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RHEOLOGICAL EVALUATION OF BITUMENIOUS BINDER CONTAINING EGG SHELL POWDER

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A thesis submitted to the school of Research and Graduate Studies of Bahir Dar Institute of Technology, BDU in partial fulfillment of the requirements for the degree of

Master of Science in the post graduate program in

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Advisor: Habtamu Melese (PhD, PE)

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Declaration

I hereby declare that research work titled "RHEOLOGICAL EVALUATION OF BITUMENIOUS BINDER CONTAINING EGG SHELL POWDER" is my original work. The work has not been presented elsewhere for assessment and award of any degree. Statements and scientific concepts, which have been used from other sources, have been properly acknowledged.

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ABSTRACT

Engineers particularly in advanced nations have been using modified binders to improve the performance of flexible pavements. They use different modifiers to augment the rheological properties of asphalt binder. And specification is done by applying superpave binder grading system which requires performance-based binder tests. In Ethiopia while one of the major pavement distresses is rutting, there is no substantial effort to minimize this high temperature distress by using modified binder of improved visco-elastic property, in addition the binder grading system does not comprise performance-based tests. This thesis was conducted by performing a number of laboratory tests on virgin and modified asphalt by adding ESP by weight of asphalt to evaluate the rheological property of asphalt bitumen. Three percentage of egg shell powder was investigated (3%, 6% and 9%) by weight of bitumen with three samples for each percentage. For both RTFO aged and unaged asphalt binder for conventional tests addition of ESP has decrease the penetration and ductility value however when the bitumen become aged at 9% ductility values are not under the desired value which is stated in ERA 2013, whereas softening point value become increase as ESP content increases.

The rheological binder tests were conducted on both RTFO aged and unaged binders for both original and ESP modified bitumen. The LVER of all samples were determined with the help of AST test at 10°C, 21.1°C, 37.8°C, and 54.4 °C. And the limiting strain was 0.38% which was used as an input for FST. Amplitude sweep test was conducted on RTFO aged and unaged bitumen by using a Dynamic Shear Rheometer (DSR). From the frequency sweep test (FST) result, the master curve shows that addition of egg shell powder (ESP) improves the behavior of asphalt binder. Addition of 3% and 6% modified asphalt binder increases the stiffening property of asphalt binder at high temperature and low loading frequency as compared to 9% ESP modified binder for unaged asphalt binder. From the performance grade test, the result shows that improvement of performance grade from PG58-YY to PG64-YY for both unaged and RTFO aged bitumen with the additions of 3%,6% and 9% ESP. The rutting performance improved as showed from MSCR test having lower total strain, lower non-recoverable creep compliance (Jnr) and higher Percent recovery for ESP modified binders.

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ABBREVIATIONS

AASHTO= American Association State of Highway and Transportation Officials

ANOVA= Analysis of Variance

AST= Amplitude Sweep Test

ASTM= American Society of Testing and Materials

BBR=Bending Beam Rheometer

DSR= Dynamic Shear Rheometer

DTT=Direct Tension Tester

ESP= Egg Shell Powder

FST= Frequency Sweep Test

HMA=Hot Mix Asphalt

LVE=Linear Visco Elastic

MSCR=Multiple Stress Creep Recovery

PAV= Pressure aging vessel

PG=Performance Grade

PR=Percent Recovery

RTFO=Rolling Thin Film Oven

SHRP= Strategic Highway Research Program

SPSS= Statistical Package for Social Science

TFOT= Thin Film Oven Test

US= United State

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1. INTRODUCTION

1.1 Background

Asphalt bitumen is the most common form of bitumen is the residue from the refining of crude oil after the more volatile material has been distilled off. It is essentially a very viscous liquid comprising many long-chain organic molecules. For use in roads it is practically solid at ambient temperatures but can be heated sufficiently to be poured and sprayed. Some natural bitumen's can be found worldwide that are not distilled from crude oil but the amounts are very small in comparison.

Asphalt binder is defined by the American Society for Testing and Materials (ASTM) as a dark brown to black Cementous material in which the predominating constituents are bitumen that occur in nature or are obtained in petroleum processing. In the crude oil refineries, the Cementous material is in the bottom of the vacuum distillation columns. Hot mix asphalt which is composed of 95% aggregate and 5% bitumen is used as the top layer in road construction. The overall cost of HMA is ever-increasing because bitumen is a by-product of a depleting petroleum resource (Kara & Karacasu, 2015).

Binding material called asphalt binder has been widely applied in the construction of asphalt pavement structure. Its physical properties and temperature susceptibility characteristics both at high and low field operating temperature influence the performance asphalt mixture. Therefore, modification of base asphalt is necessary to improve the performance of pavement structures (Kumneger, 2018).

A few years ago, many studies towards the use of alternative materials for road construction and some of them have shown positive and encouraging results. This promoted the use of organic and inorganic materials as modifiers or additives in asphaltic pavement such as plastics, polymers, glass, oils, fibers, etc. With this, not only can solve the problem of the lack of original material in asphaltic pavement construction, but also give alternatives by recycling process (Erfen & Yunus, 2015).

Ethiopian government losses huge amount of money because of frequent maintenance and due to traffic accidents related to those pavement conditions every year. As road condition affects the economy of the country, urgent response should be given.

Several road pavement distresses are related to bitumen properties. Rutting and fatigue cracking are the major distress that lead to permanent failures in pavement construction. Bitumen is a viscoelastic material; its rheological properties are very sensitive to temperature and rate of loading Premature failures such as permanent deformation or rutting is one of the failures occur now a days at higher temperature zones. The causes of permanent deformation could be weak subgrade or because of the weak binder. The conventional binder fails to withstand the adverse effect of traffic load and environment condition, leading for search of modified binder. Aging is known as a factor influencing the performance and characteristics of bitumen binder. Many factors might contribute to this hardening of the bitumen such as oxidation, volatilization, polymerization, and thixotropy (Ali, Mashaan, & Karim, 2013). The main reasons that asphalt modification has become more accepted are the traffic factors, which have increased including heavier loads, higher volumes and higher tire pressures.

It is estimated that roughly 100 million tons of hen egg shell is generated throughout the world every year without realizing that it has many uses in other areas, perhaps most distinguishes the eggshell is fit on mineral salts such as calcium, phosphorus, manganese, molybdenum, iron, copper, chromium, fluorine, zinc. The researcher chose this material in order to investigate its effect on bitumen as long as it's having an effect on the chemically cement material. These egg-shells wastes are non- biodegradable and the majority of eggshell waste is deposited as landfills. Egg-shell waste in landfills attracts vermin due to the attached membrane in landfills and causes problems associated with human health and environment. So, this egg-shell waste is useless as a landfill material (Dixit, 2016).

In Ethiopia, chickens are wide spread and almost every rural family owns chicken. The total chicken population in the country is estimated at 38.1 million. Rural chicken in Ethiopia represents a significant part of the national economy in general and the rural economy in particular and contribute to 98.5% of the national egg (Tadelle., 1996).

The research has studied the rheological properties of asphalt mix containing egg shell powder to improve the stiffness and rutting performance of binder The egg shells was added to the asphalt binder directly with proportions of (3%, 6%, and 9%) by weight of asphalt. In the laboratory test, control group binder was tested and compared by those modified by egg shell powder.

1.2 Statement of the problem

Many of paved roads constructed in the last decades in Ethiopia are being deteriorated without attaining the expected design period due to many reasons. Some of these are quality, proper designation and characterization of materials have a significant effect on it. Thus, the type and the extent of pavement deterioration and distress simulate the cause of failure, which is important to take remedial measures (Zemichael, 2007). In Ethiopia next to gravel road flexible pavement roads are constructed in each part of nation. The surface of flexible pavement (bitumen) is high cost and spend largest budget in road construction.

Binding materials called bitumen has been extensively applied in the construction of bituminous pavement structures due to its flexibility and binding property. However, the performance depends on the stresses due to traffic loads (loading time) and temperature. Due to these limitations, the performance of asphalt needs to be enhanced. Therefore, the modification of base asphalt is necessary to improve the material's performance. Due to the increased performance related requirements on asphalt pavements, the asphalt containing conventional bitumen does not always perform as expected. Lately the more advanced asphalt binder specification method using the Multiple Stress Creep Recovery (MSCR) test has been developed. This MSCR method is becoming popular since it best predicts rutting and convenient to consider different levels of traffic. But in Ethiopia still conventional binder testing and specification system is in use. Therefore, it is necessary to bring a change towards methods of binder characterization, which will help to prepare specification of asphalt binders in accordance with performance grade and also in order to improve the asphalt properties modified bitumen has been adopted (Atnafu A., 2017). Researchers are exploring alternative materials which could replace natural bitumen in highway construction.

A few years ago, many studies towards the use of alternative material for road construction and some of them have shown helpful and encouraging results. This endorsed the use of organic and inorganic material as a modifiers or additives. In asphaltic pavement such as plastic, polymers, glass, oils, fibers, Portland cement, steel slag etc, showed to improve the performance of asphalt mixtures. With this, not only can solve the problem of the lack of original material in asphaltic pavement construction, but also give alternatively by recycling. Egg shell powder contains greatest amount of lime, lime has many complex qualities as a construction product which includes adhesive property (adhesive binders). This research has the interest to introduce the egg shell powder as a sustainable modifier to fulfill the objective of reducing rutting by improving elastic behavior and making stiffer of asphalt binders.

From the previous research, application of egg shell powder on asphalt binder as a modifier was done, the outcome of the research shows the stiffness, temperature susceptibility, strength property (marshal stability) significantly improved with the increase in additives. However, they were not done further rheological test mostly focus on marshal test.

A conventional bitumen 60/70 penetration grade is commonly used in Ethiopia for hot climate zones, and additionally, it is subjected to the high traffic load. Thus, the current increasing of traffic load and adverse effect of sever environmental condition directly affects the road performance. As a result, pavements were deteriorated and permanent deformation is the major road failure in hot area.

1.3 Objective of study

1.3.1 General objective

To study the rheological properties of asphalt binder containing egg shell powder to improve the stiffness and rutting performance of binder.

1.3.2 Specific objectives

- To study the conventional properties of the neat and modified asphalt binder i.e. penetration, softening point and ductility.
- To determine visco-elastic ranges of egg shell powder modified asphalt binder using Amplitude Sweep Test (AST).
- To analyze rheological properties of asphalt binder containing egg shell powder by conducting FST and MSCR

- To determine Performance Grades (PG) of both neat and Egg shell powder modified asphalt binder.

1.4 Limitation of the study

- Due to availability of functional test equipment's, the study doesn't conduct important rheological testes like Bending Beam Rheometer (BBR) and Pressure Aging Vessel (PAV).
- The chemistry of the binder containing egg shell powder was not studied therefore it was not possibly knowing how the binder acts.
- In addition to conventional and rheological properties, compatibility of blends between egg shell powder and bitumen should be checked (Morphology, Optical microscopy).

1.5 Organization

The first chapter describe the overall importance of the problem areas and offers an introduction into what the research is all about, chapter two deals with literatures on basic asphalt bitumen concepts and pavement materials and past studies and works on pavements using egg shell powder as a construction material. Chapter three describes how the experimental work is done with detailed procedures and the results are analyzed and discussed in chapter four. Conclusions derived from experimental results and recommendations for this study and other further studies are presented in chapter five.

2. LITERATURE REVIEW

2.1 Introduction

The performance of asphalt concrete pavement depends on the bitumen properties, asphalt concrete mixtures volumetric properties and external factors such as traffic volume and environment. Bitumen is a viscoelastic material where temperature and rate of load application have a great influence on its behavior. Conventional bitumen is exposed to a wide range of loading and weather conditions; it is soft in a hot environment and brittle in cold weather. Higher traffic volume produces high stress within pavement layer, which is one of the main causes for pavement distress. Fatigue cracking and permanent deformation is considered as most serious distresses associated with flexible pavements. These distresses reduce the service life of the pavement and increase the maintenance cost. To reduce the pavement distresses there are different solutions such as adopting new mix design or by using asphalt additives. Using of asphalt additives in highway construction is known to give the conventional bitumen better engineering properties as well as it is helpful to extend the life span of asphalt concrete pavement (Hamed, 2010).

2.2 Asphalt pavement distresses

Structural damage in flexible pavements appears in two main forms: cracking and deformation, both of which are due to load repetitions or adverse environmental conditions. Different factors affect pavement performance and lead to pavement distress such as magnitude and frequency of loads density, duration of load cycle and variation of temperatures. Asphalt pavement distresses are categorized into three main types cracking, surface deformation, and surface defects. There are many reasons for asphalt concrete damage such as mixtures disintegration, fracture and visco-plastic flow (Miller & Bellinger, 2003).

2.2.1 Permanent Deformation

Permanent deformation is the distress which is non- recoverable deformation of a surface cross section. One of the most common permanent deformations is wheel path rutting (FHWA, 1995). The Distress Identification Manual for the Long-Term Pavement

Performance Project defines rut "rut is a longitudinal surface depression in the wheel path which may have associated transverse displacement" (Miller J. S., 2003).

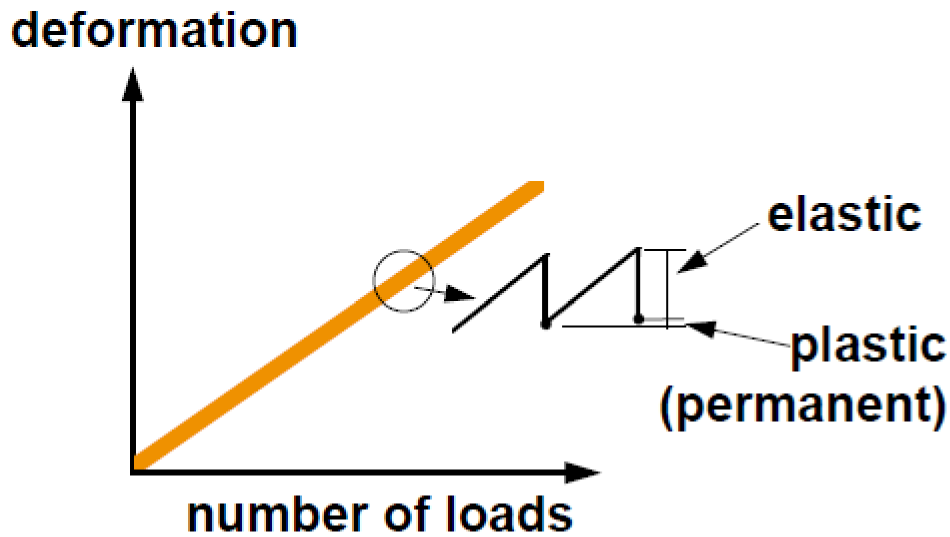

Figure 1: Deformation Due to Loading

2.2.1.1 Causes of Permanent Deformation (Rutting)

There are two main causes of rutting:

- Too much-repeated stress being applied to the native soil

When there is too much-repeated applied stress on the native soil or on layers below the asphalt layers such as subgrade or sub base or base, there will be rutting. Basically, the asphalt pavement has not enough strength to reduce such applied stress to an acceptable limit. If pavement layers have been exposed to moisture, it will weaken the layers and may be another cause for rutting. This type of rutting usually considered as structural problem not as material's problem and the deformation happens in the underlying layers not in the top asphalt layers.

- Accumulated deformations in the asphalt layers

The second cause for asphalt layer deformation which is the main focus of this research would be weak asphalt mixture which has not sufficient shear strength to resist the heavy repeated loads. When each truck passes, this weak mixture will accumulate small permanent deformation. In time, this accumulated deformation result in forming rutting with downward and lateral movement of the mixture. Weak asphalt pavement mixture rutting happens at high pavement temperature period of the year. It might seem that rutting is exclusively asphalt binder's problem, but considering both asphalt cement and mineral aggregate to address rutting would be more appropriate. Since asphalt binder is a visco-elastic material; to resist rutting asphalt binder should be both stiff and elastic (FHWA, 1995).

2.3 Asphalt Binder

The word "asphalt" comes from the Greek word “asphaltos," meaning "secure". In the US asphalt industry, the word “asphalt” or “asphalt cement” is used for the residue material which is obtained during the distillation process of crude oils. However, in the world this material is typically called bitumen. To avoid the confusion liquid asphalt is often referred to as “asphalt cement” more generically as „asphalt binder.” Asphalt binder is a dark brown to black (solid, semisolid, or viscous) cementitious materials in which the predominant constituents are bitumens, it is either found in natural asphalt deposits or it is produced by the distillation of petroleum. Natural asphalts range from a relatively soft material to a hard-black material found in the veins of rock formation (ASTM).

2.3.1 Asphalt binder Constitution

The configuration of the internal structure of asphalt binder is largely determined by the chemical constitution of the molecular species present. It is a complex chemical mixture of molecules that are predominantly hydrocarbons with a small amount of structurally analogous heterocyclic species and functional groups containing Sulphur, nitrogen and oxygen atoms. And also, it contains trace quantities of metals such as vanadium, nickel, iron, magnesium and calcium, which occur in the form of inorganic salts and oxides or in porphyrin structures (Read & Whiteoak, 2003).

- carbon 82-88%
- hydrogen 8-1 1 %
- Sulphur 0-6%
- oxygen 0-1.5 %

- nitrogen 0-1 %

Asphalt binder can be separated into the four groups: asphaltenes, resins, aromatics and saturates. The main characteristics of these four broad component groups and the metallic constituents are now discussed as follows

Asphaltenes

These are n-heptane insoluble black or brown amorphous solids containing, in addition to carbon and hydrogen, some nitrogen, Sulphur and oxygen. Asphaltenes are generally considered to be highly polar and complex aromatic materials of fairly high molecular weight. Different methods of determining molecular weights have led to different values ranging widely from 600 to 300000 depending on the separation technique employed. However, the majority of test data indicates that the molecular weights of asphaltenes range from 1000 to 100000; they have a particle size of 5 to 30nm and a hydrogen/carbon (H/C) atomic ratio of about 1.1. The asphaltene content has a large effect on the rheological characteristics of a bitumen. Increasing the asphaltene content produces a harder, more viscous asphalt binder with a lower penetration, higher softening point and, consequently, higher viscosity (Read & Whiteoak, 2003).

Resins

Resins are soluble in 12-heptane. Like asphaltenes, they are largely composed of hydrogen and carbon and contain small amounts of oxygen, sulphur and nitrogen. They are dark brown in color, solid or semi-solid and, being polar in nature, they are strongly adhesive. Resins are dispersing agents or peptised for the asphaltenes. The proportion of resins to asphaltenes governs, to a degree, the solution (SOL) or gelatinous (GEL)type character of the bitumen. Resins separated from bitumen are found to have molecular weights ranging from 500 to 50 000, a particle size of 1 to 5 nm and a H/C atomic ratio of 1.3 to 1.4.

Aromatics

Aromatics comprise the lowest molecular weight naphthenic aromatic compounds in the bitumen and represent the major proportion of the dispersion medium for the peptised asphaltenes. They constitute 40 to 65% of the total bitumen and are dark brown viscous liquids. The average molecular weight range is in the region of 300 to 2000. They consist of non-polar carbon chains in which the unsaturated ring systems(aromatics) dominate and they have a high dissolving ability for other high molecular weight hydrocarbons.

Saturates

Saturates consist of straight and branch chain aliphatic hydrocarbons together with alkyl-naphthene and some alkyl-aromatics. They are nonpolar viscous oils which are straw or white in color. The average molecular weight range is similar to that of aromatics and the components.

2.3.2 Behaviour of Asphalt binder

Asphalt is a viscoelastic material. This term means that asphalt has the properties of both a viscous material, such as motor oil, or more realistically, water, and an elastic material, such as a rubber. However, the property that asphalt exhibits, whether viscous, elastic, or most often, a combination of both, depends on temperature and time of loading. The flow behavior of an asphalt could be the same for one hour at 60°C or 10 hours at 25°C.In other words, the effects of time and temperature are related; the behavior at high temperatures over short time periods is equivalent to what occurs at lower temperatures and longer times. This is often referred to as the time-temperature shift or superposition concept of asphalt cement. (FHWA, 1995).

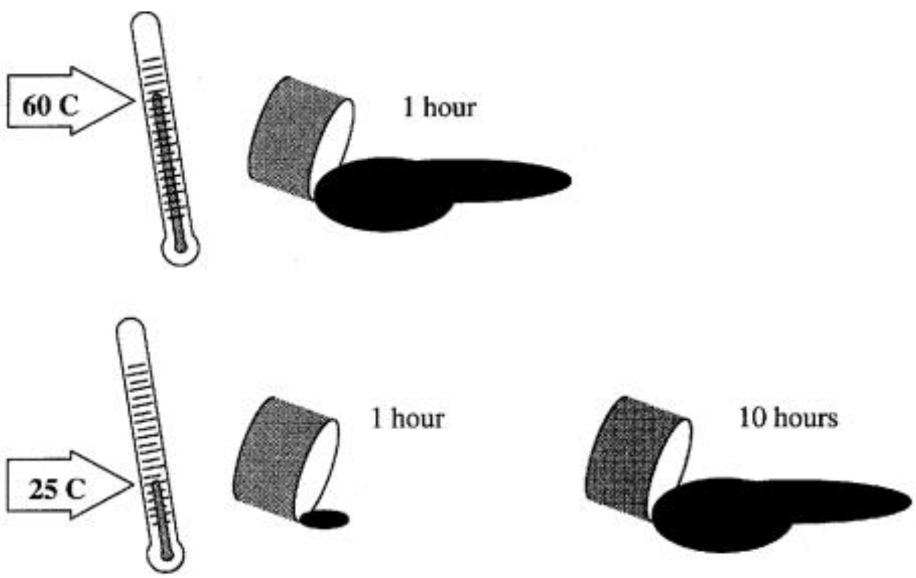

Figure 2 Asphalt cement flow Behavior

The physical properties of asphalt binder vary considerably with temperature. At high temperatures, asphalt binder is a fluid with low consistency similar to that of oil. At room temperature most asphalt binders will have the consistency of soft rubber. At low temperatures, asphalt binder can become very brittle (Mezger, 2011)

1.High temperature

In hot conditions (like., desert climate) or under sustained loads (like., slow moving trucks), asphalts cements behave like viscous liquids and flow. Viscosity is the material characteristic used to describe the resistance of liquids to flow. Viscous liquids like hot asphalt are sometimes called plastic because once they start flowing, they do not return to their original position. This is why in hot weather, some asphalt pavements flow under repeated wheel loads and wheel path ruts form. However, rutting in asphalt pavements during hot weather is also influenced by aggregate properties and it is probably more correct to say that the asphalt mixture is behaving like a plastic.

2.Low Temperature

In cold conditions such as winter temperature, or when subjected to rapidely applied loads such as fast-moving trucks, an asphalt binder behaves like an elastic solid. Elastic solids are like rubber bands; when loaded they deform, and unloaded, they return to their orginal shape.

3.Intermediate Temperature

At intermediate temperature,asphalt binders exhibit the characterstics of both viscous liquids and elastic solids. When heated, asphalt acts as a lubricant, allowing the aggregate to be mixed, coated, and tightly compacted to form a smooth, dense surfac. After cooling, the asphalt acts as the glue to hold the aggregate together in a solid matrix. In this finished state, the behavior of the asphalt is viscoelastic, meaning is has both elastic and viscous charactrstics, depending on the temperature and rate of loading.

2.3.2.1 Visco-Elastic Characterization of Bitumen

The visco-elastic nature of asphalt binder varies with the variation in temperature which requires to be characterized with the best technology available. Visco-elastic means that it simultaneously shows the behavior of an elastic material (e.g. rubber band) and a viscous material (e.g. molasses). The relationship between these two properties is used to measure the ability of the binder to resist permanent deformation and fatigue cracking. To resist rutting, a binder needs to be stiff and elastic; to resist fatigue cracking, the binder needs to be flexible and elastic. The balance between these two needs is a critical one.

The most mysterious property of an asphalt binder is its temperature susceptibility which makes it desirable and tricky at the same time. i.e., its measured properties are very dependent on its temperature. Asphalt cement is sometimes referred to as a visco-elastic material because it simultaneously displays both viscous and elastic characteristics. At high temperatures, asphalt cement acts almost as a viscous fluid. In other words, when heated to a high enough temperature (e.g., > 100 °C), it displays the consistency of a lubricating fluid such as motor oil. At very low temperatures (e.g., < 0 °C), asphalt cement behaves mostly like an elastic solid. i.e., it acts like a rubber band. When loaded it stretches or compresses to a different shape. When unloaded, it easily returns to its original shape. At intermediate temperatures, which also happen to be those in which pavements are expected to function, asphalt cement has characteristics of both a viscous fluid and an elastic solid (FHWA, 1995).

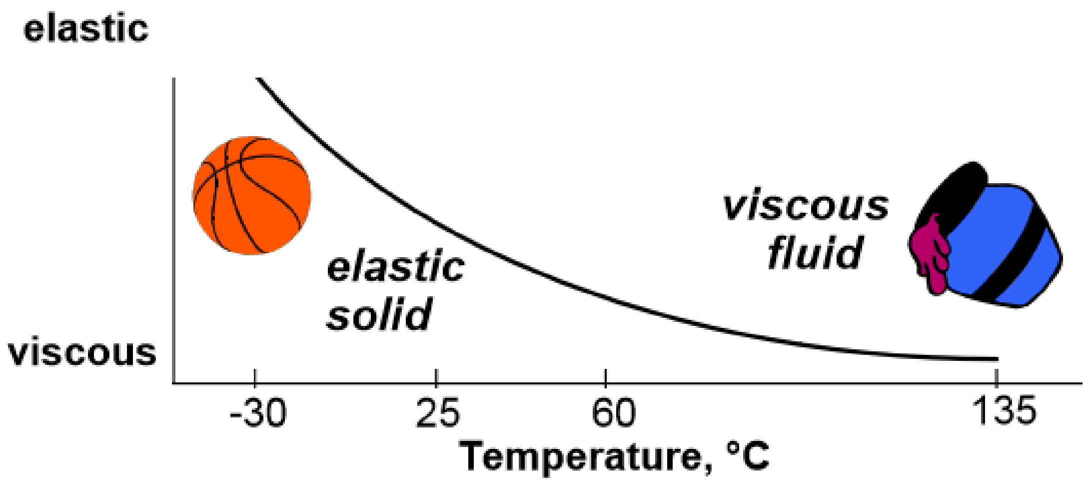

Figure 3:Visco-Elastic Characteristics of Asphalt Binder

2.4 Bitumen Modification

A few years ago, many studies towards the use of alternative materials for road construction and some of them have shown positive and encouraging results. This promoted the use of organic and inorganic materials as modifiers or additives in asphaltic pavement such as plastics, polymers, glass, oils, fibers, etc. With this, not only can solve the problem of the lack of original material in asphaltic pavement construction, but also give alternatives by recycling process (Erfen & Yunus, 2015). Conventional bitumen has a limited range of rheological properties and durability that are not sufficient to resist pavement distresses. Therefore, the conventional bitumen needs to be improved in regards with performance related properties, such as resistance to permanent deformation (rutting), fatigue cracking and low temperature cracking (Atnafu A., 2017).

There are many researchers looking for the reasons to modify bituminous materials. The main reasons to modify bituminous materials with different type of additives could be summarized as follows (Lewandowski, 1994).

- To get softer blends at low service temperatures and reduce cracking,
- To reach stiffer blends at high temperatures and reduce rutting,
- To increase the stability and the strength of mixtures,
- To improve fatigue resistance of blends,
- To reduce structural thickness of pavements.

The core advantage of using modified bitumen is the effect on the pavement performance in terms of permanent deformation, fatigue cracking, and moisture susceptibility. The stiffer asphalt concrete mixture is considered to be more resistance to permanent deformation (Rutting).

2.4.1Different modifiers

Researchers have been conducted on different bitumen modifiers in order to improve pavement distress resistance. One of the researchers conducted is, Bone, which is a composite material which consist both fluid and solid segments. Chemical composition of bone mineral is complex which is made up of calcium, phosphate as well as hydroxyl ions, but which might further more hold a small amount of cationic, magnesium in addition to strontium replacing calcium in addition to bicarbonate, replacing hydroxyl anions. Roughly 65–70% of a bone's dry weight is calcium and phosphate**.** The result shows that addition of crushed cattle bone on asphalt binder increases the stiffening property of asphalt binder at high temperatures and low loading frequencies (susceptible for rutting). The performance grade determination also depicted PG improvement from PG64-yy to PG70-

yy (Hana, 2019). However, in this research the aging effect of conventional test has not been studied.

Other modifier Portland cement have also studied. Portland cement and asphalt are adhesive binders usually used for Portland cement concrete and hot-mix asphalt mixtures. Portland cement may be used as a filler or additive to improve many properties of asphalt binders and hot-mix asphalt mixtures. Made an investigation in to the effect of cement additive on some properties of asphalt binder using Superpave testing methods. Result showed that, the increase in C/A ratio increased the stiffness of asphalt binders represented by the complex shear modulus (G*) value. The increase in the C/A ratio improved the rutting parameter, G*/sin δ value, at all temperatures. The increase in C/A ratio improved the Superpave high PG temperature (the high temperature at which the asphalt binder passed the Superpave criteria for G*/sin δ value) (Ghazi G & Nabil M, 2010).These research do not consider MSCR test, because MSCR test is the latest improved Superpave Performance Graded (PG) Asphalt Binder specification which uses creep and recovery test concept to evaluate asphalt binder’s resistance to permanent deformation by measuring non-recoverable creep compliance (Jnr) and also MSCR is more perfectly indicates the rutting performance of the asphalt binder it has to be considered to properly address rutting. But my research includes MSCR test.

And also, addition of Molasses has affected rheological behavior of asphalt binder thereby making the asphalt binder stiffer at high temperatures which results in a durable binder. From test result obtained from FST, the master curve shows an improving behavior for asphalt binder up on addition of Molasses. Addition of Molasses on asphalt binder increases the stiffening property of asphalt binder at high temperatures (low loading frequencies). Therefore, addition of Molasses improves the resistance of asphalt pavements to rutting. The replacement of asphalt binder with Molasses at optimum binder content of 5%, decreased the stability, flow, unit weight and the Va% of the HMA, while the VMA and VFA percentages decreased as the percentage of Molasses increased. The increment and reduction value of these properties of HMA up to 10% Molasses is within the Marshall criteria for heavy traffic (Timaj, 2016). However, in this research performance grade (PG) test has not been done so we can't know up to how much temperature can we use the molasses for rutting resistance.

Moreover, using crumb rubber from scrap tyres as asphalt modifier helps to improve the pavement performance, in addition solves serious environmental problem. In addition to the control specimen, three binders were obtained by mixing the asphalt binder with three different percentages of crumb rubber by weight of asphalt binder (i.e. 15%, 17.5% and 20%). Addition of Crumb Rubber up to 20% improves the rheological properties, aging effect and rutting performance of binders at high temperature ranges so, it can be used areas where rutting is more critical (Girma, 2017).

2.5 Bitumen Aging

Asphalt/bitumen properties change over time on exposure to high temperature and the atmosphere. This process is referred to as ageing. It is an effect of asphalt hardening with time caused by oxidation, heat, UV light. Over the lifetime of the road, an asphalt binder oxidizes and subsequently hardens eventually causing failure of the road. Because they are composed of organic molecules, they like to react with oxygen from the environment. This reaction is called oxidation and it changes the structure and composition of asphalt molecules. Amore brittle structure always results and that is the origin of the terms "oxidative hardening" or "age hardening." Oxidative hardening happens at a relatively slow rate in a pavement although it occurs at a faster rate in a hot, desert climate when compared to cool climate. Likewise, oxidative hardening is seasonal since it happens more in summer than in winter. Because of this type of hardening, old asphalt pavements are more susceptible to cracking. Even relatively new asphalt pavements may be excessively prone to oxidative hardening if not thoroughly compacted. In this case, the lack of adequate compaction causes high air voids, which allow a greater amount of air to permeate the asphalt mixture causing a greater degree of oxidative hardening (SHRP, 1994).

Asphalt binder ageing is usually split up into two categories:

- Short-term ageing: This occurs when bitumen is mixed with hot aggregates i.e., during production and construction
- Long-term ageing: This occurs after HMA pavement construction and is generally due to environmental exposure and loading i.e., during the life of the pavement.

2.6 Superpave Fundamentals

Strategic Highway Research Program (SHRP) was approved in 1987 to improve the performance and durability of Us roads and to make those roads safer. Asphalt technologies have had various degree of success in overcoming the three main asphalt pavement distress

- Permanent Deformation (Rutting)
- Fatigue cracking which leads to alligator cracking
- Low temperature cracking

A final product of the SHRP asphalt research is the Superpave asphalt mixture design and analysis system. Superpave is an acronym for Superior Performing Asphalt Pavements. Superpave represents an improved, performance-based system for specifying asphalt binders and mineral aggregates, performing asphalt mixture design, and analyzing pavement performance.

A unique feature of the Superpave system is that its tests are performed at temperatures and aging conditions that more realistically represent those encountered by in-service pavements. If the pavement distresses addressed by Superpave (rutting, fatigue cracking, and low temperature cracking) do occur in the pavement, they do so at relatively typical stages in a pavement's life and under relatively common temperature conditions. The Superpave performance graded (PG) binder specification makes use of these tendencies to test the asphalt under a project's expected climatic and aging conditions to help reduce pavement distress.

2.6.1 Pre-Superpave Asphalt Property Measurements

Because of its chemical complexities, asphalt specifications have been developed around physical property tests, using such tests as penetration, softening point, viscosity, and ductility. These physical property tests are performed at standard test temperatures, and the test results are used to determine if the material meets the specification criteria However, there are limitations in what these test procedures provide. Many of these tests are empirical, meaning that field experience is required before the test results yield meaningful information. Penetration is an example of this. The penetration test represents the stiffness of the asphalt, but any relationship between asphalt penetration and performance has to be gained by experience. An additional drawback of empiricism is that the relationship between the test and performance may not be very good (FHWA, 1995).

2.6.2 Superpave Binder Property Measurements

The new Superpave binder tests measure physical properties that can be related directly to field performance by engineering principles. Superpave characterizes them at the actual pavement temperatures that they will experience, and at the periods of time when the asphalt distresses are most likely to occur (SHRP, 1994).

Table 1 Superpave Binder Tests and Their Purpose

Superpave Binder Test	Purpose
Dynamic Shear Rheometer (DSR)	Measure properties at high and intermediate temperatures
Rotational Viscometer (RV)	Measure properties at high temperatures
Bending Beam Rheometer (BBR) Direct Tension Tester (DTT)	Measure properties at low temperatures
Rolling Thin Film Oven (RTFO) Pressure Aging Vessel (PAV)	Simulate hardening (durability) characteristics

2.7 Egg production

Egg production in Africa expanded by 3.8 percent per year between 2000 and 2013. As this far exceeded the global growth rate of 2.3 percent, Africa’s share of world output increased from 3.7 percent to 4.5 percent. In volume terms, production in Africa rose from 1.9 million tons to 3.1 million tons over the continuous 10 years (FAO, 2013).

Hen egg production in Ethiopia rose from 28.6 million tons to 41 million tons for the consecutive seven years. In Africa, Ethiopia is the fifteenth hen egg producer (FAO, 2013).

2.7.1 Egg shell

Eggshells are agricultural waste materials generated from chicken hatcheries, bakeries, poultry farms and fast food restaurant among others which can litter the environment and consequently constituting environmental problems or pollution which would require proper handling. In the ever-increasing efforts to convert waste to wealth, the efficacy of converting eggshell to beneficial use becomes an idea worth embracing (Hassan & Bin, 2014). Eggshell waste falls within the category of food waste, can be suitable alternative material for construction (Amu et al., 2005).Eggshell consists of several mutually growing layers of $CaCO_3$, The main ingredient in eggshells is calcium carbonate (the same brittle white stuff that chalk, limestone, cave stalactites, sea shells, coral, and pearls are made of). The shell itself is about 95% $CaCO_3$ (which is also the main ingredient in sea shells). The remaining 5% includes Magnesium, Aluminum, Phosphorous, Sodium, Potassium, Zinc, Iron, Copper, Ironic acid and Silica acid. Eggshell has a cellulosic structure and contains amino acids; thus, it is expected to be a good bio-sorbent (Samuel, 2015).

2.8 Egg shell in construction industry

I. Characterization of HMA modified with egg shell powder

Studied the effect of egg shell powder on 40-50 penetration binder on virgin and modified asphalt and to evaluate the physical properties of asphalt cement as well as the mechanical properties of hot mix asphalt mixture. The results of the study demonstrated a decrease in penetration and the rotational viscosity as well as a valuable increase in flash point versus increasing percent of the waste material hot mix asphalt mixture. Moreover, regardless of aging state the Egg Shell modified asphalt has the highest stiffness modulus (G*) compared to the neat asphalt. This indicates the improved (reduced) temperature susceptibility of the Egg Shell modified asphalt resulting in both increased flexibility at lower temperatures and increased hardness at high temperatures. The mechanical tests showed improvement in stability-flow results with increasing egg shell percent (A.K.Razzaq, Yousif, & Tayh, 2018). However, their work is limited to only one super- pave test i.e temperature sweep test only they didn’t go for further rheological test in depth.

II. Egg shell as a filler in HMA

Studies has done to evaluate the effect of using egg shell as a filler in hot mix asphalt concrete. The results of conventional samples and modified samples shows effective egg shell content in the range of 3% to 5%. In addition, the specific gravity was carried out using egg shell as a filler will reduced the specific gravity. He concludes that the egg shell is one of the substances that can be used as filler (Erfen & Yunus, 2015).

III. Strengthening of Flexible Pavement using Egg Shell as a Filler

Material combinations and modified bituminous binders have been found to result longer life for wearing courses depending upon the percentage of filler and type of fillers used. The common fillers used are lime, cement, quarry dust etc. Since the eggshell has the same composition as that of limestone, it can be used in the pavement as they are inexpensive when compared to others. Overloading of trucks and significant variations in daily and seasonal temperature of pavements have been responsible for development of distress symptoms like raveling, undulations, rutting, cracking, bleeding, shoving and potholing of bituminous surfaces. In order to find the appropriate mix proportion that will efficiently withstand the problems, optimum bitumen content (OBC) and optimum eggshell content has to be found out by experimental studies. Bituminous mixes containing eggshell as filler displayed maximum stability. The study concludes that, the use of eggshell as filler material in bituminous pavement produce positive result. This shows that eggshell is suitable to apply in the road construction (K.Kuruthiha, G.Loshini, & M.thivya, 2015).

IV. Egg shell as a partial cement replacement in concrete

Studies has done to identify the performance of oven-dried eggshell powder as a partial cement replacement in the production of concrete under both water-cured and air-cured regimes. They use eggshell powder of various amounts namely 5%, 10%, 15% and 20% by volume, was added as a replacement for ordinary Portland cement. The results showed that water-cured eggshell concrete greatly improved the compressive and flexural strength of concrete, by up to 51·1% and 57·8%, respectively. The rate of water absorption of eggshell concrete was reduced by approximately 50%, as eggshell powder filled up the existing voids, making it more impermeable. However, the compressive strength of the eggshell concrete decreases gradually when the amount of eggshell powder increased, during immersion in acid and alkali solutions, because eggshell contains a high amount of calcium, which reacts readily with acid and alkali solutions. They concluded that the optimum percentage of oven-dried eggshell powder as a partial cement replacement is 15% (Tan, Doh, & Chin, 2018).

2.9 Summary

In general, literature review describes different information about asphalt binder properties related to conventional and rheological property measurement. The material selected as a modifier (Egg shell powder) with the intention to improve binder resistance against rutting. This material has been used in different research such as a modifier in 40-50 penetration grade with the interest to introduce the egg shell powder as a sustainable modifier to fulfil the objectives of increasing flexibility at lower temperatures and increased hardness at high temperatures. From the test result, reducing the weight percentile, reducing costs and improving the economic viability of hot mix asphalt. And also, as a replacement for filler in hot mix asphalt (HMA) also for partial cement replacement in concrete and a modifier to improve different engineering materials such as bitumen, cement, and others.

After reviewing the previous studies related to utilization of ESP in the asphalt mix, materials are prepared for conducting laboratory tests.

3. METHODOLOGY AND EXPERIMENTAL WORKS

3.1 Introduction

The research was conducted based on experimental methods of analysis which have mainly three parts. The first part includes investigating quality test for both binder and egg shell powder. The second part consists of investigating conventional properties of unaged and aged binder mix containing different proportion of egg shell powder. And the third part includes investigating fundamental rheological properties of binder containing different proportion of egg shell powder.

In general, in this chapter, every test approaches, equipment and conditioning machines used to investigate binder containing different proportion of egg shell powder for both conventional and rheological properties was assessed using Dynamic shear rheometer. Rheological properties are assessed using BOHILN Dynamic Shear Rheometer

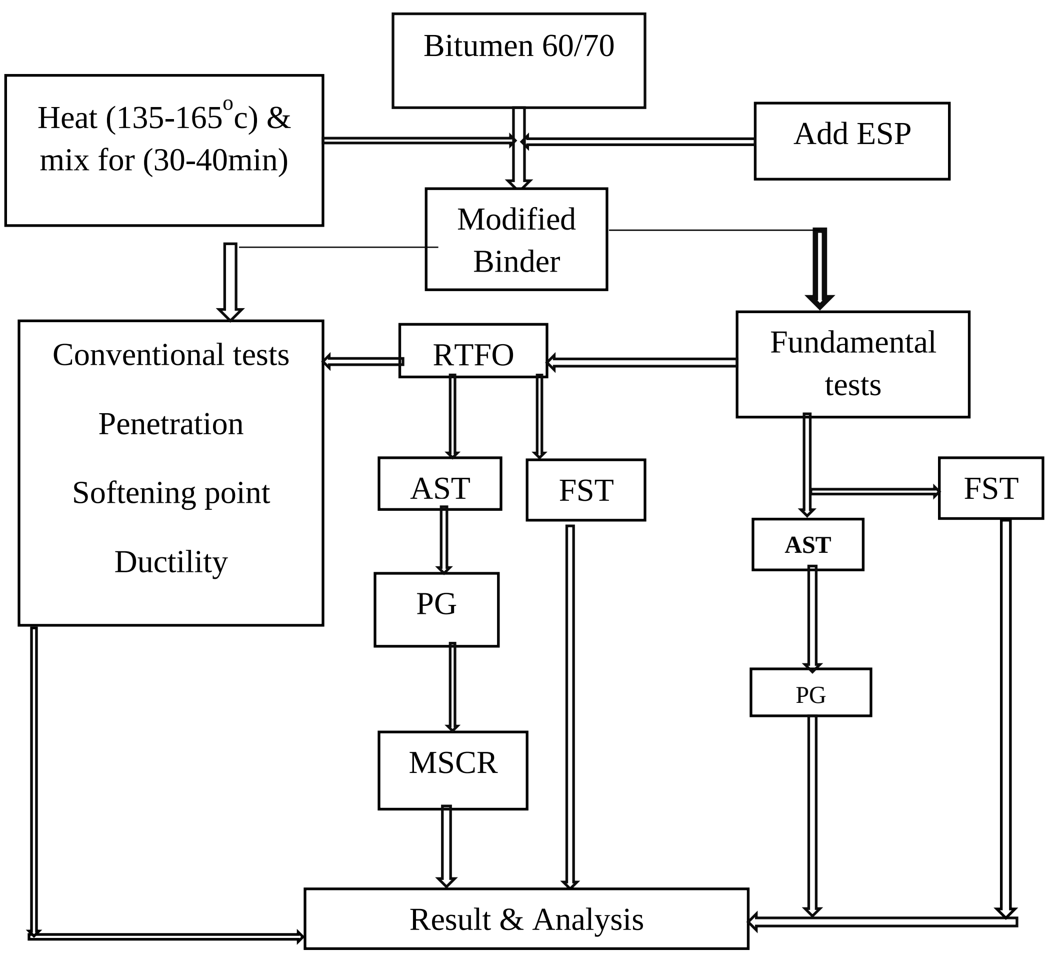

Figure 4: Flow chart diagram

3.2 Material properties

Asphalt binder with 60/70 was sampled from Amhara road works enterprise and Egg shell powder sampled from Anidassa poultry farm was used.

3.2.1 Egg shell waste processing

Broken egg shells were collected from the local sources. The shells were cleaned in normal water and dried in oven dried at constant temperature range (50^oc-60^oc) until constant weight has been observed, and they were milled by electrical mill, the eggshell powder was finally sieved through 200µm sieve size.

Figure 5 Processing of egg shell waste

Chemical Compositions of egg shell powder Complete Silicate Analysis (Geological Survey of Ethiopia) test was conducted. As shown in the table below egg shell powder have highest percentage of Cao.

Table 2 chemical composition of egg shell powder by complicate silicate analysis (Geological survey of Ethiopia)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
2.36	<0.01	<0.01	50.10	0.40	<0.01	<0.01	0.10	0.32	<0.01	1.10	46.46

3.2.2. Asphalt binder

The asphalt binder used in this study was penetration grade of 60/70. The physical properties of asphalt binder were determined according to the procedure specified by AASHTO standards. Binders were characterized by using a number of standard physical(conventional) tests such as penetration test (temperature, load and time are 25ºC, 100g and 5sec respectively), softening point test and a ductility test and also including rheological measurements (fundamental tests) by using a Dynamic Shear Rheometer.

3.3 Binder tests

3.3.1 Conventional tests

The mechanical properties of unaged and aged asphalt binder mix of containing different proportion of egg shell powder conducted conventionally using measurements of penetration, softening point and ductility. AASHTO and ASTM testing and specification was used to proceed the experimental works.

3.3.1.1 Laboratory Work Plan

The laboratory produced materials or samples were modified binders produced by blending the base asphalt with different contents of egg shell powder. Thus, in this study the evaluation was done by preparing modified binder with 3, 6 and 9 percent by weight of total binder. The sample preparation for conventional tests has been done in accordance with the respective standards of test methods.

3.3.1.1.1 Penetration Test

A sample of about 100g of asphalt binder was heated in an oven for enough time to completely soften. The asphalt binder samples were 0%, 3%, 6%, and 9% ESP modified unaged and aged binder. Then it was transferred into a 15mm penetration test cup and allowed to cool to room temperature for 1hr. The sample was then placed in a temperature-controlled water bath at a temperature of 25°C and allowed to condition for about 1 hour. It was then removed, dried quickly and placed under the needle of the penetrometer. Then three readings were taken for a single penetration cup after placing tip of the penetrometer needle precisely at the surface of the cup before the instrument was started. The average of three samples were taken for each sample and recorded (ASTMD5-06, 2006).

3.3.1.1.2 Softening point test

The softening point test is used to measure and specify the temperature at which asphalt binders begin to show fluidity. The softening point is also convenient in evaluating the uniformity of shipments or source of supply. The softening point is an indicative of the tendency of the material to flow at elevated temperature encountered in service

A sample of asphalt binder was heated in an oven for enough time to completely soften. The asphalt binder samples were 0%, 3%, 6%, and 9% ESP modified unaged and aged binder. Then it was poured in to a two brass ring and allowed to cool at room temperature for 30 minutes. A steel of 3.5g was placed on a sample of binder contained in a brass ring which was suspended in a water bath. The bath temperature was raised at 5°C per minute, the binder gradually softens and eventually deforms slowly as the ball falls through the ring. At the moment the bitumen and steel ball touch a base plate 25 mm below the ring, the temperature of the water glycerin was recorded (ASTMD36-95, 1995).

3.3.1.1.3 Ductility Test

The ductility test is used to describe the ductile and tensile behavior of asphalt binders. The test, which is normally performed at ambient temperature, is believed to reflect the homogeneity of the binder and its ability to flow.

Ductility, as a physical property, has been considered as important characteristics of asphalt binders by some engineers. The presence or absence of ductility, however, is often considered more significant than the actual degree of ductility because some asphalt binders having a high degree of ductility because some asphalt binders having a high degree of ductility have also been found to be more temperature susceptible (ASPHALTINSTITUTE, 2007).

Standard method of test for the ductility of bituminous materials is stated in ASTM D113.A heated sample of asphalt binder is then poured into the mold, slightly overfilling the mold the samples were cooled in the air for 30 minutes and then in water bath at 25 °C temperature for another 30 minutes. The sides of the molds were removed, the clips were hooked on the machine and the machine was operated. The distance up to the point of breaking of thread is the ductility value which is reported in cm (ASTMD113-07, 2007).

3.3.1.1.4 Rolling Thin film Oven test

Different binders were tested in the RTFO to age the binders to achieve the aging that can be obtained during the mixing and construction of HMA. The aging in RTFO is obtained by following the specifications in AASHTO T 240. These processes age the asphalt binder by driving off an enough amount of volatiles when exposed to elevated temperatures (AASHTOT240-13, 2013).

The Rolling Thin-Film Oven test is conducted as per AASHTO T 240, which is the standard method of test for "Effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin Film Oven Test". Preheat the oven for a minimum of 2 hrs prior to testing in such a way that the oven would equilibrate at 163± 0.5^{o}c when fully loaded and the air is on. And make sure the air flow is at a rate of o 4000 ml/min. After preparation of the oven, pour 35 ±0.5 g of asphalt binder in each bottle and immediately turn the container to a horizontal position. Then rotate the container slowly to precoat its cylindrical surface and put each container in the cooling rank to cool for at least 60min but not more than 180min. Then place sample containers in the carriage. And fill any free carriage with empty containers. Then close the door and condition the sample containers for 85 min. At the end of the testing period, remove each sample container one at a time. Then pour and scraping out as much of the remaining residue in to a collection container. The residue aged binder was used for further rheological tests by using DSR (AASHTOT240-13, Standard Method of Test for Effect of Heat and Air on a Moving Film of Asphalt Binder (Rolling Thin-Film Oven Test)., 2013).

Figure 6 Rolling thin film oven (RTFO)

3.3.2 Dynamic Shear Rheometer (DSR) Tests

3.3.3 Dynamic Shear Rheometer (DSR) Background Information

The Dynamic Shear Rheometer (DSR) is used to characterize the viscous and elastic behavior of asphalt binders. It does this by measuring the complex shear modulus (G*) and phase angle (δ) of asphalt binders. G* is a measure of the total resistance of a material to deforming when repeatedly sheared. It consists of two parts: a part that is elastic (recoverable) and a part that is viscous (non-recoverable). δ is an indicator of the relative amounts of recoverable and non-recoverable deformation. (SHRP, 1994)

Parallel plate geometry is used to test the samples, and the plate diameter is 25 mm for neat and (R)TFO-conditioned binder and 8 mm diameter plates for PAV-conditioned binder. During testing, one of the plates is oscillated with respect to the other plate. The complex shear modulus, G*, is calculated by dividing the maximum stress by the maximum strain that occurs during the loading cycle. The phase angle of the material, δ, represents the delay in the material's response to the applied load.

3.3.4 The working principle of DSR

The operation principle of the DSR is straightforward. An asphalt sample is sandwiched between an oscillating spindle and the fixed base. As shown in figure 7 below the oscillating plate (often called a "spindle") starts at point A and moves to point B. From point B the oscillating plate moves back, passing point A on the way to point C. From point

C the plate moves back to point A. This movement, from A to B to C and back to A comprises one cycle. And this is repeated for a specified number of cycles.

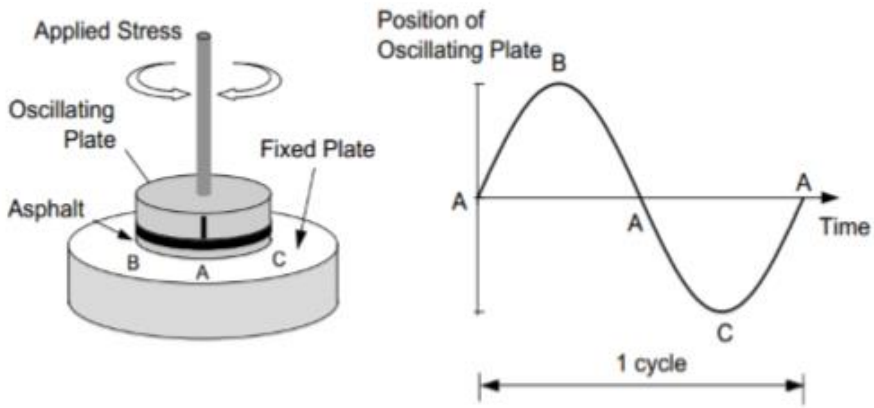

Figure 7: Dynamic Shear Rheometer Geometry

The number of cycles completed in one second is the loading frequency; typically, DSR tests that can run for specification purposes are performed at frequency of approximately 1.59 hertz (1.59 cycles per second) equivalent to 10 radians per second. Stress and strain are measured during each loading cycle and used to calculate complex shear modulus and phase angle (SHRP, 1994).

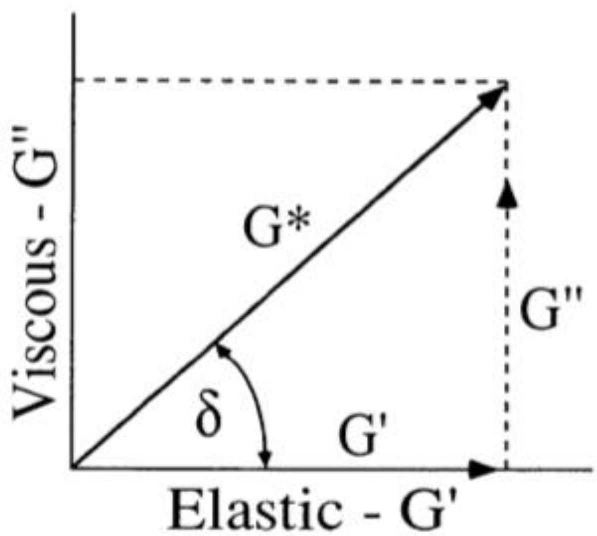

Figure 8: Graphical Representation of the Components of the Complex Shear Modulus G*

The angle (δ) is expressed in radians or degrees and is a measure of the loss and storage moduli, G" and G' which make up the complex modulus G* as shown in figure 8 above. The values of G" and G' can also be considered to be an estimate of the viscous component and the elastic and delayed elastic components of the complex stiffness modulus G*. A perfectly elastic material would exhibit a phase angle equal to zero, while a viscous material would exhibit an angle of 90°. Thus, an elastic material would exhibit maximum shear stress and maximum shear strain at the same time, while for a perfectly viscous material maximum shear stress would occur at the same time as minimum shear Strain. Asphalt tends to be elastic (δ = 0) at cold temperatures and viscous (δ = 90°) at very high temperatures.

Asphalt is sandwiched between the oscillating spindle and the fixed base. The spindle is oscillated back and forth using either a constant stress or constant strain. Constant stress means that the spindle is rotated through a certain distance until a fixed stress is achieved. Constant strain means that the spindle is rotated every time through a fixed distance, regardless of the stress achieved. While this rotation occurs, the resulting strain or stress is monitored. The relationship between the applied stress and the resulting strain provides information necessary to compute G* and δ. The diagram in Figure 9 explains this computation (SHRP, 1994).

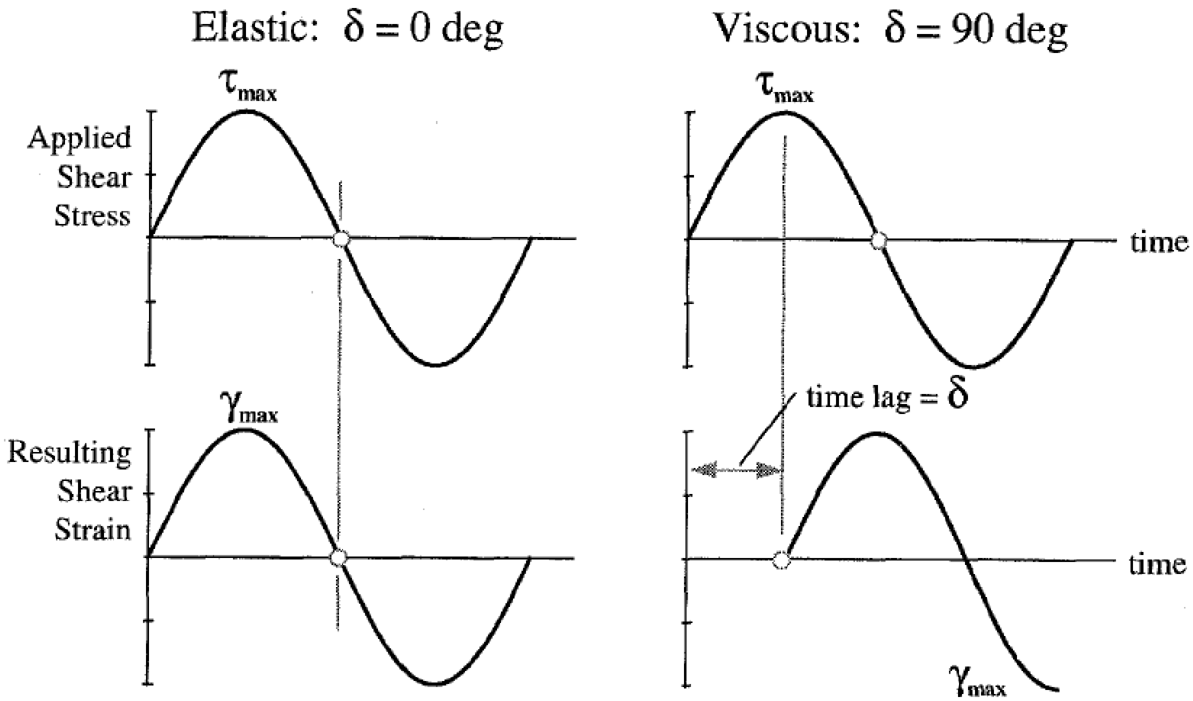

Figure 9 Stress-strain output for a constant Rheometer

G* is the ratio of maximum shear stress (Tmax) to maximum shear strain (γmax) or Tmax /γmax. The time lag between the applied stress and the resulting strain (for constant stress rheometers as shown in Figure 9**)** or the applied strain and resulting stress (constant strain rheometers) is the phase angle δ. For a perfectly elastic material, an applied load coincides with an immediate response, and the time lag or angle δ is zero. A viscous material (such as hot asphalt) has a relatively large time lag between load and response and thus, an angle that approaches 90 degrees. In the DSR, a viscoelastic material such as asphalt at normal service temperatures displays a stress-strain response that is between the two extremes as shown in Figure 10 below (SHRP, 1994).

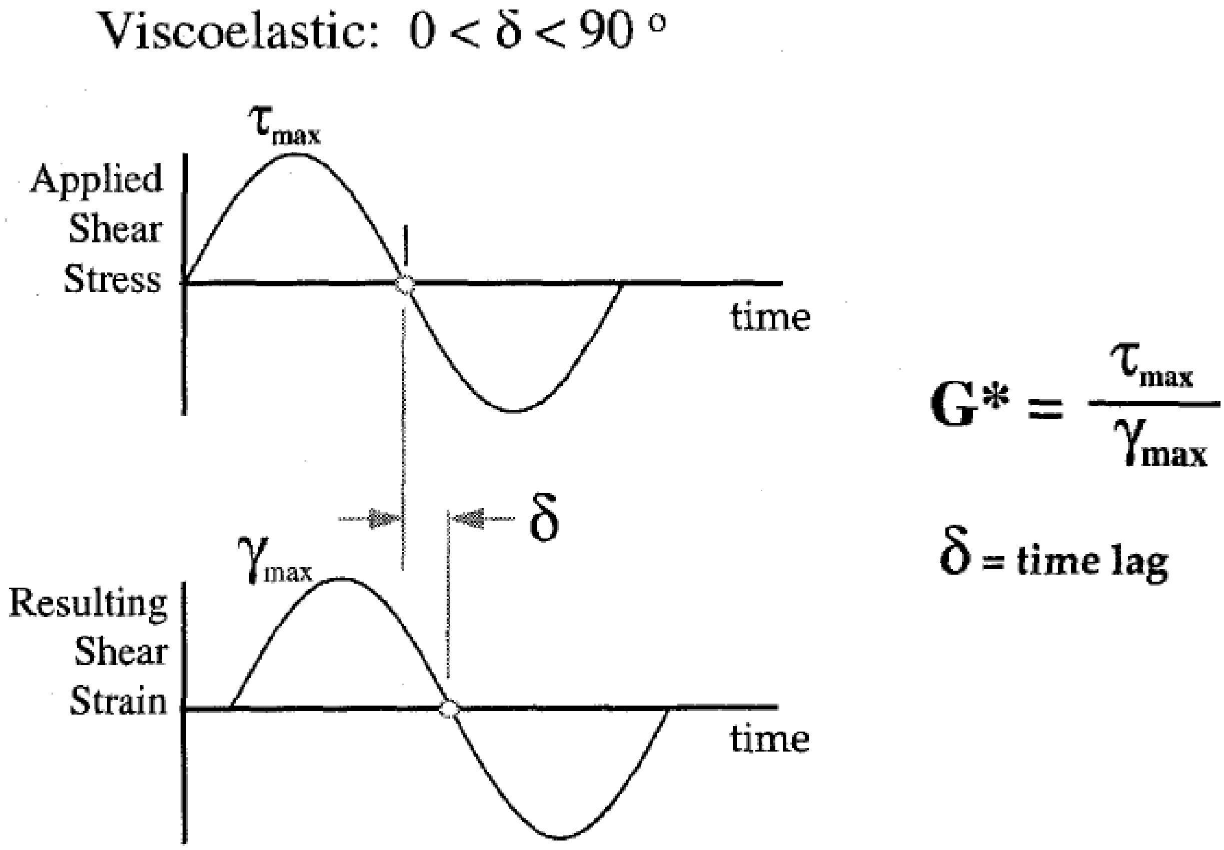

Figure 10: Stress-strain response of a viscoelastic material

The Standard test method for determining the rheological properties of asphalt binder using dynamic shear Rheometer is described in AASHTO T315-10. First the asphalt binder is heated until it is sufficiently fluid to pour and to prepare the test specimens. Then a small sample of asphalt binder is sandwiched between two plates. But before placing the sample the DSR is set to a particular temperature; this preheats the upper and lower plates, which allows the specimen to adhere to them. Depending upon the type of asphalt binder being tested the test temperature, specimen size and plate diameter varies. Using a specimen 0.04 inches (1 mm) thick and 1-inch (25 mm) in diameter, the unaged asphalt binder and RTFO residue are tested at the high temperature specification for a given performance grade (PG) binder. The measurement of a small phase angle (δ) is a result of these lower temperatures that make the specimen quite stiff. Hence, to have a measurable phase angle (δ) to be determined, a thicker sample (0.08 inches (2 mm)) with a smaller diameter (0.315 inches (8 mm)) is used. The DSR apparatus used is as shown in figure below.

Figure 11: Dynamic Shear Rheometer

For a sample 0.04 inches (1 mm) thick and 1 inch (25 mm) in diameter, test temperatures greater than 115°F (46°C) are used whereas for a sample 0.08 inches (2 mm) thick and 0.315 inches (8 mm) in diameter, test temperatures between 39°F and 104°F (4°C and 40°C) are used. To suit the desired size of specimen the upper spindle is lowered until the gap between the plates equals the test gap plus 0.002 inches (0.05 mm). Due to the compression, excess material will come out which is then trimmed around the edge of the test plates using a heated trimming tool. The test plates further moved together to the selected testing gap by eliminating the additional 0.05 gap. This creates a slight bulge in the asphalt binder specimen's perimeter. The test specimen is kept at near persistent temperature by heating and cooling a surrounding environmental chamber. The test is started up only after the specimen has been at the desired temperature for at least 10 minutes. The instrument measures the maximum applied stress, the resulting maximum strain, and the time lag between them while the top plate oscillates in a sinusoidal waveform. The calculation of the complex modulus (G*) and phase angle (δ) is done automatically with the help of the software. Based on the material being tested (e.g., unaged binder, (RTFO residue or PAV residue) the determination of a target torque at which to rotate the upper plate is carried out using the DSR software *(Kennedy T. W., 1994)*.

Amplitude sweep Test

Amplitude sweep is an oscillatory DSR test with variable stress or strain amplitude at constant frequency. The main or the sole purpose of this test is to determine the Linear Visco Elastic Range (LVR) of a visco-elastic material. The linear visco elastic part is the region where the applied oscillation is nondestructive. In most cases log-log graph on the same scale is plotted as strain in the x-axis and shear modulus in the y-axis. The complex shear modulus G* versus strain plot was used to determine the linear visco-elastic (LVE) region amplitude Sweep Test. The maximum strain of linear region was identified at the point where modulus decreased to 95% of the initial modulus. As shown in the figure 12 below, log-log scale graph of complex shear modulus versus strain was constructed as strain in the x-axis and complex shear modulus in the y-axis.

The amplitude sweep test was conducted at 10°C, 21.1°C, 37.8°C and 54.4°C temperature for unaged and RTFO aged binders with a shear stress range from 100Pa to 90kPa and at constant frequency of 1.59Hz or 10rad/s) (ASSHTO T315). The tests were run in 8mm parallel plate with 2.0mm testing gap on DSR for 10°C, 21.1°C & 37.8°C and 25mm parallel plate with 1mm testing gap for 54.4°C.

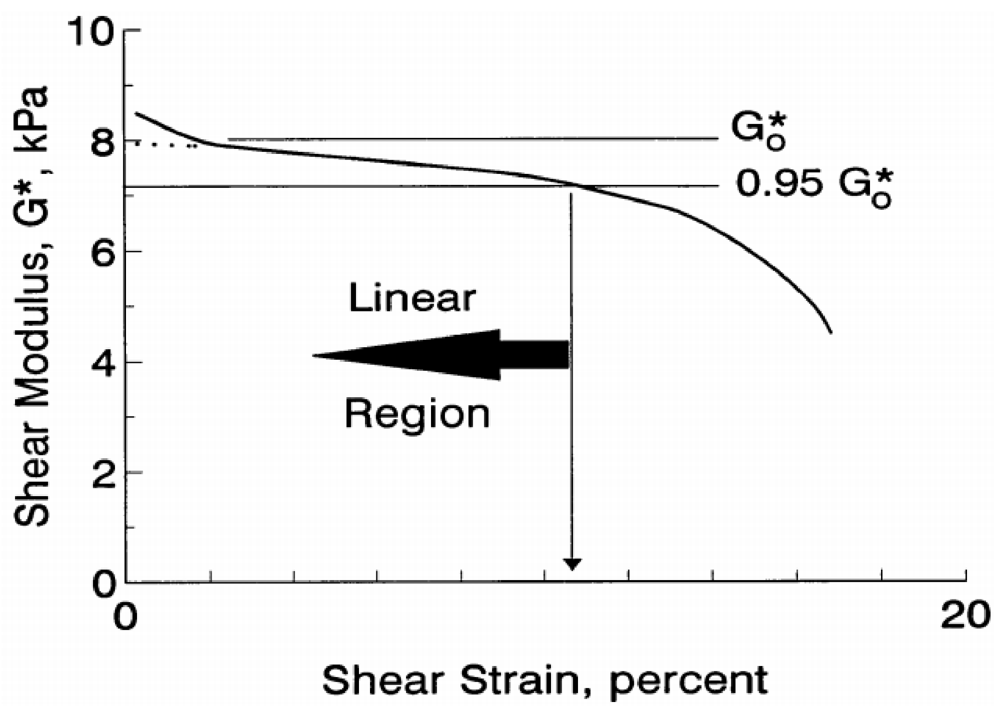

Figure 12: Linear viscoelastic range *(Ojha, 2013)*

Performance Grade Determination (PG) Test

Based on the objective of the study, the PG test is carried out at high temperature considering rutting only. According to AASHTO specifications, high temperatures are from 46 ºC to 82 ºC with 6 ºC increment. Thus, the types of samples were original binder and RTFO aged binder. And the test plates used were 25mm in diameter with a 1mm thickness of specimen. The test was conducted at 12% strain for original and 10% strain for RTFO aged samples with 10rad/sec frequency at high temperature with minimum stiffness of 1.0 kPa specification for unaged binder to guard against mixture tenderness and minimum stiffness of 2.2 kPa specification for RTFO aged binder to ensure sufficient permanent deformation resistance immediately after construction *(Kennedy T. W., 1994)*. PG performed on unaged and RTFO aged asphalt binder where by the temperature sweeps performed at 6°C increment rate on initial temperature until the test completed. The test was going to run on 25-mm parallel plates with a 1.0 mm testing gap.

3.3.4.1 Laboratory Work Design

For the purpose of this research DSR machine known as MARVEL BOHLIN INSTRUMENT, is used to characterize the viscous and elastic behavior of asphalt binder with egg shell powder at different temperatures. The DSR is used to determine both the viscous and the elastic properties of a material.

3.3.4.1.1 Testing procedure

3.3.4.1.2 Sample preparation for Fundamental tests

Commonly there are two ways to prepare the sample:

1. Asphalt can be poured directly onto the spindle in the proper quantity to provide the appropriate thickness of material
2. A mold can be used to form the asphalt disk, then the asphalt is placed between the spindle and fixed plate of the DSR

In the first method, experience is necessary to apply the proper amount of asphalt. There must not be too much or too little material. If there is too little, the test will be inaccurate. If there is too much, excess sample trimming will be required. In the second method, asphalt is heated until fluid enough to pour. The heated asphalt is poured into a silicone mold and allowed to cool until solid enough to remove the asphalt from the mold. After removal from the mold, the asphalt disk is placed between the fixed plate and the oscillating spindle of the DSR. As before, excess asphalt beyond the edge of the spindle should be trimmed (FHWA, 1995).

For the purpose of this study the binder is heated until sufficiently fluid and poured into a silicone mold and allowed to cool until solid enough to remove the asphalt from the mold. After the removal from the mold, the asphalt disk is placed between the fixed plate and the oscillating spindle of the DSR.

Figure 13: Prepared sample for DSR testing (25mm) plate

The first step on preparing the sample was heating the asphalt binder at stove around 135-165^{o}C until fluid enough to flow. Then egg shell powder was added in different volume fractions by weight of asphalt binder, i.e. 3, 6 and 9%, producing a total of 4 mix types with the controlled mix. Up on adding egg shell powder the mix was contentiously stirred for 30-40 min at a constant temperature to ensure good homogeneity. Then each sample was aged using rolling thin Film Oven (RTFO) in accordance to AASHTO T 240. Then the heated asphalt was poured into a mold and allowed to cool until solid enough to be removed from the mold. After removal from the mold, the asphalt disk was placed between the fixed plate and the oscillating spindle of the DSR for testing (AASHTOT240-13, 2013).

1.Amplitude sweep test

In this study a frequency of 10 rad/sec or 1.596 Hz was applied. The test was conducted at four different temperatures i.e. at 10,21.1, 37.8 and 54.4^{O}C using 8mm diameter plate size plate with 2.0mm testing gap on DSR for the first three temperature and 25mm diameter plate with 1.0mm testing gap on DSR for the 54.4 temperature. The dynamic rheological properties were tested by measuring the required shear stress to achieve a preset strain level for both aged and unaged mixes.

2.Frequency sweep test

For this study, frequency sweep tests were also done on the same temperatures as AST i.e. at 10^{0},21.1°, 37.8^{o} and 54.4 °C. The tests were run on 8mm and 25mm parallel plate after the samples were allowed to equilibrate for few minutes at each temperature before to testing. The shear strain applied was 0.38% for all the samples, i.e. 0%, 3%, 6% and 9%. And the frequency range used was 25Hz to 0.1Hz in an increasing damaging effect.

3.Performance grade test

During the PG grade determination, the frequency is varied while the amplitude of the deformation or alternatively the amplitude of the shear stress is kept constant. PG grade determination tests are designed to know performance grade of the binder (i.e. higher temperature grade which is the maximum pavement service temperature). This higher temperature is used for MSCR test.

In this study, PG grade determination tests were performed on unaged and aged samples at high temperature. The tests were performed on higher temperature using 25mm parallel plate diameter with 1mm gap. The starting temperature was decided based on bitumen type and test started from expected lower temperature grade, increasing until it achieves pass fail temperature. On this study bitumen grade of 60/70 penetration grade which is equivalent to PG-64 based on performance grade was used; test temperature started from 52°C. The test was conducted by applying 12% shear strain for original binder and 10% applied shear strain for RTFO aged binder and frequency of 1.59Hz for all the samples, i.e. 0%, 3%, 6% and 9% at high temperature with minimum stiffness of 1.0 kPa specification for unaged binder to guard against mixture tenderness and minimum stiffness of 2.2 kPa specification for RTFO aged binder to ensure sufficient permanent deformation resistance immediately after construction. (Kennedy T.W, 1994).

There were two main reasons for why only high temperature tests were performed. The first is because the objective of the study focuses on rutting. The second reason was the unavailability of Pressure Aging Vessel (PAV) to carry out long term ageing for intermediate and low temperature tests.

4.Multiple creep recovery test

Before conducting the MSCR test performance grade determination was conducted to decide the test temperatures for the MSCR test using the same device. Then the repeated shear creep loading test was performed. In this study a controlled-stress mode was applied. A constant shear stress of 100Pa and 3200 Pa was used to samples having 25-mm diameter using a 1-mm gap between the platens. The shear loading and unloading were applied for total of 10 seconds i.e. 1 second creep load followed by 9 seconds recovery. The test is started with the application of a low stress 100Pa for 10 creep/recovery cycles, i.e. sample conditioning then another 100Pa 10 creep/recovery cycles were repeated then the stress was increased to 3200Pa and repeated for an additional 10 cycles a total of 30 cycles or 300seconds. MSCR measure non–recoverable creep compliance (Jnr) and percent recovery (PR) by using DSR instrument. Jnr is an indicator of the resistance to permanent deformation of an asphalt binder under repeated load and percent recovery tells us how much the sample returns to its original shape after a load or stress is removed.

Nonrecoverable creep compliance (J_{nr})=$\frac{Unrecovered\ strain}{Applied\ shear\ stress}$

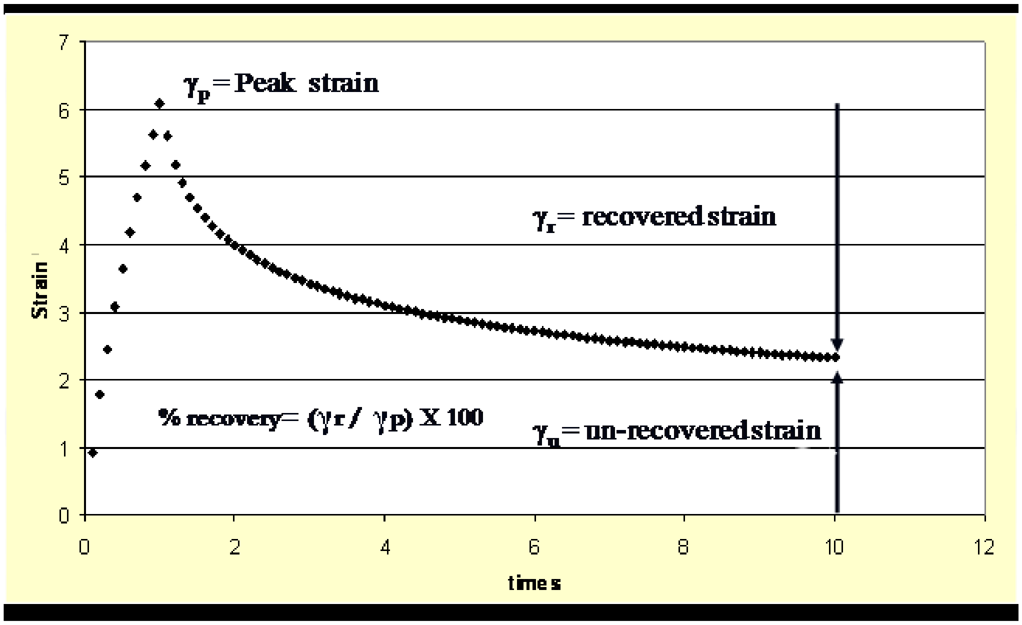

Figure 14 Strain Vs Time for MSCR

3.4 Work Plan

Table 3 Laboratory Work plan for neat and ESP modified binder conventional tests

	Description			Number of Tests		
	Sample	Sample Condition	Activity/Test	Test	Sample replicate	Total no of sample
	Type			Replicate		
1	0%,3%,6% & 9% Egg shell powder content	unaged	Softening point	3	4	12
			Penetration	3	4	12
			Ductility	3	4	12
		RTFO Aged	Penetration	3	4	12
			Ductility	3	4	12
						60

Table 4 Laboratory Work Plan for the rheological tests

	Description			**Number of Tests**		
	Sample Type	**Sample Condition**	**Activity/Test**	**Test Replicate**	**Temp. Replicates**	**No. of Tests**
1	0% egg shell powder	Un-aged	Conventional Tests			9
			AST @ 10, 21.1, 37.8 & 54.4 °C	3	4	12
			PG determination	3	4	12
			FST @ 10,21.1, 37.8 & 54.4 °C	3	4	12
		RTFO Aged	AST @ 10,21.1, 37.8 & 54.4°C	3	4	12
			FST @ 10,21.1, 37.8 & 54.4°C	3	4	12
			PG determination	3	3	9
			MSCR @ 52,58 & 64°C	3	3	9
2	3% egg shell powder	Un-aged	AST @ 10, 21.1, 37.8 & 54.4 °C	3	4	12
			FST @ 10,21.1, 37.8 & 54.4 °C	3	4	12
			PG determination	3	3	9
		RTFO Aged	AST @ 10,21.1, 37.8 & 54.4°C	3	4	12
			FST @ 10, 21.1, 37.8 & 54.4 °C	3	4	12
			PG determination	3	3	9
			MSCR @ 58,64 & 70°C	3	3	9
3	6% egg shell powder	Un-aged	AST @ 10,21.1, 37.8 & 54.4 °C	3	4	12
			FST @ 10, 21.1, 37.8 & 54.4°C	3	4	12
			PG determination	3	2	6
		RTFO Aged	AST @ 10,21.1, 37.8 & 54.4 °C	3	4	12
			FST @ 10, 21.1, 37.8 & 54.4 °C	3	4	12
			PG determination	3	3	9
			MSCR @ 58,64 & 70°C	3	3	9
4	9% egg shell powder	Un-aged	AST @ 10, 21.1, 37.8 & 54.4 °C	3	4	12
			FST @10, 21.1, 37.8 & 54.4 °C	3	4	12
			PG determination	3	3	9
		RTFO Aged	AST @10, 21.1, 37.8 & 54.4°C	3	4	12
			FST @ 10, 21.1, 37.8 & 54.4 °C	3	4	12
			PG determination	3	3	9
			MSCR @ 58,64 & 70°C	3	3	9
					Total Number of Tests	**360**

3.5 Summary

Neat 60/70 penetration grade bitumen was used as a control material and modified with 3%, 6% and 9% egg shell powder by total mass of binder. The modified samples were prepared by mixing in lab at an average temperature of 135-165^{o}c for about 30-40 minutes with manual mix.

Laboratory investigation was conducted to investigate the effect of Egg shell powder on the rheological properties of asphalt binder. Rheological tests were done in Bahir Dar institute of technology in faculty of chemical and food engineering, PG laboratory and conventional tests were done in faculty civil and water resource engineering, highway laboratory and also RTFO test were done in Addis Ababa science and technology university Highway laboratories. The fundamental tests carried out were Amplitude Sweep, Frequency Sweep, Multiple Stress Creep Recovery and Performance Grade Determination test. In addition to this conventional tests' penetration, softening and ductility test for unaged and RTFO Aged binder were performed. These tests were conducted using a DSR instrument.

4. RESULT AND ANALYSIS

This chapter presents the result on the asphalt binder properties change up on addition of egg shell powder. Then the tests results are used to draw conclusions on the performance of asphalt binder on conventional as well as fundamental rheological properties. The chapter will be divided into two main parts: (I) Analysis of conventional properties of both unaged and aged asphalt binder and (II) Analysis of rheological properties of asphalt binder.

4.1 The effect of ESP on conventional property of asphalt binder

4.1.1. Penetration Test Result

Figure 15 shown below shows that the percent proportion of egg shell powder increase the penetration of binder mix become decreases. The higher the egg shell powder content the lower the penetration was. The reason of the reduction of penetration may be free asphalt converts to fixed asphalt (absorbed asphalt) which cause the binder to be stiffen. The decrease in penetration shows the binder become harder and stiffer. Higher values of penetration shows softer consistency and the lower the value the harder the bitumen is. In other words, as ESP content increases, rutting resistance characteristics of the binder also increases. And also, the decrease in penetration value may be helpful in improving the modified binder resistance against effects of temperature.

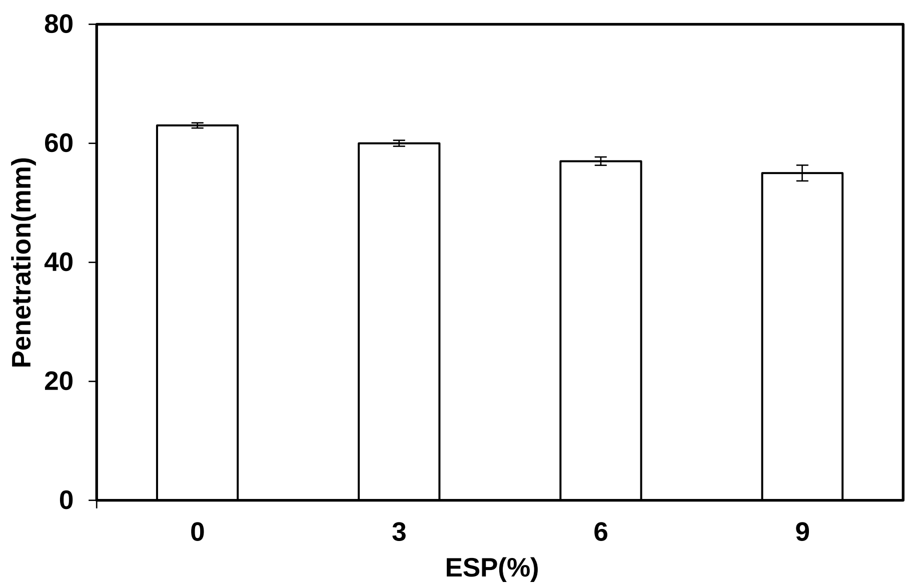

Figure 15: Penetration Test Result

4.1.2 Softening Point Test Result

Softening point test was conducted on unaged samples. As it is seen in the figure16 below, as ESP content increases, softening point temperature also increases. An increase in softening point temperature for the modified binders approves a reduction in the modified binders ability to soften easily under the influence of high temperature conditions, this will make the binders to be more resistance against rutting deformation. Higher softening point shows the lower temperature susceptibility and chosen in warm climates. And it may be less susceptible to permanent deformation or rutting.

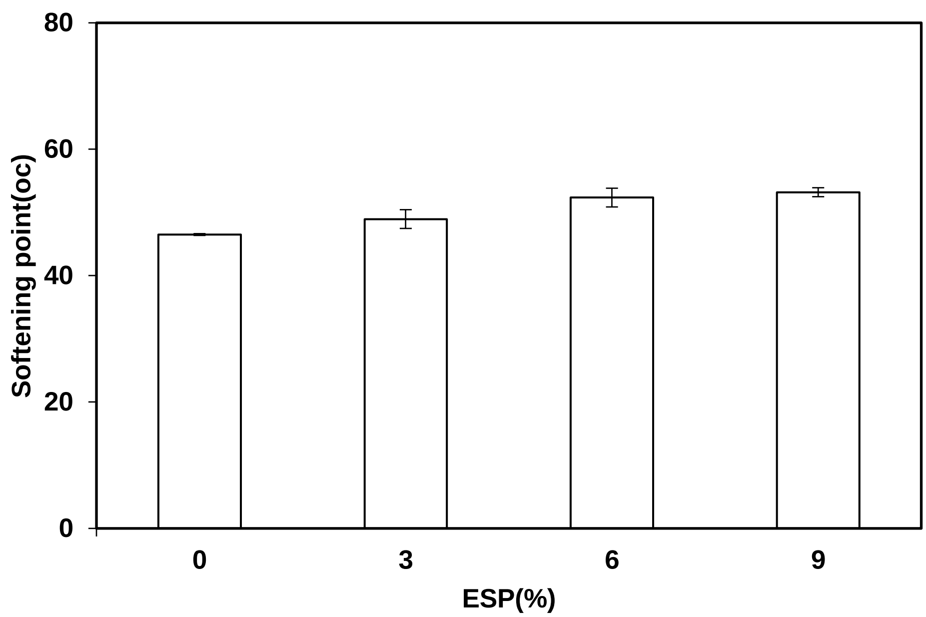

Figure 16: Softening point Test Result

4.1.3 Ductility Test Result

The result of ductility test shows that ductility value decreases as ESP content increases. The decrease in ductility value implies the breaking of the binder rapidly under a standard testing condition. And it is generally considered that a binder with a very low ductility will have poor adhesive property (SAALR.N.J.A, 1955).

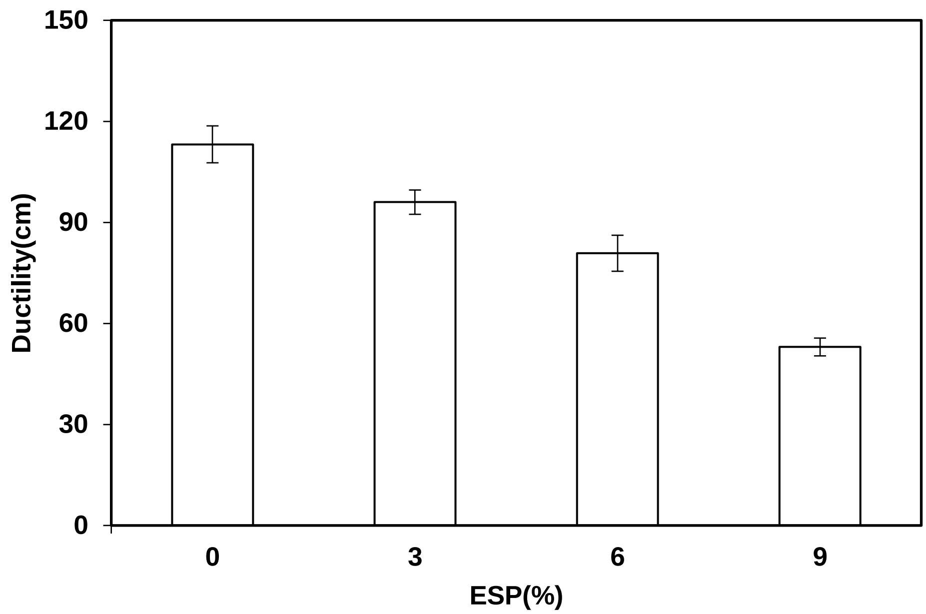

Figure 17: Ductility Test Result

4.1.4 Effect of Aging

After aging penetration and ductility test for each percentage of Egg shell powder were conducted i.e. for neat asphalt binder, 3% ESP, 6% ESP and 9% ESP. After aging the result of penetration result decreased this is because of volatilization and oxidation which makes it stiffer for all mixes but the decrease in penetration value is not similar it has higher variation on the addition of 6% ESP and 9% ESP. The ductility value shows slight decrease in ductility as compared to unaged asphalt binder and up to 6% ESP addition the values are under the desired value which is stated in ERA 2013 but at 9% ESP modified binder ductility values are not under the desired value which is stated in ERA.

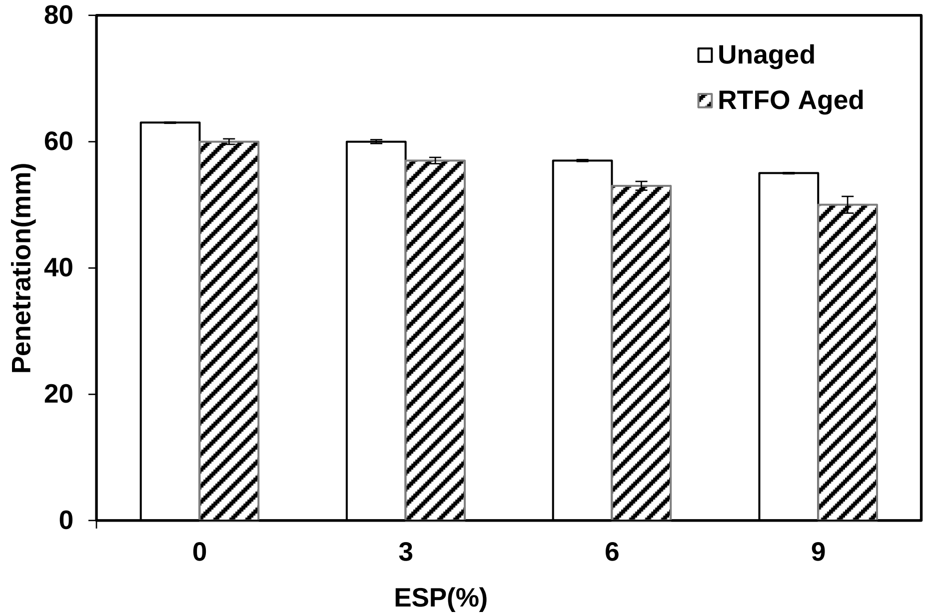

Figure 18 Comparison for penetration between unaged and aged binder

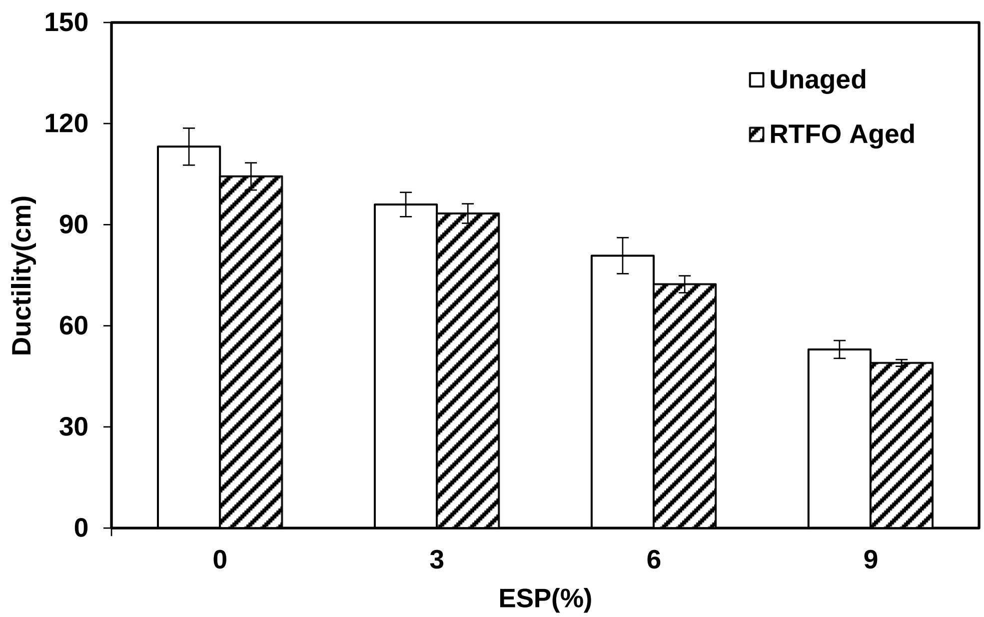

Figure 19 Comparison for ductility between unaged and aged binder

4.2 The effect of ESP on rheological (DSR) Test Results

4.2.1. Amplitude sweep test

The result obtained from amplitude sweep test, linear viscoelastic range, used for as an input for frequency sweep test. Thus, asphalt binder is viscoelastic material which expressed as a function of time and temperature, conducting a test within linear viscoelastic range assures the test repeatable and can be easily correlated with mathematical models. And the results are presented graphically with Log-Log scale of complex shear modulus |G*| in the Y-axis and Strain in percent in the X-axis. The test results are represented graphically in Appendix A. Using AST test results, the linear visco-elastic limit of the linear region was defined as the point at which the measured value of complex modulus decreased to 95 percent of its initial value. So, the limiting stain value would be the point which 0.95G* on the graph. For the purpose of this study, RTFO aged bitumen with highest content of ESP (9%) at lowest temperature (10 ^{o}C) was selected to determine linear visco-elastic range as an input for FST testing. Because of at 9% ESP modified and at the lowest test temperature binder, higher stiffness was attained which make the linear visco elastic range narrower therefore to be within linear visco elastic range for all the tests at 9% ESP percentage strain 0.38 was gained. Therefore, percentage stain up to 0.38% has been selected for both RTFO aged and unaged bitumen.

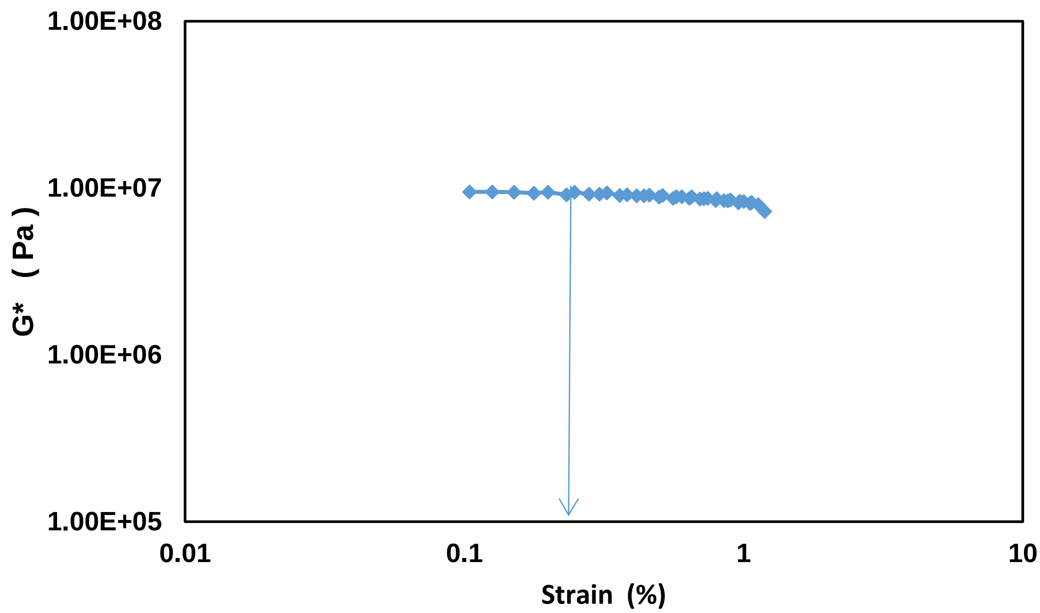

Figure 20: A typical LVE range for 9% aged Egg Shell Powder @ 10°C

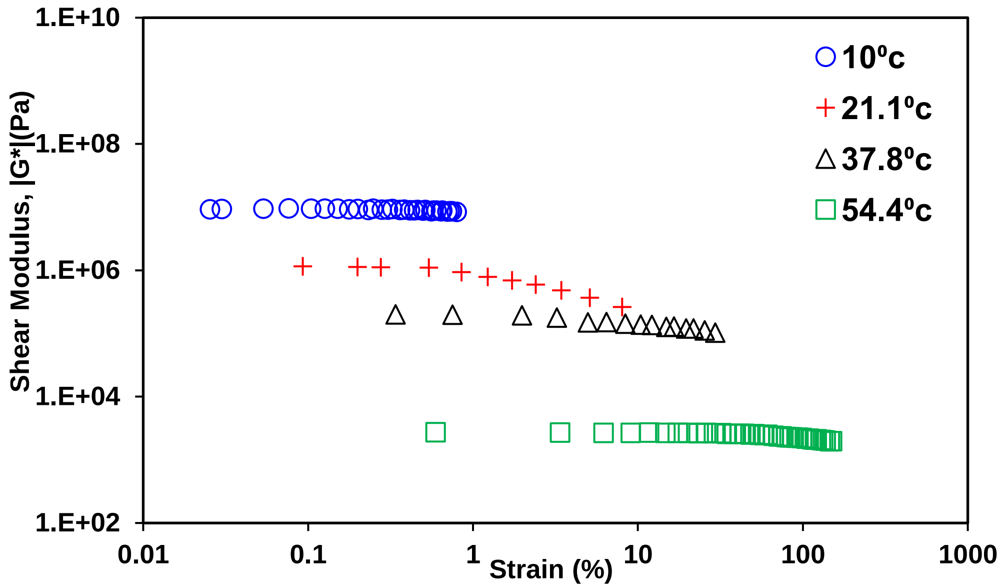

Figure 21: Linear Visco-elastic Range of RTFO Aged 9% ESP Modified

The amplitude sweep test result shows that both temperature and ESP affect the asphalt binder stiffness and strain. As shown in the figure 21 above as the temperature decrease, there is an increase in complex modulus that indicates asphalt binder stiffer at low temperature. As the temperature increase, the stiffness of the asphalt binder decreases, which means the binder become susceptible to temperature, the data shows the reduction of G* due to temperature increase from 10°c,21.1°c,37.8°c, and 54.4°c.This because the bitumen become viscous as temperature increases, this means complex modules value increases with addition Egg Shell Powder and the linear region become narrower which is binder with 9% ESP have highest complex modules value and lowest linear visco-elastic range at constant temperature. On the contrary, the bitumen with lower complex modulus value shows a larger linear visco-elastic range; indicating it is more flexible and has better performance for the lower temperature regions.

Table 5 Summary of limiting strain value

Test Temperature(°C)	Sample (ESP %)	Unaged	RTFO Aged
		Strain in (%)	Strain in (%)
10	3	0.51	0.49
	6	0.65	0.45
	9	0.69	0.38
21.1	3	11.50	11.2
	6	10.00	9.02
	9	5.80	0.55
37.8	3	25.00	23.9
	6	35.00	30.8
	9	4.89	2.47
54.4	3	45.00	32.22
	6	43.25	34.08
	9	38.00	25.54

4.2.2. Frequency sweep test (FST)

Using an input from AST which is LVE-range of 0.38%, FST test was conducted for all temperature (10°C, 21.1°C, 37.8°C, 54.4°C). The frequency varies from 25Hz to 0.1Hz to represent the damaging effect of the traffic load. The major rheological parameters determined from the FST tests are the complex shear modulus(G*) and the phase angle (δ). These parameters were organized in four ways as:

- Black space diagram in semi log graphs for phase angle
- Isothermal plots with log-log scale for complex shear modulus
- Complex modulus master curve
- Phase angle master curve

Black space diagram

A black space diagram is a diagram with a value of the complex modulus (G*), versus the phase angle (δ) acquired from the frequency sweep test. The frequency and the temperature therefore eliminated from the plot which allows all the data of complex modulus and phase angle to be presented in one chart. The black space diagram is used to evaluate the quality of the test data and tells weather the binder is modified or not.

The parameter obtained from FST (complex modulus and phase angle) is manipulated by constructing black space diagram which also evaluate the effect of modifying agent on the binder.

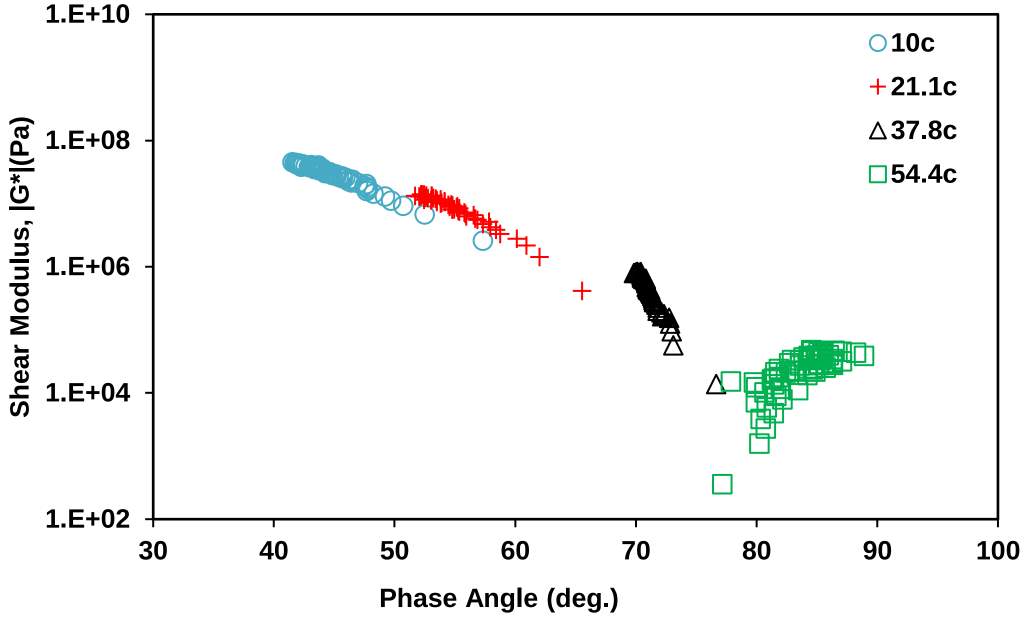

Figure 22 Typical black space diagram @ 9% ESP modified Unaged binder

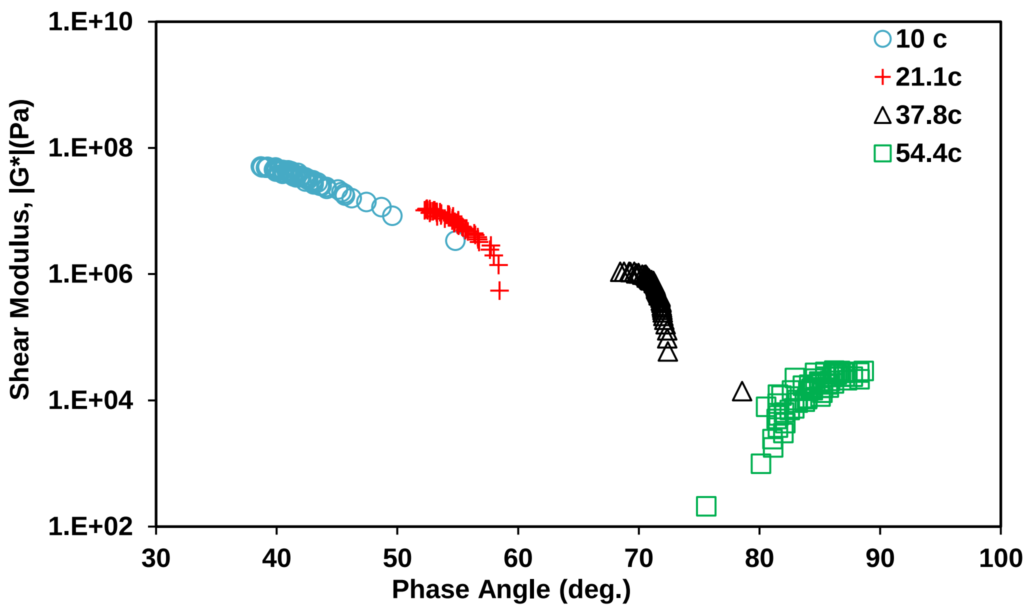

Figure 23 Typical black space diagram @ 9% ESP modified RTFO Aged binder

The figure shown from the above is a black space diagram that is plotted on a semi-log scale with complex modulus in the y-axis and phase angle on x-axis. From the above figure it is possible to conclude that the data point shown in the figure are consistent or the point stay close since the data of each graph was not dispersed (spread) except the test result at temperature of 54.4°C. The reason is that the stress resolution of the DSR for both 8mm and 25mm was different. We have used 25 mm plate diameter for 54.4 °C temperature only. And also, from the diagram, when the temperature increases the phase angle increase and the complex modulus decreases, this indicates that the binder becomes more elastic means that better to resist rutting. On the other hand, when the temperature decreases the phase angle decrease and complex modulus increase, this indicates that the binder turns into more viscous. Using black space diagram, the complex shear modulus as a function of phase angle for all binders were represented in Appendix B.

Isothermal plots

Isothermal plots are the graph between the complex shear modulus versus frequencies at constant temperature. The complex modulus plot in the y-axis and the frequency in the x-axis with the log-log scale. Using isothermal plots, the complex shear modulus as a function of frequency for all binders were represented in Appendix B. See figure 23 below as a sample.

The figure shown below presents the isothermal diagram for 6% RTFO Aged ESP modified binder. We conclude that by simply looking at the isothermal diagram considering temperature, complex shear modulus, frequency and content of modifiers.

- As the temperature increases the shear stiffness decreases for all the binders and as the temperature decreases the complex modulus increases that indicates the increment of stiffness in contrast when the temperature increases the stiffness of the binder decrease.
- The increase in content of ESP increases the complex modulus
- When the frequency increases the complex modulus increases, this indicates that the loading rate has an effect on the stiffness of the binder.

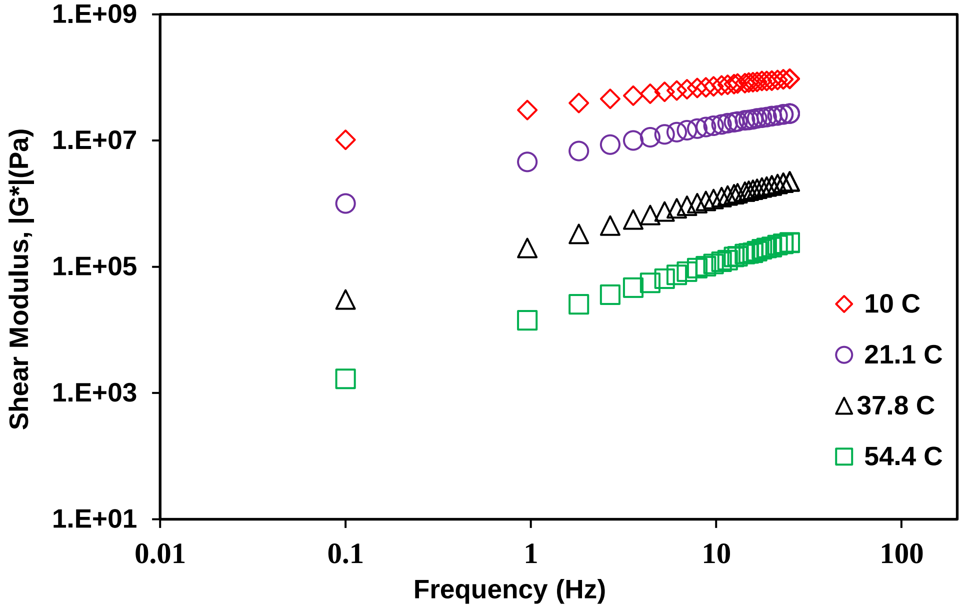

Figure 24 Isothermal plots of 6% ESP modified Unaged binder

Complex modulus master curve

FST data results are mainly used for constructing Master Curve using time- temperature superposition principle, the change in complex modulus and phase angle can be investigated and shifted to a defined reference temperature, in this case 21.1^{O}C. The data at any other temperatures were shifted with respect to time until various curves overlap almost perfectly to form a single master curve using time-temperature superposition principles. Binder master curve graph extrapolate the test result up to very high frequency and very low.

To construct master curves, horizontal shifting was used which is based on frequency and temperature equivalency. The temperature dependency of the visco-elastic behavior of bitumen is represented using shift factors and expressed as

$$a(T) = \frac{f}{fr} \tag{1}$$

Where: a(T)= is the shift factor as a function of temperature,

f =is reduced frequency at the testing temperature

f_r =is the reduced frequency at the reference temperature

T=temperature of interest (Thakre N, Mangrulkar D, Janbanghu M, & Saxena J, 2016).

The shifting was done based on visco-elastic function like |G*| and δ. The temperature shift factors fitted by a certain function like Williams-Landel-Ferry (WLF) functions. In this study WLF function as shown by equation below was used to estimate the shift factors

$$\log a(T) = \frac{-C1(T-TR)}{C2+(T-TR)} \tag{2}$$

Where, C_1 and C_2 are empirically determined constants;

T is the test temperature in ^{o}C; and

T_R is reference temperature in ^{o}C.

The master curve for binders can be represented by a sigmoidal function defined by equation frequency indicating the whole transition of asphalt binder from glassy region to fluid viscous. For the purpose of these research the master curves is constructed fitting a sigmoidal function to the measured complex modulus test data using nonlinear least squares regression, which can be done using the Solver Function in the Excel spreadsheet. The shifting could be done by solving shift factors simultaneously with the coefficients of the sigmoidal function, using any available shifting function to solve reduced frequency (f_r) as a function of temperature.

$$\text{Log}/G*/= \delta + \frac{\alpha}{\left(1+e^{\beta+\gamma(\log fr)}\right)} \tag{3}$$

Where: G* = Complex shear Modulus as a function of frequency and temperature,

δ = Minimum complex shear modulus of the asphalt binder,

α = Difference between Maximum and Minimum complex shear modulus of the binder,

f_r = Reduced frequency (Hz) at reference temperature,

ß,γ = S-shaped function parameters depend on characteristics of asphalt binder and describes the shape of sigmoid functions,β = describes the point of the turning curve half way between minimum and maximum value of G* or horizontal position of the turning point, and γ describes the steepness of the family of curves (slope of the curve) (Yuosoff N.I.MD, Chailleux E, & Airey G.D, 2011).

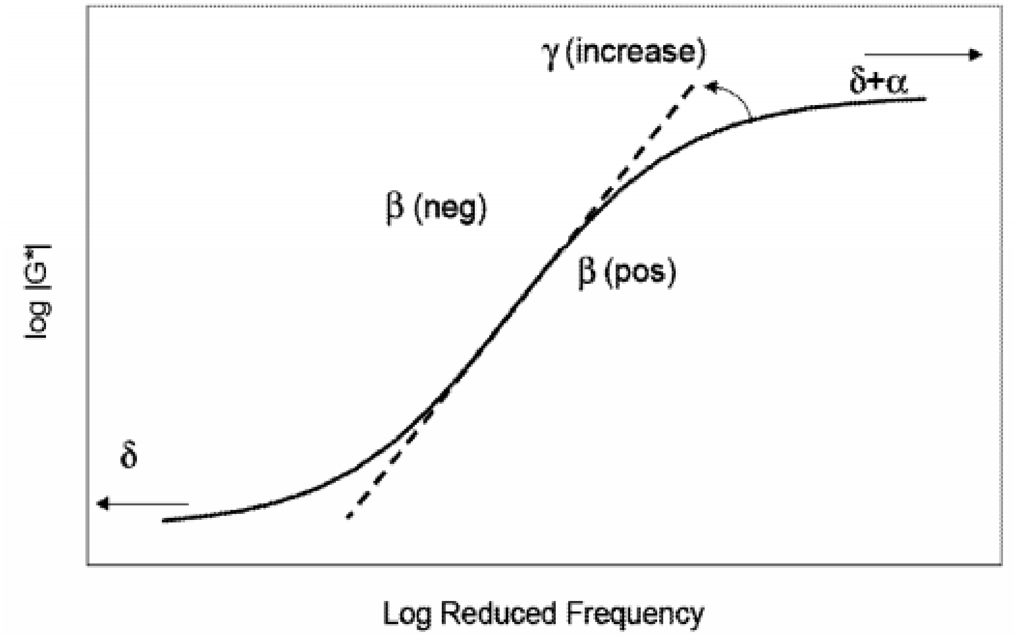

Figure 25 Definition of Sigmoid Function *(Yuosoff N.I.MD, Chailleux E, & Airey G.D, 2011)*.

In this study reference temperature of 21.1°C was used for constructing a master curve, time-temperature superposition was employed. Complex shear modulus values measured at 10 °C, 21.1 °C, 37.8 °C and 54.4 °C temperatures were plotted as the logarithm of the shear modulus versus the logarithm of frequency. And the resulting data were shifted horizontally to form a master curve. Shift factors were calculated to transfer the data from 10, 37.8 and 54.4°C to 21.1°C. Shift factor helps in moving the curves plotted at different temperatures to a reference temperature. Temperature shift factors and sigmoid coefficients of complex modulus master curve for all binders are listed below in the table 6 which have been developed and analyzed by using Microsoft excel related to the solver function.

Table 6 Shift factors for complex modulus master curve for RTFO Aged and unaged binder

Aging Condition	ESP (%)	α	β	Δ	γ	a10	a21.1	a37.8	a54.4
Unaged	0	11.993	-2.060	-2.906	0.230	1.507	0	-1.640	-3.348
	3	22.615	-2.502	-5.702	0.159	1.456	0	-1.420	-3.245
	6	6.391	-1.280	-0.493	0.582	1.315	0	-1.982	-2.538
	9	31.462	-3.120	-22.596	0.206	2.684	0	-1.420	-2.148
RTFO Aged	0	11.903	-1.924	-2.895	0.242	0.391	0	-1.063	-3.391
	3	31.498	-2.385	-10.823	0.210	1.414	0	-1.762	-4.292
	6	21.834	-2.374	-5.792	0.150	1.462	0	-1.798	-3.931
	9	32.028	-21.834	-21.834	0.136	0.56	0	-2.637	-4.254

Figure 26 and Figure 27 below present the complex modulus master curves for unaged and aged binders.

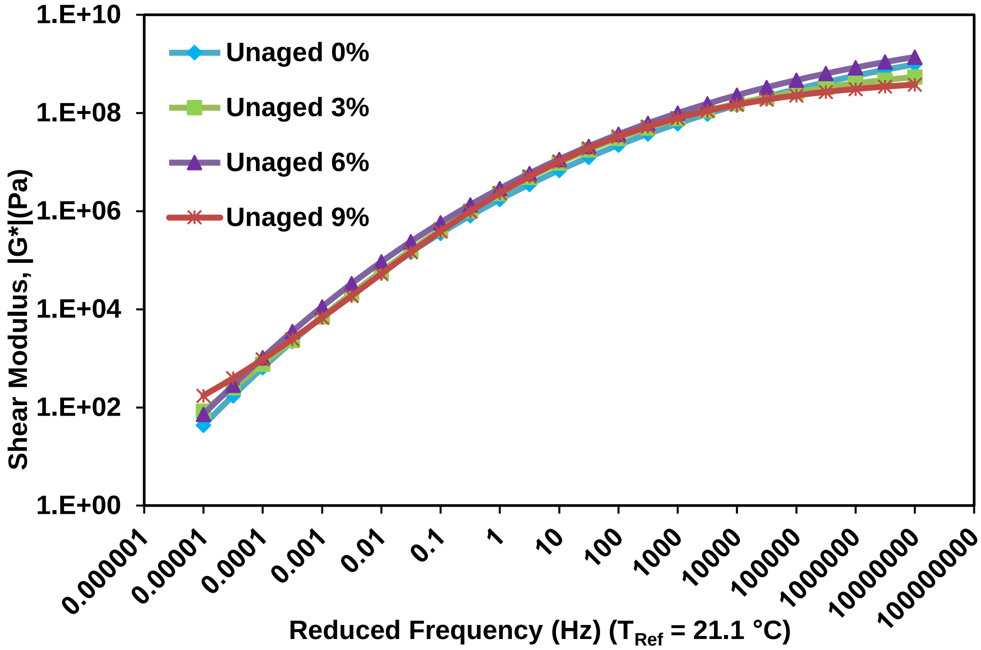

Figure 26 Complex modulus master curve for unaged binder mix

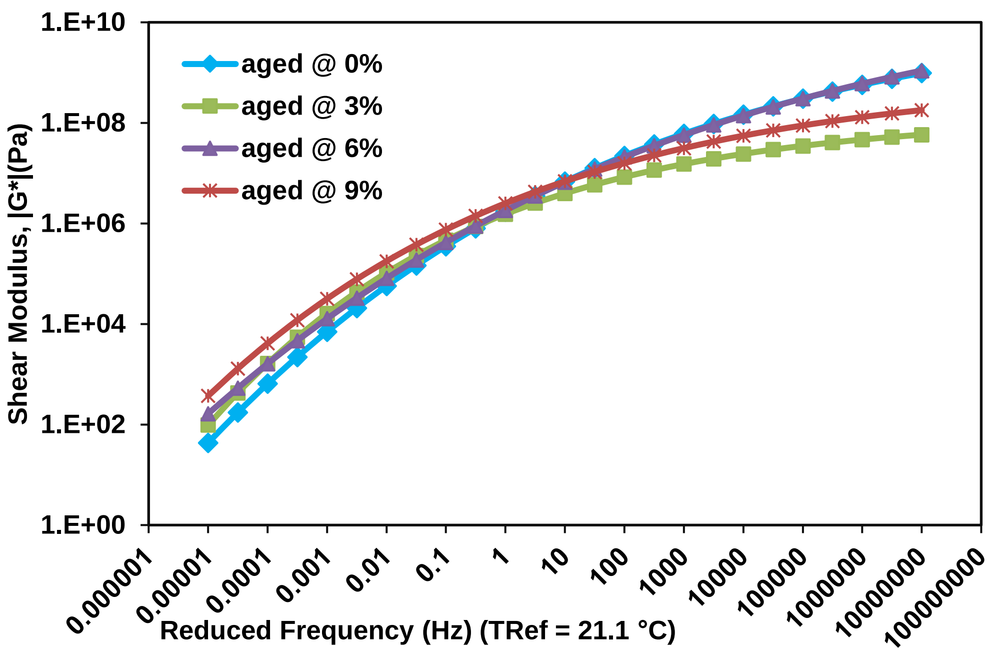

Figure 27 complex modulus master curve for RTFO Aged binder mix

The above figures 26 and 27 shows the complex shear modulus master curve for all temperatures and both unaged and RTFO aged condition of asphalt binder.

For both master curve it is observed that at low frequency and high temperature, the complex modulus becomes low for all percent of ESP. This is because the increment in temperature makes the asphalt binder less stiff which is susceptible to deformation, and the decrement of frequency indicates low-stress application of load, which has a higher damaging effect than high frequency.

The quantity of modifiers also affects the complex modulus value. It can be seen that for unaged binder mix at high temperature and low loading frequency addition of 6 percentage of egg shell powder have highest stiffness as compared to other, this implies the addition of modifier improves stiffness of the binder. Which makes better resistance to rutting. And for RTFO Aged binder mix the addition of modifier is appreciably increase stiffness than unaged binder mix. And at high temperature and low loading frequency addition of 9 percentage of egg shell powder have highest stiffness as compared to other binder mix. Also, for all binders in almost similar pattern shear stiffness decreases as temperature increases. In general, the modifier improves the complex shear modulus of the neat binder at higher temperatures. Rutting is a serious problem at high temperature due to slow moving traffic and as observed the modifier improves the pavement performance against rutting by increasing the stiffness of the binder.

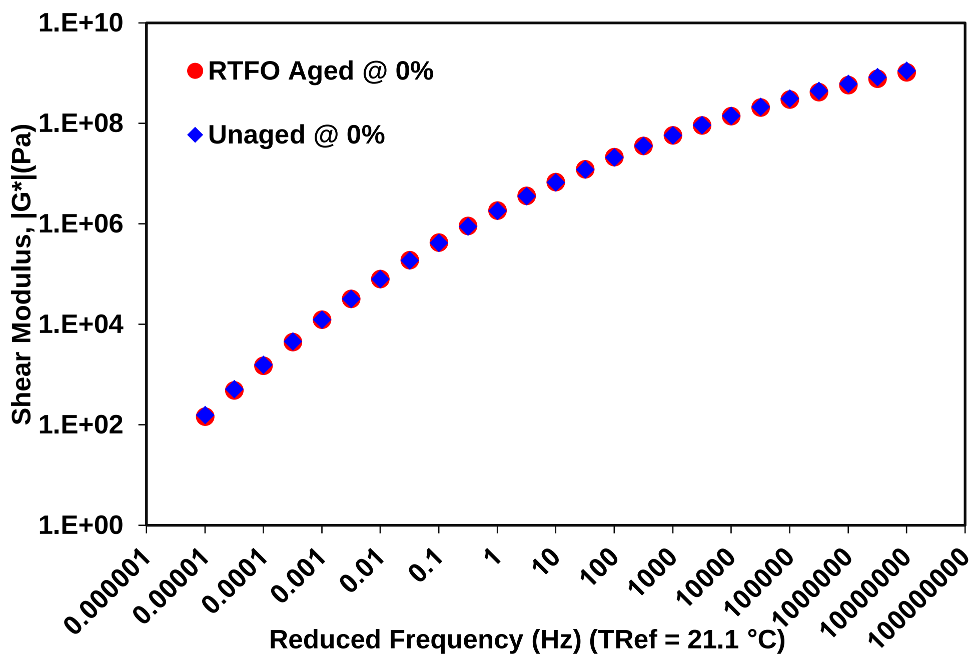

Figure 28 Shear modulus master curve neat binder for Unaged and RTFO Aged

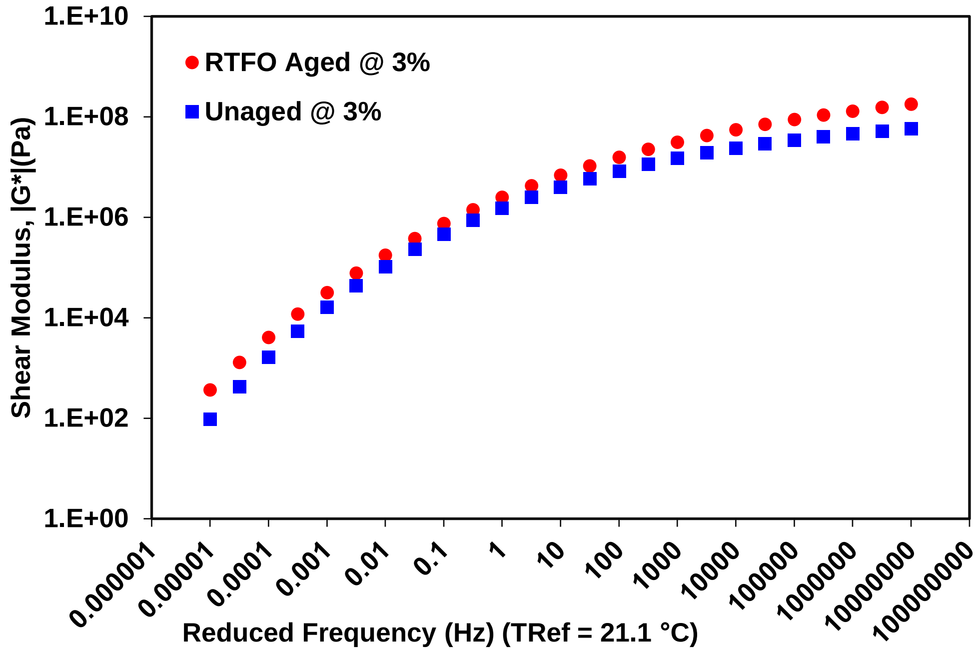

Figure 29 Shear modulus master curve 3% ESP modified binder

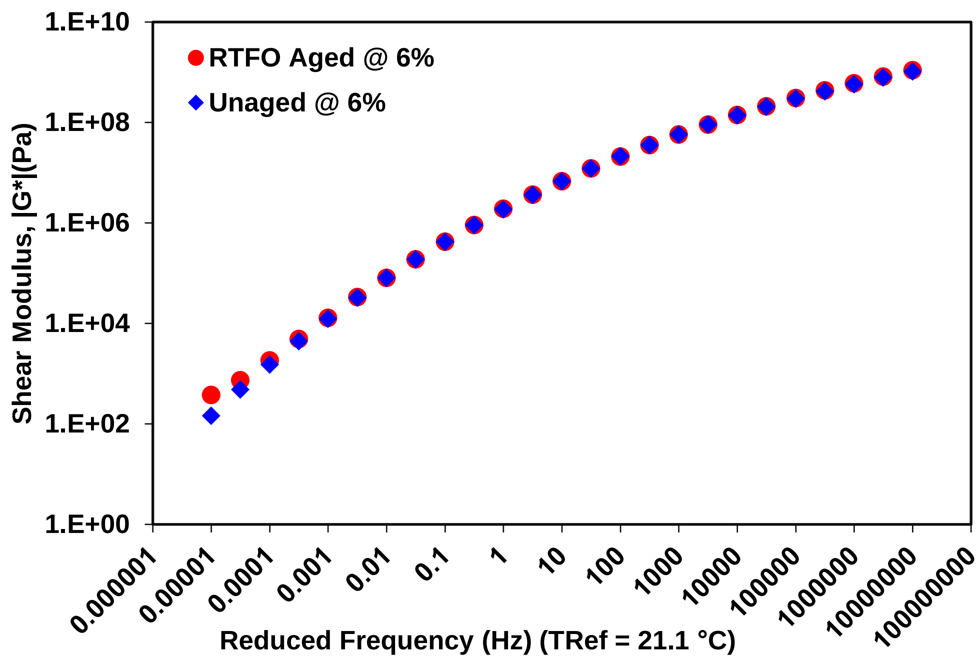

Figure 30 Shear modulus master curve 6% ESP modified binder

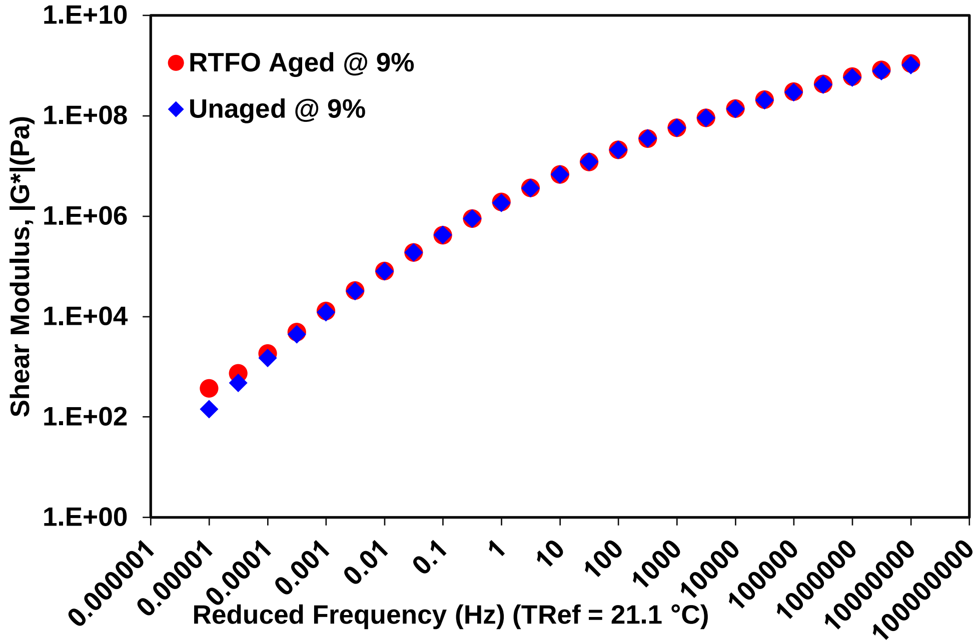

Figure 31 Shear modulus master curve 9% ESP modified binder

As we have seen from the above figure 28 to 31**,** RTFO Aged has increased shear modulus at low frequency and high temperature. For all neat and ESP modified binder, when the bitumen becomes RTFO Aged the master curve proves that it has better rutting resistance ability than Unaged asphalt binder.

Phase angle master curve

In similar style phase angle master curve can be constructed using the same idea as complex modulus master curve. Using the excel solver the sigmoid function parameters were obtained and tabulated below in table 7 including the temperature shift factors. All phase angle master curves are presented in Appendix 6.4

$$\delta = 90 * \alpha\beta \frac{e^{(\gamma+\beta(\log fr))}}{1+\left(1+e^{\gamma+\beta(\log fr)}\right)^2} \quad (4)$$

Where: α, β and γ: Sigmoid function constants,

f_r: Reduced frequency,

δ: Phase angle

By using Microsoft excel the obtained shifting factors and sigmoid function coefficients are presented in the figure below.

Table 7 Shift factors for Phase angle master curve for aged and unaged binder

Aging Condition	ESP (%)	A	β	γ	a10	a21.1	a37.8	a54.4
Unaged	0	12.803	1.215	0.189	1.985	0	-2.456	-5.283
	3	15.236	1.267	0.233	2.197	0	-2.569	-45.297
	6	15.710	1.300	0.205	2.198	0	-3.215	-7.452
	9	15.299	1.254	0.154	2.259	0	-3.118	-7.023
RTFO Aged	0	12.358	1.101	0.174	2.023	0	-2.356	-5.052
	3	15.996	1.305	0.258	2.206	0	-3.110	-7.692
	6	16.258	1.293	0.098	2.299	0	-3.217	-7.905
	9	16.568	1.322	0.154	2.368	0	-3.292	-8.889

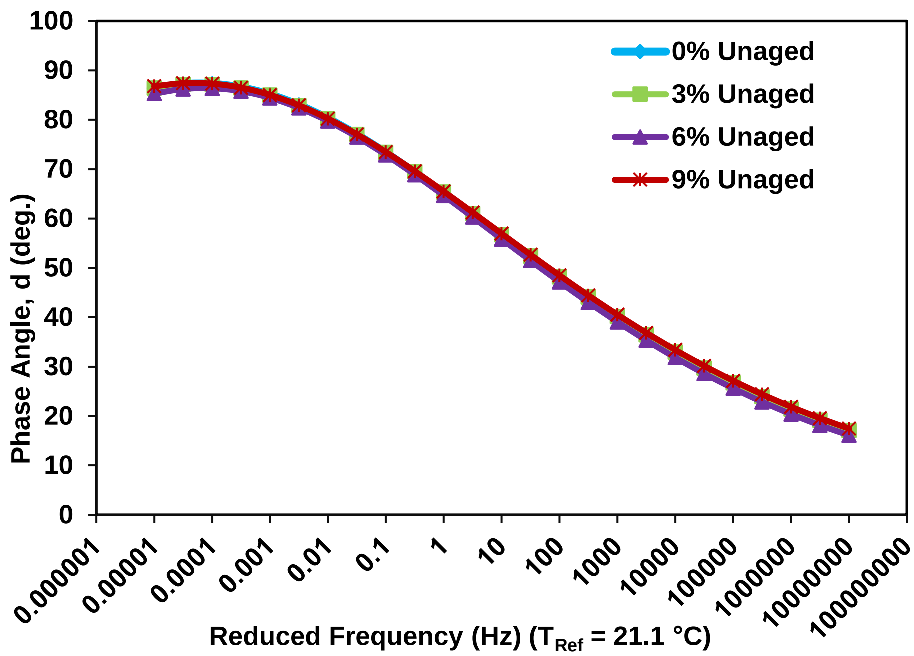

Figure 32 Phase angle master curve for Unaged binder mixes

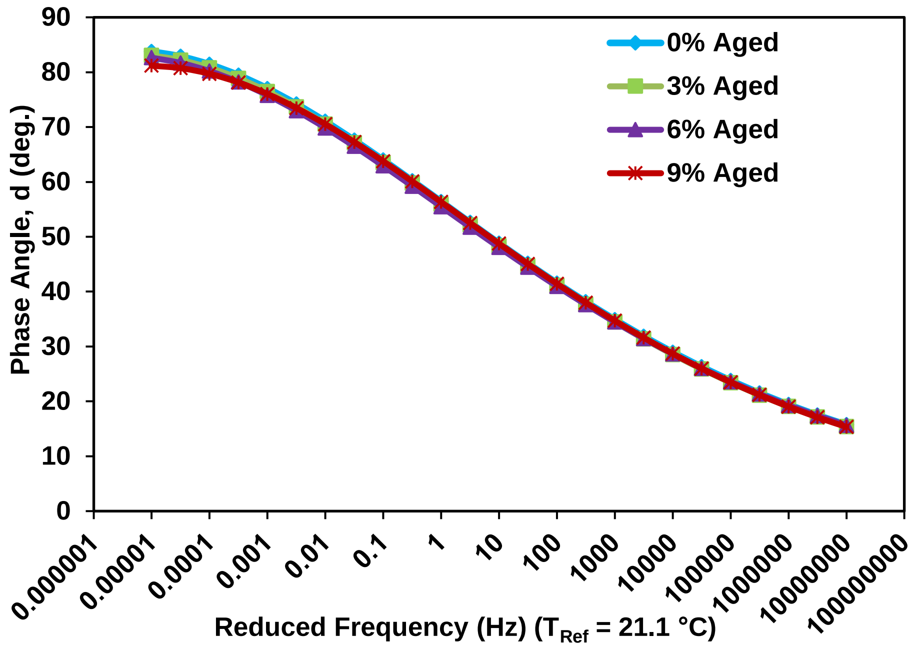

Figure 33 Phase angle master curve for aged binder mixes

As we have seen from the figure 32 and 33 above, at high temperature and low loading conditions binder containing egg shell powder have lower phase angle than neat binder. This makes the binder stiff and elastic to prevent rutting. With the increased in percent modifier at low frequency and high temperature, the value of phase angle tends to decrease relative to the neat binder, which implies the elastic property of the binder improves.

4.2.2.1 Statistical analysis

Having the above FST test result data, statistical analysis was done to evaluate the significance of addition of ESP on stiffness as well as the effect of aging. The statistical analysis F test is using to test a hypothesis concerning the means of the group on the dependent variable, the technique is called analysis of variance (commonly abbreviated as ANOVA).

The effect of egg shell powder and loading rate on stiffness of the binder was performed. This consists of two independents variable ESP content and reduced frequency and one dependent variable complex modulus. The null hypothesis (Ho) alternate hypothesis (H1) as follow:

- H0: There is no interaction effect between loading rate and egg shell powder content on stiffness of binder.
- H1: There is an interaction effect between loading rate and egg shell powder content on stiffness of binder.
- H0: There is no difference in means of binder stiffness using different loading rate.
- H1: There is a difference in means of binder stiffness using different loading rate.
- H0: There is no difference in means of binder stiffness using different proportion of egg shell powder
- H1: There is a difference in means of binder stiffness using different proportion of egg shell powder

Testing of the hypothesis is done at 0.05 level of significance, at three frequencies (25, 10 and 0.1 Hz) and different aging condition. Details of ANOVA hypothesis testing results are presented in Appendix C. The ANOVA test results are as follows:

- From hypothesis test result of reduced frequency and ESP content, P-value = 0.000 is less than the given significant level α= 0.05 there is difference in mean. Reject the null hypothesis so, the rate of loading, egg shell powder and interaction of them have a significant effect on stiffness of the binder.
- And multiple comparisons test result for reduced frequency by Tukey HSD indicates, the P-value = 0.000 is lower than the significant level 0.05, there is a significant effect of loading rate on asphalt binder stiffness in 95% confidence interval.
- And also, the multiple comparisons test result of ESP content by Tukey HSD indicates, the P-value = 0.845 which is higher than the significant level 0.05, fail to reject the null hypothesis means addition of 9% ESP have no significant effect on asphalt binder stiffness in Unaged condition at 95% confidence interval.
- More over from hypothesis test result of aged condition and ESP content, the P-value = 0.000 is less than the given significant level α= 0.05, there is difference in mean. Reject the null hypothesis so; RTFO Aged, egg shell powder and interaction of them have a significant effect on stiffness of the binder at 10Hz reduced frequency.

4.2.3. Performance Grade Determination

To determine the performance grades of the given samples, high temperature oscillatory DSR test was employed. According to the standard test procedure, AASHTO T 315, 12% strain for original and 10% strain for RTFO aged samples were used. The performance grade of the binder is designated as PG xx-yy, where xx represents the average seven days maximum temperature often called as "high temperature grade." This means that the binder would have sufficient physical properties to resist rutting at least up to xx °C and -yy ^{o}C represents the minimum temperature often called as "low temperature grade'' and it means that the binder would have sufficient physical properties to resist thermal cracking at least down to –yy °C (Hafeez I, et al., 2013).

PG determination test is conducted at high temperature with a 6^{o}C temperature increment. If the sample passes the first selected temperature, then it will continue to test for the next adjusted temperature. The final rheological parameter G*/sinδ, was believed to indicate the resistance of asphalt binder for permanent deformation at high environmental temperature before MSCR test was discovered. This study uses the parameter G*/sinδ to identify the maximum temperature that the asphalt binder could meet the minimum criteria of AASHTO M-320.

4.2.3.1Analysis and Evaluation of PG Determination

The objective of this study was set by using available laboratory equipment to evaluate ESP modified bitumen performance at high temperature only. There is no pressure aging vessel, bending beam Rheometer and direct tension testing equipment for low temperature binder characterization.

The result shows that improvement of performance grade from PG58-YY to PG64-YY for both unaged and RTFO aged bitumen with the additions of 3% ESP. This means that the binder would have sufficient physical properties to resist rutting at least up to 64°C.The higher the performance grade implies the stiffer the binder and the more rut resistance. With all the modifier contents (3%,6%, and 9%) the effect is the same for the PG grade.

Table 8 Determining High Temperature Performance Grade (AASHTO M 320)

ESP (%)	Unaged	RTFO Aged	AASHTO M320 PG Category
0	58	58	PG 58-yy
3	64	64	PG 64-yy
6	64	64	PG 64-yy
9	64	64	PG 64-yy

Table 9 PG test result of Unaged binder

ESP (%)	Test Temperature (°C)	G*/sinδ (KPa)	Remark	Pass-Fail Temperature (°C)	PG
0%	52	4.38	Pass	62.3	58-YY
	58	1.71	Pass		
	64	0.72	Fail		
3%	58	4.14	Pass	65.3	64-YY
	64	1.91	Pass		
	70	0.73	Fail		
6%	58	4.84	Pass	65.95	64-YY
	64	2.34	Pass		
	70	0.75	Fail		
9%	58	5.28	Pass	68.45	64-YY
	64	2.51	Pass		
	70	0.93	Fail		

Table 10 PG Test results of RTFO Aged binder

ESP(%)	Test Temperature (°C)	G*/sinδ (KPa)	Remark	Pass-Fail Temperature (°C)	PG
0%	52	5.79	Pass	63.8	58-YY
	58	4.14	Pass		
	64	1.96	Fail		
3%	58	9.03	Pass	66.05	64-YY
	64	3.07	Pass		
	70	1.85	Fail		
6%	58	9.71	Pass	66.9	64-YY
	64	4.57	Pass		
	70	1.88	Fail		
9%	58	9.91	Pass	69.2	64-YY
	64	4.37	Pass		
	70	1.86	Fail		

As shown in table 9 and 10 above it can be seen, both before and after RTFO aging, the modified binders have higher G*/sinδ for ESP modified binders at different temperatures. As can be seen ,both before and after RTFO aging, the modified binders have higher G*/sinδ values compared to the control binders, this indicates that modified binders will have higher resistance to rutting deformation since G*/sinδ indicates better rutting resistance .And also the performance grade of base asphalt binder upgrade due to the addition of ESP on both RTFO aged and Unaged binders. The higher the performance grade implies the stiffer the binder and the more rut resistance. For unaged and RTFO Aged binder the PG of neat binder was PG 58-YY (YY indicates the low temperature performance of the binder) with pass-fail temperature of 62.3 and 63.8 respectively, when ESP added to the neat binder by the amount of three different percentage (3%,6%, and 9%) the PG values also increase to 64-YY same of all modified binder with the increment of pass-fail temperature with the fulfilling the minimum criteria of G*/sinδ. Moreover, the addition of 9% of ESP has maximum effect relating to the resistance of rutting relative to the other percentage by observing the pass-fail temperature for both unaged and RTFO Aged binder.

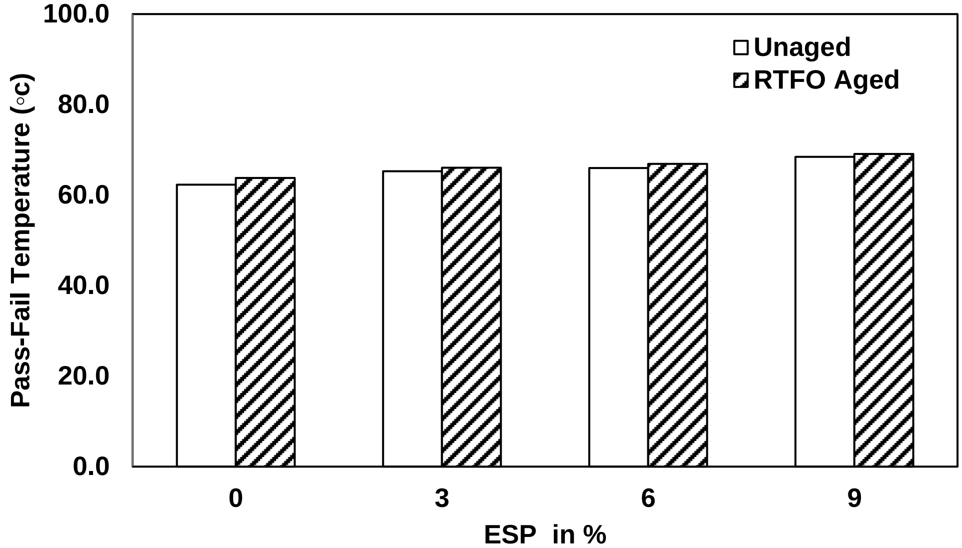

Figure 34: PG Pass- Fail Temperature for Comparison

As shown in the figure 34 above aging has an effect on the pass-fail temperature with regard to the addition of ESP compared to unaged modified binder. Even if the modified binder improves the PG grade in one grade (from 58-YY to 64-YY for both Unaged and RTFO Aged binder), the pass-fail temperature on each modified binder is different from each other. Additionally, the aged binder has a high pass-fail temperature than that of an Unaged binder which evidently implies stiffer to resist the permanent deformation.

4.2.4 Multiple stress creep recovery (MSCR)

MSCR test is used to evaluate permanent deformation resistance of asphalt binders under varying stress level. The repeated shear creep test is performed using the Dynamic Shear Rheometer (DSR) by applying a controlled shear stress (100 and 3200Pa) using a load for 1 second followed by a 9-second rest period. During each cycle, the asphalt binder reaches a peak strain and then recovers before the next cycle stress is applied again. It gives very important practical information regarding asphalt binder. Since flexible asphalt pavements are designed to be flexible, they must quickly return to their original configuration after removal of loading.

For the purpose of this study, repeated shear creep testing was conducted at four temperatures 52°, 58°, 64° and 70 °C after determination of performance grade for each binder mix. Test result for Performance Grade determination is presented on Appendix F.

Table 11 MSCR Test Temperature based on PG

ESP (%)	PG	Test temperature(^oC)		
0	58	52	58	64
3	64	58	64	70
6	64	58	64	70
9	64	58	64	70

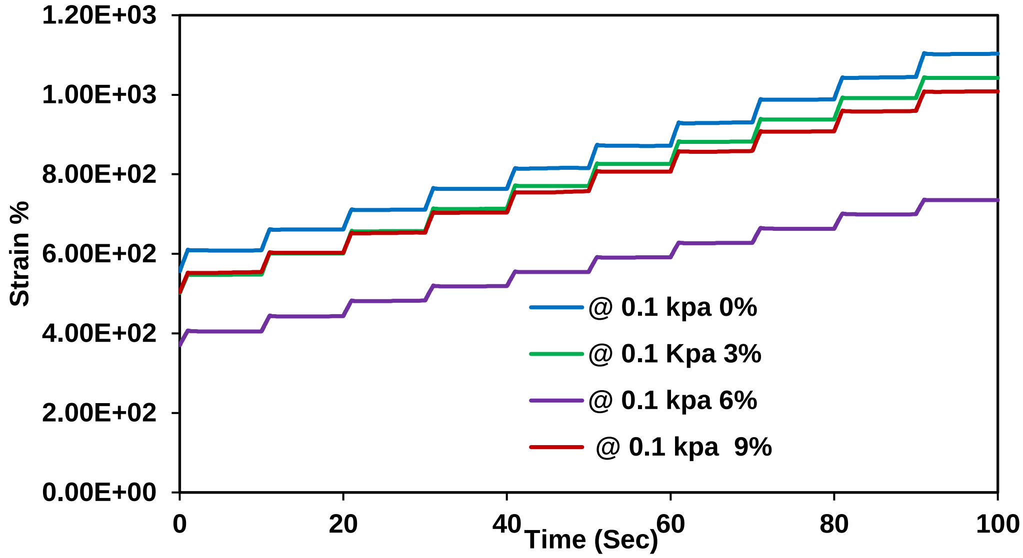

Figure 35: Effect of ESP on strain @ 0.1 kpa (64^0C)

Figure 35 shows the plot of total strain recorded as a function of time. The total strain was influenced by the addition of egg shell powder. It can be shown that as the ESP increase the total strain value decreases except for 9% ESP. Thus, using ESP as a modifier is effective to improve stiffness by decreasing the strain based on the result of MSCR test. This showing improvement on rutting performance behavior. The smallest total strain was gained from 6% followed by 9% and 3%. The addition of 6% is effective in improving rutting resistance. This indicates a favorable effect of addition of ESP on permanent deformation resistance of asphalt binder.

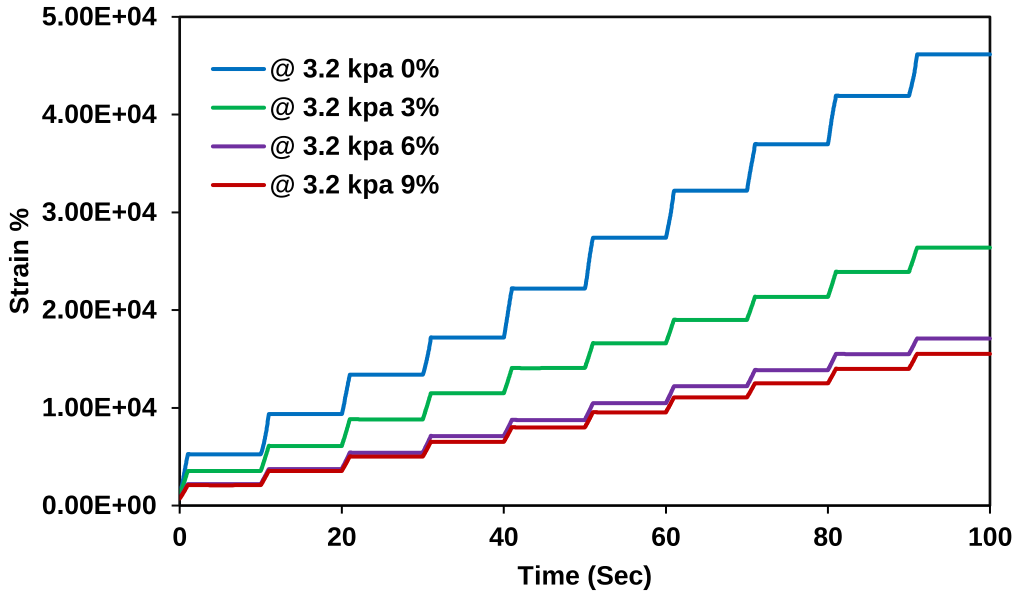

Figure 36 Effect of ESP on strain @ 3.2 kpa (64^0C)

Figure 36 shows that as the ESP percentage increases the total strain value decreases showing improving behavior. This indicates that a favorable effect of addition of ESP on permanent deformation resistance of asphalt binder. The smallest total strain values were obtained for ESP of 9%, followed by the 6% and 3% ESP. However, level of improvement after addition of 6% ESP is not significant and at the same time to be economical.

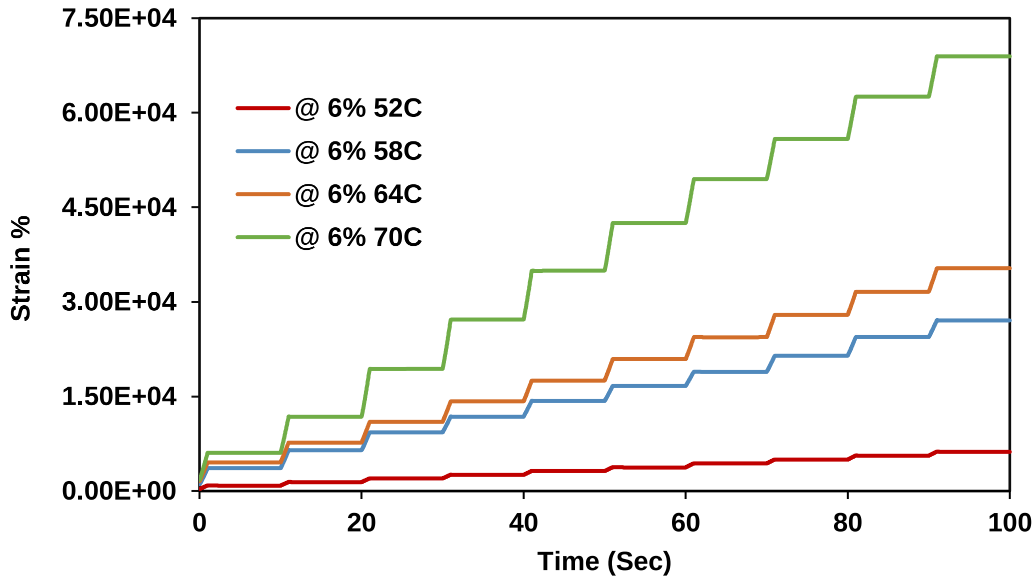

Figure 37 Effect of temperature on MSCR for 6% ESP

Figure 37 shows that total strain affected by test temperature used, which increases as the temperature increases rutting resistance characteristics of a binder decreases because the bitumen becomes viscous. The smallest total strain gained at a test temperature 52^0c followed by the other three temperatures.

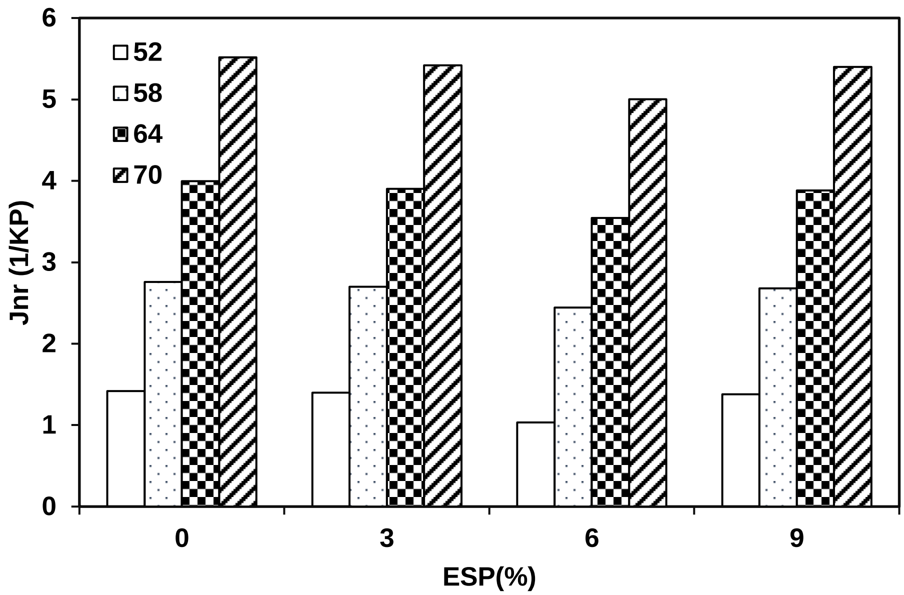

Figure 38 Non recoverable creep compliance (Jnr) @ 0.1 K pa

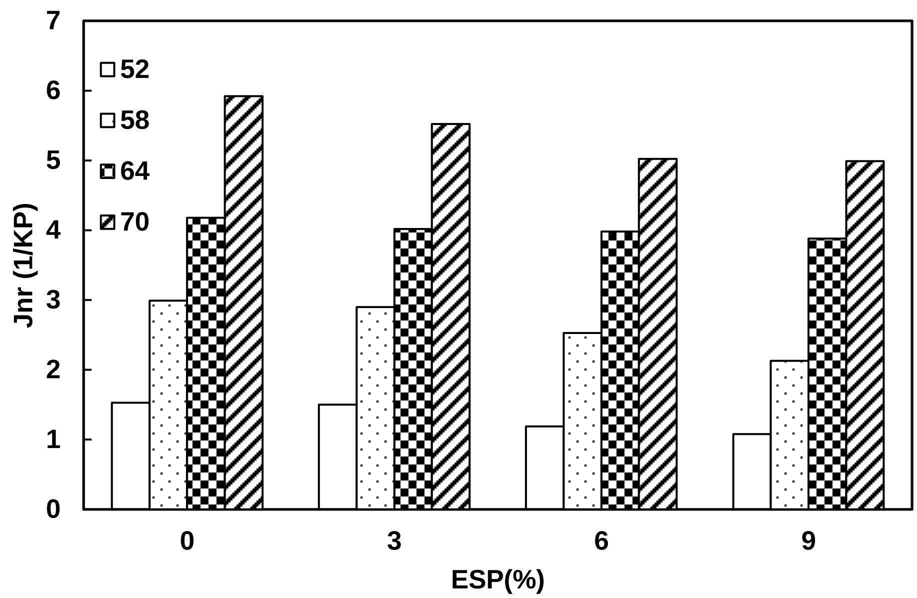

Figure 39 Non recoverable creep compliance (Jnr) @ 3.2Kpa

It is shown that the rutting parameter (J_{nr}) decreases as the percentage of ESP increase. From all, asphalt binder with neat binder has a higher Jnr value, as the amount of ESP binder increases the J_{nr} value decreases, this shows that an improvement of the resistance of modified binder to permanent deformation. The reason behind this is that the nonrecoverable creep compliance (J_{nr}) refers to the ratio between accumulated strain and applied stress which is an indicator of rutting resistance. And non-recoverable compliance (J_{nr}) increases as the temperature increases for both loading condition (3.2 and 0.1 Kpa). This is due to the behavior of asphalt binder is more viscous at higher temperatures. Which results a higher permanent strain under stress. Especially the addition of 6% have lower Jnr value as compared to 3% and 9% ESP. This means that addition of ESP could improve the resistance of asphalt pavements to rutting. Lesser Jnr indicates that higher strains are recovered during the recovery phase of MSCR test moreover lower Jnr shows that the binder is less prone to the accumulation of permanent strain in the field. As a result, the role of ESP to the resistance of the asphalt to rutting is greater.

4.2.5 Statistical analysis

Based on the laboratory procedures and data collection described in this study, the above MSCR results were gained using those data statistical analysis was performed to evaluate the significance of addition of ESP on non-recoverable creep compliance. The statistical analysis F test is using to test a hypothesis concerning the means of the group on the dependent variable; the technique is called analysis of variance (commonly abbreviated as ANOVA). Hypothesis testing was done at 0.05 significance level with three test temperatures (52,58, 64, and 70°C). Details of ANOVA hypothesis testing results are presented in Appendix C.

From hypothesis test result of test temperature and ESP content, P-value = 0.000 is less than the given significant level α= 0.05 there is difference in mean. Reject the null hypothesis so; the test temperature, Egg shell powder and interaction of them have a significant effect on non-recoverable creep compliance of the binder at 0.1 kpa stress level. Also, multiple comparisons test result for test temperature by Tukey HSD indicates, the P-value = 0.000 is lower than the significant level 0.05, there is a significant effect of test temperature on asphalt binder non-recoverable creep compliance in 95% confidence interval 0.1 kpa stress level. In addition, the multiple comparisons test result of ESP content by Tukey HSD indicates, the P-value = 0.582 which is higher than the significant level 0.05, fail to reject the null hypothesis means addition of 9% ESP have no effect on asphalt binder non-recoverable creep compliance at 0.1 kpa stress level with 95% confidence interval. However, additions of ESP content at 6% and 3% have a significant effect on the non-recoverable creep compliance of binder as the P-value indicates.

From hypothesis test result of test temperature and ESP content, P-value = 0.000 is less than the given significant level α= 0.05 there is difference in mean. Reject the null hypothesis so; the test temperature, Egg shell powder and interaction of them have a significant effect on non-recoverable creep compliance of the binder at 3.2 kpa stress level. And also multiple comparisons test result for test temperature by Tukey HSD indicates, the P-value = 0.000 is lower than the significant level 0.05, there is a significant effect of test temperature on asphalt binder non-recoverable creep compliance in 95% confidence interval 3.2 kpa stress level.in addition the multiple comparisons test result of ESP content by Tukey HSD indicates, the P-value = 0.582 which is higher than the significant level 0.05, fail to reject the null hypothesis means addition of 9% ESP have no significant effect as compared on asphalt binder non-recoverable creep compliance at 3.2 kpa stress level with 95% confidence

Percent recovery shows the elastic response of the asphalt binder. It tells us how much the sample returns to its original shape after a load or stress is removed. Generally, percent recovery value is used to provide a means for determining the elastic response and stress dependence of polymer modified and unmodified asphalt binders and no actual specification was established for the percent recovery in the MSCR test. Percent recovery increase with the non-recoverable value decrease, this shows the elasticity of asphalt binder increase due to the addition of egg shell powder.

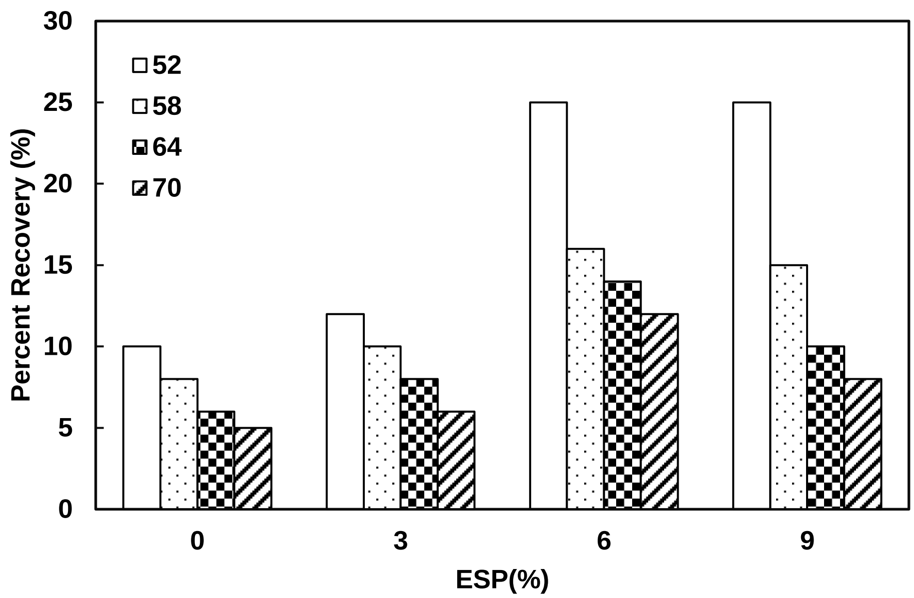

Figure 40 Percent Recovery @ 3.2kpa

The change in delayed elastic response of the binder with the addition of ESP modifier is seen by evaluating the percent recovery from MSCR test. As shown from the figure 40 due to the addition of ESP on the neat binder the percent recovery improves, showing a significant improvement of network in the binder which implies that the binder elasticity also improves and will improve the rut resistance. From figure the maximum percent recovery is score at 9% ESP modified asphalt binder. Moreover, since the bitumen become viscous as temperature increases and its elasticity decreases.

4.3 Summary

In this section the test result of conventional and rheological tests was organized and analyzed. From the test data analysis, it shows that penetration and ductility value of binder decrease and softening point increase with the increase of ESP.

From the amplitude sweep test the LVER is obtained at the minimum strain of 0.38%. The strength of asphalt binder is affected by temperature, rate of loading and aging. The complex shear modulus master curve shows, for all binders in almost similar pattern shear stiffness decreases as temperature increases. And at low frequency and high temperature the modulus increases as modifier increases. Generally, we can observe that, the modifier improves the complex shear modulus of the neat binder at higher temperatures. Since rutting is a serious problem at high temperature due to slow moving traffic; modifier improves the pavement performance against rutting by increasing the stiffness of the binder. The result of PG determination test shows improvement of performance grade from PG58-YY to PG64-YY for both unaged and RTFO aged bitumen with the additions of 3% ESP modified binder.

The analysis of MSCR test shows that the increase in percent recovery and decrease non-recoverable compliance with an increase of ESP this implies in improvement to the resistance of binder to permanent deformation.

5.CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The purpose of this study was to evaluate the effect of egg shell powder as a modifier material on asphalt binder to investigate the effectiveness of the material on the improvement of the properties of the asphalt binder. By using standard testing methods of conventional as well as rheological properties of the modified and unmodified binder was examined. Based on the results obtained from this study, the following conclusions can be made

- For both aged and unaged asphalt binder for conventional tests addition of ESP has decrease the penetration and increase in softening point temperature. This indicates improvement in hardness and resistance to temperature effect in the modified binders.
- For both aged and unaged asphalt binder ductility value decreases however when the bitumen become aged at 9% ductility values are not under the desired value which is stated in ERA 2013.
- The LVER of all samples were determined with the help of AST test. And the limiting strain was 0.38%.
- From the frequency sweep test (FST) result, the master curve (MC) displays that addition of egg shell powder (ESP) improves the behavior of asphalt binder. Addition of 3% and 6% modified asphalt binder increases the stiffening property of asphalt binder at high temperature and low loading frequency as compared to 9% for unaged asphalt binder.
- From the performance grade test, the result shows that improvement of performance grade from PG58-YY to PG64-YY for both unaged and RTFO aged bitumen with the additions of 3% ESP. With all the modifier contents (3%,6%, and 9%) the effect is the same for the PG grade but slight difference in pass-fail temperature.
- From the test result obtained from MSCR as the percent of ESP increase the total strain result decrease, which shows the increase in stiffness. Also, the J_{nr} at 3.2 value decrease as the percent of the modifier increase. This shows that the addition of ESP in to the

neat binder improves the rutting resistance by decreasing the J_{nr} value. The maximum effect was observed at 6% of the modified binder.

5.2 Recommendation

- Generally, from this study it is possible to recommend that the performance grade of a binder of 60/70 enhanced with addition of 6% ESP, thus it is recommendable to use this modified binder in Ethiopia around wide areas with pavement design temperature 64^{o}C and below.
- 6% ESP can be used a modifier for asphalt binder in order to improve rutting performance at intermediate and high temperature.
- In addition to the rutting performance of the binder, it is recommended to study the modifier binder for fatigue and low temperature performance
- It is advantageous to recognize such waste material to improve the properties of asphalt binders and apply them in real road construction work rather than keeping them as a research.
- For ERA, it is recommended to include the superpave grading system in to their manual for better characterization of the asphalt binder based on the environmental temperature, local loading condition and other.

5.3 Future studies

Future research work may include:

- The effect of Egg shell powder on rheological properties of binder conducted on this paper using only asphalt binder penetration grade 60/70. Further studies are needed for other penetration grade asphalt binder.

- Compatibility of blends between egg shell powder and bitumen should be checked (Morphology, Optical microscopy)

- Life cycle cost analysis must be conducted for roads constructed using egg shell powder mix asphalt binder in comparison to those constructed using neat asphalt binder.

- Characterize the chemistry of egg shell powder as well as binder molecular interaction containing egg shell powder using Fourier transform infrared spectroscopy (FTIR).
- Fatigue resistance of the modified binder
- Binder testing is not enough to know the effect of ESP on rutting. So Superpave asphalt mixture tests are also required.

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APPENDIX A- AMPLITUDE SWEEP TEST (AST) RESULTS

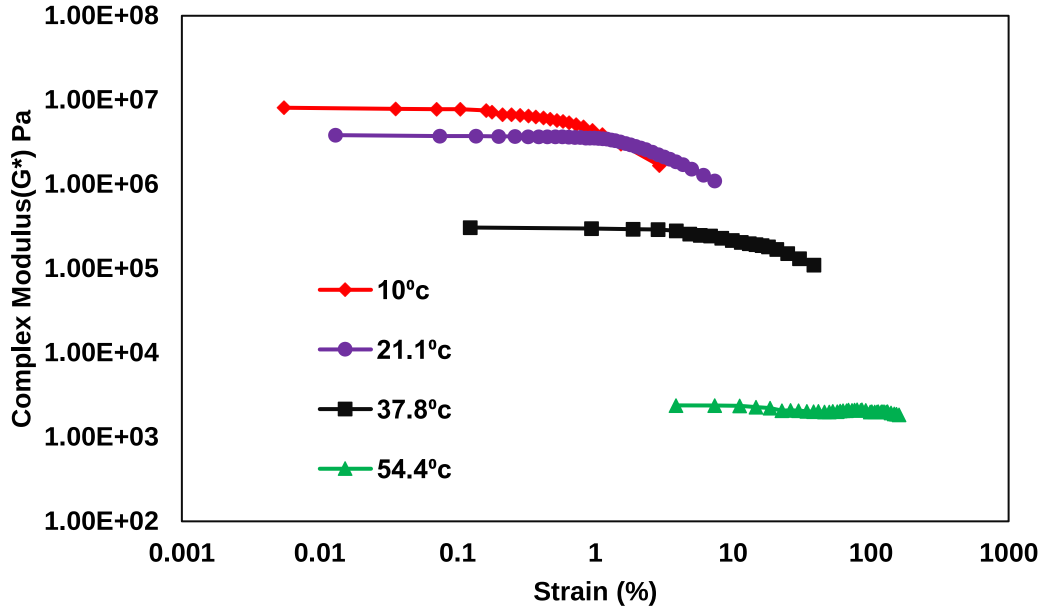


Figure: A.1 Linear Visco-Elastic Range of Unaged neat binder

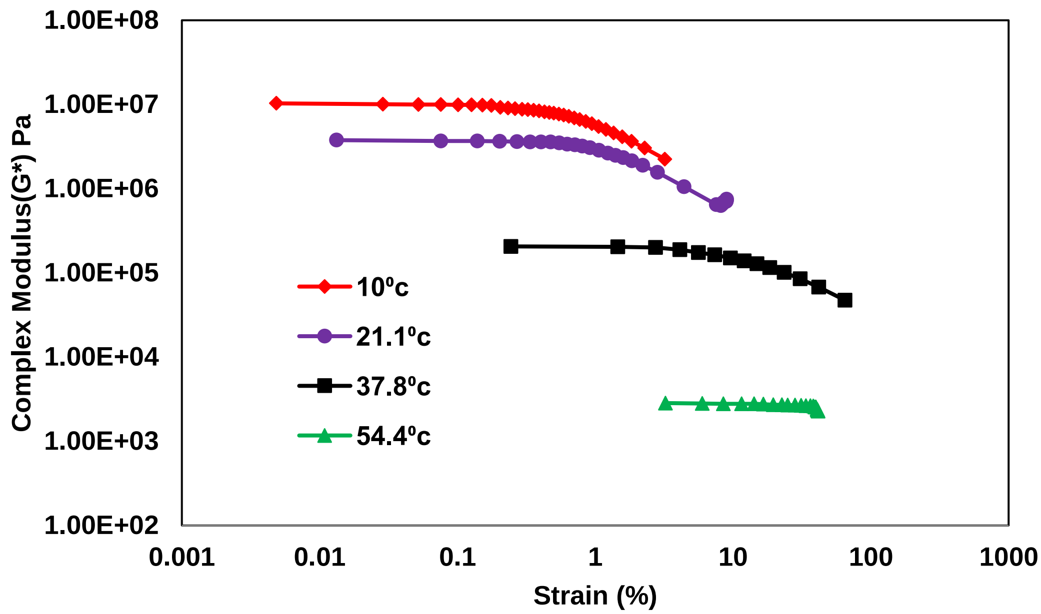


Figure: A.2 Linear Visco-Elastic Range of RTFO Aged neat binder

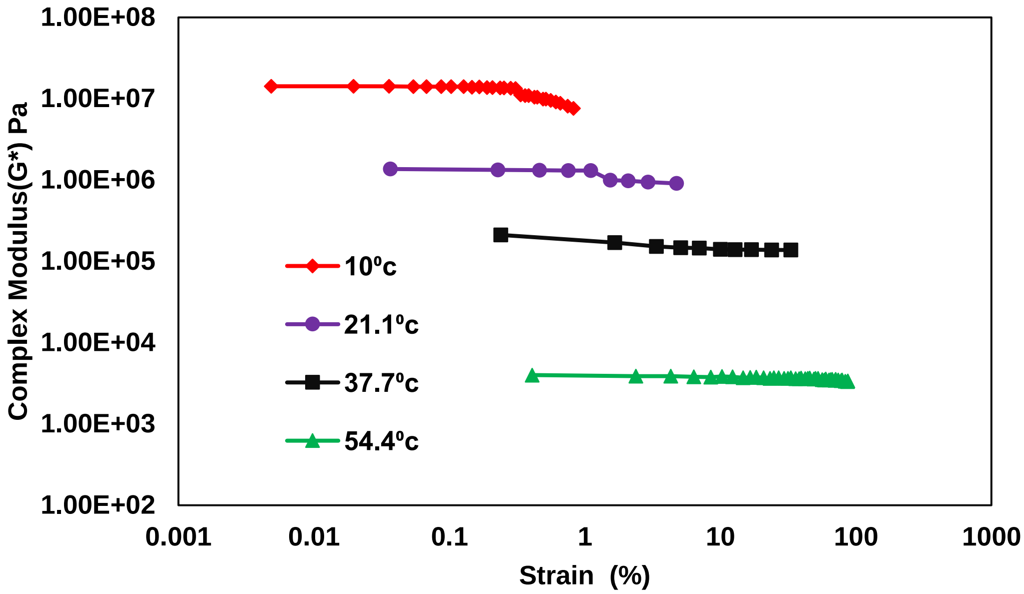


Figure: A.3 Linear Visco-Elastic Range of Unaged 3% ESP Modified binder

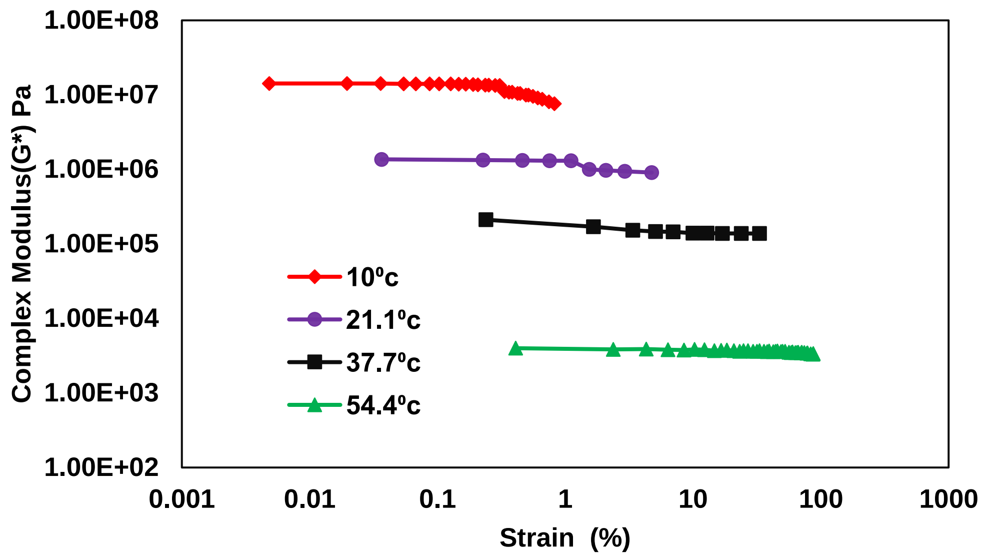


Figure: A.4 Linear Visco-Elastic Range of RTFO Aged 3% ESP Modified binder

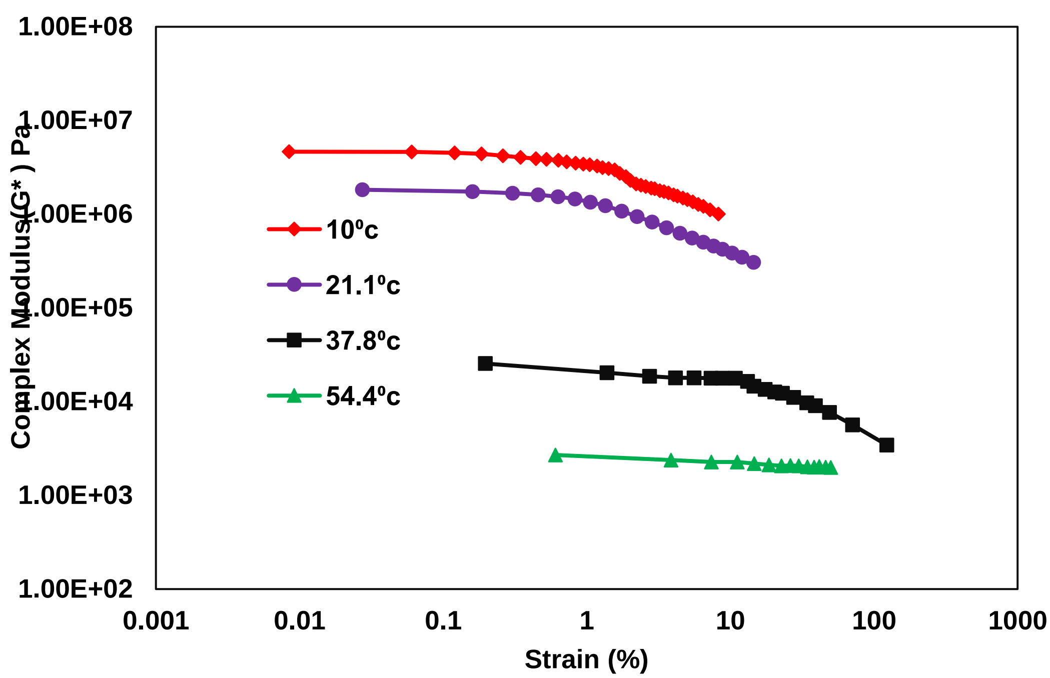


Figure: A.5 Linear Visco-Elastic Range of Unaged 6% ESP Modified binder

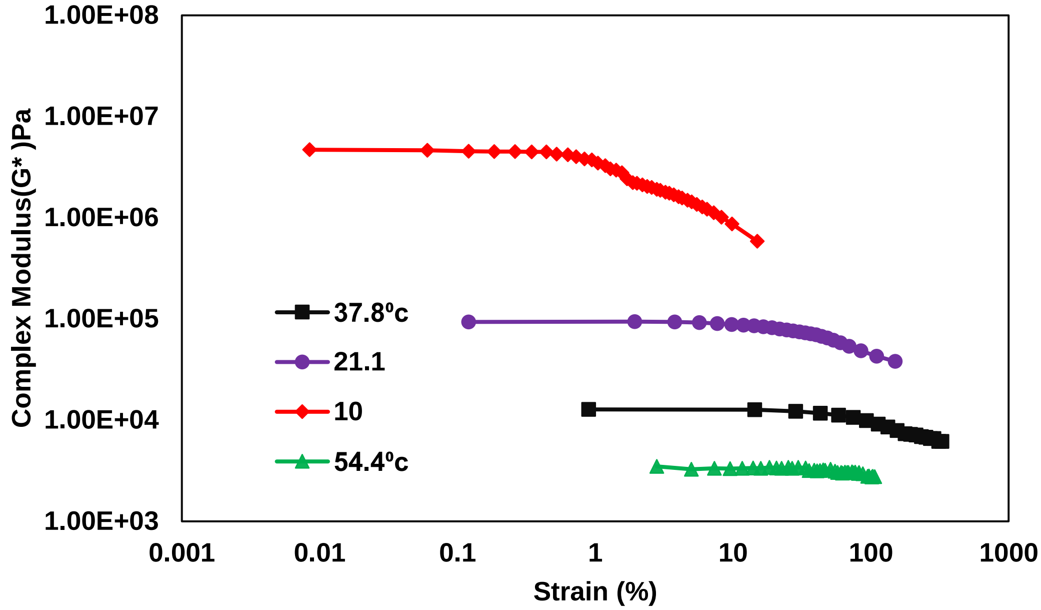


Figure: A.6 Linear Visco-Elastic Range of RTFO Aged 6% ESP Modified

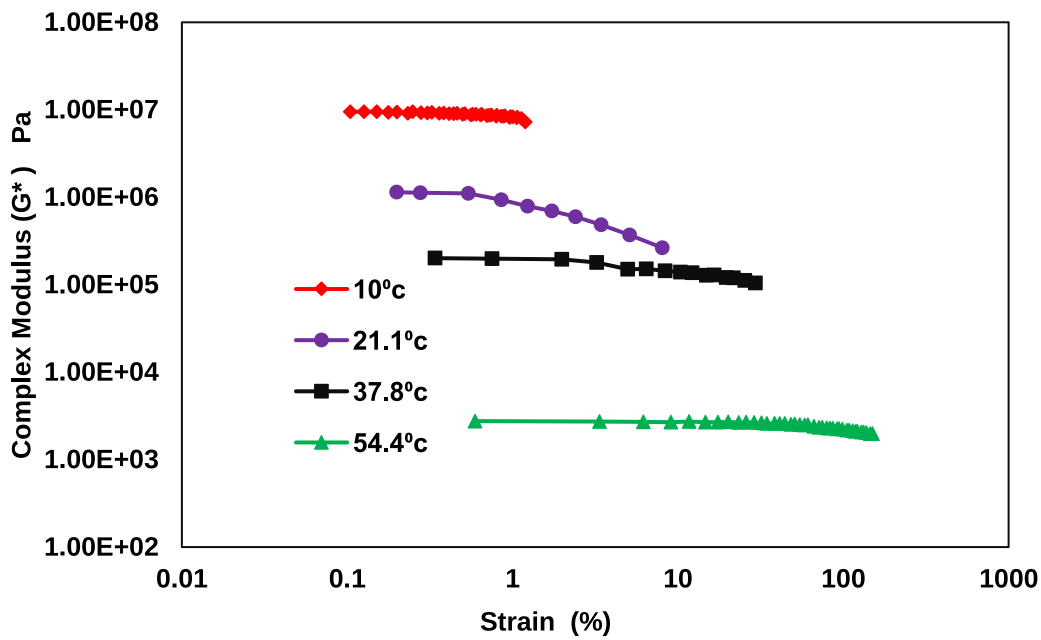
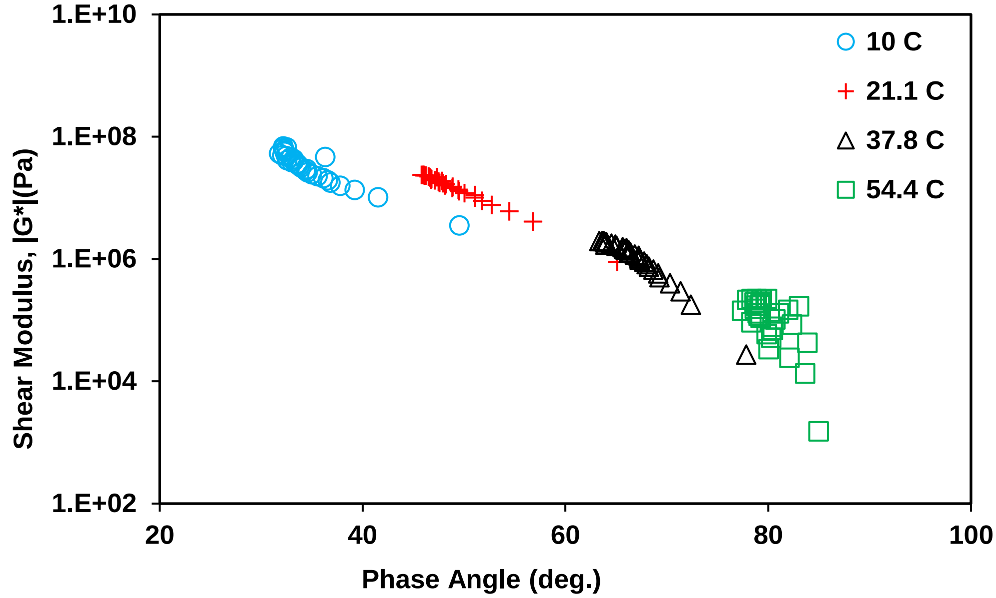
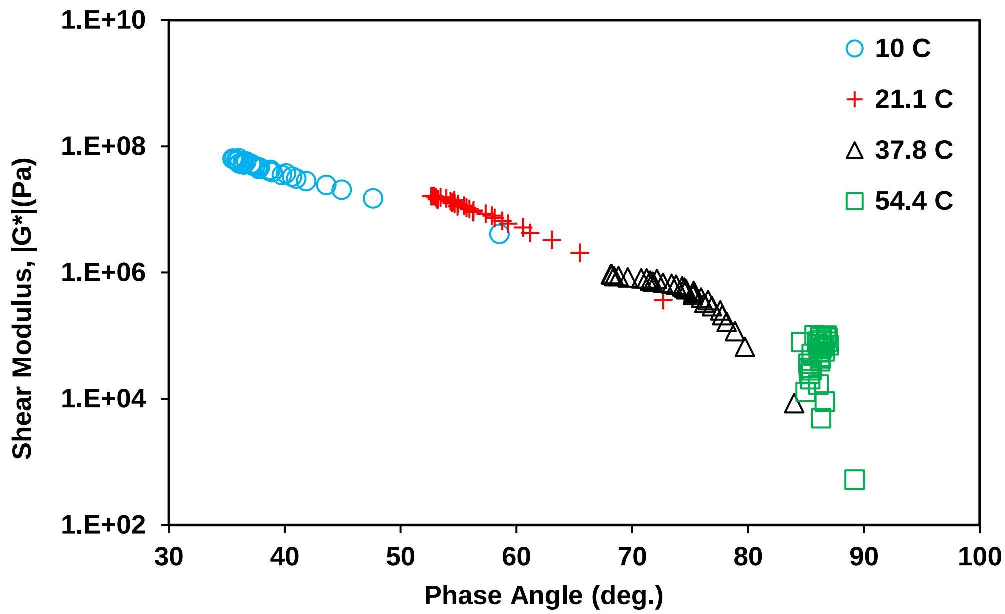


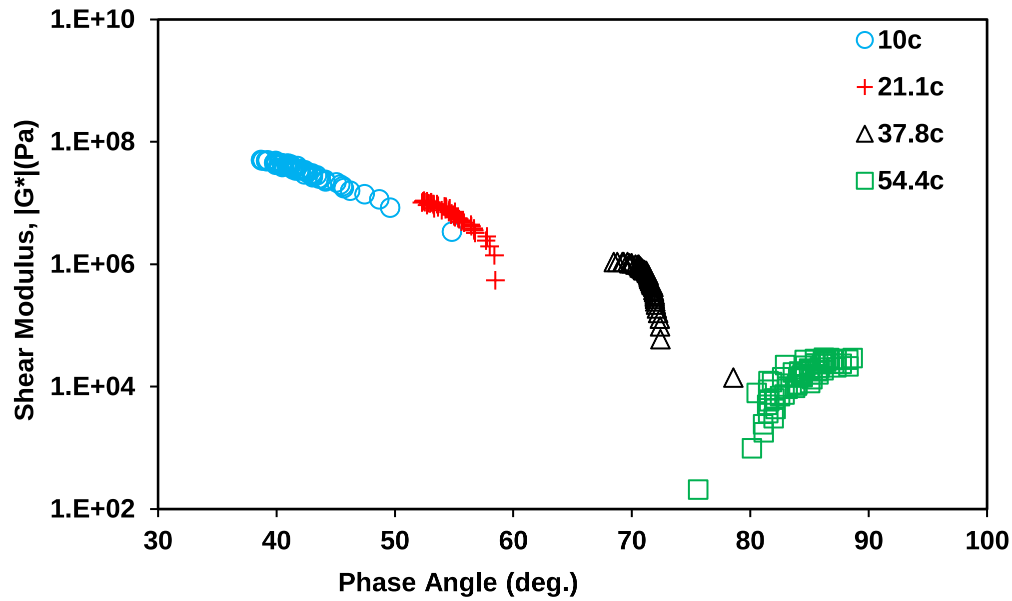
Figure: A.7 Linear Visco-Elastic Range of RTFO Aged 9% ESP Modified

APPINDIX B- FREQUENCY SWEEP TEST RESULTS

I) Black space diagram







II) Isothermal plots

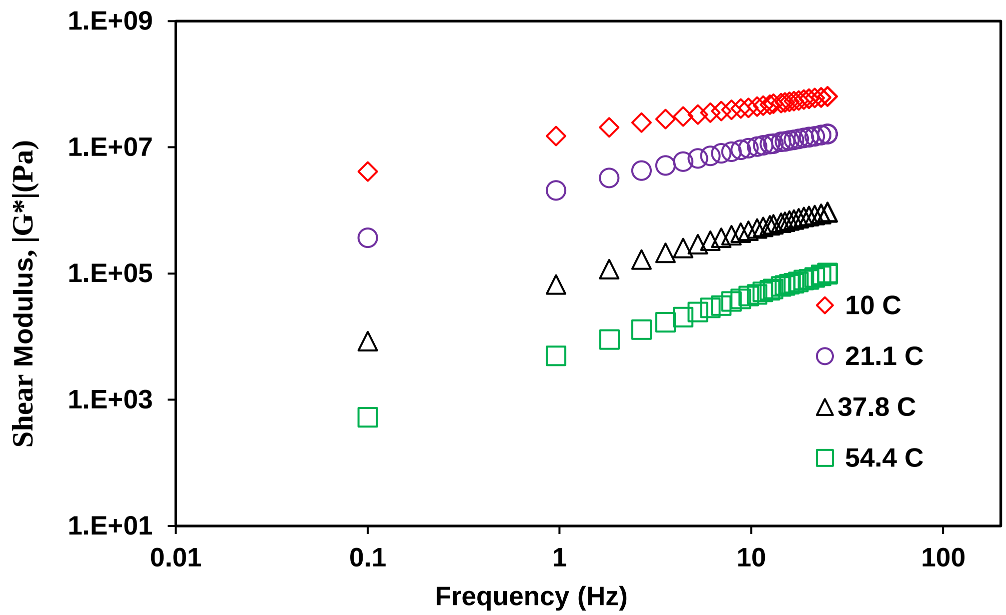


Figure B.4 Effect of temperature on unaged neat binder

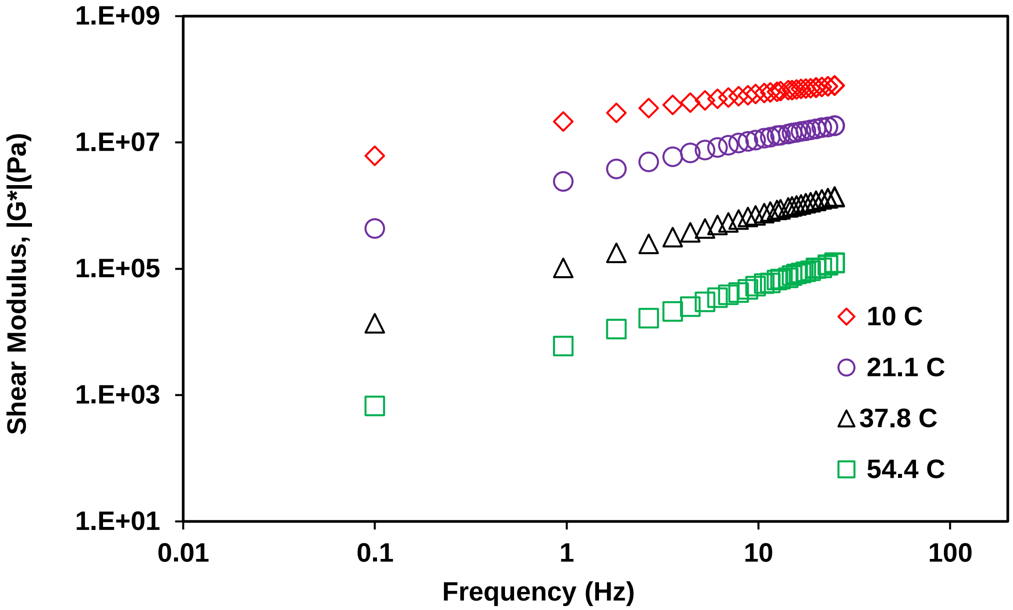


Figure B.5 Effect of temperature on unaged 3% ESP

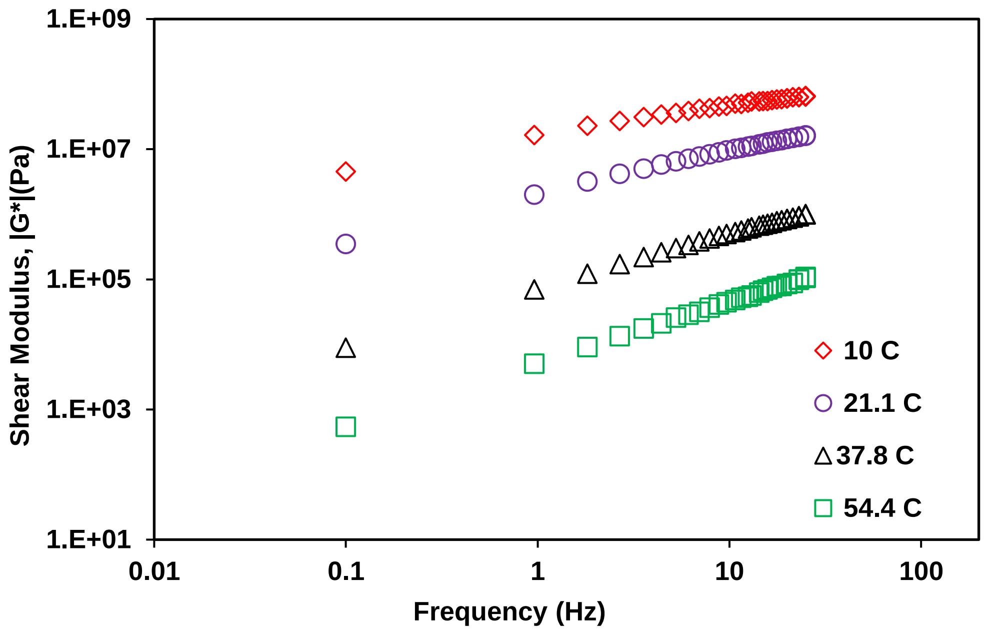


Figure B.6 Effect of temperature on unaged 6% ESP binder

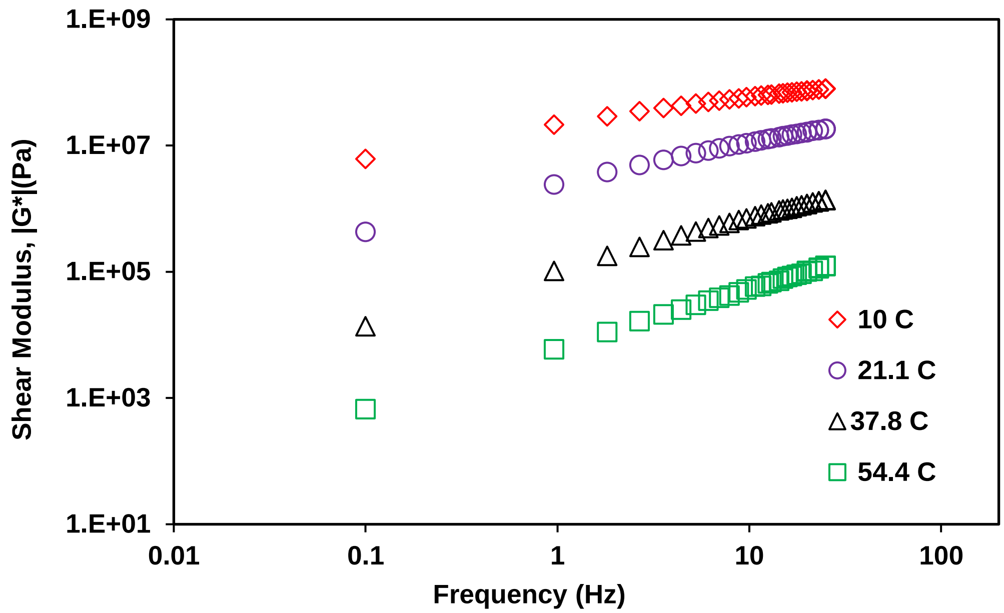


Figure B.7 Effect of temperature on aged 0% neat binder

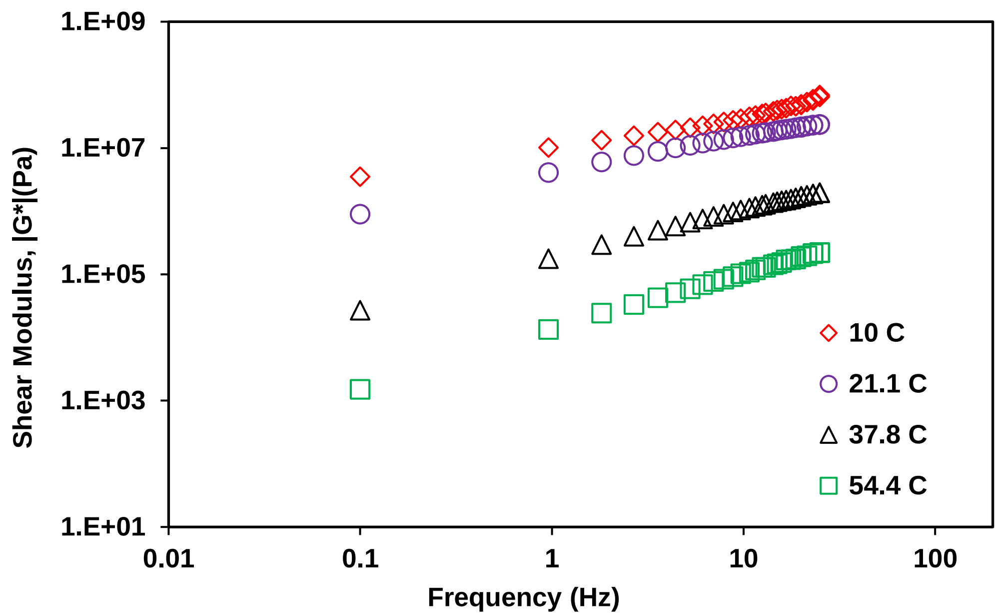


Figure B.8 Effect of temperature on aged 3% ESP binder

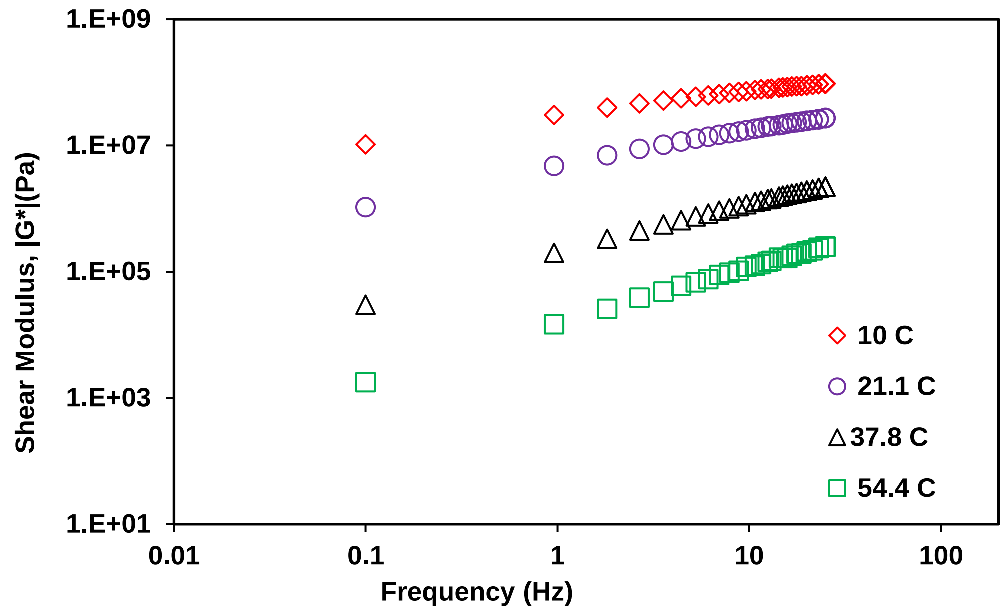


Figure B.9 Effect of temperature on aged 6% ESP binder

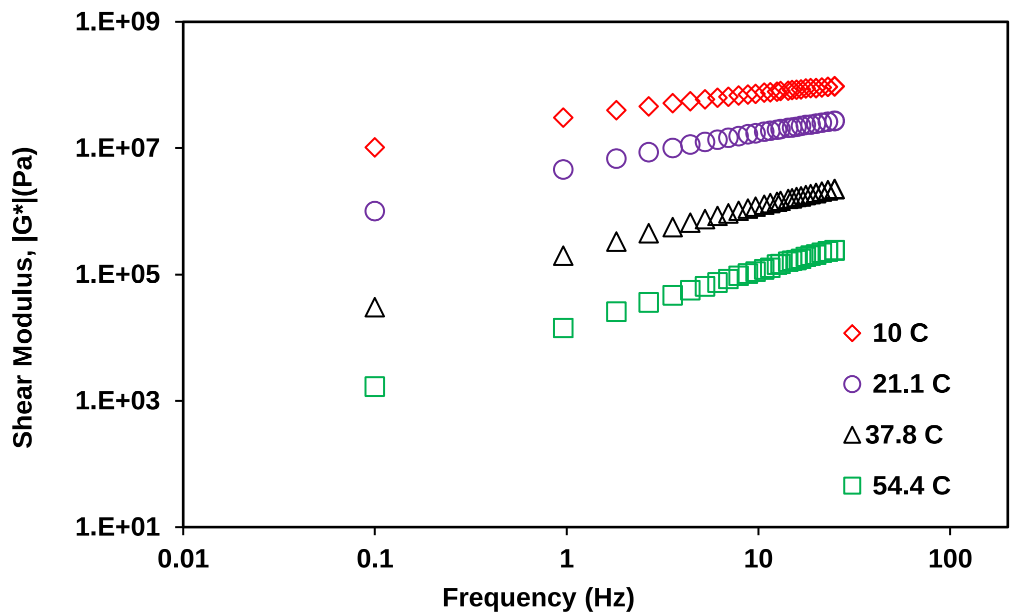


Figure B.10 Effect of temperature on aged 6% ESP binder

APPINDIX C- STATISTICAL ANALYSIS USING ANOVA

I) Statistical analysis for master curves

Table C.1 Hypothesis test result of reduced frequency and ESP content (RTFOT aged)

Tests of Between-Subjects Effects

Dependent Variable: Complex modulus

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	5.405E15[a]	9	4.913E14	288.466	.000
Intercept	3.057E15	1	3.057E15	1.116E3	.000
ESP	3.981E15	1	1.327E15	769.056	.000
Reduced frequency HZ	3.192E14	3	1.596E14	100.843	.000
ESP*Reduced frequency HZ	1.104E15	3	1.840E14	150.998	.000
Error	1.537E13	18	.		
Total	8.462E15	24			
Corrected Total	5.405E15	23			

a. R Squared = .998 (Adjusted R Squared = .997)

Table C.2 Multiple comparison test result for reduced frequency

Multiple Comparisons

Dependent Variable: Complex modulus

Tukey HSD

	(I) Reducedfrequency	(J) Reducedfrequency	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
	0.1HZ	10	-6.81970E6*	1.71458E6	.000	-1.1607E7	-2.0326E6
		25	-1.06264E7*	1.71458E6	.000	-1.5414E7	-5.8393E6
	10HZ	0.1	6.81970E6*	1.71458E6	.000	2.0326E6	1.1607E7
		25	-3.80672E6*	1.71458E6	.000	-8.5938E6	980405.5736
	25HZ	0.1	1.06264E7*	1.71458E6	.000	5.8393E6	1.5414E7
		10	3.80672E6*	1.71458E6	.000	-9.8041E5	8.5938E6

*. The mean difference is significant at the 0.05 level.

Multiple Comparisons

Complex modulus

Tukey HSD

(I) ESP	(J) ESP	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
0	3	-4.24683E7*	1.08912E7	.009	-7.7346E7	-7.5910E6
	6	-4.2468367E7*	1.08912E7	.000	-3.4976E7	3.4779E7
	9	-1.14833E6*	1.08912E7	.000	-3.6026E7	3.3729E7
3	0	4.24683E7*	1.08912E7	.009	7.5910E6	7.7346E7
	6	4.23700E7*	1.08912E7	.000	7.4927E6	7.7247E7
	9	4.13200E7*	1.08912E7	.000	6.4427E6	7.6197E7
6	0	98333.33333*	1.08912E7	.000	-3.4779E7	3.4976E7
	3	-4.23700E7*	1.08912E7	.000	-7.7247E7	-7.4927E6
	9	-1.05000E6	1.08912E7	.845	-3.5927E7	3.3827E7
9	0	1.14833E6*	1.08912E7	.000	-3.3729E7	3.6026E7
	3	-4.13200E7*	1.08912E7	.000	-7.6197E7	-6.4427E6
	6	4.35000E7	1.08912E7	.845	-7.9827E7	7.7227E7

*. The mean difference is significant at the 0.05 level.

Table C.3 Hypothesis test result of reduced frequency and ESP content (after RTFO)

Tests of Between-Subjects Effects

Dependent Variable:Complex modulus

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2.861E14[a]	7	5.236E13	50.666	.000
Intercept	4.633E15	1	4.687E15	3.843E3	.000
Aging effect	3.127E14	1	2.718E14	215.622	.000
ESP	8.082E13	3	2.684E13	20.909	.000
Aging effect * ESP	3.255E13	3	1.098E13	8.269	.000
Error	1.892E13	15	1.260E12		
Total	4.938E15	21			
Corrected Total	3.950E14	20			

a. R Squared = .996 (Adjusted R Squared = .993)

Table C.4 Multiple comparison test result for reduced frequency

Multiple Comparisons

Dependent Variable:Complex modulus

Tukey HSD

	(I) Reducedfrequency	(J) Reducedfrequency	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
	0.1HZ	10HZ	-6.81970E6*	1.71458E6	.000	-1.1607E7	-2.0326E6
		25HZ	-1.06264E7*	1.71458E6	.000	-1.5414E7	-5.8393E6
	10HZ	0.1HZ	6.81970E6*	1.71458E6	.000	2.0326E6	1.1607E7
		25HZ	-3.80672E6*	1.71458E6	.000	-8.5938E6	980405.5736
	25HZ	0.1HZ	1.06264E7*	1.71458E6	.000	5.8393E6	1.5414E7
		10HZ	3.80672E6*	1.71458E6	.000	-9.8041E5	8.5938E6

*. The mean difference is significant at the 0.05 level.

Table C.5 Multiple comparison test result for ESP content (after RTFO)

Multiple Comparisons

Dependent Variable: Complex modulus

Tukey HSD

(I) ESP	(J) ESP	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
0	3	-3.72815E6*	4.59380E6	.000	-1.8439E7	1.0983E7
	6	-3.10979E6*	4.59380E6	.000	-1.7821E7	1.1601E7
	9	-4.11982E6*	4.59380E6	.000	-1.8831E7	1.0591E7
3	0	3.72815E6*	4.59380E6	.000	-1.0983E7	1.8439E7
	6	6.18364E5*	4.59380E6	.000	-1.4093E7	1.5329E7
	9	-3.91670E5*	4.59380E6	.000	-1.5103E7	1.4319E7
6	0	3.10979E6*	4.59380E6	.000	-1.1601E7	1.7821E7
	3	-6.18364E5*	4.59380E6	.000	-1.5329E7	1.4093E7
	9	-1.01003E6*	4.59380E6	.000	-1.5721E7	1.3701E7
9	0	4.11982E6	4.59380E6	.000	-1.0591E7	1.8831E7
	3	3.91670E5*	4.59380E6	.000	-1.4319E7	1.5103E7
	6	1.01003E6*	4.59380E6	.000	-1.3701E7	1.5721E7

Multiple Comparisons

Phase angle
Tukey HSD

(I) ESP	(J) ESP	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
0%	3%	.3811*	.06552	.000	.2004	.5618
	6%	.6433*	.06552	.000	.4626	.8241
	9%	-.0844	.06552	.579	-.2652	.0963
3%	0%	-.3811*	.06552	.000	-.5618	-.2004
	6%	.2622*	.06552	.003	.0815	.4430
	9%	-.4656*	.06552	.000	-.6463	-.2848
6%	0%	-.6433*	.06552	.000	-.8241	-.4626
	3%	-.2622*	.06552	.003	-.4430	-.0815
	9%	-.7278*	.06552	.000	-.9085	-.5470
9%	0%	.0844	.06552	.579	-.0963	.2652
	3%	.4656*	.06552	.000	.2848	.6463
	6%	.7278*	.06552	.000	.5470	.9085

Based on observed means.

The error term is Mean Square(Error) = .019.

*. The mean difference is significant at the .05 level.

II) Statistical analysis for MSCR

Multiple Comparisons

Jnr
Tukey HSD

(I) Temperature	(J) Temperature	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
52	58	-0.68722	.04942	.000	-0.8345	-1.0289
	64	-1.0192*	.04942	.000	-1.3176	-1.1208
	70	-2.7759*	.04942	.000	-3.9542	-3.7574
58	52	0.68722	.04942	.000	0.5134	0.7345
	64	-1.2192*	.04942	.000	1.1208	1.3176
	70	-3.9558*	.04942	.000	-2.7351	-2.5383
64	52	1.0192*	.04942	.000	0.9812	1.0989
	58	1.2192*	.04942	.000	1.1574	1.3542
	70	-2.6367*	.04942	.000	-2.7383	-2.5351
70	52	2.7759*	.04942	.000	2.6378	2.9876
	58	3.9558*	.04942	.000	3.7574	3.9542
	64	2.6367*	.04942	.000	2.5383	2.7456

Based on observed means.

The error term is Mean Square(Error) = .009.

*. The mean difference is significant at the .05 level.

Multiple Comparisons

Jnr
Tukey HSD

(I) ESP	(J) ESP	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
0%	3%	-.0344*	.34821	.000	-.1558	.1125
	6%	.2778*	.34821	.000	.2012	.4155
	9%	.4967*	.34821	.000	.4058	.6531
3%	0%	.0344*	.34821	.000	-.1514	-.1935
	6%	.3022*	.34821	.003	.1924	.4596
	9%	-.6152*	.34821	.000	.4026	-.6935
6%	0%	-.2778*	.34821	.000	-.3922	-.1535
	3%	-.3022*	.34821	.003	-.4355	-.1678
	9%	.2611	.34821	. 582	.1524	.3956
9%	0%	.4967*	.34821	.000	-.6535	-.3925
	3%	-.6152*	.34821	.000	-.6525	-.4025
	6%	-.2611	.34821	. 582	-.3922	-.1526

Based on observed means.

The error term is Mean Square(Error) = .019.

*. The mean difference is significant at the .05 level.

Multiple Comparisons

Jnr
Tukey HSD

(I) Temperature	(J) Temperature	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
52	58	-0.68722	.04942	.000	-0.8345	-1.0289
	64	-1.0192*	.04942	.000	-1.3176	-1.1208
	70	-2.7759*	.04942	.000	-3.9542	-3.7574
58	52	0.68722	.04942	.000	0.5134	0.7345
	64	-1.2192*	.04942	.000	1.1208	1.3176
	70	-3.9558*	.04942	.000	-2.7351	-2.5383
64	52	1.0192*	.04942	.000	0.9812	1.0989
	58	1.2192*	.04942	.000	1.1574	1.3542
	70	-2.6367*	.04942	.000	-2.7383	-2.5351
70	52	2.7759*	.04942	.000	2.6378	2.9876
	58	3.9558*	.04942	.000	3.7574	3.9542
	64	2.6367*	.04942	.000	2.5383	2.7456

Based on observed means.

The error term is Mean Square(Error) = .009.

*. The mean difference is significant at the .05 level.

APPINDIX D- PHASE ANGLE MASTER CURVE

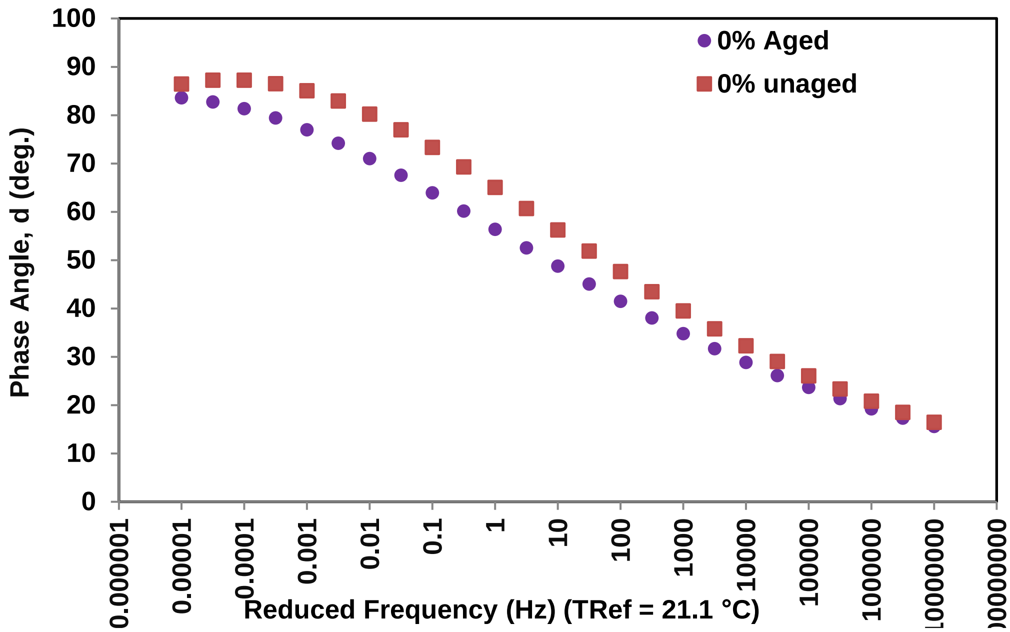


Figure D.1 Phase angle master curve for 0% neat binder for Unaged and RTFO Aged

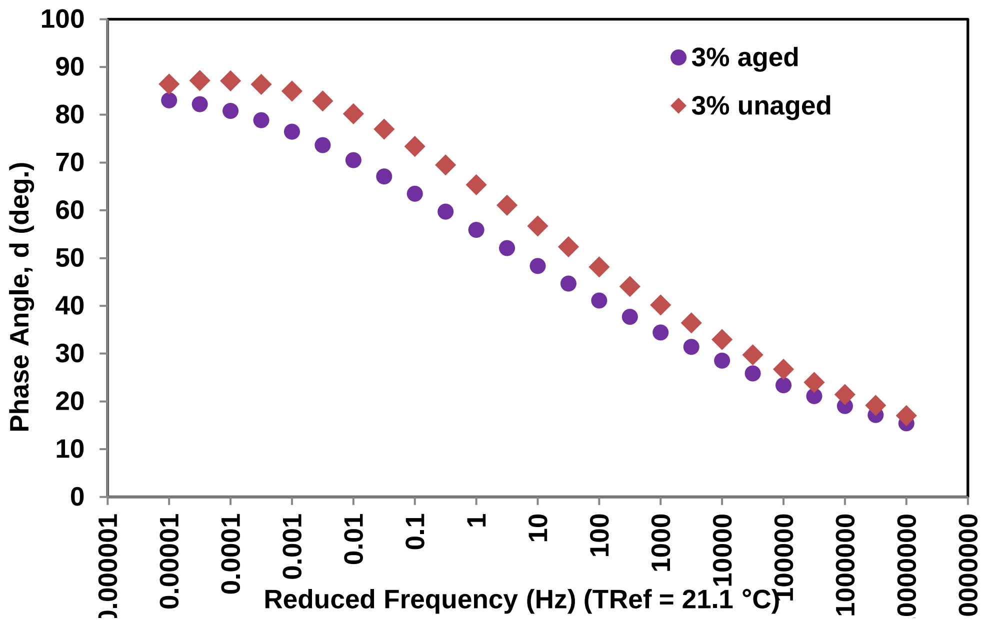


Figure D.2 Phase angle master curve for 3% modified binder Unaged and RTFO Aged

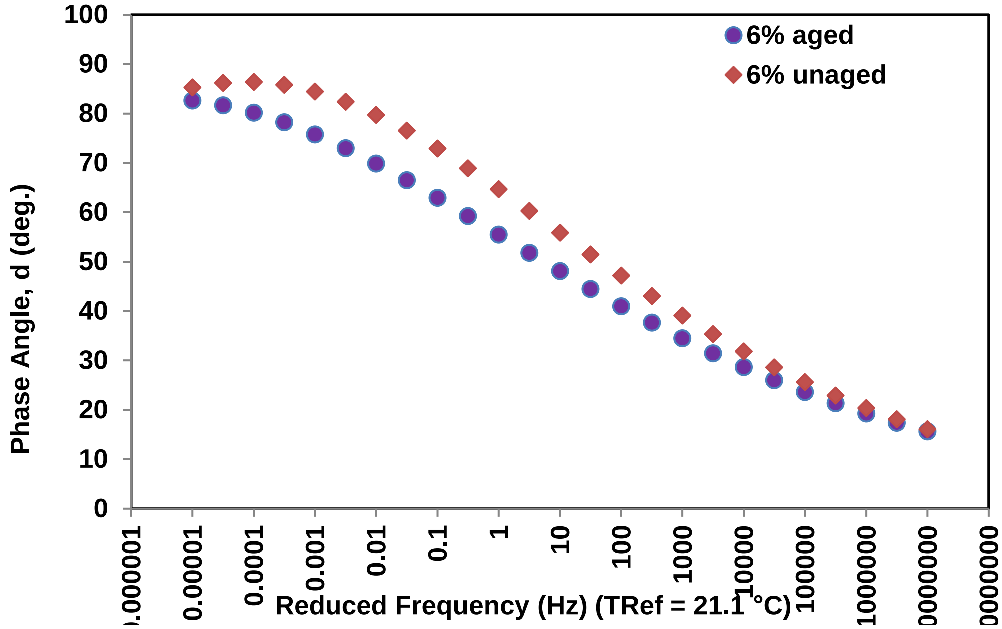


Figure D.3 Phase angle master curve for 6% modified binder Unaged and RTFO Aged

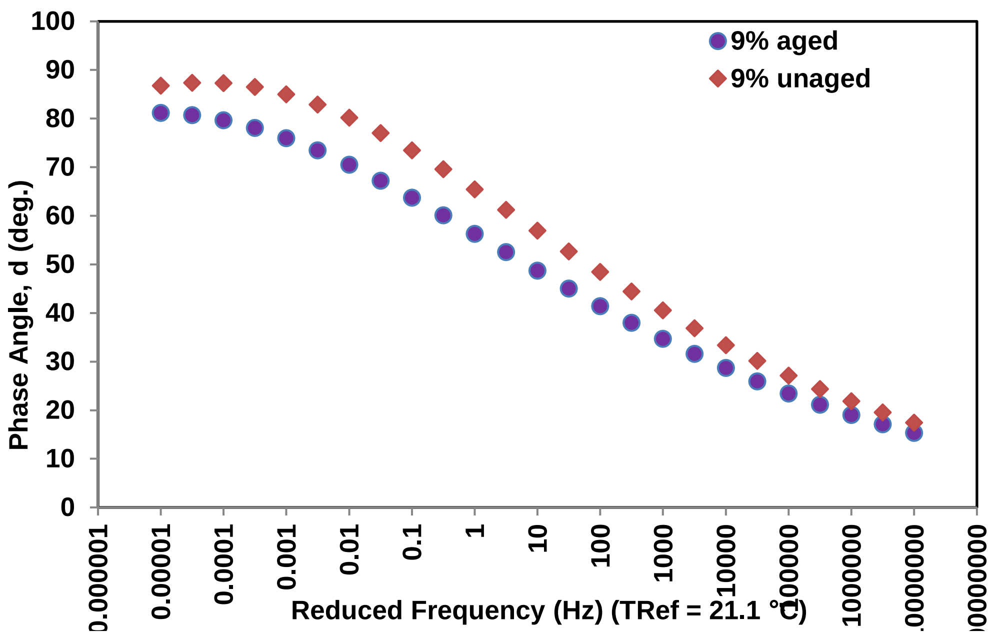


Figure D.4 Phase angle master curve for 9% modified binder Unaged and RTFO Aged

APPINDIX E- PERFORMANCE GRADE TEST RESULT

Table E.1 Performance Grade Determination Test Result for Unaged Bitumen

Sample (% ESP)	0			3			6			9		
test Temperature (oc)	52	58	64	58	64	70	58	64	70	58	64	70
Crieria G*/sin delta @10rad/sec. kPa	>= 1.0kPa			>= 1.0kPa						>= 1.0kPa		
Sample-01 G*/sin delta @10rad/sec. kPa	4.05	1.61	0.67	5.90	2.70	0.70	5.95	2.75	0.81	4.50	2.08	0.90
Sample -02 G*/sin delta @10rad/sec. kPa	4.70	1.81	0.77	2.38	1.11	0.75	3.72	1.93	0.69	6.05	2.93	0.95
Average G*/sin delta @10rad/sec. kPa	4.38	1.71	0.72	4.14	1.91	0.73	4.84	2.34	0.75	5.28	2.51	0.93
Phase angle (sample-01). Deg	82.33	83.77	83.46	83.42	84.46	84.43	83.92	84.00	77.20	82.73	84.00	85.49
Phase angle (sample-02). Deg	83.90	84.07	84.60	83.57	84.54	84.75	83.90	84.20	84.50	83.00	84.40	85.78
Phase angle (Average). Deg	83.12	83.92	84.03	83.50	84.43	84.59	83.91	84.10	80.85	82.87	84.20	85.64
Pass Fail Temp. (sample-01).oc	62.40			65.70			66.20			68.10		
Pass Fail Temp. (sample-02).oc	62.20			64.90			65.70			68.80		
Pass Fail Temp. (Average). Oc	62.30			65.30			65.95			68.45		
Temp Pass. Oc	**58.00**			**64.00**			**64.00**			**64.00**		

Table E.2 Performance Grade Determination Test Result for RTFO Aged Bitumen

Sample (% ESP)	0			3			6			9		
test Temperature (oc)	52	58	64	58	64	70	58	64	70	58	64	70
Crieria G*/sin delta @10rad/sec. kPa	>= 2.2kPa			>= 2.2kPa			>= 2.2kPa			>= 2.2kPa		
Sample-01 G*/sin delta @10rad/sec. kPa	6.53	4.28	2.03	9.58	3.13	1.98	9.71	4.50	1.82	10.02	4.95	1.92
Sample -02 G*/sin delta @10rad/sec. kPa	5.05	3.99	1.89	8.47	3.01	1.72	9.71	4.63	1.93	9.80	3.78	1.80
Average G*/sin delta @10rad/sec. kPa	5.79	4.14	1.96	9.03	3.07	1.85	9.71	4.57	1.88	9.91	4.37	1.86
Phase angle (sample-01). Deg	82.94	83.92	84.66	82.97	83.02	84.41	82.73	84.40	85.78	82.54	84.20	86.38
Phase angle (sample-02). Deg	83.05	84.24	84.29	83.00	83.13	84.46	82.73	84.10	85.71	82.90	84.60	85.50
Phase angle (Average). Deg	83.00	84.08	84.48	83.08	83.08	84.44	82.73	84.25	85.75	82.72	84.40	85.94
Pass Fail Temp. (sample-01).oc	63.40			66.20			66.80			69.20		
Pass Fail Temp. (sample-02).oc	64.20			65.90			67.00			69.00		
Pass Fail Temp. (Average). Oc	63.80			66.05			66.90			69.10		
Temp Pass. Oc	**58.00**			**64.00**			**64.00**			**64.00**		

APPINDIX F- MSCR TEST RESULT

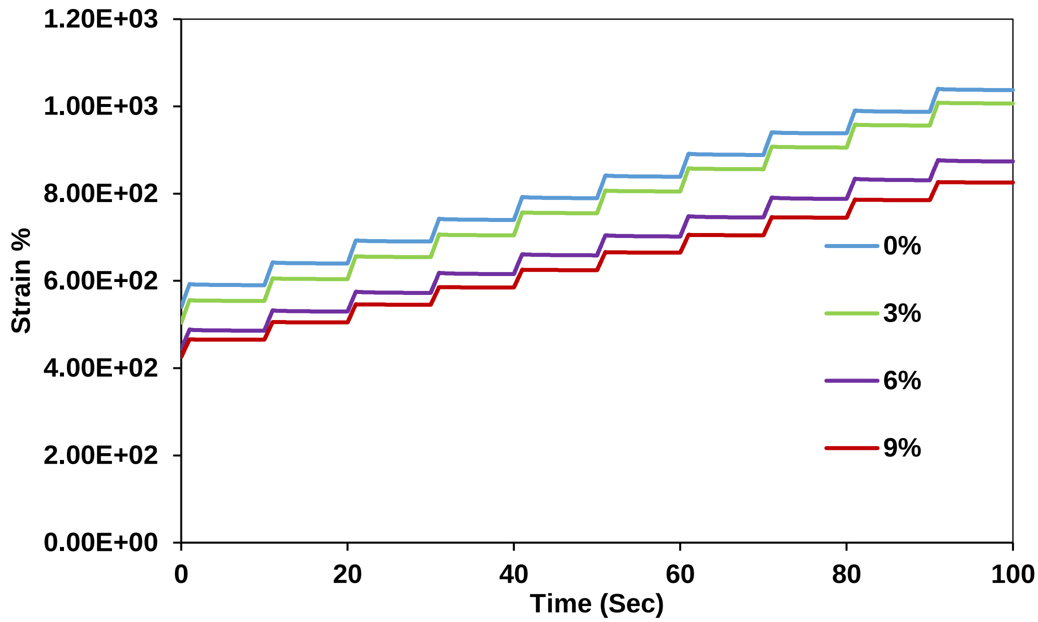


Figure F.1 Effect of ESP on Strain at 0.1 kpa (52 ^{o}C)

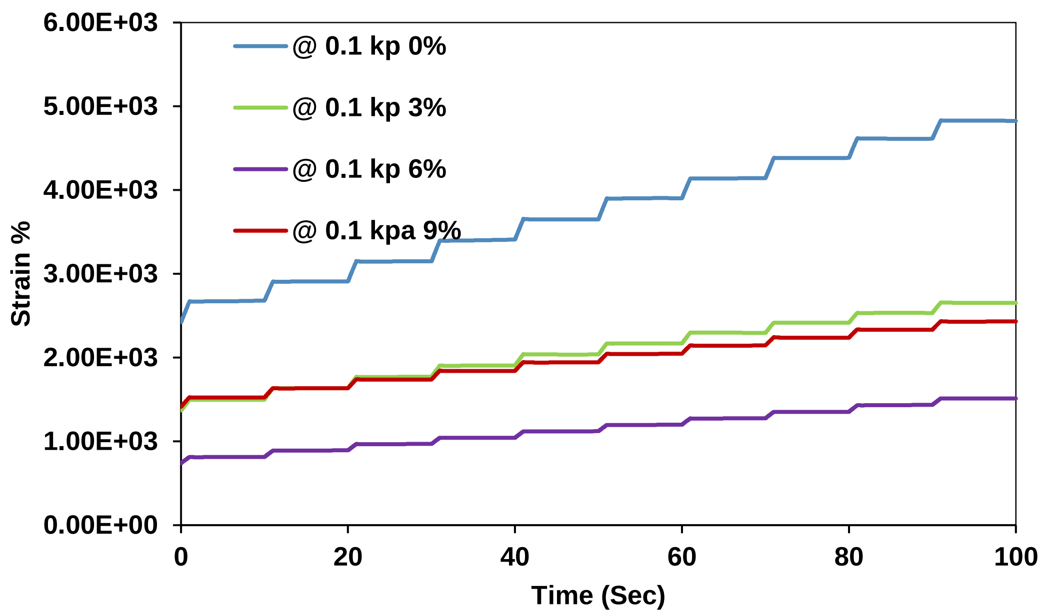


Figure F.2 Effect of ESP on Strain at 0.1 kpa (58 ^{o}C)

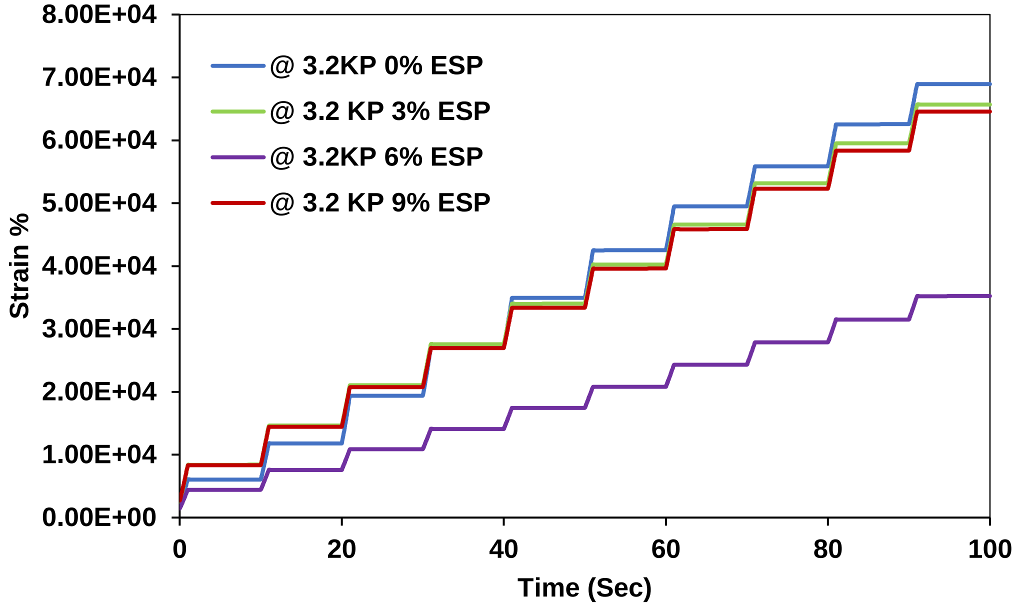


Figure F.3 Effect of ESP on Strain at 3.2 kpa (58 ºC)

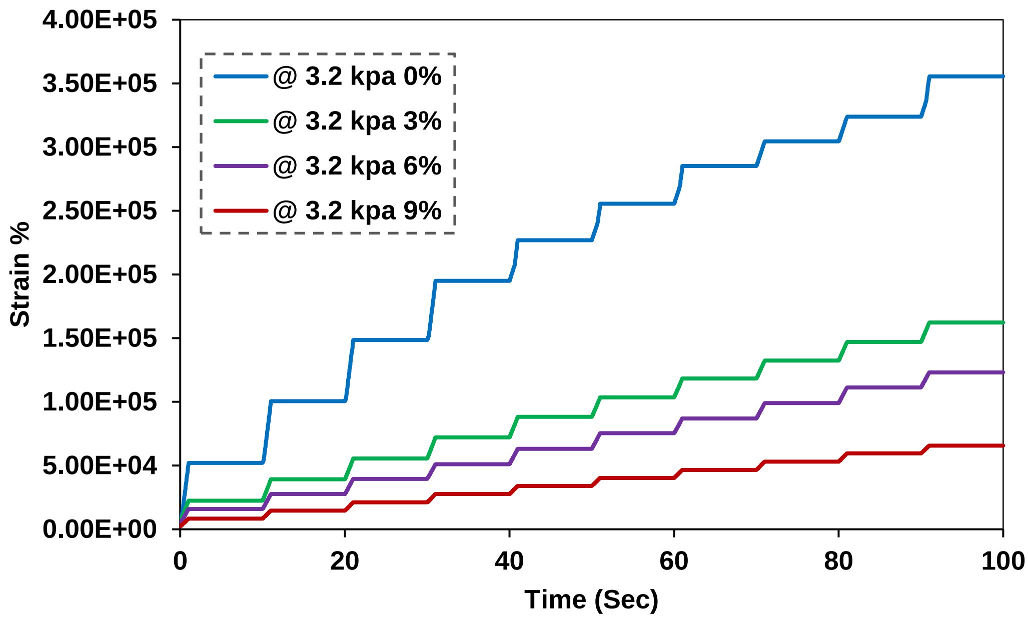


Figure F.4 Effect of ESP on Strain at 3.2 kpa (70 ºC)

APPINDIXG-MATERIAL QUALITY TEST

TableG.1 chemical composition of egg shell powder by complicate silicate analysis (Geological survey of Ethiopia)

Sio2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
2.36	<0.01	<0.01	50.10	0.40	<0.01	<0.01	0.10	0.32	<0.01	1.10	46.46