50\$

Cost of 7.5% CRA Rejuvenating Agent by Weight of Bitumen = 4,007.82\$ + 4.22\$ = 4,012.04\$

Daily Personnel Cost = 350.00\$ + 100.00\$ = 450.00\$

Daily Energy Cost = 500.00\$ + 100.00\$ = 600.00\$

Daily Maintenance Cost: 50.00\$

Therefore for 1 ton of base course AC including 25% of CRA Rejuvenated RAP,

$$\text{AC} + \text{RAP (CRA)} = (3,286.41\$ + 22,668.75\$ + 112.50\$ + 4,012.04\$ + 450\$ + 600\$ + 50\$) / 1500 = 20.79\$/\text{ton}$$

For base course AC including WEO and WVO, the calculations are as follows:

For 1500 tons daily manufacturing of neat base course AC including 25% of WEO Rejuvenated RAP:

Aggregates Cost (Including Transportation) + Bitumen Cost (Including Transportation) + RAP Cost (Including Transportation) + WEO Rejuvenating Agent Cost + Daily Personnel Cost + Daily Energy Cost (Including Rejuvenation) + Daily Maintenance Cost

$$\text{Aggregates Cost (Including Transportation)} = 3,206.25\$ + 80.16\$ = 3,286.41\$$$

$$\text{Bitumen Cost (Including Transportation)} = 22,500.00\$ + 168.75.00\$ = 22,668.75\$$$

$$\text{RAP Cost (Including Transportation)} = 0.00\$ + 112.50\$ = 112.50\$$$

$$\text{Cost of 5\% WEO Rejuvenating Agent by Weight of Bitumen} = 890.63\$ + 2.82\$ = 893.45\$$$

$$\text{Daily Personnel Cost} = 350.00\$ + 100.00\$ = 450.00\$$$

$$\text{Daily Energy Cost} = 500.00\$ + 100.00\$ = 600.00\$$$

$$\text{Daily Maintenance Cost: } 50.00\$$$

Therefore for 1 ton of base course AC including 25% of CRA Rejuvenated RAP,

$$\text{AC} + \text{RAP (WEO)} = (3,286.41\$ + 22,668.75\$ + 112.50\$ + 893.45\$ + 450\$ + 600\$ + 50\$) / 1500 = 18.71\$/\text{ton}$$

For 1500 tons daily manufacturing of neat base course AC including 25% of WVO Rejuvenated RAP:

Aggregates Cost (Including Transportation) + Bitumen Cost (Including Transportation) + RAP Cost (Including Transportation) + WVO Rejuvenating Agent Cost + Daily Personnel Cost + Daily Energy Cost (Including Rejuvenation) + Daily Maintenance Cost

Aggregates Cost (Including Transportation) = 3,206.25\$ + 80.16\$ = 3,286.41\$

Bitumen Cost (Including Transportation) = 22,500.00\$ + 168.75.00\$ = 22,668.75\$

RAP Cost (Including Transportation) = 0.00\$ + 112.50\$ = 112.50\$

Cost of 5% WVO Rejuvenating Agent by Weight of Bitumen = 1,125.00\$ + 2.82\$ = 1,127.82\$

Daily Personnel Cost = 350.00\$ + 100.00\$ = 450.00\$

Daily Energy Cost = 500.00\$ + 100.00\$ = 600.00\$

Daily Maintenance Cost: 50.00\$

Therefore for 1 ton of base course AC including 25% of CRA Rejuvenated RAP,

AC + RAP (WVO) = (3,286.41\$ + 22,668.75\$ + 112.50\$ + 893.45\$ + 450\$ + 600\$ + 50\$) / 1500 = 18.86\$/ton

5.2 Rejuvenation Feasibility

In this section, the economic feasibility of rejuvenation in an asphalt plant with 1500 tons capacity has been investigated using simple engineering economics. In order to manufacture rejuvenated RAP in an asphalt facility, necessary plant modification is required. For a typical drum plant with 1500 tons manufacturing capacity, approximate RAP facility installation cost is foreseen to be 350,000.00\$. If an asphalt plant with 1500 tons of capacity is assumed to be active for just 20 days in a month, then the monthly production will be:

Monthly Plant Production = 1500 tons x 20 days = 30,000 tons

The differences for neat base course and base courses including CRA, WEO and WVO rejuvenated RAP materials are as follows:

CRA Rejuvenated RAP Saving / ton = 2.95\$

WEO Rejuvenated RAP Saving / ton = 5.03\$

WEO Rejuvenated RAP Saving / ton = 4.88\$

Monthly (20 days active) CRA Rejuvenated RAP Saving = 88,500.00\$

Monthly (20 days active) WEO Rejuvenated RAP Saving = 150,900.00\$

Monthly (20 days active) WVO Rejuvenated RAP Saving = 146,400.00\$

Based on this initial investment cost, a simple feasibility calculation reveals that the period of redemption for the most expensive rejuvenating agent (Commercial Rejuvenating Agent) is approximately 4 months for such an investment. The Internal Return Rate (IRR) can be calculated as more than 200% for CRA rejuvenation investment. For WEO and WVO rejuvenated RAP, the initial investment cost can be redeemed in two to three months.

CHAPTER SIX

CONCLUSIONS

The literature review unveils that numerous studies have been conducted by academics as well as sector stakeholders to introduce recycling and rejuvenating techniques. The economic and environmental benefits of recycling are great enough to investigate, develop and introduce novel technologies to asphalt industry. One of these novel technologies is use of rejuvenating agents to improve aged bitumen properties of RAP material. By improving the properties of aged RAP bitumen, more efficient recycling can be ensured. As discussed within this dissertation, there are commercial rejuvenating agents which can allegedly restore the aged bitumen characteristics to its original state to some extent. Many of these commercial rejuvenating agents are oils from various sources. Besides, waste oils can be used to modify bitumen to ease the stiffness problem of aged bitumen.

Within the scope of this study, waste oils together with a commercial rejuvenator produced locally were used to rejuvenate aged RAP bitumen. The new-born rejuvenated bitumen samples were investigated from rheological, morphological and chemical aspects. In addition to investigation conducted on bitumen samples, mixture testing was also performed to study the effectiveness of the used rejuvenating agents within bituminous mixtures.

Experimental studies on rejuvenated and non-rejuvenated bitumen samples included conventional testing and DSR testing for rheological studies, AFM imaging for morphological studies and FTIR spectra analysis for chemical studies. Mixture testing consisted of Marshall and IDT testing.

According to conventional tests conducted on neat, aged RAP and WEO, WVO and CRA rejuvenated bitumen samples, all rejuvenating agents including commercial product and waste oils are able to increase the penetration grade of aged bitumen to desired level through adding adequate amounts of additive. Softening points decrease when rejuvenating agents are used. Viscosity of aged RAP bitumen decrease in a

desired manner by implementing rejuvenators. The aging indices developed using viscosity values show that all three rejuvenators are effective to restore the properties of aged RAP bitumen to some extent. WEO and WVO rejuvenating agents perform very similar to each other in terms of conventional test results since CRA can be more effective than these two waste oils.

DSR testing showed that the rheological properties of aged RAP bitumen can be improved significantly by use of WEO, WVO and CRA. WEO and WVO similarly improve the rutting performance, phase angle and complex viscosity of aged RAP bitumen since CRA performs even better to accomplish this task. By using waste oils, although it is not actually possible to obtain a rejuvenated bitumen sample having the same rheological properties with neat bitumen but both recycling additives have impressive influence on aged bitumen. By adding 7.5% of CRA and 5% of waste oils, the rheology of aged bitumen changes dramatically. This change is obvious in all temperatures and frequency levels. By increasing in rejuvenating agent content the change in rheology also increases significantly.

CRA is more successful to restore the rheological characteristics of aged bitumen to its original properties since the after RFTO aging performance of CRA rejuvenated bitumen is not at the same level with neat virgin bitumen. All rejuvenating agents can restore the PG of aged bitumen sample to the PG of neat bitumen. All rejuvenated bitumen samples are capable of fulfilling the after RTFO specification limit although the after RTFO properties are not as better as the virgin neat bitumen properties.

When AFM images of rejuvenated and non-rejuvenated bitumen samples are investigated, bee structures observable on the surface of neat virgin bitumen can not be viewed on the micro surface of age bitumen. Tiny bee structures appear on rejuvenated bitumen samples. It can be concluded that all rejuvenating agents significantly change the micro topography and stiffness of aged bitumen. Although AFM images of virgin neat bitumen are significantly different from rejuvenated bitumen samples, similarities in terms of stiffness and bee structures can be observed compared to aged RAP bitumen. The change in polarity by adding different contents

of rejuvenating agents helps the bee structures appear on the surface of the rejuvenated bitumen in some extent.

Comparing the rejuvenators with each other, both waste oils images are very closed to each other in terms of topography with some differences in disperse phase and medium consistency. The topographic image of CRA is different from the rejuvenated waste oil samples. It can be understood that the stiffness of the bitumen changes considerably by rejuvenation with CRA. The consistency of the dispersion medium of rejuvenated bitumen samples appears to be better than that of the WEO and WVO rejuvenated bitumen samples.

FTIR spectra analysis show that at the same wavenumbers, the absorbance level of aged RAP bitumen sample is more than virgin bitumen. Rejuvenation with commercial rejuvenating agent and waste oils can help to approach lower absorbance between neat and aged bitumen sample at almost all ranges of wavenumbers. CRA seems to modify aged RAP bitumen sample more effectively than WEO and WVO. Peaks at specific wavenumbers associated with aging of bitumen can be observed. The effect of each rejuvenating agent's spectrum can also be viewed on rejuvenated bitumen spectrums. For example, the CRA and WVO rejuvenating agents which have vegetable bases, make the rejuvenated bitumen samples show peaks at specific wavenumbers associated with the ester carbonyl functional group found in vegetable oils.

As part of this study, mixture testing was performed. The results showed that the amount of RAP applicable in bituminous mixtures significantly increase by use of rejuvenators. Flow rate is the determining factor in selecting the largest amount of RAP in the mixtures. All rejuvenated samples tested and verified according to Turkish specification limits can fulfill the criteria. Implementing rejuvenators makes it possible to involve high amounts of RAP within HMA wearing courses.

By rejuvenation technology up to 70-80% of RAP can be utilized without adverse effects (as per Turkish specifications) within the surface courses. Rejuvenated mixtures are convenient to process in terms of mixing and compaction. Rejuvenators

can ease the compaction at standard HMA application temperature ranges. Investigations present that rejuvenated RAP mixtures are less brittle and more durable than non-rejuvenated RAP mixtures. Aging indices of rejuvenated mixtures are improved compared to non-rejuvenated mixtures.

All test results linked to binder and mixture testing show high consistency and conformity with each other. Rheological, morphological and chemical results support each other in terms of all bitumen samples. Waste oils can be used as rejuvenating agents. CRA is able to improve aged RAP bitumen and increase the amount of RAP significantly.

It is highly recommended that the low temperature properties of rejuvenated bitumen samples and recycled mixtures should be investigated carefully. Within the scope of this study, low temperature properties of the rejuvenated bitumen samples are not addressed adequately. The most concerned deterioration reference for recycled asphalt materials is fatigue and low temperature cracking. Thus, it is highly recommended to investigate the low temperature characteristics of rejuvenated bitumen samples using tests such as BBR and DTT.

In terms of rheological studies, other studies should be performed on aged, neat and rejuvenated bitumen samples such as Linear Amplitude Sweep (LAS), Multiple Stress Creep Recovery (MSCR) and Zero Shear Viscosity (ZSV) and the results should be compared to each other to fully identify the effect of rejuvenating agents on the rheological and creep behavior of aged bitumen.

As recommended within the literature, also based on RV and DSR results, the usability of all rejuvenating agents as WMA additives should be investigated. The oily based rejuvenating agents can decrease the viscosity and thus the mixing and compaction temperatures of modified samples. This fact generates the idea of using these additives as WMA additives.

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APPENDIX

Rheological Test Results



Table A.1 Rheological test results of WEO rejuvenated samples at different temperatures for 0.01 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (3%)	40	84.11	3.2241E+03	3.3086E+02	3.2071E+03	5.1312E+04	1.61E+01	5.00E-03	3.241253E+03
WEO (3%)	50	87.65	4.9824E+02	2.0430E+01	4.9782E+02	7.9294E+03	2.48E+00	4.97E-03	4.986552E+02
WEO (3%)	60	88.56	8.4186E+01	2.1156E+00	8.4160E+01	1.3398E+03	4.29E-01	5.09E-03	8.421304E+01
WEO (3%)	70	88.72	2.0704E+01	4.6248E-01	2.0698E+01	3.2950E+02	9.96E-02	4.81E-03	2.070877E+01
WEO (4%)	40	84.5	2.3991E+03	2.2995E+02	2.3881E+03	3.8182E+04	1.18E+01	4.90E-03	2.410227E+03
WEO (4%)	50	86.96	3.7408E+02	1.9838E+01	3.7355E+02	5.9534E+03	1.86E+00	4.98E-03	3.746026E+02
WEO (4%)	60	86.7	7.4044E+01	4.2622E+00	7.3921E+01	1.1784E+03	3.59E-01	4.85E-03	7.416650E+01
WEO (4%)	70	84.05	1.9338E+01	2.0046E+00	1.9234E+01	3.0776E+02	9.33E-02	4.82E-03	1.944252E+01
WEO (5%)	40	81.59	2.7249E+03	3.9854E+02	2.6956E+03	4.3368E+04	1.34E+01	4.90E-03	2.754568E+03
WEO (5%)	50	86.61	3.8458E+02	2.2741E+01	3.8391E+02	6.1206E+03	1.89E+00	4.91E-03	3.852553E+02
WEO (5%)	60	88.22	7.2078E+01	2.2389E+00	7.2044E+01	1.1471E+03	3.52E-01	4.88E-03	7.211324E+01
WEO (5%)	70	88.08	1.7433E+01	5.8409E-01	1.7424E+01	2.7745E+02	8.61E-02	4.94E-03	1.744315E+01
WEO (6%)	40	85.44	1.2683E+03	1.0083E+02	1.2643E+03	2.0185E+04	6.32E+00	4.98E-03	1.272332E+03
WEO (6%)	50	87.75	2.1946E+02	8.6160E+00	2.1929E+02	3.4927E+03	1.11E+00	5.05E-03	2.196301E+02
WEO (6%)	60	84.48	4.9811E+01	4.7915E+00	4.9580E+01	7.9275E+02	2.37E-01	4.76E-03	5.004331E+01
WEO (6%)	70	84.32	1.5697E+01	1.5536E+00	1.5620E+01	2.4982E+02	8.28E-02	5.27E-03	1.577430E+01
WEO (7%)	40	85.70	1.0356E+03	7.7645E+01	1.0326E+03	1.6481E+04	5.12E+00	4.95E-03	1.038484E+03
WEO (7%)	50	87.69	1.8098E+02	7.2945E+00	1.8083E+02	2.8803E+03	9.02E-01	4.98E-03	1.811246E+02
WEO (7%)	60	86.29	4.0694E+01	2.6331E+00	4.0608E+01	6.4764E+02	2.00E-01	4.91E-03	4.077916E+01
WEO (7%)	70	84.42	9.9085E+00	9.6346E-01	9.8615E+00	1.5769E+02	4.72E-02	4.77E-03	9.955664E+00
Neat Bitumen	40	87.62	8.6933E+00	3.6100E-01	8.6858E+00	1.3835E+02	4.47E+04	5.14E+03	8.700786E+00
Neat Bitumen	50	89.58	1.3208E+00	9.6815E-03	1.3207E+00	2.1020E+01	5.74E+03	4.35E+03	1.320788E+00
Neat Bitumen	60	86.75	2.4831E-01	1.4077E-02	2.4791E-01	3.9519E+00	1.26E+03	5.08E+03	2.487119E-01
Neat Bitumen	70	85.76	8.3114E-02	4.3105E-02	7.1063E-02	1.3228E+00	3.38E+02	4.07E+03	9.720951E-02
Aged RAP	40	76.97	1.1567E+04	2.6079E+03	1.1269E+04	1.8409E+05	5.61E+01	4.85E-03	1.187284E+04
Aged RAP	50	84.1	1.5810E+03	1.6252E+02	1.5727E+03	2.5162E+04	7.92E+00	5.01E-03	1.589458E+03
Aged RAP	60	87.14	2.7513E+02	1.3728E+01	2.7478E+02	4.3786E+03	1.38E+00	5.02E-03	2.754698E+02
Aged RAP	70	89.23	5.7409E+01	7.7150E-01	5.7404E+01	9.1367E+02	2.93E-01	5.10E-03	5.741453E+01

Table A.2 Rheological test results of WEO rejuvenated samples at different temperatures for 0.1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pa.s)	(Pa)	()	(Pa)
WEO (3%)	40	77.37	2.6158E+04	5.7196E+03	2.5525E+04	4.1631E+04	1.29E+02	4.94E-03	2.680689E+04
WEO (3%)	50	83.42	4.6024E+03	5.2739E+02	4.5721E+03	7.3247E+03	2.29E+01	4.97E-03	4.632901E+03
WEO (3%)	60	87.11	8.5348E+02	4.3031E+01	8.5239E+02	1.3583E+03	4.29E+00	5.03E-03	8.545665E+02
WEO (3%)	70	86.03	2.0048E+02	1.3880E+01	2.0000E+02	3.1907E+02	9.96E-01	4.97E-03	2.009646E+02
WEO (4%)	40	78.29	1.9525E+04	3.9627E+03	1.9119E+04	3.1074E+04	9.77E+01	5.00E-03	1.993987E+04
WEO (4%)	50	83.93	3.4356E+03	3.6330E+02	3.4164E+03	5.4678E+03	1.72E+01	5.02E-03	3.455007E+03
WEO (4%)	60	86.2	7.2740E+02	4.8207E+01	7.2580E+02	1.1576E+03	3.59E+00	4.94E-03	7.289981E+02
WEO (4%)	70	85.42	1.7645E+02	1.4090E+01	1.7589E+02	2.8083E+02	8.73E-01	4.95E-03	1.770189E+02
WEO (5%)	40	73.72	2.0032E+04	5.6157E+03	1.9229E+04	3.1882E+04	9.92E+01	4.95E-03	2.086919E+04
WEO (5%)	50	81.64	3.3993E+03	4.9423E+02	3.3632E+03	5.4100E+03	1.69E+01	4.96E-03	3.435822E+03
WEO (5%)	60	86.12	7.0068E+02	4.7413E+01	6.9907E+02	1.1151E+03	3.52E+00	5.02E-03	7.022870E+02
WEO (5%)	70	88.53	1.7777E+02	4.5604E+00	1.7771E+02	2.8292E+02	8.61E-01	4.84E-03	1.778284E+02
WEO (6%)	40	79.82	1.0885E+04	1.9238E+03	1.0713E+04	1.7323E+04	5.37E+01	4.93E-03	1.105891E+04
WEO (6%)	50	84.78	2.0915E+03	1.9028E+02	2.0828E+03	3.3286E+03	1.05E+01	5.01E-03	2.100202E+03
WEO (6%)	60	85.87	4.7502E+02	3.4211E+01	4.7378E+02	7.5599E+02	2.37E+00	4.99E-03	4.762540E+02
WEO (6%)	70	82.14	1.2673E+02	1.7331E+01	1.2554E+02	2.0170E+02	6.40E-01	5.05E-03	1.279365E+02
WEO (7%)	40	80.00	8.8183E+03	1.5313E+03	8.6843E+03	1.4034E+04	4.40E+01	4.99E-03	8.954318E+03
WEO (7%)	50	84.89	1.6968E+03	1.5113E+02	1.6901E+03	2.7005E+03	8.44E+00	4.98E-03	1.703600E+03
WEO (7%)	60	86.47	3.9804E+02	2.4508E+01	3.9728E+02	6.3348E+02	2.00E+00	5.02E-03	3.987962E+02
WEO (7%)	70	84.67	1.0704E+02	9.9432E+00	1.0658E+02	1.7036E+02	5.37E-01	5.02E-03	1.075057E+02
Neat Bitumen	40	84.27	8.3935E+01	8.3801E+00	8.3516E+01	1.3358E+02	4.23E+05	5.04E+03	8.435660E+01
Neat Bitumen	50	87.34	1.3443E+01	6.2386E-01	1.3428E+01	2.1394E+01	6.66E+04	4.95E+03	1.345719E+01
Neat Bitumen	60	88.63	2.6696E+00	6.3827E-02	2.6689E+00	4.2487E+00	1.30E+04	4.87E+03	2.670383E+00
Neat Bitumen	70	82.55	7.1212E-01	9.2335E-02	7.0611E-01	1.1333E+00	3.56E+03	5.00E+03	7.181867E-01
Aged RAP	40	68.44	7.4409E+04	2.7343E+04	6.9202E+04	1.1842E+05	3.67E+02	4.93E-03	8.000639E+04
Aged RAP	50	77.76	1.2623E+04	2.6762E+03	1.2336E+04	2.0090E+04	6.33E+01	5.01E-03	1.291670E+04
Aged RAP	60	83.16	2.5721E+03	3.0633E+02	2.5538E+03	4.0935E+03	1.28E+01	4.97E-03	2.590554E+03
Aged RAP	70	87.29	5.8670E+02	2.7739E+01	5.8604E+02	9.3373E+02	2.93E+00	4.99E-03	5.873520E+02

Table A.3 Rheological test results of WEO rejuvenated samples at different temperatures for 1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pa.s)	(Pa)	()	(Pa)
WEO (3%)	40	69.06	1.7022E+05	6.0837E+04	1.5898E+05	2.7091E+04	8.57E+02	5.03E-03	1.822624E+05
WEO (3%)	50	77.11	3.6179E+04	8.0707E+03	3.5267E+04	5.7578E+03	1.80E+02	4.99E-03	3.711396E+04
WEO (3%)	60	82.75	7.7689E+03	9.8043E+02	7.7068E+03	1.2364E+03	3.87E+01	4.98E-03	7.831556E+03
WEO (3%)	70	85.45	1.8726E+03	1.4855E+02	1.8667E+03	2.9803E+02	9.33E+00	4.98E-03	1.878529E+03
WEO (4%)	40	70.22	1.3201E+05	4.4673E+04	1.2422E+05	2.1009E+04	6.58E+02	4.98E-03	1.402855E+05
WEO (4%)	50	77.71	2.7828E+04	5.9234E+03	2.7190E+04	4.4287E+03	1.39E+02	4.99E-03	2.848021E+04
WEO (4%)	60	82.81	6.4499E+03	8.0727E+02	6.3992E+03	1.0265E+03	3.21E+01	4.98E-03	6.501047E+03
WEO (4%)	70	85.4	1.6328E+03	1.3095E+02	1.6275E+03	2.5986E+02	8.14E+00	4.99E-03	1.638050E+03
WEO (5%)	40	65.63	1.1821E+05	4.8776E+04	1.0768E+05	1.8813E+04	5.96E+02	5.04E-03	1.297707E+05
WEO (5%)	50	74.23	2.4988E+04	6.7911E+03	2.4047E+04	3.9768E+03	1.25E+02	5.01E-03	2.596513E+04
WEO (5%)	60	80.80	6.1590E+03	9.8470E+02	6.0797E+03	9.8020E+02	3.07E+01	4.99E-03	6.239228E+03
WEO (5%)	70	85.17	1.7149E+03	1.4439E+02	1.7088E+03	2.7292E+02	8.61E+00	5.02E-03	1.720978E+03
WEO (6%)	40	72.29	7.5668E+04	2.3018E+04	7.2082E+04	1.2043E+04	3.80E+02	5.02E-03	7.943245E+04
WEO (6%)	50	79.19	1.7350E+04	3.2540E+03	1.7042E+04	2.7612E+03	8.66E+01	4.99E-03	1.766322E+04
WEO (6%)	60	83.40	4.2871E+03	4.9275E+02	4.2587E+03	6.8230E+02	2.14E+01	5.00E-03	4.315734E+03
WEO (6%)	70	84.81	1.1544E+03	1.0443E+02	1.1497E+03	1.8372E+02	5.77E+00	5.00E-03	1.159161E+03
WEO (7%)	40	72.70	6.2503E+04	1.8587E+04	5.9675E+04	9.9473E+03	3.13E+02	5.00E-03	6.546454E+04
WEO (7%)	50	79.50	1.4192E+04	2.5863E+03	1.3954E+04	2.2587E+03	7.10E+01	5.01E-03	1.443375E+04
WEO (7%)	60	83.68	3.7047E+03	4.0782E+02	3.6822E+03	5.8960E+02	1.85E+01	5.00E-03	3.727341E+03
WEO (7%)	70	85.49	1.0079E+03	7.9255E+01	1.0048E+03	1.6041E+02	5.07E+00	5.03E-03	1.011035E+03
Neat Bitumen	40	79.13	6.9572E+02	1.3120E+02	6.8323E+02	1.1072E+02	3.50E+06	5.03E+03	7.084287E+02
Neat Bitumen	50	84.02	1.2029E+02	1.2532E+01	1.1964E+02	1.9145E+01	6.03E+05	5.01E+03	1.209529E+02
Neat Bitumen	60	86.73	2.5986E+01	1.4823E+00	2.5944E+01	4.1357E+00	1.30E+05	5.00E+03	2.602868E+01
Neat Bitumen	70	86.5	6.6190E+00	4.0408E-01	6.6066E+00	1.0534E+00	3.31E+04	5.00E+03	6.631348E+00
Aged RAP	40	58.81	3.8786E+05	2.0086E+05	3.3179E+05	6.1727E+04	1.91E+03	4.94E-03	4.533908E+05
Aged RAP	50	69.78	8.3438E+04	2.8838E+04	7.8296E+04	1.3279E+04	4.19E+02	5.02E-03	8.891733E+04
Aged RAP	60	77.02	2.0299E+04	4.5595E+03	1.9781E+04	3.2307E+03	1.01E+02	4.99E-03	2.083176E+04
Aged RAP	70	82.66	5.3386E+03	6.8204E+02	5.2949E+03	8.4964E+02	2.67E+01	5.01E-03	5.382712E+03

Table A.4 Rheological test results of WEO rejuvenated samples at different temperatures for 10 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (3%)	40	54.8	8.7776E+05	5.0597E+05	7.1726E+05	1.3970E+04	6.18E+03	7.04E-03	1.074179E+06
WEO (3%)	50	68.62	2.3682E+05	8.6333E+04	2.2052E+05	3.7690E+03	1.20E+03	5.07E-03	2.543207E+05
WEO (3%)	60	76.41	5.9604E+04	1.4005E+04	5.7935E+04	9.4859E+02	2.98E+02	4.99E-03	6.132063E+04
WEO (3%)	70	81.25	1.5947E+04	2.4259E+03	1.5761E+04	2.5379E+02	8.06E+01	5.05E-03	1.613444E+04
WEO (4%)	40	57.77	7.0664E+05	3.7687E+05	5.9776E+05	1.1246E+04	3.59E+03	5.09E-03	8.353603E+05
WEO (4%)	50	70.01	1.8595E+05	6.3567E+04	1.7474E+05	2.9594E+03	9.43E+02	5.07E-03	1.978684E+05
WEO (4%)	60	76.97	4.9811E+04	1.1231E+04	4.8529E+04	7.9275E+02	2.49E+02	4.99E-03	5.112777E+04
WEO (4%)	70	81.29	1.3982E+04	2.1174E+03	1.3821E+04	2.2253E+02	7.01E+01	5.01E-03	1.414523E+04
WEO (5%)	40	55.50	5.7024E+05	3.2299E+05	4.6995E+05	9.0754E+03	2.85E+03	5.00E-03	6.919334E+05
WEO (5%)	50	66.51	1.5058E+05	6.0020E+04	1.3810E+05	2.3965E+03	7.53E+02	5.00E-03	1.641888E+05
WEO (5%)	60	73.71	4.4031E+04	1.2351E+04	4.2264E+04	7.0076E+02	2.38E+02	5.41E-03	4.587294E+04
WEO (5%)	70	79.73	1.4150E+04	2.5228E+03	1.3924E+04	2.2521E+02	7.07E+01	4.99E-03	1.438090E+04
WEO (6%)	40	61.89	4.3398E+05	2.0448E+05	3.8279E+05	6.9068E+03	2.44E+03	5.63E-03	4.920169E+05
WEO (6%)	50	71.69	1.1906E+05	3.7404E+04	1.1303E+05	1.8948E+03	6.04E+02	5.07E-03	1.254094E+05
WEO (6%)	60	77.77	3.3911E+04	7.1835E+03	3.3141E+04	5.3969E+02	1.70E+02	5.02E-03	3.469823E+04
WEO (6%)	70	81.82	9.9442E+03	1.4149E+03	9.8430E+03	1.5826E+02	4.84E+01	4.86E-03	1.004640E+04
WEO (7%)	40	63.23	3.6444E+05	1.6415E+05	3.2538E+05	5.8000E+03	1.88E+03	5.17E-03	4.081859E+05
WEO (7%)	50	72.48	9.9218E+04	2.9869E+04	9.4616E+04	1.5791E+03	4.92E+02	4.96E-03	1.040448E+05
WEO (7%)	60	78.18	2.9622E+04	6.0677E+03	2.8994E+04	4.7143E+02	1.49E+02	5.03E-03	3.026349E+04
WEO (7%)	70	82.46	8.8703E+03	1.1639E+03	8.7936E+03	1.4117E+02	4.58E+01	5.16E-03	8.947650E+03
Neat Bitumen	40	73.39	5.0147E+03	1.4335E+03	4.8054E+03	7.9809E+01	3.67E+05	7.32E+01	5.233050E+03
Neat Bitumen	50	78.95	9.8491E+02	1.8877E+02	9.6665E+02	1.5675E+01	5.29E+06	5.37E+03	1.003518E+03
Neat Bitumen	60	83.29	2.2902E+02	2.6760E+01	2.2745E+02	3.6449E+00	1.15E+06	5.04E+03	2.306006E+02
Neat Bitumen	70	85.69	6.2780E+01	4.7181E+00	6.2603E+01	9.9915E-01	3.13E+05	4.98E+03	6.295817E+01
Aged RAP	40	42.35	1.4806E+06	1.0942E+06	9.9739E+05	2.3563E+04	1.17E+04	7.91E-03	2.197786E+06
Aged RAP	50	60.42	4.5058E+05	2.2242E+05	3.9185E+05	7.1709E+03	2.23E+03	4.95E-03	5.181039E+05
Aged RAP	60	69.52	1.3184E+05	4.6128E+04	1.2351E+05	2.0982E+03	6.33E+02	4.80E-03	1.407351E+05
Aged RAP	70	75.95	4.0551E+04	9.8445E+03	3.9338E+04	6.4537E+02	2.04E+02	5.02E-03	4.180136E+04

Table A.5 Rheological test results of WVO rejuvenated samples at different temperatures for 0.01 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WVO (3%)	40	85.22	2.0096E+03	1.6746E+02	2.0026E+03	3.1983E+04	9.94E+00	4.95E-03	2.016634E+03
WVO (3%)	50	88.3	3.2798E+02	9.7301E+00	3.2784E+02	5.2199E+03	1.56E+00	4.76E-03	3.281289E+02
WVO (3%)	60	89.66	6.2904E+01	3.7328E-01	6.2902E+01	1.0011E+03	3.22E-01	5.12E-03	6.290469E+01
WVO (3%)	70	89.5	1.5251E+01	1.3309E-01	1.5250E+01	2.4272E+02	7.14E-02	4.68E-03	1.525149E+01
WVO (4%)	40	86.09	1.3007E+03	8.8697E+01	1.2977E+03	2.0701E+04	6.49E+00	4.99E-03	1.303776E+03
WVO (4%)	50	87.98	2.9888E+02	1.0535E+01	2.9869E+02	4.7566E+03	1.49E+00	4.99E-03	2.990622E+02
WVO (4%)	60	88.96	4.5348E+01	8.2309E-01	4.5341E+01	7.2172E+02	2.28E-01	5.02E-03	4.535597E+01
WVO (4%)	70	80.16	1.1152E+01	1.9058E+00	1.0988E+01	1.7748E+02	5.84E-02	5.24E-03	1.131850E+01
WVO (5%)	40	83.95	1.7218E+03	1.8147E+02	1.7122E+03	2.7403E+04	8.83E+00	5.13E-03	1.731475E+03
WVO (5%)	50	87.72	2.8354E+02	1.1280E+01	2.8332E+02	4.5126E+03	1.41E+00	4.98E-03	2.837666E+02
WVO (5%)	60	84.42	4.0557E+01	3.9436E+00	4.0365E+01	6.4547E+02	1.73E-01	4.27E-03	4.075029E+01
WVO (5%)	70	83.05	1.6813E+01	2.0344E+00	1.6689E+01	2.6757E+02	8.16E-02	4.86E-03	1.693702E+01
WVO (6%)	40	86.50	5.6258E+02	3.4345E+01	5.6153E+02	8.9534E+03	2.78E+00	4.93E-03	5.636294E+02
WVO (6%)	50	87.87	1.0617E+02	3.9462E+00	1.0610E+02	1.6898E+03	5.11E-01	4.81E-03	1.062473E+02
WVO (6%)	60	86.63	2.4887E+01	1.4629E+00	2.4844E+01	3.9607E+02	1.25E-01	5.03E-03	2.493001E+01
WVO (6%)	70	85.18	6.3980E+00	5.3760E-01	6.3754E+00	1.0182E+02	3.26E-02	5.10E-03	6.420704E+00
WVO (7%)	40	87.29	5.0028E+02	2.3654E+01	4.9972E+02	7.9620E+03	2.52E+00	5.03E-03	5.008420E+02
WVO (7%)	50	88.68	8.9332E+01	2.0579E+00	8.9309E+01	1.4217E+03	4.40E-01	4.93E-03	8.935614E+01
WVO (7%)	60	88.56	2.0491E+01	5.1495E-01	2.0485E+01	3.2612E+02	1.05E-01	5.13E-03	2.049791E+01
WVO (7%)	70	88.54	5.5516E+00	1.4145E-01	5.5498E+00	8.8354E+01	2.68E-02	4.83E-03	5.553441E+00
Neat Bitumen	40	87.62	8.6933E+00	3.6100E-01	8.6858E+00	1.3835E+02	4.47E+04	5.14E+03	8.700786E+00
Neat Bitumen	50	89.58	1.3208E+00	9.6815E-03	1.3207E+00	2.1020E+01	5.74E+03	4.35E+03	1.320788E+00
Neat Bitumen	60	86.75	2.4831E-01	1.4077E-02	2.4791E-01	3.9519E+00	1.26E+03	5.08E+03	2.487119E-01
Neat Bitumen	70	85.76	8.3114E-02	6.1450E-03	8.2887E-02	1.3228E+00	3.38E+02	4.07E+03	8.334246E-02
Aged RAP	40	76.97	1.1567E+04	2.6079E+03	1.1269E+04	1.8409E+05	5.61E+01	4.85E-03	1.187284E+04
Aged RAP	50	84.1	1.5810E+03	1.6252E+02	1.5727E+03	2.5162E+04	7.92E+00	5.01E-03	1.589458E+03
Aged RAP	60	87.14	2.7513E+02	1.3728E+01	2.7478E+02	4.3786E+03	1.38E+00	5.02E-03	2.754698E+02
Aged RAP	70	89.23	5.7409E+01	7.7150E-01	5.7404E+01	9.1367E+02	2.93E-01	5.10E-03	5.741453E+01

Table A.6 Rheological test results of WVO rejuvenated samples at different temperatures for 0.1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WVO (3%)	40	78.95	1.6815E+04	3.2228E+03	1.6503E+04	2.6761E+04	8.36E+01	4.97E-03	1.713247E+04
WVO (3%)	50	84.46	3.1142E+03	3.0064E+02	3.0996E+03	4.9562E+03	1.56E+01	5.02E-03	3.128768E+03
WVO (3%)	60	87.53	6.8659E+02	2.9590E+01	6.8595E+02	1.0927E+03	3.47E+00	5.05E-03	6.872314E+02
WVO (3%)	70	88.39	1.5557E+02	4.3708E+00	1.5551E+02	2.4759E+02	7.59E-01	4.88E-03	1.556292E+02
WVO (4%)	40	80.73	1.1222E+04	1.8077E+03	1.1075E+04	1.7860E+04	5.58E+01	4.97E-03	1.137038E+04
WVO (4%)	50	83.73	2.9699E+03	3.2435E+02	2.9521E+03	4.7266E+03	1.48E+01	4.97E-03	2.987778E+03
WVO (4%)	60	87.64	4.5872E+02	1.8889E+01	4.5833E+02	7.3005E+02	2.28E+00	4.96E-03	4.591077E+02
WVO (4%)	70	87.31	1.1908E+02	5.5887E+00	1.1895E+02	1.8952E+02	5.84E-01	4.91E-03	1.192129E+02
WVO (5%)	40	76.50	1.4273E+04	3.3319E+03	1.3879E+04	2.2715E+04	7.11E+01	4.98E-03	1.467849E+04
WVO (5%)	50	83.29	2.6131E+03	3.0532E+02	2.5952E+03	4.1587E+03	1.30E+01	4.99E-03	2.631076E+03
WVO (5%)	60	85.71	3.1570E+02	2.3616E+01	3.1481E+02	5.0243E+02	1.70E+00	5.38E-03	3.165845E+02
WVO (5%)	70	86.86	1.5409E+02	8.4402E+00	1.5385E+02	2.4523E+02	7.72E-01	5.01E-03	1.543180E+02
WVO (6%)	40	82.88	5.0150E+03	6.2160E+02	4.9764E+03	7.9814E+03	2.50E+01	4.99E-03	5.053995E+03
WVO (6%)	50	85.96	1.0224E+03	7.2029E+01	1.0198E+03	1.6271E+03	5.11E+00	4.99E-03	1.024915E+03
WVO (6%)	60	87.14	2.5002E+02	1.2475E+01	2.4971E+02	3.9790E+02	1.25E+00	5.01E-03	2.503292E+02
WVO (6%)	70	86.67	6.7351E+01	3.9122E+00	6.7237E+01	1.0719E+02	3.26E-01	4.84E-03	6.746480E+01
WVO (7%)	40	83.16	4.6510E+03	5.5392E+02	4.6179E+03	7.4021E+03	2.31E+01	4.98E-03	4.684356E+03
WVO (7%)	50	86.82	8.8137E+02	4.8892E+01	8.8001E+02	1.4027E+03	4.40E+00	4.99E-03	8.827275E+02
WVO (7%)	60	88.37	2.1746E+02	6.1856E+00	2.1737E+02	3.4609E+02	1.05E+00	4.83E-03	2.175475E+02
WVO (7%)	70	88.55	6.3663E+01	1.6110E+00	6.3643E+01	1.0132E+02	3.22E-01	5.05E-03	6.368356E+01
Neat Bitumen	40	84.27	8.3935E+01	8.3801E+00	8.3516E+01	1.3358E+02	4.23E+05	5.04E+03	8.435660E+01
Neat Bitumen	50	87.34	1.3443E+01	6.2386E-01	1.3428E+01	2.1394E+01	6.66E+04	4.95E+03	1.345719E+01
Neat Bitumen	60	88.63	2.6696E+00	6.3827E-02	2.6689E+00	4.2487E+00	1.30E+04	4.87E+03	2.670383E+00
Neat Bitumen	70	82.55	7.1212E-01	9.2335E-02	7.0611E-01	1.1333E+00	3.56E+03	5.00E+03	7.181867E-01
Aged RAP	40	68.44	7.4409E+04	2.7343E+04	6.9202E+04	1.1842E+05	3.67E+02	4.93E-03	8.000639E+04
Aged RAP	50	77.76	1.2623E+04	2.6762E+03	1.2336E+04	2.0090E+04	6.33E+01	5.01E-03	1.291670E+04
Aged RAP	60	83.16	2.5721E+03	3.0633E+02	2.5538E+03	4.0935E+03	1.28E+01	4.97E-03	2.590554E+03
Aged RAP	70	87.29	5.8670E+02	2.7739E+01	5.8604E+02	9.3373E+02	2.93E+00	4.99E-03	5.873520E+02

Table A.7 Rheological test results of WVO rejuvenated samples at different temperatures for 1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WVO (3%)	40	71.39	1.1558E+05	3.6884E+04	1.0954E+05	1.8394E+04	5.77E+02	4.99E-03	1.219551E+05
WVO (3%)	50	78.47	2.5677E+04	5.1324E+03	2.5159E+04	4.0865E+03	1.29E+02	5.03E-03	2.620592E+04
WVO (3%)	60	83.34	6.3730E+03	7.3912E+02	6.3299E+03	1.0143E+03	3.19E+01	5.01E-03	6.416251E+03
WVO (3%)	70	86.48	1.5198E+03	9.3309E+01	1.5169E+03	2.4187E+02	7.59E+00	4.99E-03	1.522646E+03
WVO (4%)	40	73.51	8.0989E+04	2.2989E+04	7.7658E+04	1.2889E+04	4.05E+02	5.00E-03	8.446306E+04
WVO (4%)	50	77.51	2.3033E+04	4.9813E+03	2.2488E+04	3.6656E+03	1.15E+02	5.00E-03	2.359097E+04
WVO (4%)	60	84.31	4.2333E+03	4.1971E+02	4.2124E+03	6.7372E+02	2.11E+01	4.98E-03	4.254222E+03
WVO (4%)	70	86.56	1.1638E+03	6.9830E+01	1.1617E+03	1.8521E+02	5.84E+00	5.02E-03	1.165863E+03
WVO (5%)	40	68.76	9.1056E+04	3.2987E+04	8.4870E+04	1.4492E+04	4.58E+02	5.03E-03	9.769170E+04
WVO (5%)	50	76.42	2.0602E+04	4.8373E+03	2.0026E+04	3.2788E+03	1.02E+02	4.96E-03	2.119424E+04
WVO (5%)	60	84.48	9.2479E+03	8.8959E+02	9.2050E+03	1.4718E+03	4.65E+01	5.03E-03	9.291019E+03
WVO (5%)	70	85.32	1.4586E+03	1.1901E+02	1.4538E+03	2.3214E+02	7.28E+00	4.99E-03	1.463509E+03
WVO (6%)	40	76.28	3.8662E+04	9.1698E+03	3.7559E+04	6.1531E+03	1.94E+02	5.01E-03	3.979786E+04
WVO (6%)	50	81.83	8.9990E+03	1.2789E+03	8.9077E+03	1.4322E+03	4.49E+01	4.99E-03	9.091309E+03
WVO (6%)	60	85.23	2.3489E+03	1.9533E+02	2.3408E+03	3.7383E+02	1.18E+01	5.01E-03	2.357075E+03
WVO (6%)	70	86.64	6.5051E+02	3.8126E+01	6.4940E+02	1.0353E+02	3.26E+00	5.02E-03	6.516350E+02
WVO (7%)	40	76.60	3.5846E+04	8.3072E+03	3.4870E+04	5.7048E+03	1.79E+02	4.99E-03	3.684891E+04
WVO (7%)	50	82.14	7.8635E+03	1.0754E+03	7.7896E+03	1.2515E+03	3.94E+01	5.01E-03	7.938098E+03
WVO (7%)	60	85.73	2.1045E+03	1.5669E+02	2.0986E+03	3.3493E+02	1.05E+01	4.99E-03	2.110346E+03
WVO (7%)	70	87.28	6.4086E+02	3.0412E+01	6.4014E+02	1.0199E+02	3.22E+00	5.02E-03	6.415819E+02
Neat Bitumen	40	79.13	6.9572E+02	1.3120E+02	6.8323E+02	1.1072E+02	3.50E+06	5.03E+03	7.084287E+02
Neat Bitumen	50	84.02	1.2029E+02	1.2532E+01	1.1964E+02	1.9145E+01	6.03E+05	5.01E+03	1.209529E+02
Neat Bitumen	60	86.73	2.5986E+01	1.4823E+00	2.5944E+01	4.1357E+00	1.30E+05	5.00E+03	2.602868E+01
Neat Bitumen	70	86.5	6.6190E+00	4.0408E-01	6.6066E+00	1.0534E+00	3.31E+04	5.00E+03	6.631348E+00
Aged RAP	40	58.81	3.8786E+05	2.0086E+05	3.3179E+05	6.1727E+04	1.91E+03	4.94E-03	4.533908E+05
Aged RAP	50	69.78	8.3438E+04	2.8838E+04	7.8296E+04	1.3279E+04	4.19E+02	5.02E-03	8.891733E+04
Aged RAP	60	77.02	2.0299E+04	4.5595E+03	1.9781E+04	3.2307E+03	1.01E+02	4.99E-03	2.083176E+04
Aged RAP	70	82.66	5.3386E+03	6.8204E+02	5.2949E+03	8.4964E+02	2.67E+01	5.01E-03	5.382712E+03

Table A.8 Rheological test results of WVO rejuvenated samples at different temperatures for 10 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WVO (3%)	40	59.79	6.4644E+05	3.2527E+05	5.5864E+05	1.0288E+04	3.35E+03	5.18E-03	7.480312E+05
WVO (3%)	50	70.97	1.7418E+05	5.6793E+04	1.6466E+05	2.7720E+03	8.72E+02	5.01E-03	1.842465E+05
WVO (3%)	60	77.25	4.9947E+04	1.1023E+04	4.8716E+04	7.9491E+02	2.42E+02	4.84E-03	5.120995E+04
WVO (3%)	70	82.12	1.3228E+04	1.8136E+03	1.3104E+04	2.1053E+02	6.72E+01	5.08E-03	1.335454E+04
WVO (4%)	40	63.22	4.8255E+05	2.1742E+05	4.3079E+05	7.6797E+03	2.42E+03	5.01E-03	5.405218E+05
WVO (4%)	50	67.22	1.4316E+05	5.5431E+04	1.3199E+05	2.2784E+03	7.17E+02	5.01E-03	1.552726E+05
WVO (4%)	60	78.82	3.4093E+04	6.6104E+03	3.3446E+04	5.4259E+02	1.71E+02	5.02E-03	3.475274E+04
WVO (4%)	70	82.94	1.0340E+04	1.2709E+03	1.0261E+04	1.6456E+02	5.22E+01	5.05E-03	1.041882E+04
WVO (5%)	40	59.05	4.8099E+05	2.4737E+05	4.1251E+05	7.6550E+03	3.17E+03	6.60E-03	5.608453E+05
WVO (5%)	50	65.45	1.2572E+05	5.2235E+04	1.1435E+05	2.0008E+03	6.57E+02	5.23E-03	1.382144E+05
WVO (5%)	60	74.81	4.8290E+04	1.2653E+04	4.6603E+04	7.6854E+02	2.40E+02	4.97E-03	5.003827E+04
WVO (5%)	70	80.26	1.2150E+04	2.0555E+03	1.1975E+04	1.9337E+02	6.07E+01	5.00E-03	1.232778E+04
WVO (6%)	40	68.24	2.4952E+05	9.2501E+04	2.3174E+05	3.9711E+03	1.25E+03	5.01E-03	2.686602E+05
WVO (6%)	50	75.53	6.7591E+04	1.6889E+04	6.5447E+04	1.0757E+03	3.38E+02	5.00E-03	6.980518E+04
WVO (6%)	60	81.05	1.9627E+04	3.0534E+03	1.9388E+04	3.1237E+02	9.76E+01	4.97E-03	1.986908E+04
WVO (6%)	70	84.06	6.0582E+03	6.2695E+02	6.0257E+03	9.6417E+01	3.02E+01	4.98E-03	6.090931E+03
WVO (7%)	40	68.76	2.3263E+05	8.4275E+04	2.1683E+05	3.7023E+03	1.18E+03	5.07E-03	2.495820E+05
WVO (7%)	50	76.05	5.9637E+04	1.4377E+04	5.7878E+04	9.4912E+02	2.98E+02	5.00E-03	6.144904E+04
WVO (7%)	60	80.99	1.7852E+04	2.7958E+03	1.7632E+04	2.8412E+02	8.93E+01	5.00E-03	1.807512E+04
WVO (7%)	70	84.16	6.0246E+03	6.1301E+02	5.9933E+03	9.5882E+01	3.01E+01	5.00E-03	6.056040E+03
Neat Bitumen	40	73.39	5.0147E+03	1.4335E+03	4.8054E+03	7.9809E+01	3.67E+05	7.32E+01	5.233050E+03
Neat Bitumen	50	78.95	9.8491E+02	1.8877E+02	9.6665E+02	1.5675E+01	5.29E+06	5.37E+03	1.003518E+03
Neat Bitumen	60	83.29	2.2902E+02	2.6760E+01	2.2745E+02	3.6449E+00	1.15E+06	5.04E+03	2.306006E+02
Neat Bitumen	70	85.69	6.2780E+01	4.7181E+00	6.2603E+01	9.9915E-01	3.13E+05	4.98E+03	6.295817E+01
Aged RAP	40	42.35	1.4806E+06	1.0942E+06	9.9739E+05	2.3563E+04	1.17E+04	7.91E-03	2.197786E+06
Aged RAP	50	60.42	4.5058E+05	2.2242E+05	3.9185E+05	7.1709E+03	2.23E+03	4.95E-03	5.181039E+05
Aged RAP	60	69.52	1.3184E+05	4.6128E+04	1.2351E+05	2.0982E+03	6.33E+02	4.80E-03	1.407351E+05
Aged RAP	70	75.95	4.0551E+04	9.8445E+03	3.9338E+04	6.4537E+02	2.04E+02	5.02E-03	4.180136E+04

Table A.9 Rheological test results of CRA rejuvenated samples at different temperatures for 0.01 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
CRA (5.5%)	40	84.65	2.0384E+01	1.9006E+00	2.0295E+01	3.2441E+02	9.97E+04	4.89E+03	2.047322E+01
CRA (5.5%)	50	87.3	3.5190E+00	1.6577E-01	3.5151E+00	5.6005E+01	1.77E+04	5.04E+03	3.522913E+00
CRA (5.5%)	60	87.83	7.0133E-01	2.6555E-02	7.0082E-01	1.1162E+01	3.48E+03	4.96E+03	7.018296E-01
CRA (5.5%)	70	87.06	1.7706E-01	9.0817E-03	1.7683E-01	2.8180E+00	8.90E+02	5.03E+03	1.772976E-01
CRA (6.5%)	40	84.91	1.6516E+01	1.4653E+00	1.6451E+01	2.6286E+02	8.19E+04	4.96E+03	1.658177E+01
CRA (6.5%)	50	87.1	2.7786E+00	1.4058E-01	2.7751E+00	4.4222E+01	1.37E+04	4.94E+03	2.782210E+00
CRA (6.5%)	60	87.85	5.8093E-01	2.1794E-02	5.8053E-01	9.2456E+00	2.84E+03	4.89E+03	5.813433E-01
CRA (6.5%)	70	84.25	1.5915E-01	1.5945E-02	1.5835E-01	2.5329E+00	7.69E+02	4.83E+03	1.599589E-01
CRA (7.5%)	40	87.06	8.1733E+00	4.1921E-01	8.1626E+00	1.3008E+02	4.03E+04	4.92E+03	8.184087E+00
CRA (7.5%)	50	88.08	1.4637E+00	4.9039E-02	1.4629E+00	2.3295E+01	7.13E+03	4.87E+03	1.464509E+00
CRA (7.5%)	60	87.82	3.2331E-01	1.2299E-02	3.2308E-01	5.1456E+00	1.65E+03	5.12E+03	3.235489E-01
CRA (7.5%)	70	85.01	8.6432E-02	7.5180E-03	8.6104E-02	1.3756E+00	4.51E+02	5.22E+03	8.676070E-02
CRA (8.5%)	40	86.67	7.9790E+00	4.6347E-01	7.9655E+00	1.2699E+02	3.97E+04	4.97E+03	7.992455E+00
CRA (8.5%)	50	87.63	1.4193E+00	5.8691E-02	1.4181E+00	2.2588E+01	6.84E+03	4.82E+03	1.420504E+00
CRA (8.5%)	60	87.72	3.0530E-01	1.2146E-02	3.0506E-01	4.8589E+00	1.51E+03	4.95E+03	3.055456E-01
CRA (8.5%)	70	88.14	8.4940E-02	2.7569E-03	8.4895E-02	1.3518E+00	4.35E+02	5.12E+03	8.498464E-02
CRA (9.5%)	40	88.47	8.7037E-01	2.3239E-02	8.7006E-01	1.3852E+01	4.30E+03	4.94E+03	8.706801E-01
CRA (9.5%)	50	88.82	1.9437E-01	4.0027E-03	1.9433E-01	3.0934E+00	1.01E+03	5.18E+03	1.944103E-01
CRA (9.5%)	60	84.89	5.4004E-02	4.8100E-03	5.3789E-02	8.5947E-01	2.06E+02	3.82E+03	5.421941E-02
CRA (9.5%)	70	78.72	2.9454E-02	5.7614E-03	2.8885E-02	4.6877E-01	1.63E+02	5.53E+03	3.003461E-02
Neat Bitumen	40	87.62	8.6933E+00	3.6100E-01	8.6858E+00	1.3835E+02	4.47E+04	5.14E+03	8.700786E+00
Neat Bitumen	50	89.58	1.3208E+00	9.6815E-03	1.3207E+00	2.1020E+01	5.74E+03	4.35E+03	1.320788E+00
Neat Bitumen	60	86.75	2.4831E-01	1.4077E-02	2.4791E-01	3.9519E+00	1.26E+03	5.08E+03	2.487119E-01
Neat Bitumen	70	85.76	8.3114E-02	6.1450E-03	8.2887E-02	1.3228E+00	3.38E+02	4.07E+03	8.334246E-02
Aged RAP	40	76.97	1.1567E+04	2.6079E+03	1.1269E+04	1.8409E+05	5.61E+01	4.85E-03	1.187284E+04
Aged RAP	50	84.1	1.5810E+03	1.6252E+02	1.5727E+03	2.5162E+04	7.92E+00	5.01E-03	1.589458E+03
Aged RAP	60	87.14	2.7513E+02	1.3728E+01	2.7478E+02	4.3786E+03	1.38E+00	5.02E-03	2.754698E+02
Aged RAP	70	89.23	5.7409E+01	7.7150E-01	5.7404E+01	9.1367E+02	2.93E-01	5.10E-03	5.741453E+01

Table A.10 Rheological test results of CRA rejuvenated samples at different temperatures for 0.1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pa.s)	(Pa)	()	(Pa)
CRA (5.5%)	40	78.84	1.6720E+02	3.2362E+01	1.6404E+02	2.6611E+02	8.31E+05	4.97E+03	1.704267E+02
CRA (5.5%)	50	83.87	3.3498E+01	3.5770E+00	3.3306E+01	5.3311E+01	1.68E+05	5.02E+03	3.369016E+01
CRA (5.5%)	60	86.79	6.9414E+00	3.8869E-01	6.9305E+00	1.1047E+01	3.48E+04	5.01E+03	6.952315E+00
CRA (5.5%)	70	87.2	1.7423E+00	8.5112E-02	1.7402E+00	2.7729E+00	8.29E+03	4.76E+03	1.744401E+00
CRA (6.5%)	40	79.31	1.3709E+02	2.5430E+01	1.3471E+02	2.1819E+02	6.80E+05	4.96E+03	1.395152E+02
CRA (6.5%)	50	84.39	2.5601E+01	2.5027E+00	2.5478E+01	4.0744E+01	1.28E+05	4.99E+03	2.572431E+01
CRA (6.5%)	60	86.62	5.6587E+00	3.3362E-01	5.6488E+00	9.0058E+00	2.84E+04	5.02E+03	5.668554E+00
CRA (6.5%)	70	86.71	1.5381E+00	8.8271E-02	1.5356E+00	2.4479E+00	7.69E+03	5.00E+03	1.540638E+00
CRA (7.5%)	40	82.75	7.2874E+01	9.1966E+00	7.2291E+01	1.1598E+02	3.62E+05	4.97E+03	7.346129E+01
CRA (7.5%)	50	86.34	1.4270E+01	9.1093E-01	1.4241E+01	2.2711E+01	7.13E+04	5.00E+03	1.429907E+01
CRA (7.5%)	60	87.81	3.2850E+00	1.2553E-01	3.2826E+00	5.2281E+00	1.65E+04	5.04E+03	3.287430E+00
CRA (7.5%)	70	87.35	9.1849E-01	4.2466E-02	9.1750E-01	1.4618E+00	4.51E+03	4.91E+03	9.194696E-01
CRA (8.5%)	40	82.56	7.0853E+01	9.1746E+00	7.0257E+01	1.1276E+02	3.53E+05	4.99E+03	7.145461E+01
CRA (8.5%)	50	86.14	1.3678E+01	9.2081E-01	1.3647E+01	2.1769E+01	6.84E+04	5.00E+03	1.370945E+01
CRA (8.5%)	60	87.51	3.0100E+00	1.3077E-01	3.0072E+00	4.7905E+00	1.51E+04	5.02E+03	3.012884E+00
CRA (8.5%)	70	87.78	8.3460E-01	3.2330E-02	8.3397E-01	1.3283E+00	4.02E+03	4.81E+03	8.352276E-01
CRA (9.5%)	40	86.72	8.6007E+00	4.9209E-01	8.5866E+00	1.3688E+01	4.30E+04	5.00E+03	8.614811E+00
CRA (9.5%)	50	88.27	1.9988E+00	6.0343E-02	1.9979E+00	3.1811E+00	1.01E+04	5.03E+03	1.999701E+00
CRA (9.5%)	60	87.51	5.7983E-01	2.5191E-02	5.7928E-01	9.2280E-01	2.92E+03	5.03E+03	5.803780E-01
CRA (9.5%)	70	89.45	1.8291E-01	1.7558E-03	1.8290E-01	2.9110E-01	9.54E+02	5.21E+03	1.829184E-01
Neat Bitumen	40	84.27	8.3935E+01	8.3801E+00	8.3516E+01	1.3358E+02	4.23E+05	5.04E+03	8.435660E+01
Neat Bitumen	50	87.34	1.3443E+01	6.2386E-01	1.3428E+01	2.1394E+01	6.66E+04	4.95E+03	1.345719E+01
Neat Bitumen	60	88.63	2.6696E+00	6.3827E-02	2.6689E+00	4.2487E+00	1.30E+04	4.87E+03	2.670383E+00
Neat Bitumen	70	82.55	7.1212E-01	9.2335E-02	7.0611E-01	1.1333E+00	3.56E+03	5.00E+03	7.181867E-01
Aged RAP	40	68.44	7.4409E+04	2.7343E+04	6.9202E+04	1.1842E+05	3.67E+02	4.93E-03	8.000639E+04
Aged RAP	50	77.76	1.2623E+04	2.6762E+03	1.2336E+04	2.0090E+04	6.33E+01	5.01E-03	1.291670E+04
Aged RAP	60	83.16	2.5721E+03	3.0633E+02	2.5538E+03	4.0935E+03	1.28E+01	4.97E-03	2.590554E+03
Aged RAP	70	87.29	5.8670E+02	2.7739E+01	5.8604E+02	9.3373E+02	2.93E+00	4.99E-03	5.873520E+02

Table A.11 Rheological test results of CRA rejuvenated samples at different temperatures for 1 Hz frequency

Frequency	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
(Hz)	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
CRA (5.5%)	40	71.15	1.1342E+03	3.6645E+02	1.0734E+03	1.8051E+02	5.69E+06	5.02E+03	1.198468E+03
CRA (5.5%)	50	77.67	2.7156E+02	5.7990E+01	2.6530E+02	4.3220E+01	1.36E+06	5.03E+03	2.779766E+02
CRA (5.5%)	60	82.82	6.2895E+01	7.8611E+00	6.2402E+01	1.0010E+01	3.14E+05	4.99E+03	6.339243E+01
CRA (5.5%)	70	85.57	1.6624E+01	1.2841E+00	1.6575E+01	2.6458E+00	8.29E+04	4.99E+03	1.667428E+01
CRA (6.5%)	40	71.89	9.4468E+02	2.9365E+02	8.9789E+02	1.5035E+02	4.70E+06	4.97E+03	9.939211E+02
CRA (6.5%)	50	78.57	2.0870E+02	4.1359E+01	2.0457E+02	3.3215E+01	1.04E+06	5.00E+03	2.129274E+02
CRA (6.5%)	60	83.23	5.1479E+01	6.0685E+00	5.1120E+01	8.1929E+00	2.57E+05	5.00E+03	5.184039E+01
CRA (6.5%)	70	85.7	1.4524E+01	1.0890E+00	1.4484E+01	2.3116E+00	7.27E+04	5.00E+03	1.456550E+01
CRA (7.5%)	40	76.34	5.5592E+02	1.3129E+02	5.4020E+02	8.8475E+01	2.78E+06	5.01E+03	5.721066E+02
CRA (7.5%)	50	81.80	1.2556E+02	1.7908E+01	1.2427E+02	1.9982E+01	6.26E+05	4.99E+03	1.268530E+02
CRA (7.5%)	60	85.39	3.1279E+01	2.5139E+00	3.1177E+01	4.9780E+00	1.56E+05	4.99E+03	3.138004E+01
CRA (7.5%)	70	87.17	9.0492E+00	4.4678E-01	9.0381E+00	1.4402E+00	4.51E+04	4.99E+03	9.060215E+00
CRA (8.5%)	40	76.08	5.4937E+02	1.3216E+02	5.3324E+02	8.7433E+01	2.77E+06	5.04E+03	5.659939E+02
CRA (8.5%)	50	81.72	1.1993E+02	1.7271E+01	1.1868E+02	1.9087E+01	6.00E+05	5.00E+03	1.211938E+02
CRA (8.5%)	60	85.51	2.8759E+01	2.2514E+00	2.8671E+01	4.5770E+00	1.43E+05	4.98E+03	2.884742E+01
CRA (8.5%)	70	86.86	8.0337E+00	4.4006E-01	8.0217E+00	1.2786E+00	4.02E+04	5.00E+03	8.045826E+00
CRA (9.5%)	40	81.84	7.6101E+01	1.0802E+01	7.5330E+01	1.2111E+01	3.81E+05	5.00E+03	7.687912E+01
CRA (9.5%)	50	85.53	1.9112E+01	1.4895E+00	1.9054E+01	3.0416E+00	9.56E+04	5.00E+03	1.917012E+01
CRA (9.5%)	60	87.28	5.7954E+00	2.7502E-01	5.7889E+00	9.2234E-01	2.92E+04	5.04E+03	5.801972E+00
CRA (9.5%)	70	88.79	2.1038E+00	4.4426E-02	2.1033E+00	3.3482E-01	1.07E+04	5.09E+03	2.104280E+00
Neat Bitumen	40	79.13	6.9572E+02	1.3120E+02	6.8323E+02	1.1072E+02	3.50E+06	5.03E+03	7.084287E+02
Neat Bitumen	50	84.02	1.2029E+02	1.2532E+01	1.1964E+02	1.9145E+01	6.03E+05	5.01E+03	1.209529E+02
Neat Bitumen	60	86.73	2.5986E+01	1.4823E+00	2.5944E+01	4.1357E+00	1.30E+05	5.00E+03	2.602868E+01
Neat Bitumen	70	86.5	6.6190E+00	4.0408E-01	6.6066E+00	1.0534E+00	3.31E+04	5.00E+03	6.631348E+00
Aged RAP	40	58.81	3.8786E+05	2.0086E+05	3.3179E+05	6.1727E+04	1.91E+03	4.94E-03	4.533908E+05
Aged RAP	50	69.78	8.3438E+04	2.8838E+04	7.8296E+04	1.3279E+04	4.19E+02	5.02E-03	8.891733E+04
Aged RAP	60	77.02	2.0299E+04	4.5595E+03	1.9781E+04	3.2307E+03	1.01E+02	4.99E-03	2.083176E+04
Aged RAP	70	82.66	5.3386E+03	6.8204E+02	5.2949E+03	8.4964E+02	2.67E+01	5.01E-03	5.382712E+03

Table A.12 Rheological test results of CRA rejuvenated samples at different temperatures for 10 Hz frequency

Frequency	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
(Hz)	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
CRA (5.5%)	40	59.94	6.3378E+03	3.1746E+03	5.4854E+03	1.0087E+02	3.19E+07	5.03E+03	7.322670E+03
CRA (5.5%)	50	70.19	1.8207E+03	6.1702E+02	1.7129E+03	2.8976E+01	9.13E+06	5.01E+03	1.935176E+03
CRA (5.5%)	60	76.75	4.8337E+02	1.1079E+02	4.7050E+02	7.6928E+00	2.43E+06	5.02E+03	4.965888E+02
CRA (5.5%)	70	81.38	1.4075E+02	2.1096E+01	1.3916E+02	2.2401E+00	7.12E+05	5.06E+03	1.423613E+02
CRA (6.5%)	40	61.8	5.3880E+03	2.5461E+03	4.7485E+03	8.5750E+01	2.72E+07	5.04E+03	6.113674E+03
CRA (6.5%)	50	71.52	1.4261E+03	4.5203E+02	1.3525E+03	2.2696E+01	7.15E+06	5.02E+03	1.503601E+03
CRA (6.5%)	60	77.5	4.0209E+02	8.7029E+01	3.9256E+02	6.3993E+00	1.93E+06	4.81E+03	4.118545E+02
CRA (6.5%)	70	81.63	1.2488E+02	1.8178E+01	1.2355E+02	1.9874E+00	6.24E+05	5.00E+03	1.262229E+02
CRA (7.5%)	40	67.33	3.6100E+03	1.3914E+03	3.3311E+03	5.7454E+01	1.82E+07	5.03E+03	3.912300E+03
CRA (7.5%)	50	75.63	9.4186E+02	2.3375E+02	9.1239E+02	1.4990E+01	4.71E+06	5.00E+03	9.722812E+02
CRA (7.5%)	60	81.77	2.5979E+02	3.7188E+01	2.5711E+02	4.1346E+00	1.28E+06	4.91E+03	2.624937E+02
CRA (7.5%)	70	84.01	8.2508E+01	8.6101E+00	8.2057E+01	1.3131E+00	4.14E+05	5.01E+03	8.296078E+01
CRA (8.5%)	40	67.25	3.5326E+03	1.3661E+03	3.2577E+03	5.6221E+01	1.80E+07	5.10E+03	3.830569E+03
CRA (8.5%)	50	75.79	9.0289E+02	2.2164E+02	8.7526E+02	1.4369E+01	4.62E+06	5.12E+03	9.313858E+02
CRA (8.5%)	60	80.64	2.4018E+02	3.9063E+01	2.3699E+02	3.8225E+00	1.20E+06	5.01E+03	2.434253E+02
CRA (8.5%)	70	83.94	7.3656E+01	7.7758E+00	7.3244E+01	1.1722E+00	3.70E+05	5.03E+03	7.406973E+01
CRA (9.5%)	40	73.51	6.0179E+02	1.7082E+02	5.7704E+02	9.5775E+00	2.88E+06	4.78E+03	6.276043E+02
CRA (9.5%)	50	80.46	1.5975E+02	2.6476E+01	1.5754E+02	2.5424E+00	8.00E+05	5.01E+03	1.619885E+02
CRA (9.5%)	60	83.84	5.4254E+01	5.8217E+00	5.3941E+01	8.6345E-01	3.94E+05	7.26E+03	5.456897E+01
CRA (9.5%)	70	86.37	2.6502E+01	1.6779E+00	2.6449E+01	4.2178E-01	1.81E+05	6.84E+03	2.655562E+01
Neat Bitumen	40	73.39	5.0147E+03	1.4335E+03	4.8054E+03	7.9809E+01	3.67E+05	7.32E+01	5.233050E+03
Neat Bitumen	50	78.95	9.8491E+02	1.8877E+02	9.6665E+02	1.5675E+01	5.29E+06	5.37E+03	1.003518E+03
Neat Bitumen	60	83.29	2.2902E+02	2.6760E+01	2.2745E+02	3.6449E+00	1.15E+06	5.04E+03	2.306006E+02
Neat Bitumen	70	85.69	6.2780E+01	4.7181E+00	6.2603E+01	9.9915E-01	3.13E+05	4.98E+03	6.295817E+01
Aged RAP	40	42.35	1.4806E+06	1.0942E+06	9.9739E+05	2.3563E+04	1.17E+04	7.91E-03	2.197786E+06
Aged RAP	50	60.42	4.5058E+05	2.2242E+05	3.9185E+05	7.1709E+03	2.23E+03	4.95E-03	5.181039E+05
Aged RAP	60	69.52	1.3184E+05	4.6128E+04	1.2351E+05	2.0982E+03	6.33E+02	4.80E-03	1.407351E+05
Aged RAP	70	75.95	4.0551E+04	9.8445E+03	3.9338E+04	6.4537E+02	2.04E+02	5.02E-03	4.180136E+04

Table A.13 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 0.01 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(Delta)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (5%)	40	81.59	2.7249E+03	3.9854E+02	2.6956E+03	4.3368E+04	1.34E+01	4.90E-03	2.754568E+03
WEO (5%)	50	86.61	3.8458E+02	2.2741E+01	3.8391E+02	6.1206E+03	1.89E+00	4.91E-03	3.852553E+02
WEO (5%)	60	88.22	7.2078E+01	2.2389E+00	7.2044E+01	1.1471E+03	3.52E-01	4.88E-03	7.211324E+01
WEO (5%)	70	88.08	1.7433E+01	5.8409E-01	1.7424E+01	2.7745E+02	8.61E-02	4.94E-03	1.744315E+01
WVO (5%)	40	83.95	1.7218E+03	1.8147E+02	1.7122E+03	2.7403E+04	8.83E+00	5.13E-03	1.731475E+03
WVO (5%)	50	87.72	2.8354E+02	1.1280E+01	2.8332E+02	4.5126E+03	1.41E+00	4.98E-03	2.837666E+02
WVO (5%)	60	84.42	1.2481E+02	1.2136E+01	1.2422E+02	1.9863E+03	5.33E-01	4.27E-03	1.254018E+02
WVO (5%)	70	83.05	1.6813E+01	2.0344E+00	1.6689E+01	2.6757E+02	8.16E-02	4.86E-03	1.693702E+01
CRA (7.5%)	40	87.06	8.1733E+00	4.1921E-01	8.1626E+00	1.3008E+02	4.03E+04	4.92E+03	8.184087E+00
CRA (7.5%)	50	88.08	1.4637E+00	4.9039E-02	1.4629E+00	2.3295E+01	7.13E+03	4.87E+03	1.464509E+00
CRA (7.5%)	60	87.82	3.2331E-01	1.2299E-02	3.2308E-01	5.1456E+00	1.65E+03	5.12E+03	3.235489E-01
CRA (7.5%)	70	85.01	8.6432E-02	7.5180E-03	8.6104E-02	1.3756E+00	4.51E+02	5.22E+03	8.676070E-02
Neat Bitumen	40	87.62	8.6933E+00	3.6100E-01	8.6858E+00	1.3835E+02	4.47E+04	5.14E+03	8.700786E+00
Neat Bitumen	50	89.58	1.3208E+00	9.6815E-03	1.3207E+00	2.1020E+01	5.74E+03	4.35E+03	1.320788E+00
Neat Bitumen	60	86.75	2.4831E-01	1.4077E-02	2.4791E-01	3.9519E+00	1.26E+03	5.08E+03	2.487119E-01
Neat Bitumen	70	85.76	8.3114E-02	4.3105E-02	7.1063E-02	1.3228E+00	3.38E+02	4.07E+03	9.720951E-02
Aged RAP	40	76.97	1.1567E+04	2.6079E+03	1.1269E+04	1.8409E+05	5.61E+01	4.85E-03	1.187284E+04
Aged RAP	50	84.1	1.5810E+03	1.6252E+02	1.5727E+03	2.5162E+04	7.92E+00	5.01E-03	1.589458E+03
Aged RAP	60	87.14	2.7513E+02	1.3728E+01	2.7478E+02	4.3786E+03	1.38E+00	5.02E-03	2.754698E+02
Aged RAP	70	89.23	5.7409E+01	7.7150E-01	5.7404E+01	9.1367E+02	2.93E-01	5.10E-03	5.741453E+01

Table A.14 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 0.1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pa.s)	(Pa)	()	(Pa)
WEO (5%)	40	73.72	2.0032E+04	5.6157E+03	1.9229E+04	3.1882E+04	9.92E+01	4.95E-03	2.086919E+04
WEO (5%)	50	81.64	3.3993E+03	4.9423E+02	3.3632E+03	5.4100E+03	1.69E+01	4.96E-03	3.435822E+03
WEO (5%)	60	86.12	7.0068E+02	4.7413E+01	6.9907E+02	1.1151E+03	3.52E+00	5.02E-03	7.022870E+02
WEO (5%)	70	88.53	1.7777E+02	4.5604E+00	1.7771E+02	2.8292E+02	8.61E-01	4.84E-03	1.778284E+02
WVO (5%)	40	76.5	1.4273E+04	3.3319E+03	1.3879E+04	2.2715E+04	7.11E+01	4.98E-03	1.467849E+04
WVO (5%)	50	83.29	2.6131E+03	3.0532E+02	2.5952E+03	4.1587E+03	1.30E+01	4.99E-03	2.631076E+03
WVO (5%)	60	85.71	1.8039E+03	1.3494E+02	1.7988E+03	9.7086E+02	9.70E+00	5.38E-03	6.089396E+02
WVO (5%)	70	86.86	1.5409E+02	8.4402E+00	1.5385E+02	2.4523E+02	7.72E-01	5.01E-03	1.543180E+02
CRA (7.5%)	40	82.75	7.2874E+01	9.1966E+00	7.2291E+01	1.1598E+02	3.62E+05	4.97E+03	7.346129E+01
CRA (7.5%)	50	86.34	1.4270E+01	9.1093E-01	1.4241E+01	2.2711E+01	7.13E+04	5.00E+03	1.429907E+01
CRA (7.5%)	60	87.81	3.2850E+00	1.2553E-01	3.2826E+00	5.2281E+00	1.65E+04	5.04E+03	3.287430E+00
CRA (7.5%)	70	87.35	9.1849E-01	4.2466E-02	9.1750E-01	1.4618E+00	4.51E+03	4.91E+03	9.194696E-01
Neat Bitumen	40	84.27	8.3935E+01	8.3801E+00	8.3516E+01	1.3358E+02	4.23E+05	5.04E+03	8.435660E+01
Neat Bitumen	50	87.34	1.3443E+01	6.2386E-01	1.3428E+01	2.1394E+01	6.66E+04	4.95E+03	1.345719E+01
Neat Bitumen	60	88.63	2.6696E+00	6.3827E-02	2.6689E+00	4.2487E+00	1.30E+04	4.87E+03	2.670383E+00
Neat Bitumen	70	87.55	7.1212E-01	9.2335E-02	7.0611E-01	1.1333E+00	3.56E+03	5.00E+03	7.181867E-01
Aged RAP	40	68.44	7.4409E+04	2.7343E+04	6.9202E+04	1.1842E+05	3.67E+02	4.93E-03	8.000639E+04
Aged RAP	50	77.76	1.2623E+04	2.6762E+03	1.2336E+04	2.0090E+04	6.33E+01	5.01E-03	1.291670E+04
Aged RAP	60	83.16	2.5721E+03	3.0633E+02	2.5538E+03	4.0935E+03	1.28E+01	4.97E-03	2.590554E+03
Aged RAP	70	87.29	5.8670E+02	2.7739E+01	5.8604E+02	9.3373E+02	2.93E+00	4.99E-03	5.873520E+02

Table A.15 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pa.s)	(Pa)	()	(Pa)
WEO (5%)	40	65.63	1.1821E+05	4.8776E+04	1.0768E+05	1.8813E+04	5.96E+02	5.04E-03	1.297707E+05
WEO (5%)	50	74.23	2.4988E+04	6.7911E+03	2.4047E+04	3.9768E+03	1.25E+02	5.01E-03	2.596513E+04
WEO (5%)	60	80.8	6.1590E+03	9.8470E+02	6.0797E+03	9.8020E+02	3.07E+01	4.99E-03	6.239228E+03
WEO (5%)	70	85.17	1.7149E+03	1.4439E+02	1.7088E+03	2.7292E+02	8.61E+00	5.02E-03	1.720978E+03
WVO (5%)	40	68.76	9.1056E+04	3.2987E+04	8.4870E+04	1.4492E+04	4.58E+02	5.03E-03	9.769170E+04
WVO (5%)	50	76.42	2.0602E+04	4.8373E+03	2.0026E+04	3.2788E+03	1.02E+02	4.96E-03	2.119424E+04
WVO (5%)	60	84.48	9.2479E+03	8.8959E+02	9.2050E+03	1.4718E+03	4.65E+01	5.03E-03	9.291019E+03
WVO (5%)	70	85.32	1.4586E+03	1.1901E+02	1.4538E+03	2.3214E+02	7.28E+00	4.99E-03	1.463509E+03
CRA (7.5%)	40	76.34	5.5592E+02	1.3129E+02	5.4020E+02	8.8475E+01	2.78E+06	5.01E+03	5.721066E+02
CRA (7.5%)	50	81.80	1.2556E+02	1.7908E+01	1.2427E+02	1.9982E+01	6.26E+05	4.99E+03	1.268530E+02
CRA (7.5%)	60	85.39	3.1279E+01	2.5139E+00	3.1177E+01	4.9780E+00	1.56E+05	4.99E+03	3.138004E+01
CRA (7.5%)	70	87.17	9.0492E+00	4.4678E-01	9.0381E+00	1.4402E+00	4.51E+04	4.99E+03	9.060215E+00
Neat Bitumen	40	79.13	6.9572E+02	1.3120E+02	6.8323E+02	1.1072E+02	3.50E+06	5.03E+03	7.084287E+02
Neat Bitumen	50	84.02	1.2029E+02	1.2532E+01	1.1964E+02	1.9145E+01	6.03E+05	5.01E+03	1.209529E+02
Neat Bitumen	60	86.73	2.5986E+01	1.4823E+00	2.5944E+01	4.1357E+00	1.30E+05	5.00E+03	2.602868E+01
Neat Bitumen	70	86.50	6.6190E+00	4.0408E-01	6.6066E+00	1.0534E+00	3.31E+04	5.00E+03	6.631348E+00
Aged RAP	40	58.81	3.8786E+05	2.0086E+05	3.3179E+05	6.1727E+04	1.91E+03	4.94E-03	4.533908E+05
Aged RAP	50	69.78	8.3438E+04	2.8838E+04	7.8296E+04	1.3279E+04	4.19E+02	5.02E-03	8.891733E+04
Aged RAP	60	77.02	2.0299E+04	4.5595E+03	1.9781E+04	3.2307E+03	1.01E+02	4.99E-03	2.083176E+04
Aged RAP	70	82.66	5.3386E+03	6.8204E+02	5.2949E+03	8.4964E+02	2.67E+01	5.01E-03	5.382712E+03

Table A.16 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 10 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (5%)	40	55.5	5.7024E+05	3.2299E+05	4.6995E+05	9.0754E+03	2.85E+03	5.00E-03	6.919334E+05
WEO (5%)	50	66.51	1.5058E+05	6.0020E+04	1.3810E+05	2.3965E+03	7.53E+02	5.00E-03	1.641888E+05
WEO (5%)	60	73.71	4.4031E+04	1.2351E+04	4.2264E+04	7.0076E+02	2.38E+02	5.41E-03	4.587294E+04
WEO (5%)	70	79.73	1.4150E+04	2.5228E+03	1.3924E+04	2.2521E+02	7.07E+01	4.99E-03	1.438090E+04
WVO (5%)	40	62.05	4.4562E+05	2.0886E+05	3.9364E+05	7.0920E+03	2.26E+03	5.07E-03	5.044598E+05
WVO (5%)	50	71.84	1.1831E+05	3.6874E+04	1.1242E+05	1.8829E+03	5.93E+02	5.02E-03	1.245117E+05
WVO (5%)	60	77.94	3.3648E+04	7.0302E+03	3.2905E+04	5.3550E+02	1.68E+02	5.00E-03	3.440706E+04
WVO (5%)	70	81.69	1.0217E+04	1.4766E+03	1.0109E+04	1.6260E+02	5.11E+01	5.00E-03	1.032496E+04
CRA (7.5%)	40	67.33	3.6100E+03	1.3914E+03	3.3311E+03	5.7454E+01	1.82E+07	5.03E+03	3.912300E+03
CRA (7.5%)	50	75.63	9.4186E+02	2.3375E+02	9.1239E+02	1.4990E+01	4.71E+06	5.00E+03	9.722812E+02
CRA (7.5%)	60	81.77	2.5979E+02	3.7188E+01	2.5711E+02	4.1346E+00	1.28E+06	4.91E+03	2.624937E+02
CRA (7.5%)	70	84.01	8.2508E+01	8.6101E+00	8.2057E+01	1.3131E+00	4.14E+05	5.01E+03	8.296078E+01
Neat Bitumen	40	73.39	5.0147E+03	1.4335E+03	4.8054E+03	7.9809E+01	3.67E+05	7.32E+01	5.233050E+03
Neat Bitumen	50	78.95	9.8491E+02	1.8877E+02	9.6665E+02	1.5675E+01	5.29E+06	5.37E+03	1.003518E+03
Neat Bitumen	60	83.29	2.2902E+02	2.6760E+01	2.2745E+02	3.6449E+00	1.15E+06	5.04E+03	2.306006E+02
Neat Bitumen	70	85.69	6.2780E+01	4.7181E+00	6.2603E+01	9.9915E-01	3.13E+05	4.98E+03	6.295817E+01
Aged RAP	40	42.35	1.4806E+06	1.0942E+06	9.9739E+05	2.3563E+04	1.17E+04	7.91E-03	2.197786E+06
Aged RAP	50	60.42	4.5058E+05	2.2242E+05	3.9185E+05	7.1709E+03	2.23E+03	4.95E-03	5.181039E+05
Aged RAP	60	69.52	1.3184E+05	4.6128E+04	1.2351E+05	2.0982E+03	6.33E+02	4.80E-03	1.407351E+05
Aged RAP	70	75.95	4.0551E+04	9.8445E+03	3.9338E+04	6.4537E+02	2.04E+02	5.02E-03	4.180136E+04

Table A.17 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 0.01 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (5%) RTFO	40	83.58	7.8612E+03	8.7901E+02	7.8119E+03	1.2511E+05	7.89E+01	1.00E-02	7.910847E+03
WEO (5%) RTFO	50	87.29	1.2343E+03	5.8358E+01	1.2329E+03	1.9644E+04	1.10E+01	8.88E-03	1.235664E+03
WEO (5%) RTFO	60	87.99	2.2016E+02	7.7219E+00	2.2003E+02	3.5039E+03	1.92E+00	8.71E-03	2.202965E+02
WEO (5%) RTFO	70	88.84	4.3946E+01	8.8967E-01	4.3937E+01	6.9941E+02	3.83E-01	8.71E-03	4.395544E+01
WVO (5%) RTFO	40	82.24	5.3729E+03	7.2547E+02	5.3237E+03	8.5510E+04	3.54E+01	6.59E-03	5.422584E+03
WVO (5%) RTFO	50	86.29	8.7524E+02	5.6634E+01	8.7341E+02	1.3929E+04	4.79E+00	5.47E-03	8.770807E+02
WVO (5%) RTFO	60	87.38	1.5838E+02	7.2399E+00	1.5822E+02	2.5206E+03	8.62E-01	5.44E-03	1.585469E+02
WVO (5%) RTFO	70	88.79	3.2507E+01	6.8645E-01	3.2500E+01	5.1735E+02	1.72E-01	5.28E-03	3.251436E+01
CRA (7.5%) RTFO	40	88.33	3.1473E+03	9.1723E+01	3.1460E+03	5.0090E+04	2.14E+01	6.79E-03	3.148677E+03
CRA (7.5%) RTFO	50	88.28	5.3950E+02	1.6193E+01	5.3926E+02	8.5862E+03	2.85E+00	5.29E-03	5.397452E+02
CRA (7.5%) RTFO	60	87.84	1.0051E+02	3.7883E+00	1.0044E+02	1.5996E+03	5.33E-01	5.30E-03	1.005822E+02
CRA (7.5%) RTFO	70	88.84	2.0406E+01	4.1310E-01	2.0401E+01	3.2476E+02	1.09E-01	5.35E-03	2.040982E+01
Neat Bitumen RTFO	40	87.51	9.0319E+02	3.9239E+01	9.0234E+02	1.4374E+04	6.07E+00	6.72E-03	9.040480E+02
Neat Bitumen RTFO	50	88.49	1.5440E+02	4.0686E+00	1.5434E+02	2.4572E+03	7.58E-01	4.91E-03	1.544498E+02
Neat Bitumen RTFO	60	87.63	3.7657E+01	1.5572E+00	3.7625E+01	5.9932E+02	1.83E-01	4.85E-03	3.768963E+01
Neat Bitumen RTFO	70	88.35	8.1651E+00	2.3511E-01	8.1617E+00	1.2995E+02	4.26E-02	5.22E-03	8.168473E+00
Aged RAP	40	76.97	1.1567E+04	2.6079E+03	1.1269E+04	1.8409E+05	5.61E+01	4.85E-03	1.187284E+04
Aged RAP	50	84.1	1.5810E+03	1.6252E+02	1.5727E+03	2.5162E+04	7.92E+00	5.01E-03	1.589458E+03
Aged RAP	60	87.14	2.7513E+02	1.3728E+01	2.7478E+02	4.3786E+03	1.38E+00	5.02E-03	2.754698E+02
Aged RAP	70	89.23	5.7409E+01	7.7150E-01	5.7404E+01	9.1367E+02	2.93E-01	5.10E-03	5.741453E+01

Table A.18 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 0.1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (5%) RTFO	40	77.63	5.8716E+04	1.2578E+04	5.7353E+04	9.3446E+04	5.28E+02	9.00E-03	6.011132E+04
WEO (5%) RTFO	50	83.84	9.4710E+03	1.0163E+03	9.4163E+03	1.5073E+04	8.67E+01	9.16E-03	9.526029E+03
WEO (5%) RTFO	60	85.78	1.8831E+03	1.3857E+02	1.8780E+03	2.9969E+03	1.72E+01	9.13E-03	1.888190E+03
WEO (5%) RTFO	70	87.39	4.8848E+02	2.2244E+01	4.8798E+02	7.7742E+02	4.14E+00	8.47E-03	4.889909E+02
WVO (5%) RTFO	40	77.53	4.0052E+04	8.6484E+03	3.9107E+04	6.3743E+04	2.21E+02	5.51E-03	4.101979E+04
WVO (5%) RTFO	50	82.66	7.0863E+03	9.0533E+02	7.0283E+03	1.1278E+04	3.91E+01	5.52E-03	7.144874E+03
WVO (5%) RTFO	60	85.43	1.4821E+03	1.1809E+02	1.4774E+03	2.3588E+03	8.06E+00	5.44E-03	1.486845E+03
WVO (5%) RTFO	70	87.35	3.3886E+02	1.5667E+01	3.3850E+02	5.3930E+02	1.79E+00	5.28E-03	3.392240E+02
CRA (7.5%) RTFO	40	83.78	2.3881E+04	2.5874E+03	2.3740E+04	3.8007E+04	1.26E+02	5.26E-03	2.402240E+04
CRA (7.5%) RTFO	50	87.17	4.4867E+03	2.2152E+02	4.4812E+03	7.1406E+03	2.40E+01	5.35E-03	4.492200E+03
CRA (7.5%) RTFO	60	87.98	9.5718E+02	3.3739E+01	9.5658E+02	1.5233E+03	5.04E+00	5.27E-03	9.577713E+02
CRA (7.5%) RTFO	70	87.98	2.1537E+02	7.5913E+00	2.1523E+02	3.4276E+02	1.13E+00	5.25E-03	2.155008E+02
Neat Bitumen RTFO	40	86.63	4.8511E+03	2.8517E+02	4.8427E+03	7.7205E+03	2.33E+01	4.81E-03	4.859499E+03
Neat Bitumen RTFO	50	87.56	1.5185E+03	6.4648E+01	1.5171E+03	2.4167E+03	7.57E+00	4.98E-03	1.519890E+03
Neat Bitumen RTFO	60	87.70	3.7375E+02	1.4999E+01	3.7345E+02	5.9483E+02	1.83E+00	4.89E-03	3.740547E+02
Neat Bitumen RTFO	70	87.42	8.6812E+01	3.9078E+00	8.6724E+01	1.3816E+02	4.26E-01	4.91E-03	8.690047E+01
Aged RAP	40	68.44	7.4409E+04	2.7343E+04	6.9202E+04	1.1842E+05	3.67E+02	4.93E-03	8.000639E+04
Aged RAP	50	77.76	1.2623E+04	2.6762E+03	1.2336E+04	2.0090E+04	6.33E+01	5.01E-03	1.291670E+04
Aged RAP	60	83.16	2.5721E+03	3.0633E+02	2.5538E+03	4.0935E+03	1.28E+01	4.97E-03	2.590554E+03
Aged RAP	70	87.29	5.8670E+02	2.7739E+01	5.8604E+02	9.3373E+02	2.93E+00	4.99E-03	5.873520E+02

Table A.19 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 1 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (5%) RTFO	40	72.26	2.7946E+05	8.5151E+04	2.6617E+05	4.4476E+04	2.64E+03	9.44E-03	2.934126E+05
WEO (5%) RTFO	50	77.49	6.6443E+04	1.4392E+04	6.4866E+04	1.0574E+04	5.98E+02	9.00E-03	6.805906E+04
WEO (5%) RTFO	60	82.07	1.6339E+04	2.2542E+03	1.6183E+04	2.6004E+03	1.48E+02	9.05E-03	1.649715E+04
WEO (5%) RTFO	70	85.39	4.1286E+03	3.3183E+02	4.1153E+03	6.5707E+02	3.89E+01	9.43E-03	4.142016E+03
WVO (5%) RTFO	40	71.18	2.1250E+05	6.8551E+04	2.0114E+05	3.3819E+04	1.20E+03	5.64E-03	2.244989E+05
WVO (5%) RTFO	50	77.03	4.7165E+04	1.0586E+04	4.5962E+04	7.5063E+03	2.69E+02	5.70E-03	4.840000E+04
WVO (5%) RTFO	60	81.65	1.1961E+04	1.7369E+03	1.1834E+04	1.9036E+03	6.59E+01	5.51E-03	1.208897E+04
WVO (5%) RTFO	70	85.11	3.0868E+03	2.6313E+02	3.0756E+03	4.9126E+02	1.69E+01	5.47E-03	3.098077E+03
CRA (7.5%) RTFO	40	76.78	1.2974E+05	2.9671E+04	1.2630E+05	2.0648E+04	7.10E+02	5.47E-03	1.332727E+05
CRA (7.5%) RTFO	50	82.45	3.1984E+04	4.2024E+03	3.1706E+04	5.0902E+03	1.74E+02	5.45E-03	3.226322E+04
CRA (7.5%) RTFO	60	86.14	8.0042E+03	5.3883E+02	7.9861E+03	1.2739E+03	4.29E+01	5.36E-03	8.022412E+03
CRA (7.5%) RTFO	70	87.63	2.0129E+03	8.3238E+01	2.0112E+03	3.2035E+02	1.08E+01	5.35E-03	2.014621E+03
Neat Bitumen RTFO	40	83.56	3.8597E+04	4.3292E+03	3.8354E+04	6.1428E+03	1.92E+02	4.98E-03	3.884242E+04
Neat Bitumen RTFO	50	84.28	1.3939E+04	1.3892E+03	1.3869E+04	2.2184E+03	6.94E+01	4.98E-03	1.400850E+04
Neat Bitumen RTFO	60	86.28	3.6511E+03	2.3689E+02	3.6434E+03	5.8108E+02	1.83E+01	5.01E-03	3.658833E+03
Neat Bitumen RTFO	70	87.56	8.4637E+02	3.6033E+01	8.4560E+02	1.3470E+02	4.26E+00	5.03E-03	8.471376E+02
Aged RAP	40	58.81	3.8786E+05	2.0086E+05	3.3179E+05	6.1727E+04	1.91E+03	4.94E-03	4.533908E+05
Aged RAP	50	69.78	8.3438E+04	2.8838E+04	7.8296E+04	1.3279E+04	4.19E+02	5.02E-03	8.891733E+04
Aged RAP	60	77.02	2.0299E+04	4.5595E+03	1.9781E+04	3.2307E+03	1.01E+02	4.99E-03	2.083176E+04
Aged RAP	70	82.66	5.3386E+03	6.8204E+02	5.2949E+03	8.4964E+02	2.67E+01	5.01E-03	5.382712E+03

Table A.20 Rheological test results of rejuvenated samples with optimum contents at different temperatures for 10 Hz frequency

	Temperature	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(δ)
Bitumen Sample	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
WEO (5%) RTFO	40	72.26	2.7946E+05	8.5151E+04	2.6617E+05	4.4476E+04	2.64E+03	9.44E-03	2.934126E+05
WEO (5%) RTFO	50	77.49	6.6443E+04	1.4392E+04	6.4866E+04	1.0574E+04	5.98E+02	9.00E-03	6.805906E+04
WEO (5%) RTFO	60	82.07	1.6339E+04	2.2542E+03	1.6183E+04	2.6004E+03	1.48E+02	9.05E-03	1.649715E+04
WEO (5%) RTFO	70	85.39	4.1286E+03	3.3183E+02	4.1153E+03	6.5707E+02	3.89E+01	9.43E-03	4.142016E+03
WVO (5%) RTFO	40	71.18	2.1250E+05	6.8551E+04	2.0114E+05	3.3819E+04	1.20E+03	5.64E-03	2.244989E+05
WVO (5%) RTFO	50	77.03	4.7165E+04	1.0586E+04	4.5962E+04	7.5063E+03	2.69E+02	5.70E-03	4.840000E+04
WVO (5%) RTFO	60	81.65	1.1961E+04	1.7369E+03	1.1834E+04	1.9036E+03	6.59E+01	5.51E-03	1.208897E+04
WVO (5%) RTFO	70	85.11	3.0868E+03	2.6313E+02	3.0756E+03	4.9126E+02	1.69E+01	5.47E-03	3.098077E+03
CRA (7.5%) RTFO	40	76.78	1.2974E+05	2.9671E+04	1.2630E+05	2.0648E+04	7.10E+02	5.47E-03	1.332727E+05
CRA (7.5%) RTFO	50	82.45	3.1984E+04	4.2024E+03	3.1706E+04	5.0902E+03	1.74E+02	5.45E-03	3.226322E+04
CRA (7.5%) RTFO	60	86.14	8.0042E+03	5.3883E+02	7.9861E+03	1.2739E+03	4.29E+01	5.36E-03	8.022412E+03
CRA (7.5%) RTFO	70	87.63	2.0129E+03	8.3238E+01	2.0112E+03	3.2035E+02	1.08E+01	5.35E-03	2.014621E+03
Neat Bitumen RTFO	40	83.56	3.8597E+04	4.3292E+03	3.8354E+04	6.1428E+03	1.92E+02	4.98E-03	3.884242E+04
Neat Bitumen RTFO	50	84.28	1.3939E+04	1.3892E+03	1.3869E+04	2.2184E+03	6.94E+01	4.98E-03	1.400850E+04
Neat Bitumen RTFO	60	86.28	3.6511E+03	2.3689E+02	3.6434E+03	5.8108E+02	1.83E+01	5.01E-03	3.658833E+03
Neat Bitumen RTFO	70	87.56	8.4637E+02	3.6033E+01	8.4560E+02	1.3470E+02	4.26E+00	5.03E-03	8.471376E+02
Aged RAP	40	58.81	3.8786E+05	2.0086E+05	3.3179E+05	6.1727E+04	1.91E+03	4.94E-03	4.533908E+05
Aged RAP	50	69.78	8.3438E+04	2.8838E+04	7.8296E+04	1.3279E+04	4.19E+02	5.02E-03	8.891733E+04
Aged RAP	60	77.02	2.0299E+04	4.5595E+03	1.9781E+04	3.2307E+03	1.01E+02	4.99E-03	2.083176E+04
Aged RAP	70	82.66	5.3386E+03	6.8204E+02	5.2949E+03	8.4964E+02	2.67E+01	5.01E-03	5.382712E+03

Table A.21 PG test results for neat, aged RAP and rejuvenated bitumen samples

	Temp.	Phase Angle	Complex Modulus	Elastic Modulus	Viscous Modulus	Complex Viscosity	Shear Stress	Strain	G*/Sin(Delta)
	(°C)	(°)	(Pa)	(Pa)	(Pa)	(Pas)	(Pa)	()	(Pa)
Neat	52	86.21	7.4045E+03	4.8943E+02	7.3883E+03	7.4045E+02	8.9172E+02	1.204298E-01	7.420709E+03
	58	87.49	3.2079E+03	1.4049E+02	3.2048E+03	3.2079E+02	3.8447E+02	1.198510E-01	3.210980E+03
	64	88.42	1.4294E+03	3.9413E+01	1.4289E+03	1.4294E+02	1.7149E+02	1.199719E-01	1.429961E+03
	70	88.91	7.0361E+02	1.3385E+01	7.0348E+02	7.0361E+01	8.4250E+01	1.197395E-01	7.037381E+02
Aged RAP	52	73.59	3.8119E+04	1.0769E+04	3.6567E+04	3.8119E+03	4.6742E+03	1.226199E-01	3.973817E+04
	58	77.4	1.6257E+04	3.5464E+03	1.5866E+04	1.6257E+03	1.9712E+03	1.212492E-01	1.665862E+04
	64	80.63	7.1510E+03	1.1643E+03	7.0556E+03	7.1510E+02	8.6480E+02	1.209334E-01	7.247746E+03
	70	83.07	3.2671E+03	3.9420E+02	3.2433E+03	3.2671E+02	3.9562E+02	1.210909E-01	3.291177E+03
	76	84.57	1.6501E+03	1.5614E+02	1.6426E+03	1.6501E+02	1.9633E+02	1.189840E-01	1.657492E+03
	82	85.21	9.5255E+02	7.9541E+01	9.4922E+02	9.5255E+01	1.0967E+02	1.151336E-01	9.558840E+02
5.4% WEO	52	75.01	1.8625E+04	4.8173E+03	1.7991E+04	1.8625E+03	2.2220E+03	1.193047E-01	1.92807E+04
	58	78.31	9.0595E+03	1.8356E+03	8.8716E+03	9.0595E+02	1.0890E+03	1.202047E-01	9.25144E+03
	64	81.16	4.3472E+03	6.6806E+02	4.2956E+03	4.3472E+02	5.2177E+02	1.200237E-01	4.39948E+03
	70	83.37	2.0405E+03	2.3559E+02	2.0268E+03	2.0405E+02	2.4626E+02	1.206882E-01	2.05420E+03
	76	84.59	1.0220E+03	9.6357E+01	1.0175E+03	1.0220E+02	1.2280E+02	1.201555E-01	1.02658E+03
	82	84.95	5.0895E+02	4.4801E+01	5.0698E+02	5.0895E+01	6.1833E+01	1.214902E-01	5.10938E+02
5.1% WVO	52	74.38	1.7217E+04	4.6357E+03	1.6581E+04	1.7217E+03	2.0360E+03	1.182578E-01	1.78768E+04
	58	77.56	8.0853E+03	1.7417E+03	7.8955E+03	8.0853E+02	9.8200E+02	1.214546E-01	8.27972E+03
	64	80.18	3.9968E+03	6.8167E+02	3.9383E+03	3.9968E+02	4.8500E+02	1.213459E-01	4.05627E+03
	70	83.07	1.7710E+03	2.1369E+02	1.7581E+03	1.7710E+02	2.1429E+02	1.209982E-01	1.78405E+03
	76	84.28	9.3699E+02	9.3387E+01	9.3233E+02	9.3699E+01	1.1284E+02	1.204279E-01	9.41681E+02
	82	84.77	4.6728E+02	4.2594E+01	4.6534E+02	4.6728E+01	5.6770E+01	1.214902E-01	4.69234E+02
7.5% CRA	52	85.94	8.1779E+03	5.7900E+02	8.1574E+03	8.1779E+02	9.8486E+02	1.204298E-01	8.198451E+03
	58	86.26	3.9291E+03	2.5629E+02	3.9208E+03	3.9291E+02	4.7091E+02	1.198510E-01	3.937514E+03
	64	87.38	1.7586E+03	8.0388E+01	1.7567E+03	1.7586E+02	2.1098E+02	1.199719E-01	1.760418E+03
	70	88.04	8.2078E+02	2.8072E+01	8.2030E+02	8.2078E+01	9.8280E+01	1.197395E-01	8.212623E+02