# ENGINEERING CHARACTERIZATION OF SULPHUR MODIFIED BITUMINOUS BINDERS

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# ENGINEERING CHARACTERIZATION OF SULPHUR MODIFIED BITUMINOUS BINDERS

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# Certificate

This is to certify that the work in the thesis entitled. "**ENGINEERING CHARACTERIZATION OF SULPHUR MODIFIED BITUMINOUS BINDERS**" by **Aditya Kumar Das** is a record of an original work carried out by him under my supervision and guidance in partial fulfillment of the requirements for the award of the degree of **Master of Technology** in **Department of Civil Engineering** with specialization in **Transportation Engineering**. Neither this project nor any part of it has been submitted for any degree or academic award elsewhere.

Prof. Mahabir Panda
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Dedicated to
My Father, Mother
And
My guide Prof. M. Panda

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# Abstract

The demand on bituminous flexible pavement, as a result of growth in heavy traffic loads and their tyre contact pressure with adverse climatic conditions, fatigue and rutting performance has resulted in an interest towards the modified bituminous binders. There are various popular modified binders already available worldwide. These modifiers significantly alter the rheological and morphological properties of the binder, as characterized by rheological testing methods along with the morphological rather than the conventional methods, to enhance the performance of the binder. This study is intended towards the modification of the conventional viscosity grade VG 30 bitumen and applications of commercial sulphur available in local market to modify the VG 30 bitumen and to evaluate the rheological characteristics of unaged and aged samples of these two binders using a Dynamic Shear Rheometer (DSR). Attempt has been made to decide the appropriate conditions for binder development such as mixing/blending time and temperature to ensure proper modification, through the rheological parameters of phase angle and complex modulus. This development ultimately helps to influence the fatigue and rutting resistances of bituminous mixes. The modification of bitumen with sulphur at six different mixing temperature such as 100ºC, 110ºC, 120ºC, 130ºC, 140ºC, 150ºC and 160ºC, each made at five different mixing times such as 5 min, 10 min, 15 min, 20 min, 30 min. has also been carried out. The optimum modification level has been evaluated considering unaging and aging criteria for five sulphur contents such as 1%, 2%, 3%, 4% and 5% by weight of the bitumen. It is observed that the addition of 2% sulphur by weight with bitumen blended at 140ºC temperature for about 30 min., results in the best modification of VG 30 bitumen in terms of the rheological properties, and satisfying the requirements of conventional properties.



**Key words*:*** *Bitumen, Rheology, Viscosity, Elasticity, Phase angle, Complex Shear Modulus*

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# List of Abbreviations

PAV: Pressure Ageing Vessel

TFO: Thin Film Oven

SHRP: Strategic Highway Research Program

DSR: Dynamic Shear Rheometer

AASHTO: American Association of State Highway and Transportation Officials

G' : Storage modulus [Pa]

G" : Loss Modulus [Pa]

G* : complex modulus

$\tau max$ : Absolute value of the peak-to-peak shear stress(Pa)

$\gamma max$ : Absolute value of the peak-to-peak shear strain (%)

Tmax : Maximum applied torque (load) (Pa)

$\theta max$ : Maximum deflection angle (rad)

r : Radius of specimen plate (mm)

h : Specimen height (mm)

# Thesis Structure

The thesis structure is arranged as follows:

- ✓ **Chapter 1** a brief background on structural and chemical properties of bitumen are that normally used in bituminous mixture are summarized. The problem statement and research objectives are also included.
- ✓ **Chapter 2** deals with the literature review of bitumen composition, its physical, chemical, rheological, morphological and thermal characteristics.
- ✓ **Chapter 3** deals with theoretical and basic concept of bitumen rheology, morphology and its thermal behavior. The fundamentals of dynamic shear rheometer and explains two important parameters i.e. Complex shear modulus (G*) and phase angle ($\delta$).
- ✓ **Chapter 4** describes the experimental section and various types of materials used in this study, the dynamic mechanical analysis using DSR tests, the morphological and thermal analysis of the un modified and modified bitumen. Also includes the ageing tests of binders.
- ✓ **Chapter 5** deals with the analysis of various test results for modified and unmodified binder.
- ✓ **Chapter 6** summarizes the conclusions obtained from this study and presents several potential recommendations for future work.

# CHAPTER-1

# INTRODUCTION

## 1.1 Background of the study

In India roads and highways are preferred as primary modes of transportation. Roads constructed with flexible pavements always given more importance due to its smooth riding quality and less construction costs than in case of rigid pavements. Bituminous materials along with aggregates are utilized for the construction of flexible pavement roads. The Indian road transportation infrastructure is a great challenge in development of National Highways Development Programs (NHDP), Pradhan Mantri Gram Sadak Yojana (PMGSY) and State Highways Improvement Programs (SHIPs) etc. where huge money is being invested by the Government of India in order to empower the pavement performance.

Bitumen is a civil engineering material used for construction of highways in terms of Flexible pavement. One of the advantage of bitumen as an engineering construction material is its great versatility. Bitumen is a strong binding material that has very high adhesive property and highly waterproof and durable, making it useful in road Constructions. It is also highly resistive to the actions of most acids, alkalis, and salts **[Minnesota Asphalt Pavement Association, 2003].**

The principle of use of bitumen is as a binder in the road construction where it is mixed with aggregate to produce bituminous mixture. This mixture is then laid as the structural pavement layers as base and surface course of a road. The main function of these 'bitumen-bound' layers is to transfer upcoming traffic loads evenly over the unbound pavement layers of the road and natural sub-grade to prevent failure due to overstressing**[Airey, 2009].**Bitumen being a viscoelastic material is effectively used as a binder. VG-30 and VG-10 grades of bitumen are commonly used as depending on the climatic conditions. In addition to increase the performance in terms of stiffness and elasticity, bituminous mixture must be able to resist the most and primary modes of flexible pavement distress types , namely, fatigue cracking and permanent deformation, known as rutting failure. As the mechanical properties of bituminous mixture are strongly dependent upon the properties of the binder, it has to fulfil certain mechanical and rheological requirements to ensure the integrity of the road**[Lesueur, 2009].**

Generally two characteristics of bitumen affect the service life of flexible pavement. First it has to stiff enough to resist rutting deformation at the highest pavement service temperature nearby 60ºC, depending on the climate. Second, it should be elastic enough at lower temperatures down to 20ºC depending on the local climate to resist fatigue cracking. But due to the increase in heavy traffic loading and adverse climatic conditions the conventional VG-30 bitumen is not fulfilling the performance criteria in the improvement of service life of the flexible pavement. To enhance the flexible pavement performance regarding fatigue and rutting resistance of the bitumen so as the pavement, bitumen need to be modified with some additives whose tendency is to empower the bitumen performance. Several modifiers are available in the market. But in this study sulphur in powder form has been utilized as a modifier by considering its huge production in industries.

The performance related study of bitumen is also known as bitumen rheology and its analysis is conducted by Dynamic Mechanical Analysis. The rheological properties of bitumen are typically determined in terms of dynamic mechanical analysis (DMA) utilizing a dynamic shear rheometer (DSR) tests. The test is lead within the linear viscoelastic (LVE) region **[Airey, 2002a].** The rheological properties of bitumen has been growing and importance in specifications in the USA since the early 1990's following the **Strategic Highway Research Program (SHRP).** The DSR instrument, however, does have its limitations where the measured rheological data are exposed to the measurement error particularly at low temperatures and/or high frequencies.

Apart from these the modification process has a great importance on the homogeneity of the modification of bitumen with sulphur modifier. The morphological analysis provides an idea about the disperse medium of unmodified and modified bitumen, as a result of which the homogeneity of the blending of bitumen with modifier can be observed properly.

**1.2 Problem Statement**

In India, the type of bitumen grade used is based on the penetration test, which is conducted to know the softness of bitumen at a temperature of 25°C. The pavement failure is due to heavy traffic loads and seasonal variations, which are directly, affect the durability and performance of pavements. The most common problem associated with the performance of bituminous pavements is low temperature distress type or fatigue cracking and Permanent failure or rutting failure.

The pavement surface temperature on hot summer season is within 60°C, which makes bitumen soft and results in permanent deformation as rutting in pavement. It usually occurs along longitudinal direction of the flexible pavement under the traffic wheel path accompanied by small upheavals to the sides. At low temperature in winter season, bituminous pavements become too brittle and there exist fatigue cracking. Fatigue cracking is processed as cumulative damage resulting from repeated traffic loading. Therefore, to minimize the distresses of the flexible pavement some measures can be performed such as:

- Improving the mix design of bituminous mixtures.
- By improving the construction methods and maintenance techniques.
- Introduce modification of bitumen so as to improving the bituminous mixture.

The one effective approach for pavement distresses minimization is to explore and use a new modified binder with the help of an additive. There are several additives available to develop modification of bitumen by using different additives. Sulphur has also been used as a common method to enhance the bitumen characteristics and is found to have wide range of application and potential for use as a good modifier. It is also very important to study the thermal behavior and morphology, which will provide information regarding the changes those usually occur due to modification, which affect the chemical compositions of conventional bitumen binder.

### 1.3 Objectives of Research

In general, this research is lead to explicate better understanding of the rheological properties of modified and unmodified bitumen binders. Considering the problem statement above, the main objectives of this research are summarized as follows:

1. The aim of this study is to explore the use of modified binder to improve the performance of flexible pavements.
2. The dynamic shear rheometer (DSR) is used to determine the rheological characteristics of bitumen binder over a wide range of temperature and rate of loading conditions.
3. Comparing the rheological properties at high, medium, low temperatures for unmodified bitumen and modified bitumen by Dynamic Mechanical Analysis.
4. The effect of sulphur on modification of bitumen in terms of rheological, storage stability and morphology has been studied.
5. The effect of ageing on unmodified and modified bitumen rheology and morphology using Rolling Thin Film Oven (RTFO) and Pressure Aging Vessel(PAV) and FESEM respectively.

## 1.4 Scope of the study

The scope of this study is to focus on the characterization of sulphur modified bituminous binder. The evaluation of rheological properties of VG-30 bitumen binder without and with modification with sulphur from dynamic mechanical analysis followed by morphological and thermal analysis is the main aim of this study. The rheological properties, creep recovery tests, morphology and thermal analysis are conducted using dynamic shear rheometer (DSR) and field emission scanning electron microscope (FESEM), apparatus. Ageing of bitumen and modified binders has been understand using the Rolling Thin Film Oven (RTFO) for short term ageing and Pressure Aging Vessel (PAV) for long term ageing and effect of ageing on the rheological, morphological and thermal parameters are studied.

# CHAPTER-2

## LITERATURE REVIEW

### 2.1 Introduction

The aim of this study is to gather some knowledge of previous research work those has been already executed, which are related to rheology, morphology and thermal characteristics of unmodified and modified bitumen binder. This chapter covers five sections through which related literatures of this research work has been included.

**Section 1** deals with the literature review concerning constitution of bitumen with its elementary analysis and chemical composition.

**Section 2** the literature survey gives a brief description of the Empirical tests as softening point, penetration and viscosity.

**Section 3** described the bituminous binder rheology, its viscoelastic characteristics, effect of ageing, chemical composition, thermal analysis and morphology of bituminous binder.

**Section 4**this section deals some common distresses inflexible pavement.

**Section 5** deals with the modification of bitumen. The review included various types of modifiers and different modification mechanism.

### 2.2 Bitumen constitution (chemical composition)

#### 2.2.1 Elemental Composition of Bitumen

The bitumen is a dark brown to black in color, sticky and viscous material which is composed of high molecular weight hydrocarbons. It is obtained from the bottom of the vacuum distillation columns in the crude oil refineries.

Elementary analysis of bitumen manufactured from the various crude oils has varying physical properties. It is predominately consists of carbon and hydrogen. Most bitumen binder contains heteroatom such as carbon, hydrogen, sulphur, oxygen and nitrogen **[Whiteoak(1990)].**The elementary analysis of the bitumen binder is summarized below in Table 2.1.

Table 2.1 Elemental Analysis of Bitumen **[Whiteoak.(1990)]**

| **Component of Bitumen** | **Percentage** |
|---|---|
| Carbon | 82% -88% |
| Hydrogen | 8% -11% |
| Sulphur | 0% -6% |
| Oxygen | 0% -1.5% |
| Nitrogen | 0% -1% |

### 2.2.2 Chemical Groups of Bitumen

Bitumen consists of two major chemical groups known as asphaltenes and maltenes. Further the Maltenes has three groups under it. The major chemical composition for bitumen is shown below [figure 2.1]**[Robert et al (2000)].**

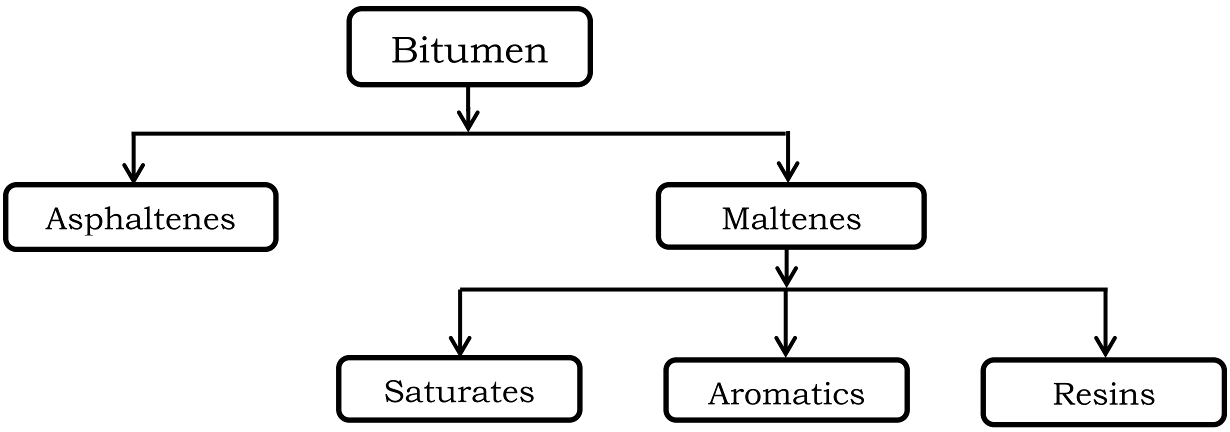



Figure 2.1ChemicalGroups of Bitumen **[Robert et al (2000)]**

**Asphaltenes:** Asphaltenes are dark brown in color friable solids with highest polarity and insoluble in non-polar solvents. Bitumen with higher asphaltenes content will have more viscosity and lower penetration.

**Maltenes:** Maltenes is divided into three groups, such as saturates, aromatics and resins.

**Saturates:** Saturates are straight and branched chain hydrocarbons. These are non-polar viscous oils, white in color, with similar molecular weight range to aromatics.

**Resin:** Resins are dark semi solid material having lower molecular weight. At high temperature it is liquid and brittle at low temperature. It acts as dispersing agents to the asphaltenes.

**Aromatics:** These are the lowest molecular weight compounds in bitumen. These are dark brown color viscous liquids.

## 2.3 Empirical testing

### 2.3.1 Penetration test

The penetration test is conducted in terms of determination of consistency and indirect determination of the viscosity of the bitumen at 25ºC, to identify the grade of bitumen. The penetration value is described in tenths of a millimeter (decimillimetre, dmm).

For penetration value less than 30 dmm, the bitumen sample is to be known as hard. On the contrary the penetration values more than 100 dmm relate to soft bitumen. For example, 40/60 penetration grade bitumen has a penetration value at 25 ºC ranging from 40 to 60 in units dmm. Therefore, a variety of bitumens can be easily graded and specified based on the penetration results **[Lesueur, (2009)].**

### 2.3.2 Softening point test

The softening point test is generally conducted by ring and ball apparatus to determine the consistency of bitumen by measuring the temperature at the commencement of the fluidity of bitumen.

Water is used for bitumen with a softening point of 80ºC or below. Meanwhile, glycerin is used for softening points greater than 80ºC **[Read and Whiteoak, (2003)].** The softening point is used to determine the temperature when bitumen softens and deforms slowly. Under this state, typical value of paving grade bitumen ranges between 35°C and 65°C. A hard bitumen usually has a softening point nearby 60°C while a softer grade of bitumen will have a softening temperature around 40°C **[ Lesueur, (2009) ]**.

### 2.3.3 Viscosity tests

Resistance to flow of a liquid is known as Viscosity and also can be defined as the ratio between the applied shear stress and the rate of shear strain. It is well known as a fundamental characteristic of bitumen. Viscosity of a fluid are defined in two ways as absolute and kinematic viscosity. In general, specifications are based on determination of absolute viscosity at 60°C and kinematic viscosity at 135°C using Dynamic shear rheometer and vacuum tube capillary viscometers respectively. Absolute viscosity can also be measured using a fundamental method known as vacuum capillary tube viscometer.

The rotational viscometer test according to ASTM D 4402-02 is presently considered to be the most practical means of determining the viscosity of bitumen. The thermocel system based Brookfield rotational viscometer, allows the testing of bitumen over a wide range of high temperatures. The viscosity of bitumen is determined for various shear rates according to the variation in R.P.M. of the spindle. To get very accurate viscosity torque of the spindle should be at least 10%. The torque on the rotating spindle is used to determine the relative resistance to rotation of the binder at a particular temperature and shear rate. The torque value is then changed by means of calibration factors to yield the viscosity of the bitumen **[Airey, (2009)]**.

## 2.4 Fundamentals of Bitumen Rheology

The performance of bituminous pavements are not characterized only by physical properties as they are subjected to complex environmental and loading conditions. In addition to this modified bituminous binders also cannot essentially be characterized only by the empirical properties. It is very much important to understand stress-strain behavior of bituminous binders under a wide range of loading time and temperatures conditions. Thus, fundamental tests were established to investigate dynamic mechanical properties and viscoelastic behavior of binders under various environmental conditions. The Strategic Highways Research Program (SHRP), United States of America, developed a synchronized effort to produce binder specifications which are classified based on performance-grade system in accordance with the fundamental testing results**[Petersen et al., 1994; Anderson et al., 1994].**

### 2.4.1 Rheology

Rheology is a part of continuum mechanics and the study of flow and deformation. Rheology is the description of the dynamic mechanical properties for different materials under various deformation conditions **[Vinogradov et al (1980)].** The rheological properties of asphalt binder as an indicator of performance of flexible pavement, which are related to the permanent deformation and fatigue cracking of flexible pavement at high and low temperature respectively. With improved rheological properties of asphalt binder, resistance to fatigue and rutting stiffness values has been improved **[Bahia and Davies (1994)].**

The rheological properties of asphalt binder play an important role in the performance of asphalt flexible pavement. The fundamental asphalt binder rheology can be used to quantify the performance asphalt flexible pavement. Dynamic shear rheometer (DSR) apparatus is used to evaluate the rheological properties of asphalt binder [**Anderson et al (1994)**]. Dynamic shear rheometer was used to evaluate the effect of ageing on polymer modified bituminous binder rheology. The test results are used to evaluate the changes in the rheological properties of SBS polymer modified binder. He concluded that there is increase in the viscous behavior o f modified binder after aging as compared to elastic behavior of unmodified bitumen [**Airey. (1997)]**.

The dynamic shear rheometer (DSR) was used to characterize the viscoelastic behavior of bituminous binder over a wide range of temperatures. The Stress-strain behavior defines the response of binder to loading conditions. Asphalt binder exhibits both elastic and viscous behavior for which it is well known as a viscoelastic material **[Bahia and Anderson (1995)]**.

The asphalt rheology is the study and evaluation of the flow and permanent deformation for time and temperature dependent materials and results are reported in terms of complex modulus and phase angle. Also the variations of complex modulus and phase angle with respect to change in frequencies plots are used to characterize the nature of binder [**En. Nur et al (2011)]**. The rheological plots of bitumen used to characterize the behavior are presented in below:

- Master curves: - A curve of relationship between complex modulus and frequencies for various service temperatures.
- Isochronal plot: - A graph representing the behavior of complex modulus as a function of temperature of the system at a constant frequency.
- Black diagram: - A graph of complex modulus versus phase angles
- Cole-Cole diagram: - A graph of loss modulus as a function of storage modulus

Time sweep test using dynamic shear rheometer provides a method of applying repeated cyclic stress or strain loading at service temperatures and loading frequencies. Advantages of the time sweep test is that, it can be used to evaluate the fatigue life of asphalt binder based in dissipated energy approaches [**Bahia et al (1993)]**.

### 2.4.2 Viscoelastic Behavior of Bitumen

Bitumens are well known as viscoelastic materials. Behaviors of these materials are comprises of two parts: elastic behavior and viscous behavior. Materials with elastic behavior reoccurrence to their initial state after amputation of the applied loads, whereas permanent deformations persist under applied loads in viscous behavior. Most important factors affect the behavior of viscoelastic materials by means of their elastic and viscous behavior are temperature and loading conditions. Temperature is the most critical among the parameters. Viscoelastic materials possess more elastic behavior at low temperatures, whereas they perform more viscously at high temperatures. The second parameter is loading time or rate of loading. Bitumen exhibits an elastic solid at high rates of loading, whereas it behaves like a viscous liquid at long times of loading. Bitumen, therefore, possess high stiffness and brittleness at short times of loading, whereas it is related with high ductility and shows low stiffness at long loading times **[Jongepier, R., and Kuilman, B (1968)].**

This study is to quantify the "Viscoelastic properties of high-consistency asphalt". It is to be expected that the outcomes of other investigations into the flow characteristics of stiff asphalts will find their way into pattern. Such studies can ultimately lead to preparation of asphalts of a much wider range of abilities. **[A. B. Brown., et al (1957)**].

The bitumen is a viscoelastic material and its behavior depends on the rate of loading and temperature. Bitumen behaves as elastic solids at low temperature under small loading time. It behaves as liquid at high temperature under long rate of loading. But bitumen becomes more complex at intermediate temperature under intermediate rate of loading as its behavior becomes viscoelastic [**Robert (2000)**].

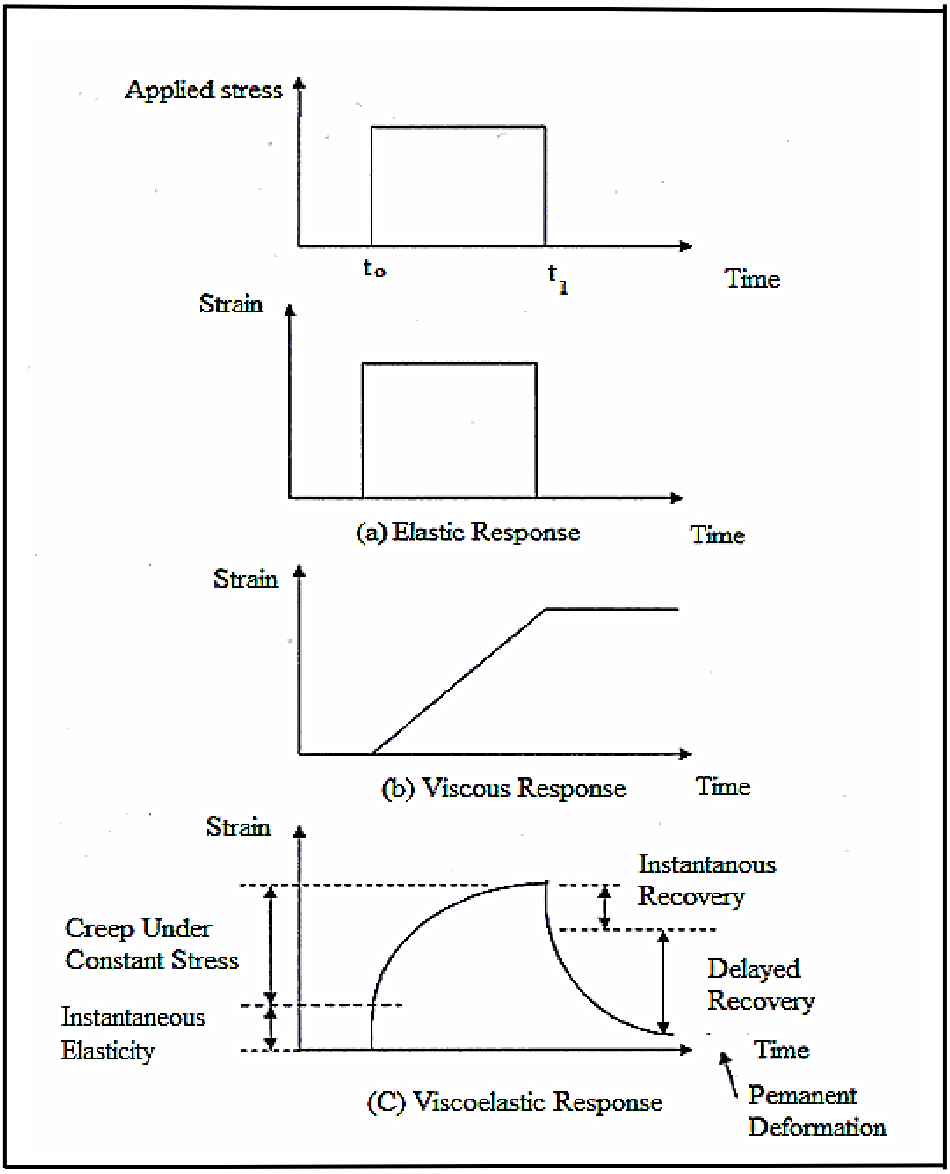



Figure-2.2-Idealized response of a material under constant stress loading**[**Source**:** Manual for CVOR 100 Bohlin Rheometer**]**

(a) elastic response of material under constant loading
(b) viscous behaviour of material over time under constant stress
(c) behaviour of a viscoelastic material under constant stress

The characterization of the relaxation modulus has been determined for an asphalt rubber mix and for a empirical dense hot mix, in terms of a set of one cycle long duration load. The tests results have been tailored in the kernel function of the generalized Maxwell elements, G(t) and K(t), demonstrating the shear and bulk relaxation modulus, respectively.[**Manuel J. C. Minhoto., et al (2005)**].

Relevant rheological modeling based on physical elements was considered for evaluation of the viscoelastic behavior of bituminous binders and mixes using complex shear modulus tests [**Herve Di Benedetto., et al (2011)]**. The characterization of the LVE response of asphalt mixture was proposed having several advantages over the conventional method of building a viscoelastic function master curve by fitting a sigmoidal function to test results [**Yanqing Zhao., et al (2013)]**.

## 2.5 Ageing of Bitumen

Aging of bituminous binders is one of the key factors determining the lifespan of a flexible pavement. The process of aging involves chemical and physical property changes that make bituminous binder stiffer and more brittle, thus increasing pavement failure. The aging-related pavement failure modes include thermal or traffic induced cracking and raveling **[Xiaohu Lu, Ulf Isacsson (2001)].**

Bitumen aging takes place in two stages, namely short-term aging at high temperature during asphalt mixing, storage and lying and long-term aging at ambient temperature during in-service period. The mechanisms of aging include evaporation, oxidation and physical hardening. Both the short term and long term aging are conducted by using Rolling Thin Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) respectively and are summarized below.

### 2.5.1 Rolling Thin Film Oven Test

The rolling thin film oven (RTFO) test is used to conduct the short term aging occur in the bituminous binder during the mixing process under high temperature. The residue from RTFO then tested for penetration, softening point and rheological properties and for Long term aging process.

### 2.5.2 Pressure Aging Vessel:

The SHRP organization developed a method to simulate the changes in the physical and chemical property of bitumen under long term aging during service life using the pressure aging vessel (PAV) compare to oxidative aging after 5 to 10 years in field. The method involves oxidation of the bitumen using RTFOT followed by the oxidation of the pressure aging vessel.

Hardening of the bituminous binder occurs in two stages. The first stage is loss of volatile components during sample preparation before lying in the field, well known as short-term aging under high temperature. The second stage is the oxidative hardening which is known as long term aging during entire service life of the pavement [**Anderson (1994)**]. There are four important mechanism of bitumen ageing:

- Oxidation
- Loss of volatile materials
- Exudative hardening ( loss of lighter bitumen fractions by absorption into the Aggregate) and Steric Hardening

Polymer modification can enhance mechanical characteristics of asphalt binder at both high and low temperatures, although the low-temperature test result is much less obvious and more indefinite. Primarily polymeric modifiers may improve an asphalt binder's ductility significantly [**YonghongRuan. et al (2003)].**

Ageing in RTFOT conditions influences bitumen and polymer bitumen chemistry and rheology. As a consequence of this, the rheological properties of BITs and PMBs dispersions are changed and their share viscosity increases. The changes of rheological properties of aged BITs and PMBs under the traffic loading conditions are connected with destroying the swelling polymer network [**Vesna Rek. Et al. (2005)**].

High pressure gel infusion chromatography and Fourier Transform Infrared spectroscopy, the aging properties of both neat asphalt and modified asphalt were calculated. The outcome of a small amount of zinc dialkyldithiophosphate (ZDDP) on the chemical and physical properties, and molecular weight dispersal of asphalts was studied. [**Chunfa Ouyang. et al (2005)**].

## 2.6 Morphology Analysis

Morphology is a branch of science deals with the study of the arrangement and structure of precise structural features of materials. This study comprises features of the appearance in terms of shape, structure, color, and pattern as well as the structure of the internal matrix.

### 2.6.1 Morphology of Bitumen

Morphology of bitumen indicates the homogeneity of the modified bituminous binder. Different researchers have conducted several morphological analyses to have a proper blending of modified binder.

The compatibility of the polymer modifier inside the bitumen mostly depends on molecular weight. As higher molecular weight polymer additives are incompatible with bitumen possessing lower molecular weight [**Whiteoak (2003)**]. Field emission scanning electron microscopy (FESEM) has been carried out to study the interior morphology of the PMB. Prominent phase separation was not observed although the viscosity of the mix was increased as being proved by penetration results, PP and LLDPE seems to be better modification establishing homogenous mix in comparison to HDPE. [**Noor Zainab Habib. et al (2010)**].

## 2.7 Distresses in bituminous pavements

Flexible pavement distresses begin to accumulate due to pavement service life and increase in repeated traffic wheel load. Low temperature Fatigue cracking progresses in flexible pavement bound layer mainly appears due to brittleness of bituminous mixes, ageing, temperature and rate of loading. Due to the complex interaction between these variables, advanced mechanics such as damage analysis, viscoelasticity, and fracture mechanics to comprehend the failure need to be introduced **[Terrel (1971)].** Structural failure of flexible pavements is in the form of fatigue cracking and permanent deformation known as rutting failures, which are established due to load repetitions, adverse environmental conditions and viscoelastic behavior of bituminous mixes. As a result of which the service life of pavement lead to structure collapse **[Monismith et al (1985)].**

Fatigue failure normally takes place in two stages. One is initiation of crack and second is propagation of crack. The pavement distress is considered as complex phenomena as several factors associate with the pavement deterioration [**Lytton et al (1994)**]. The main causes of the pavement distresses are:

- ✓ Lower quality of materials used.
- ✓ Deficiency in constructional methodology and quality control.
- ✓ Improper drainage of Surface and subsurface.
- ✓ Settlement in the pavement soil subgrade.
- ✓ Adverse effect of environment such as heavy rainfall, frost action, temperature variations.

Asphalt pavement distresses are categorized into cracking, surface deformation, and surface defects. The reasons for asphalt pavement damage are disintegration, fracture and visco plastic flow [**Miller and Bellinger (2003)**].

Flexible pavement fatigue cracking is due to the development of the horizontal tensile strain at the bottom of the asphalt mixture layer. In the crack initiation stage water percolate in the cracks which led to materials strength reduction under repeated loading action. As a result of which crack start to propagate and lead to pavement collapse [**Bahia (2006)**].

## 2.8 Modification of bituminous binders with additives

Conventional bitumen being a viscoelastic binder performs satisfactory in most of the flexible pavement. But the conventional binder is not able to sustain due to increase in heavy traffic loads, adverse effect of seasonal variations. To increase the service life of flexible pavement in terms of empowering its resistance is not sufficient enough to withstand against the most common failures of flexible pavement. Hence, to enhance the properties of bituminous binder it need to be modified so as to serve in a better way. As a result of which the resistance capacity against the fatigue and rutting failure can be improved.

The reasons for which bituminous binder is modified with various types of additives To increase the strength and the stability of bituminous mixtures is to reduce rutting failure and become stiffer blends at high temperatures and to improve fatigue resistance of blends, [**Lewandowski (1994)**].

Effect of polymer modification using scanning electron microscope images. The result showed that the modified asphalt concrete mixtures have better binder-aggregate adhesion, which led to increase in its toughness. The role of modified bitumen is to increase the resistance of asphalt to permanent deformation at high temperatures **[Bahia (1995)].**

## 2.9 Modification of bitumen with sulphur

Bitumen altered with 2% of sulfur at $160^0$ C coming about more plastic consecutive to a variety of the way of the collaboration between asphaltenes particles **[Fritchy et al (1980)].** Determination of the attainability of utilizing dune sand as a part of asphalt-concrete in hot, desert like atmospheres through use of single size crushed aggregates. Thick reviewed total and powdered sulfur were utilized within the sand asphalt mixes **[Fatani and Sultan (1982).**

Sulfur utilized as a restoration executor in recycling reclaimed asphalt pavement from a normal fizzled section of Dammam-Abu (Hadriyah Expressway) **[Arora and Rahman (1985)].** Research facility testing system was planned to measure upgrades in building properties of sulfur-asphalt-sand (SAS) blends attributable to the vicinity of sulfur in the mix considering mainly accessible sands and predominating natural conditions in eastern Saudi Arabia **[Akili (1985)].** Sulfur augmented asphalt as a real outlet for sulfur that outflanked other asphalt mixes blends in the Gulf [M**ohammed et al (2010)].**

The **Federal Highway Administration (FHWA)** finished a field study to analyze the execution of sulfur-expanded asphalt (SEA) to expected asphalt concrete (AC) . The essential decision was that there was no distinction in general execution between the SEA and AC segments. Sulfur did not build or abatement most test properties, and regularly it had no impact on a given test property of a mixture. Sulfur did diminish the imperviousness to dampness helplessness in the research facility. There were likewise minor patterns showing that with some mixtures, sulfur might decrease the helplessness to rutting and expanded the weakness to fatigue cracking.

Sulfur extended asphalt blend utilized within asphalt mix design. It finishes up that Thiopave has the potential to decrease the general obliged asphalt pavement depth while as of now controlling strain at the bottom of the asphalt **[David and Mary (2009) ].**

The convergence of the altered sulfur pellets in the mixture is intended to improve asphalt mixture properties keeping up workability and similarity. The results inferred that the asphalt mixtures holding the changed sulfur pellets were indicated enhanced execution contrasted with the customary asphalt mixtures **[Bailey and Allen (2009)].**

Flexible asphalt pavement material solidness fundamentally impacts exhaustion fatigue and rutting execution. Subsequently, picking high-modulus asphalt concrete can possibly expand the general life of the asphalt **[Richard (2010)].** It is liked to utilize an expository technique, which represents stiffness and exhaustion instead of utilizing a strategy that just uses solidness, as it can propose a layer thickness decrease, which can have a hindering impact on asphalt life **[Colange et al (2010) ].** Hot fluid sulfur was control blend and second era sulfur-modified asphalt mixtures with fluctuating binder substitution levels **[Taylor et al (2010)].**

# CHAPTER-3

# METHODOLOGY

## 3.1 Introduction

The viscoelastic behavior of bitumen is exceptionally complex to depict by basic traditional experiments of consistency, for example, penetration tests and softening point tests. Hence, the assessment of bitumen attributes ought to be focused around its performance regarding fatigue and rutting safety. Hence, new test instruments like the Dynamic Shear Rheometer (DSR), Brookfield Viscometer have been created to give rheological properties of bitumen over an extensive variety of loading and encompassing conditions.

The DSR might be acknowledged as the most compelling and complex instrument for characterization of the bitumen flow properties. It is additionally really vital to comprehend the chemical progressions of bitumen that has been made throughout change by sulphur. To study the chemical compound arrangement framing, thermal and morphological investigation of unmodified and modified bitumen, a few tests have been led utilizing new innovation instruments, for example, FESEM, TGA, DTA and FTIR Spectroscopy individually.

## 3.2 Determination of rheological properties of bitumen

Rheological properties are utilized as execution parameter has favorable circumstances and disadvantage. The point is that it permits estimation of physical properties with wide temperature range at high and low recurrence, which is prone to be accomplished in the field because of movement. Dynamic shear rheometer need qualified individual with high encounter to work the element tests and additionally to get great rheological results. In this section a concise representation of the element shear rheometer (DSR) device and in addition the geometry and example creation and example measurement will be exhibited. In this section additionally a point of interest description of all rheological test methods received for the characterization of materials are given.

The examination led for the Strategic Highway Research Program (SHRP), testing system acquainted with describe the rheological, durability and failure properties of asphalt binders totally focused around the rheological properties. The examination results were examined in four principle points:(i) The viscoelastic nature of bitumen and its connection to performance of pavement; (ii) the crucial issues identified with these tests and; the sorts of traditional estimations are utilized now (iii) the idea of selecting the new test routines and the new properties; and (iv) how to analyze the new measured properties to the traditional properties [**Bahia et al (1993)].**

### 3.3 Viscoelastic Properties of bitumen

Viscoelastic properties from Dynamic mechanical analysis through DSR indicate the reaction of a material as it is subjected to a cyclic stress. These properties may be communicated as far as dynamic storage modulus, dynamic loss modulus, and a mechanical damping term. The performance of bituminous binders is affected by viscosity and two critical rheological parameters as phase angle and complex modulus. The constraints and their effect over bitumen performance have been talked about quickly underneath **[Airey]**.

#### 3.3.1 Viscosity of bitumen

Viscosity is defined as the resistance to flow. Bitumen is a visco elastic material that is at room temperature it act as a semi solid in high temperatures over 60°C it acts as a Newtonian fluid or low viscosity liquid. Hence viscosity is the main two properties checked to categories bitumen. Viscosity of bitumen is characterized in two different ways one being absolute or dynamic and other is Kinematic Viscosity.

Absolute viscosity or dynamic viscosity (60°C) is defined as the resistance of a material when it is subjected to an external and controlled shear stress, which represents its internal resistance to the applied force and is calculated as given in equation 3.1 .

$$\text{Absolute Viscosity (Pa·s)} = K^*t \quad (3.1)$$

Where
K = calibration factor, (Pa · s/s)
t = flow time(s)

Similarly Kinematic viscosity (135°C) can be defined as the resistance of a material, when it is subjected to no external force. Generally speaking, kinematic viscosity (cSt) is associated to absolute viscosity (cP) as a function of the material's specific gravity (SG) according to the equations 3.2.

$$\text{Kinematic Viscosity (cst)} = \text{Dynamic Viscosity/ Density} \quad (3.2)$$

**3.3.1.1 Measurement of Viscosity**

Viscosity of bitumen can be measured in several methods such as through DSR, Brookfield rotational viscometer and Capillary Viscometer. The working principles of measurement are different for all the above mentioned viscometer and are briefly summarized below.

**3.3.1.2 Brookfield Viscometer**

The Brookfield rotary method shown in [Figure 3.1] is the most common method for determination of viscosity of fluid. Absolute viscosity evaluation has traditionally been used for research applications, quality control and grease analysis within the field of machinery lubrication. Its working rule is that an overall composed shaft is to pivot with legitimate unrest for proper revolution inside a bitumen filled metal tube applying shear stress and torque and consequently the resistance given by the bitumen will be calibrated and viscosity of the bitumen will be assessed.

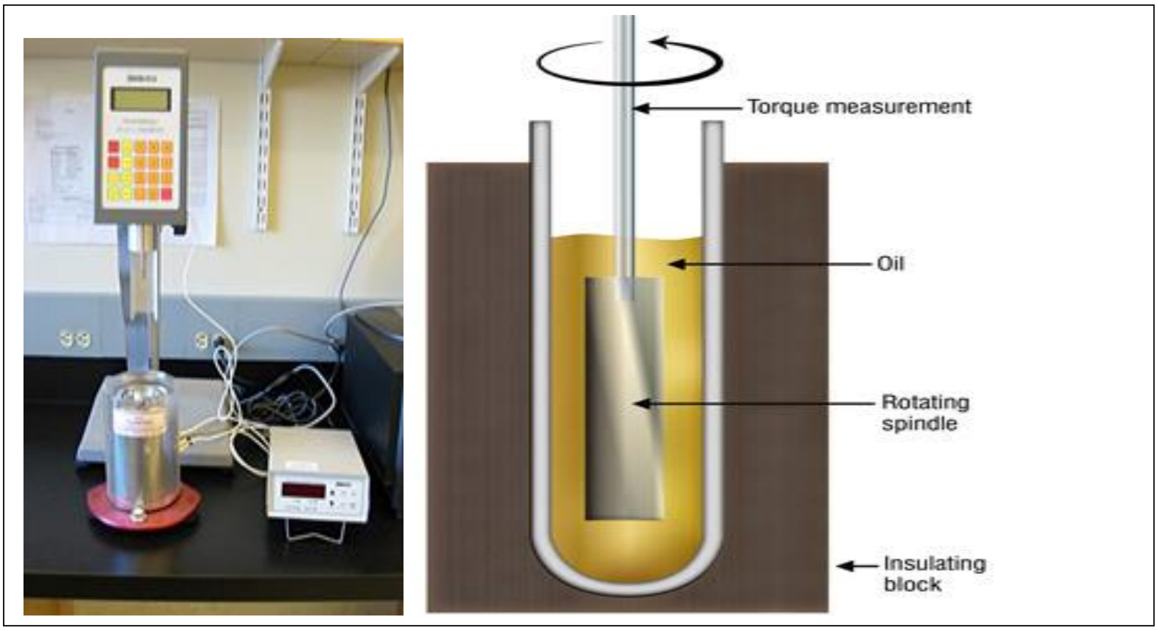



Figure 3.1: Rotational Viscometer and working principle **[**www.pavementinteractive.org**]**

This viscometer is meant for evaluation of viscosity at high temperature at least the temperature at which the sample begins to flow. Disadvantage of this instrument is that absolute or dynamic viscosity cannot be evaluated due to pivoting of spindle is not possible inside the fluid filled tube at low temperature.

**3.3.1.3 Capillary Viscometer**

This viscometer is utilized to assess both absolute and kinematic viscosity according to ASTM D 2170 and D 2171. Absolute viscosity is adjusted regarding resistance offered by the liquid against the vacuum pressure, when the liquid is inside a legitimately outlined glass tube. Correspondingly the kinematic viscosity is aligned as far as resistance of liquid to stream under gravity at specified temperature inside an exceptionally designed glass tube. The temperature is maintained by silicon oil for both the cases. The viscometer and its component parts are shown in [figure 3.2] below.

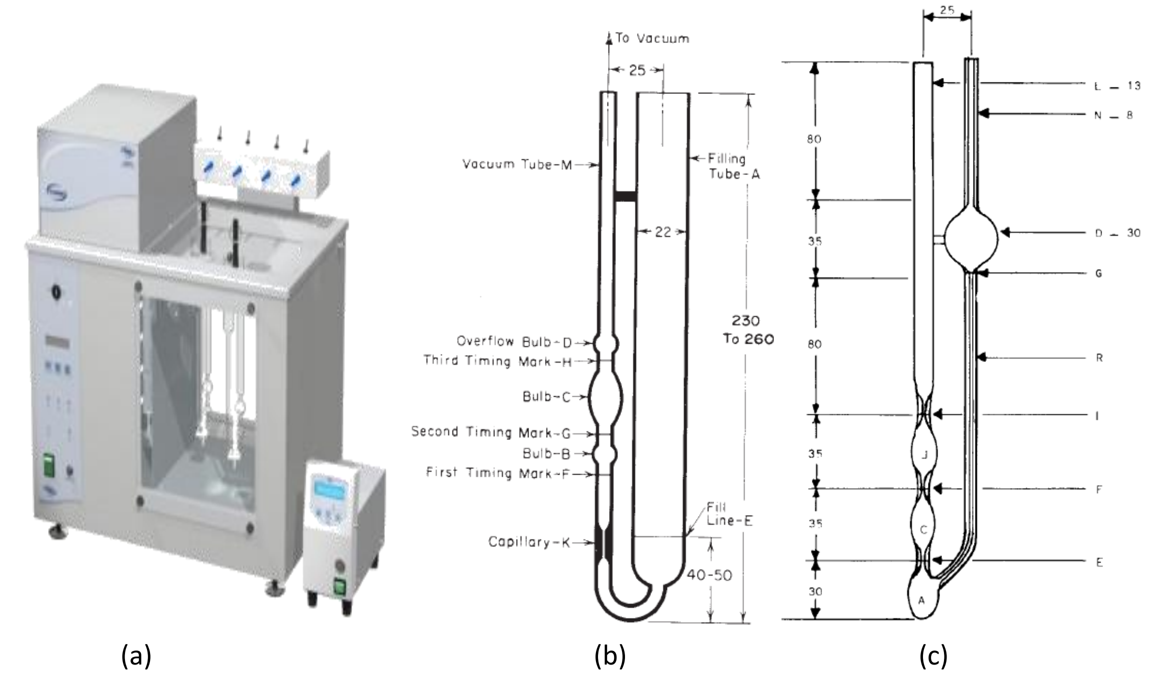



Figure 3.2: (a) Cannon Capillary Vacuum Viscometer (b) Glass tube for Absolute viscosity measurement (c) glass tube for Kinematic viscosity measurement [**ASTM D2170 & D2171]**

### 3.3.2 Dynamic Shear Rheometer

Dynamic shear rheometer (DSR) was utilized to measure visco-elastic properties, fatigue failure, splitting also rutting at low, middle and high temperature. DSR is likewise characterized as a binder characterization method and used to focus the rheological properties of asphalt binders. This gadget utilized where sinusoidal shear push or strain is connected as sinusoidal time capacity to make dynamic oscillatory burden. DSR gadget was utilized to measure different binder properties of unmodified and modified bituminous binders. The dynamic shear rheometer testing equipment and its working principle are shows in [figure3.3 and 3.4].

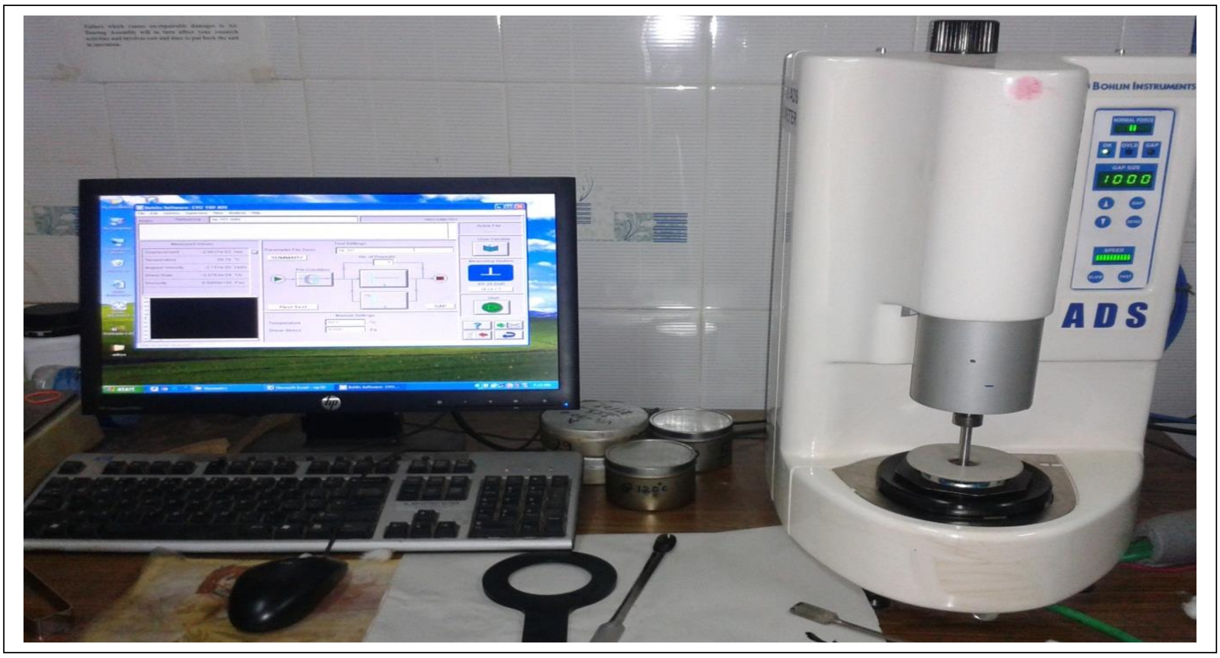



Figure 3.3: Showing Stress Controlled Dynamic Shear Rheometer (DSR)

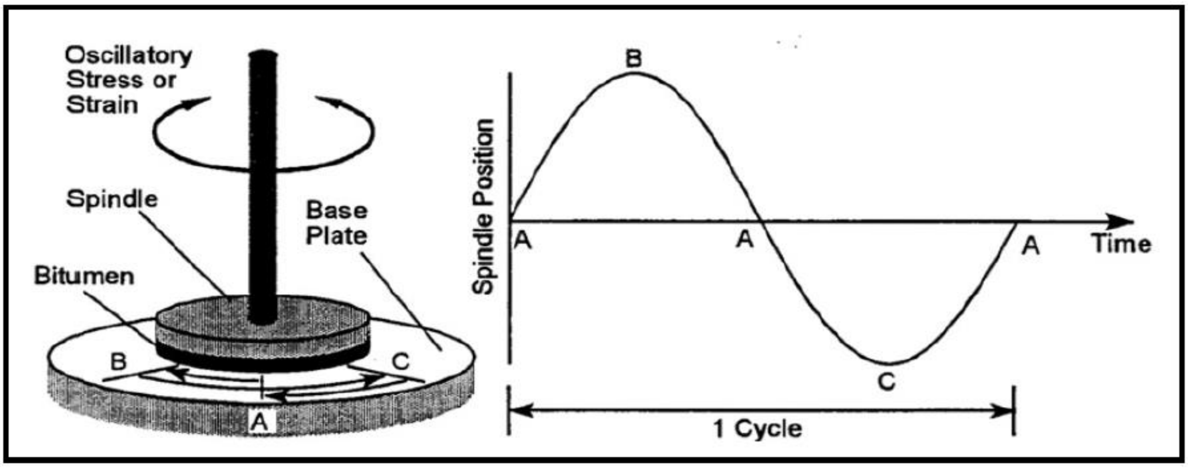



Figure 3.4: Working Principle of Dynamic Shear Rheometer**[Airey, 1997]**

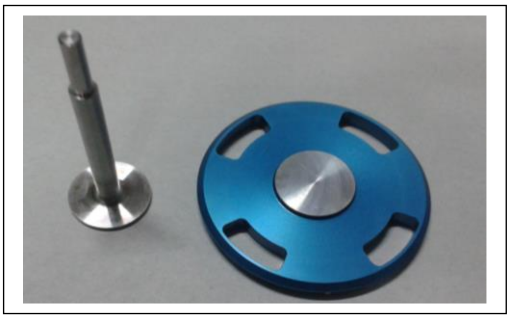
The wavering provides for one smooth, persistent cycle which might be rehashed persistently throughout the test. DSR tests are conveyed out over an extensive variety of frequencies (i.e. number of cycles for every second) and temperature. The element burden might be given as sinusoidal time capacity, which is introduced in the accompanying mathematical statement in equation 3.3.

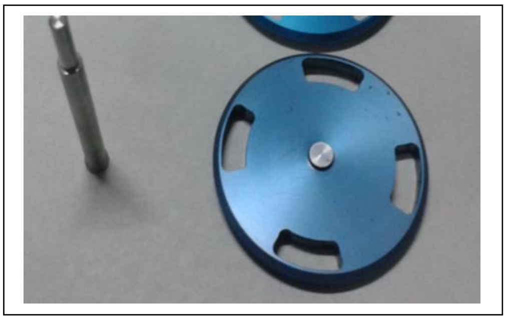
$$\tau = \tau_0 \text{ Sin } (\omega t) \qquad (3.3)$$

Dynamic shear Rheometer is utilized to focus the sample's reaction to the dynamic load. DSR tests might be completed in two testing modes, for example, controlled stress or controlled strain. In the controlled strain mode, a specified extent of shear strain is connected to the bitumen and resultant shear stress is computed. In the controlled stress mode, a specified size of shear stress is connected to the bitumen and resultant shear strain is compute.**[Airey,1997].**

### 3.3.2.1 Specimen geometry

The DSR geometry was picked as indicated by the test condition and particular. The 25mm diameter geometry with specimen thickness (1mm) utilized for high temperature test to spare the sample from dissolving. At intermediate road temperature the sample ought to have little diameter (8mm) with specimen thickness (2mm) to keep it from fatigue failure. The DSR geometry has been shown in [figure 3.5].The bitumen specimen is sandwiched between two parallel plates. The upper plate geometry is permitted to pivot about its own particular hub while base plate stays settled throughout testing.





25mm Parallel Plate 8 mm Parallel Plate

Figure 3.5: Two Types of Parallel Plate Geometry

### 3.3.2.2 Theoretical analysis and Data collection

The analysis of the tried sample could be exhibited as elastic or viscous segment as rheological parameter complex modulus and phase angle. The information securing unit records the test temperature, angular frequency, applied stress and deflection angle throughout the test cycles, which specifically sends the test information to the PC. The workstation programming figures the rheological parameters, for example, shear stress, shear strain, phase angle and complex modulus and present it in form of tables and figures.A few specialists' remarks about the rule hypothetical investigation and accumulation of yield test results utilizing Dynamic shear rheometer are outlined underneath.

The rheological tests were done concurring to the **AASTHO: T 315-08**(determining the rheological Properties of asphalt binder using a Dynamic Shear Rheometer (DSR)). This test technique holds the determination of the dynamic shear modulus and phase angle of bitumen binder when tried in dynamic (oscillatory) shear utilizing parallel plate test geometry. This test is relevant for the bitumen binder having dynamic shear modulus values in the reach from 100 Pa to 10 MPa got between 6 to 88° C with a precise angular frequency of 10 rad/s. Dynamic shear rheometer, temperature unit and information procurement unit are exhibited.

#### 3.3.2.3 Phase Angle

For an applied stress shifting sinusoidal with time, a viscoelastic material will additionally react with a sinusoidal strain for low amplitudes of stress. The sinusoidal variety in time is typically portrayed as a rate specified by the recurrence (f = Hz; $\omega$ =rad/sec). Phase angle is defined as the lag between the applied stress and resulting strain of a body when subjected to a sinusoidal shear stress. The phase angle is a critical parameter to portray the viscoelastic behavior of bitumen, which is a yield result from the dynamic mechanical examination through DSR. Typical phase angle has been illustrated below[figure 3.6].

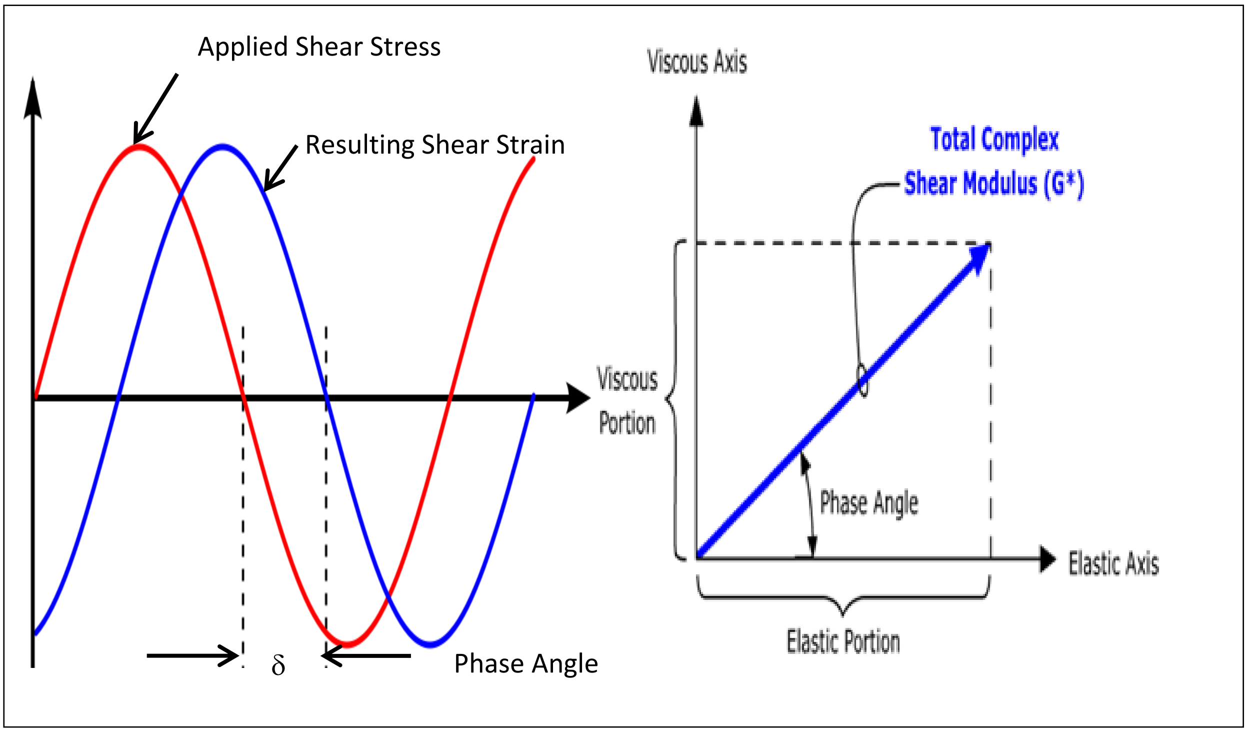



Figure3.6: Illustrating the phase angle and complex modulus**[**http://mrbhr.com]

### 3.3.2.4 Complex Modulus

Complex modulus acquired from dynamic mechanical test comprises of "genuine" and non-existent" parts. The genuine/real (storage) part portrays the capability of the material to store potential energy and discharge it upon deformity. The nonexistent/imaginary (loss) allotment is connected with energy dissemination as hotness upon distortion. The mathematical statement is revised for shear modulus as follows in equation 3.4.

$$G^* = G'^2 + G''^2 \qquad (3.4)$$

Where G′ is the storage modulus and G′′ is the loss modulus. The phase angle δ is given by equation 3.5.

$$\tan(\delta) = G''/\,G' \qquad (3.5)$$

The dynamic properties give data at the atomic level to comprehension the polymer mechanical behavior. Dynamic modulus comprised of two components such as elastic component and viscous component, which are also known as the storage modulus (G′) and loss modulus (G″).Storage modulus (G′):- storage modulus can be defined as the elastic (recoverable) component. Loss modulus (G″):- loss modulus can be defined as the viscous (non-recoverable) component.

The relationship between the complex shear modulus (G*), storage modulus (G′), loss modulus, (G″) and phase angle (δ) are given below. There will be various studies for utilizing the rheological methods to anticipate asphalt performance focused around the principle two rheological parameter complex shear modulus and phase angle**[Bahia et al (1995)].**The relationship between the loss modulus and storage modulus has been represented in graphical form [figure 3.7] their limiting values are given in equation 3.6 (a) & (b) below.

Absolutely viscous:

$$\delta = 90°;\ G' = 0;\ G'' = G^* \qquad 3.6\ (a)$$

Purely elastic:

$$\delta = 0°;\ G'' = 0;\ G' = G^* \qquad 3.6\ (b)$$

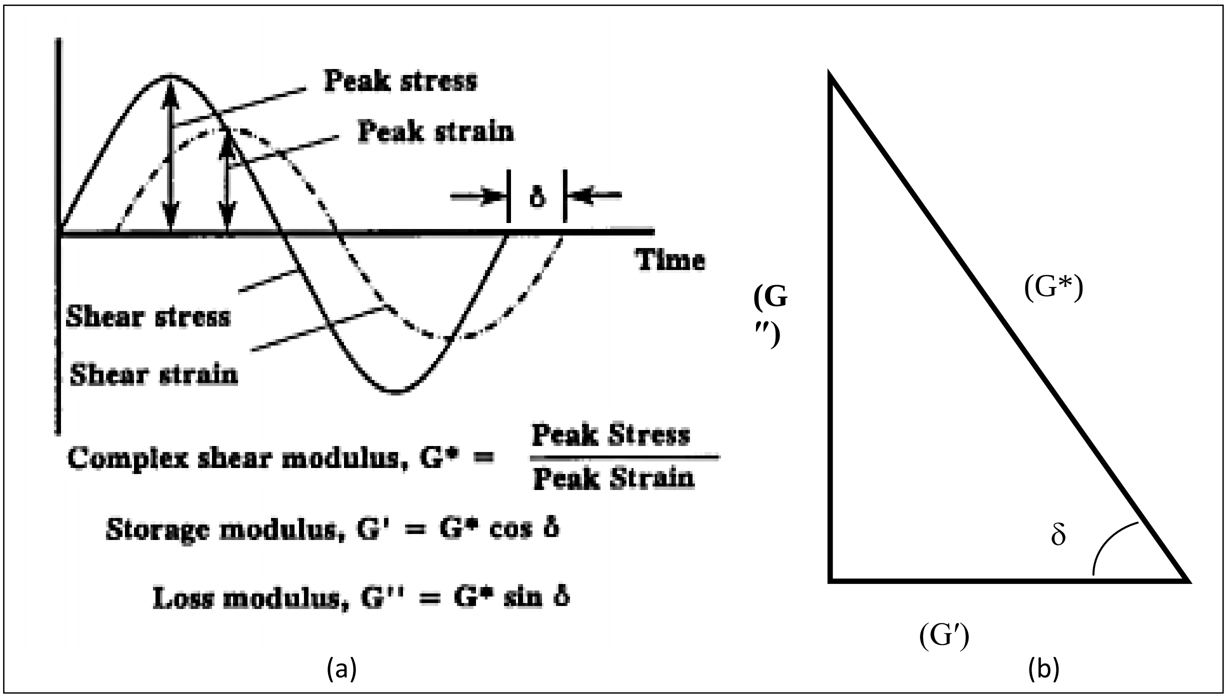



Figure 3.7: Relationship between Complex Shear Modulus (G*), Storage Modulus (G′), Loss Modulus (G″), and Phase angle (δ) **(Bahia, 1993).**

The axes of the figure speak to the bitumen behavior. The vertical pivot represents to the totally viscous behavior and the horizontal axis represents to the totally elastic behavior. At moderate to high temperature elastic behavior can be accomplished and at low temperature viscous behavior figured it out. Under typical conditions of temperature and rate of loading discovered such a behavior of bitumen which lies between the two axes and spoke to by a vector extent complex modulus (G*) and the course of phase angle(δ) degrees against clockwise from the horizontal axis. The mathematical relationship for complex modulus G* in terms of shear stress and shear strain is given by equation 3.6.

$$G^* (Pa) = (\tau_{max}-\tau_{min}) / (\gamma_{max}-\gamma_{min}) \quad (3.6)$$

Storage modulus or elastic modulus G′ and loss or viscous modulus G″ are given by equation 3.7 & 3.8 as follows.

$$G' = Cos(\delta)(\tau/\gamma) \quad (3.7)$$

$$G'' = Sin(\delta)(\tau/\gamma) \quad (3.8)$$

Maximum shear stress in terms of maximum applied torque by DSR is given as in equation 3.9.

$$\tau_{max} = 2T_{max} / \Pi r^3 \quad (3.9)$$

Maximum shear stress is calculated through deflection angle as given in equation 3.10.

$$\gamma_{max} = \theta_{max} r / h \quad (3.10)$$

Where

G’ = Storage modulus [Pa]

G" = Loss Modulus [Pa]

G* = Complex modulus [Pa]

$\tau_{max}$ = Absolute value of the peak-to-peak shear stress (Pa)

$\gamma_{max}$ = Absolute value of the peak-to-peak shear strain (%)

$T_{max}$ = Maximum applied torque (load) (Pa)

$\theta_{max}$ = Maximum deflection angle (rad)
r = Radius of specimen plate (mm) and h = Specimen height (mm)

### 3.3.3 Factors affecting dynamic shear rheometry testing

#### 3.3.3.1 Temperature

Bitumen is a material whose behavior is very much complex to understand as it changes its properties with change in temperature. Its behavior in the field along with aggregates and mineral filler after proper blending is too much difficult to understand during service period. To makes exact estimations it will be most essential, firstly, the entire test will be at the field temperature and also, the temperature ought to precisely controlled. Fluid (water) bath temperature control framework is utilized with dynamic shear rheometer **[Airey].**

#### 3.3.3.2 Stress Level, Strain Amplitude and Frequency of Oscillation

A viscoelastic material, bitumen does not carry on straightly as far as their stiffness as a function of stress or strain. Accordingly the dynamic shear modulus and phase angle rely on the size of the shear strain with both expanding and diminishing shear strain. A linear region is characterized as at little strains where the complex shear modulus is autonomous of shear strain **[Airey].**.

The point of confinement of the straight viscoelastic behavior is characterized as the measured quality of G* reductions to 95% of its zero strain esteem. The rheological tests ought to be performed with in the straight viscoelastic area of bitumen performance **[Airey].** So a strain range test was led at $60^0$c as demonstrated in [figure 3.8] for VG-30 and sulfur modified bitumen**.**

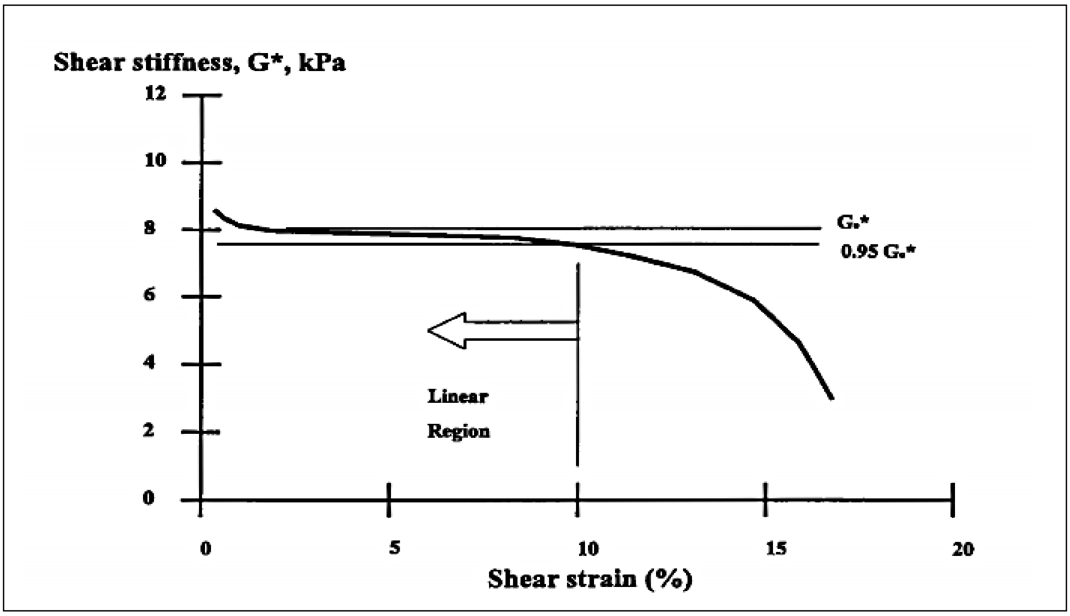



Figure 3.8: Strain sweep used to determine linear viscoelastic region **(Airey)**

### 3.3.4 Tests Performed using DSR

#### 3.3.4.1 Strategic Highway Research Program Grade Determination (SHRP)

The Strategic Highway Research Program (SHRP) grade determination, methods were optimized to grade asphalt binder so that they could be appreciated and sold appropriately as SUPERPAVE products. The SHRP testing regime contains several steps, this being only one of them. The SHRP grade determination is fully depend upon the G*/Sin(δ) value and the characterization of original binder is depend on the phase angle and complex shear modulus usually obtained from SHRP results.

#### 3.3.4.2 Frequency sweep

Frequency sweep test is used to determine the damage analysis of binder. The frequency sweep test is performed at the standard service temperature, and oscillatory shear load of constant amplitude are applied over a wide range of frequencies loading. For this test method, the frequency sweep test is selected from the DSR manufacturer's controller software, employing an applied load of 12% strain over a range of frequencies from 0.1 – 100 Hz. A typical plot [figure 3.9] for frequency sweep result data.

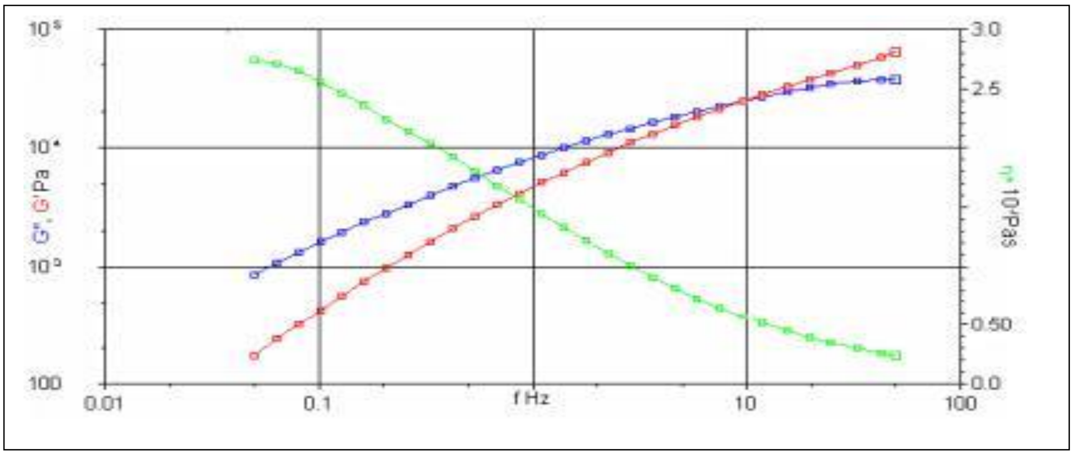



Figure 3.9: Showing master curve plot from frequency sweep data [http://www.azom.com**]**

### 3.3.4.3 Amplitude Sweep

This test is intended to evaluate the ability of bituminous binder to resist fatigue damage by employing cyclic loading at increasing amplitudes in terms of stress/ strain in order to accelerate damage. The characteristics of the rate of damage accumulation in the material can be used to identify the fatigue performance of the bituminous binder given conditions of pavement structure and/or expected amount of traffic loading using predictive modeling techniques**[AASHTO Designation: T-10**] .

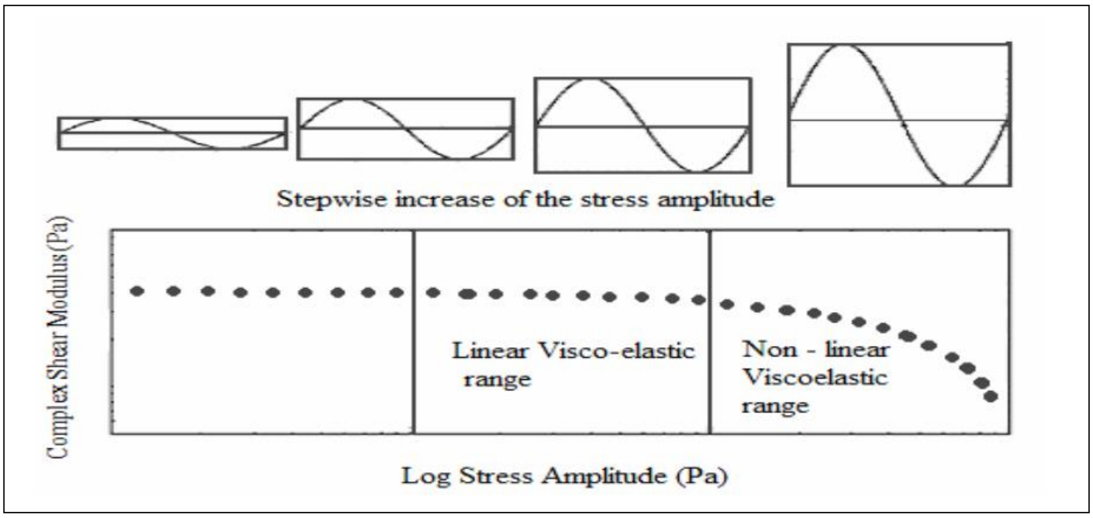



Figure 3.10: Amplitude stress sweep test **[Gebhard, 2004]**

### 3.4 Aging of bituminous binder

Aging occurs due to exposure to environmental condition under repeated action of load at field during service life. Aging may be defined as loss of volatile materials and oxidative reaction occurs due to adverse effect of climatic conditions.

#### 3.4.1 Short term aging

This type of aging in bituminous material occurs during the mixing, laying and compaction process in the field. Also short term aging includes aging occurs just after application of bituminous mixes in the field up to 3 years, which may be due to loss of volatile material due to high temperatures. Equivalent short term aged sample are generally prepared in the laboratory using Rolling Thin Film Oven Test (RTFOT) apparatus shown [figure 3.11].

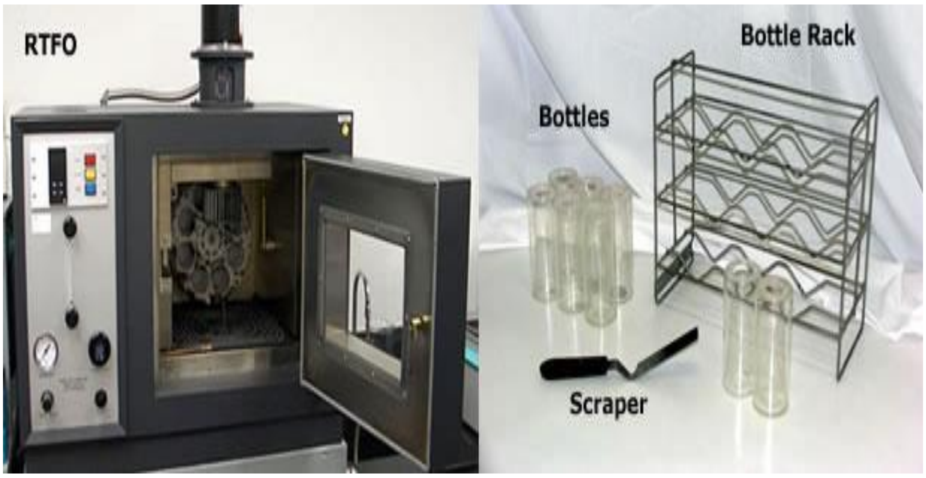



Figure 3.11: RTFOT with glass container for sample [www.pavementinteractive.org]

#### 3.4.2 Long term aging

This type of aging occurs due to the oxidation of bituminous material due to exothermic reaction takes place during service life of pavement on exposure to environment. The pressure ageing vessel (PAV) has been developed to simulate in-service ageing of asphalt binder after 5 to 10 years. The binder is exposed to high pressure and temperature for

20 hours to simulate the effect of long term oxidative ageing.PAV apparatus are shown in [figure 3.12].

Figure3.12: PAV with degasser and pan for sample storage **[**www.pavementinteractive.org]

### 3.5 Determination of physical properties of bitumen

The traditional properties is to be tried after the assessment of rheological properties on execution premise to know the progressions those may happened in the empirical properties of bitumen after modification with sulphur, so that consistency, softness, ductility, % elastic recovery and adhesion behavior could be evaluated. These properties are considered as really vital in India as these essential physical properties of bitumen are generally tested before used as a road making material.

#### 3.5.1 Penetration Test

The penetration test of a bituminous material is the separation in tenths of millimeter that a standard needle will infiltrate vertically into an example of the material under standard state of temperature, load and time. This test is the most generally adopted test on bitumen to determine the grade of the material in terms of its hardness. The Equipment for penetration test is shown in [figure 3.13] below.

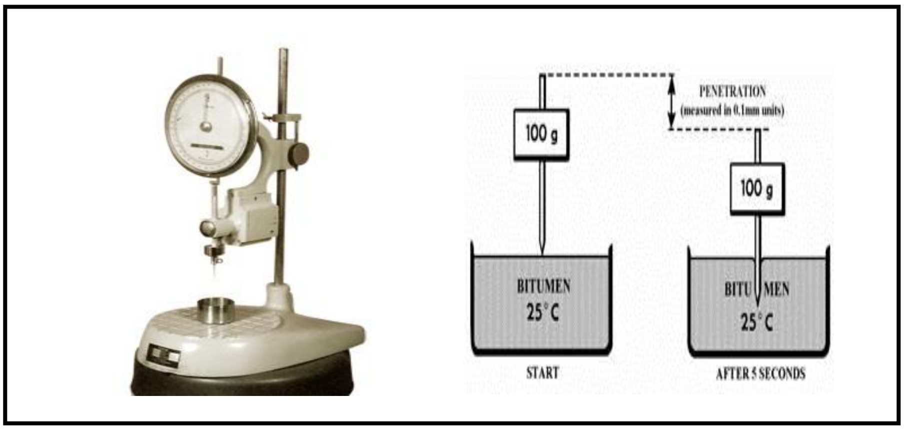


Figure 3.13: The Penetration test Apparatus [www.pavementinteractive.org]

### 3.5.2 The softening point test

The softening point is the temperature at which the material attains a particular degree of softening under specified test condition. This test is conducted to know the resistance to remain in semisolid state in high service temperature. The test setup is shown in [figure 3.14].

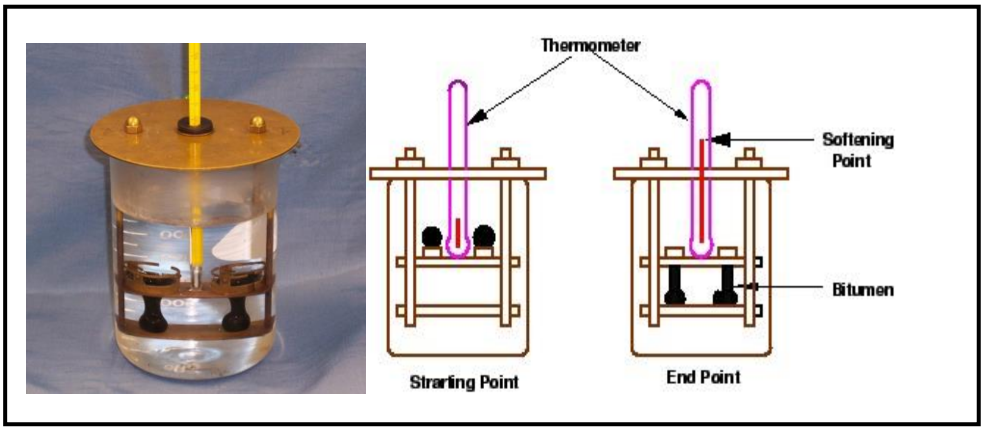


Figure3.14: Softening Point Test Apparatus [www.pavementinteractive.org]

### 3.5.3 Ductility Test

The ductility of bitumen is defined as the distance in centimeters to which the bitumen poured and levelled in a standard briquette mould elongates before the breaking of the thread of bitumen formed due to applied force to elongate under specified conditions. The testing equipment is shown in [figure 3.15]. This test is very essential to know the ductile properties of bituminous binder to use as a flexible pavement material.

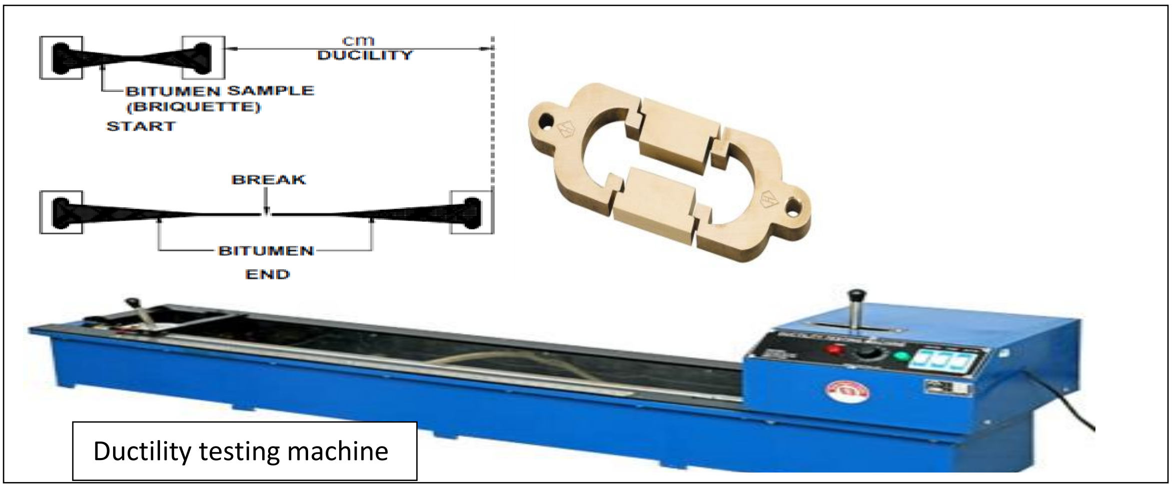



Figure 3.15: Ductility testing apparatus **[**www.pavementinteractive.org]

### 3.5.4 Elastic Recovery

The elastic recovery of the bitumen is evaluated by means of the percentage recovery of the bitumen thread formed by the stretching of bitumen specimen when it is cut down by a scissor at standard conditions. The test was carried out in Ductility testing machine as shown in [figure 3.16].

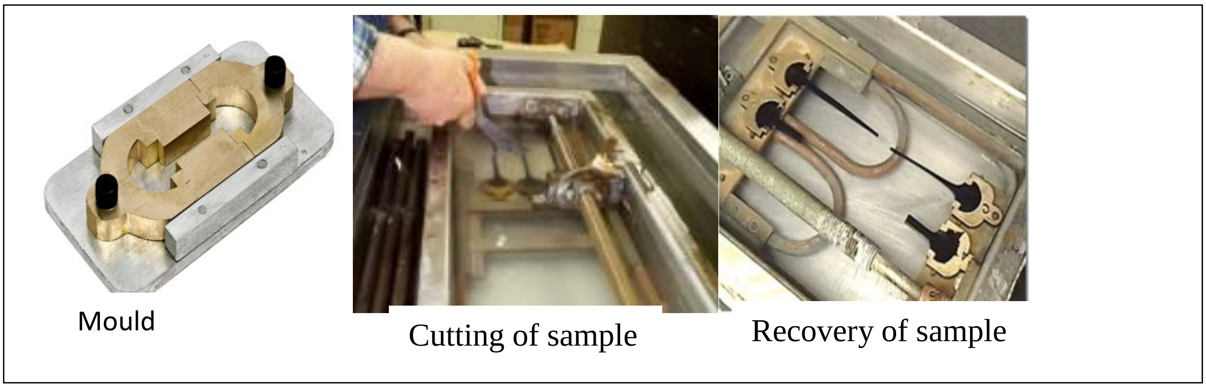



Figure 3.16: Elastic Recovery testing apparatus**[**www.priasphalt.com**]**

### 3.5.5 Adhesion properties Test

The forfeiture of bond between aggregates and bitumen that commonly starts at the lowest part of the HMA layer and progresses upward. At the point when stripping starts at the surface and progresses descending it is typically called raveling. Test arrangement is same for both the test, while the conditionings of specimen for measurement of striping value are distinctive.

Figure 3.17: Striping test of aggregate test through boiling test.

### 3.6 Storage Stability Test

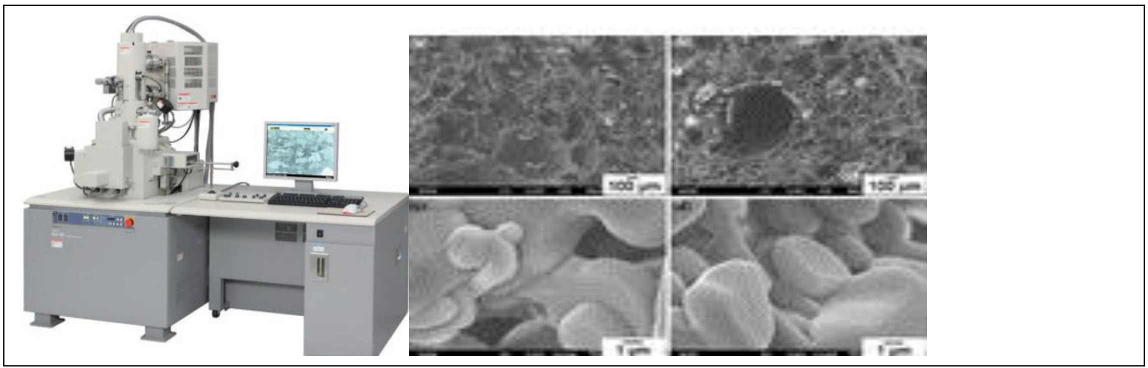
Storage stability test was carried out to evaluate the high temperature storage stability of modified bitumen and to evaluate possible differences in characteristics. The storage stability of bituminous binder are observed in terms of difference in softening point of sample from upper portion and lower portion of tube as shown in figure below [figure3.18].

Figure 3.18: Tube used in storage stability test.

If the difference of the softening points is less than2.5°C, the sample is considered to have good high-temperature storage stability, otherwise the binder was considered to be unstable.

### 3.7 Morphology

Morphology is a branch of science to study the intermolecular structural form of a material using advanced microscope instrument especially Field emission scanning electron microscope (FESEM) for a very high zoom. More or less this study helps to observe the homogeneity of the chemical component structure of a material. In this study this analysis was utilized to observe the uniformity in the sulphur in the bitumen after modification. The FESEM instrument is shown in [figure 3.21] below.



FESEM equipment                Sample photo using FESEM

Figure 3.19: FESEM testing equipment with sample photos [www.directindustry.com].

# CHAPTER-4

## EXPERIMENTAL PROGRAM

### 4.1 Introduction

The work demonstrated in this section has been partitioned into four zones. The primary zone of this study comprises the type of material used, their standard properties and sample preparation for testing. The secondary region of study depicts the operational confinements of the Viscometry and DSR in wording of connected stress levels and recoverable strain levels along with the testing conditions of samples. The third range of study examined the impact of different temperatures on the physical properties tests. The fourth zone of this study summarized with chemical, morphological and thermal analysis with testing conditions of the samples. In this study the rheological, physical, storage stability, chemical, thermal and morphological properties of both unmodified and modified bitumen, their working standards have been briefly discussed.

### 4.2 Material

It is known from the studies that the level of modification relies on upon the neat bitumen type and modifier type. Different studies have been carried out in the field of sulfur modification and there are a few descriptions for the need of utilizing modifier within bitumen industry. There are different explanations behind utilizing bitumen modifier within bitumen industry began with expansion the service life of the pavement, enhance its performance, meet the overwhelming traffic demands  and at last saving the expense of maintenance. In this test project viscosity grade bitumen VG-30 has been utilized. The physical properties of VG-30 bitumen were given in table underneath.

Table 4.1 Physical properties of VG -30 bitumen

| Properties | Result |
|---|---|
| Absolute viscosity 60°C ( Cp) | 2462 |
| Kinematic Viscosity 135°C (cst) | 365 |
| Softening point °C | 47 |
| Penetration (dmm) 25 °C | 57 |
| Ductility (cm) 25°C | >100 |
| Elastic Recovery (%) | 26 |

Observing the production and cost, Sulfur being in powder structure utilized as modifier for modification of bitumen as shown in figure below. The essential properties of sulphur have been represented in [figure 4.1] beneath

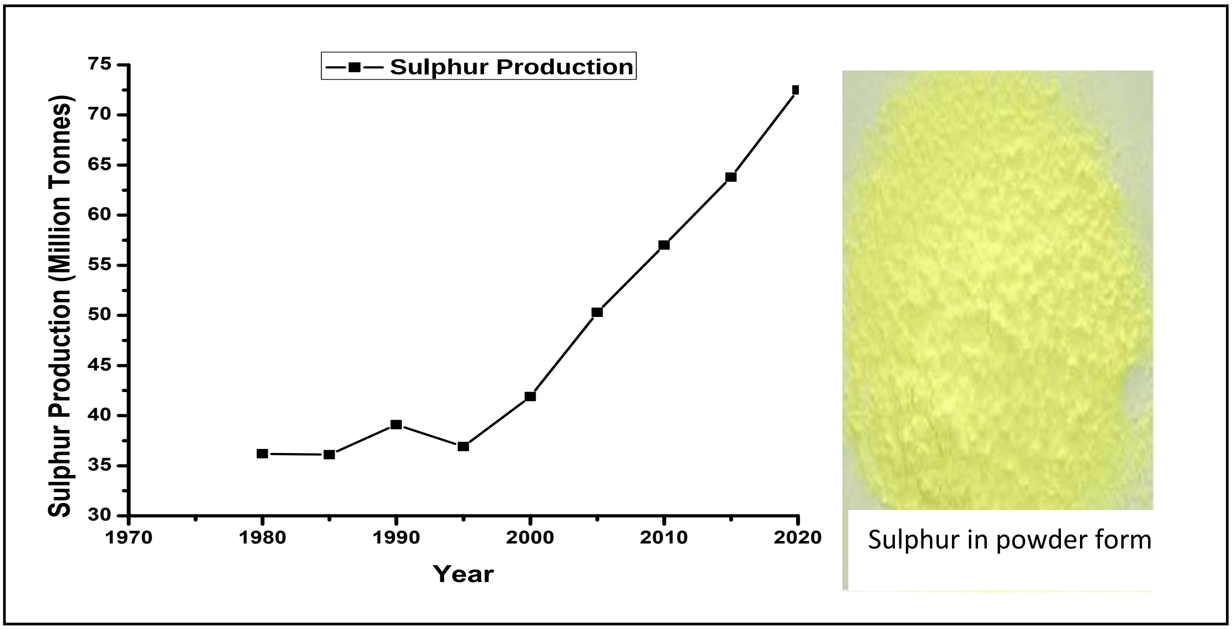



Figure 4.1: Production of sulphur over years and Elemental sulphur powder [Data Ref: http://en.wikipedia.org/wiki/Sulfur]

Table 4.2 Physical properties of elemental Sulphur [Source: www.sulphuric-acid.com]

| Properties | Result |
|---|---|
| Appearance | Yellow crystalline solid |
| Melting point | 120°C |
| Specific Gravity | 1.92 |

### 4.3 Sample Preparation

At first the Rheological properties of sulfur extended bitumen have been tested to know about the progressions in the viscoelastic properties of modified specimen. To discover a good structure for a good sulphur modified bitumen , four steps are carried out , under which a few sets of tests are to be directed with viscometry and DSR instrument to evaluate the rheological properties of sulphur extended bitumen to evaluate the optimum sulfur content and ideal condition for proper modification and are explained below. After obtaining the proper sulphur content and blending condition in terms of blending time and temperature analysis of physical, chemical, thermal and morphological properties has been carried out.

To prepare the modification of VG 30 bitumen with sulphur, about 1.0 Kg of bitumen is taken in a 3 liter metal container and heated up to fluid condition. The blending of sulphur with bitumen is carried out using a mechanical stirrer at a stirring speed of 3000 R.P.M. for temperature beyond 120 °C, but for temperature within 100°C to 120°C, speed of the stirrer for blending was kept 1500 R.P.M.

### 4.4 Aging of binder

#### 4.4.1 Short term aging

This ageing has conducted utilizing Rolling thin film oven test (RTFOT). About 35gm of bitumen sample in flow condition was taken in RTFO bottle and were properly placed inside RTF oven rack holders. After that the sample rack were rotated under hot airflow condition at $163^0$ C temperatures for 85 minutes. Some part of residue obtained from RTFO then tested for rheological, chemical, thermal and morphological properties and other part of residue was taken for long term aging test by utilizing PAV.

#### 4.4.2 Long term aging

Long term aged sample has been prepared using Pressure aging vessel (PAV). About 50 gm of RTFOT residue has been placed in a pan within a pressurized heated close air tight vessel with air blow of 2.1 MPa at $100^0$C temperature for 20 hours. After 20 hours of treatment of pressure aging the samples are removed. Degassing of PAV residue at 170° C for 30 minutes has been done and samples were used for future testing.

### 4.5 Testing program for evaluation of rheological properties

The objective of this section is to find optimum sulphur content, blending time, blending temperature for proper modification of sulphur and bitumen. To achieve the above desired conditions several steps are carried out and rheological properties were tested as choosing criteria and are briefly summarized underneath. DSR geometry used for fatigue is 8 mm parallel plate and for rutting is 25mm parallel plate.

#### 4.5.1 Determination of appropriate mixing/blending temperature for modification of bitumen by sulphur

In this section modification was done for Sulphur powder of 2% by wt. of VG-30 bitumen with blending time of 10 minutes for different blending temperatures of $100^0$C, $110^0$C, $120^0$C, $130^0$C, $140^0$C, $150^0$C & $160^0$C as a trial basis to get the ideal blending temperature utilizing the mechanical stirrer. SHRP grade determination test was executed for 10 hz frequency at testing temperature of $60^0$C for every blending temperature. Choosing criteria of blending temperature was based on the output result of SHRP test as complex modulus (G*) and phase angle (δ) value.

#### 4.5.2 Determination of appropriate mixing/blending time for modification of bitumen by sulphur

In this section modification was done for Sulphur powder of 2% by wt. of VG-30 bitumen with blending temperature resulted from above section for different blending time of 5min,10min, 15min, 20min, 25min and 30minas a trial basis to get the ideal blending temperature utilizing the mechanical stirrer. SHRP grade determination test was executed for

10 hz frequency at testing temperature of $60^0$C for every blending temperature. Choosing criteria of blending temperature was based on the output result of SHRP test as complex modulus (G*) and phase angle (δ) value.

### 4.5.3 Determination of optimum sulphur content to get a good modified bitumen binder

This section deals with modification of VG 30 bitumen binder for different Sulphur contents such as 1%, 2%, 3%, 4%, and 5% by wt. of VG-30 bitumen with blending temperature and time resulted from above two section respectively as a trial basis to get the ideal blending of bitumen and sulphur utilizing the mechanical stirrer.

To achieve ideal optimum sulphur content, both unaged and aged samples have been tested. Samples underwent both RTFOT and PAV aging under standard conditions. SHRP grade determination test was executed for 10 hz frequency at testing temperature of $60^0$C for every blending temperature. Choosing criteria of blending temperature was based on the output result of SHRP test as complex modulus (G*) and phase angle (δ) value keeping the aging condition of sample.

### 4.5.4 Sulphur extended bitumen characterization through Viscosity Tests

#### 4.5.4.1 Determination of absolute viscosity

This test is carried out utilizing the Capillary viscometer for absolute/ dynamic viscosity at $60^0$C and procedure has been carried out according to ASTM D2170.Measurement was taken to within 0.1 s the time required for the leading edge of the meniscus to pass between successive pairs of timing marks. The resistance by the sample against the vacuum pressure to lift up those timing marks inside a tube [figure 4.2(a)] has been measured and reported as dynamic viscosity of that sample. The first flow time was reported which exceeds 60 s between a pair of timing marks, noting the identification of the pair of timing marks.

#### 4.5.4.2 Determination of Kinematic viscosity

This test is carried out utilizing the Capillary viscometer for kinematic viscosity at 135$^0$C and procedure has been carried out according to ASTM D2171.Measurement was taken to within 0.1 s the time required for the leading edge of the meniscus to pass between successive pairs of timing marks.

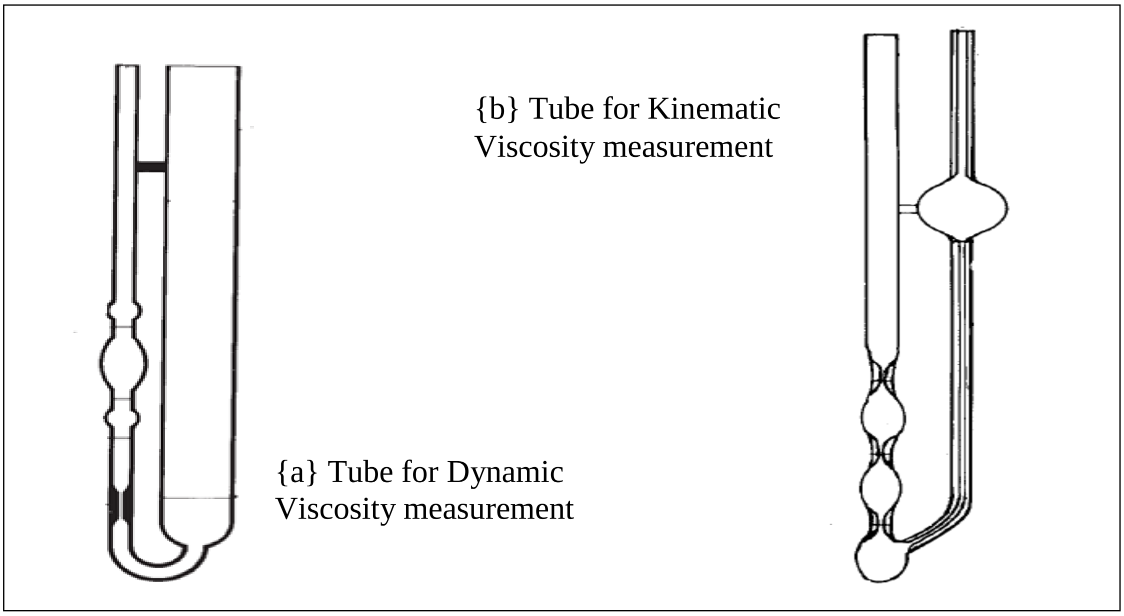



Figure 4.2: Capillary tube used for determination of absolute and kinematic viscosity **[ASTM D2170 & D2171]**

The resistance to flow under gravity and to pass those timing marks inside the tube [figure 4.2 (b)] has been reported as kinematic viscosity of that sample. The first flow time was reported which exceeds 60 s between a pair of timing marks, noting the identification of the pair of timing marks.

#### 4.5.4.3 Determination of viscosity at high temperatures

For determination of high temperature viscosity for a large variations of shear rate to predict the Newtonian behavior of binder, Brookfield viscosity tests has been carried out. In this test Rheocal software is used, which gives the viscosity directly in poise. This test is carried out for determination of viscosity for temperatures at 100$^0$C, 120$^0$C, 135$^0$C, 140$^0$C,

$150^0$C, $160^0$C & $180^0$C for unaged unmodified and modified samples. Tests are carried out for different R.P.M. to know the variations in viscosity behavior over variation of shear rate.

### 4.5.5 Sulphur extended bitumen characterization through Oscillation tests

Oscillation tests has been carried out in terms of Frequency sweep, Amplitude sweep and Temperature sweep of the un modified and modified binder for both unaged and aged binder after RTFOT and PAV to characterize the behavior of complex shear modulus of binder for different frequencies, Shear stresses and different service temperatures for optimum sulphur content.

#### 4.5.5.1 Strategic Highway Research Program Grade Determination (SHRP)

This test is utilized to find the optimum sulphur content and ideal blending condition to have a very good modification according to standard conditions of Temperature and frequency. The Characterization of Binders under Standard Conditions of SHRP is summarized below. The test was carried out under certain standard conditions given by SHRP which is used to characterize the VG 30 and sulphur modified bitumen binder in terms of complex modulus and phase angle. The following specifications are provided by SHRP for different binders:

- ✓ To control possible tenderness, the stiffness value of G*/sin (δ) of the original binder must be greater than 1.0kPa at the average pavement temperature.
- ✓ To minimize rutting criteria, the stiffness value of G*/sin (δ) of the binder after RTFO must be greater than 2.2kPa at the average pavement design temperature.
- ✓ To minimize fatigue cracking effect, the stiffness value of G* x sin (δ) of thePAV binder must be less than 5000kPa at the intermediate pavement design temperature.

#### 4.5.5.2 Frequency sweep

Frequency sweep tests were performed on unaged and aged modified and unmodified bitumen. The test was directed at 60°C temperature for unaged and RTFO aged binders and 45°C temperature for PAV matured fasteners. The greater part of the recurrence range tests were performed from 0.1 to 100 rad/s under a stress parameter taken from adequacy test. Frequency sweep test is utilized to build the master curve for the tried specimen.

#### 4.5.5.3 Amplitude Sweep

A stress sweep was initially directed to focus the straight viscoelastic region for distinctive binders. The test was led at 60°c temperature for unaged and RTFO matured fasteners and 45 ° C temperature for PAV aged binders with a shear stress range from 0.5 kpa to 15 kpa and at steady frequency of 10 Hz. The complex shear modulus G* versus stress/strain plot was utilized to focus the direct viscoelastic district (LVR). The stress focus from this test utilized as the parameter for further test.

## 4.6 Testing program for determination of physical properties

After evaluation of optimum sulphur content and ideal blending condition with respect to performance criteria in terms of rheological properties, modified binder undergone for empirical physical properties testing, so that the improvement in engineering properties of modified binder can be compared with the conventional binder.

### 4.6.1 Penetration Test

The penetration test is conducted as a determination of consistency of the bitumen at 25 ºC. In this test, a needle penetrates a sample of bitumen under a load of 100 grams at a temperature of 25 ºC for a loading time of 5 seconds. The test was conducted as per IS: 1203-1978.The penetration value is described in tenths of a millimeter (decimillimetre, dmm).

### 4.6.2 Softening point Test

In this test bitumen sample in fluid condition poured in a brass ring, levelled and kept for 30 minutes at room temperature. Glass beaker containing distilled water kept at B.O.D. incubator at 5ºC for 30 minutes. A steel ball of weight 3.5 g is placed on a bitumen sample contained in a brass ring that is suspended inside a water bath, maintaining bath temperature to be raised at 5ºC per minute. The softening point of bitumen was determined as per IS: 1205-1978.Temperature at which the bitumen sample touches the lower plate is reported as softening point of that sample.

### 4.6.3 Ductility Test

The test has been carried out at a temperature $27^0$ C and a rate of pull of 50mm/min. The experimental procedure has been followed according to ASTM D113 – 07. The distance from the starting point of bitumen thread formed to the broken point of bitumen thread was reported as ductility value.

### 4.6.4 Elastic Recovery Test

Sample preparation up to attachment of mould with sample and briquette in ductility testing machine is same as ductility testing of sample. The experimental procedure has been followed according to ASTM D6084. After that elongation of sample was done up to a distance of 10 cm at speed of 50 mm/min. Sample at that condition was for 1 hour at 15 ºC. After that the two broken sample was made to come closer and the distance was recorded. Using formula as given below in equation 4.1, the percentage elastic recovery was reported.

$$\text{Elastic recovery (\%)} = \left[\frac{10-X}{10}\right] * 100 \quad (4.1)$$

Where

X = unrecovered length in cm.

### 4.6.5 Adhesion Test

The extent of stripping was estimated visually while specimen was still under water and boil water for 15 minutes. Determination of striping value has been carried out according to IS 6241 (1971). The stripping value is the ratio of the uncovered area observed visually to the total area of the aggregates in each test expressed as a percentage.

## 4.7 Storage Stability test

The sample was poured into an aluminum tube specially made up with 30 mm internal diameter and 150 mm in height. The tube was sealed from bottom and kept vertically in an oven at 163$^{\circ}$C for a period of 48 hour, then taken out and cooled to room temperature, and the tube with sample was splices horizontally into three equal sections. The samples taken from the top and bottom sections were used to evaluate the storage stability of binder by measuring their softening points **[Haiying Fu et al]**.

## 4.8 Morphology analysis

Field emission scanning electron microscope (FESEM) instrument used for the study of the microscopic structure of VG 30 bitumen and sulphur modified bitumen with a resolution of 10µm. Its automated advance features include focus, stigmator, gun saturation, gun alignment with very good contrast and brightness.

# CHAPTER-5

## RESULTS AND ANALYSIS

### 5.1 Introduction

This section portrays the rheological properties in terms of fatigue and rutting behavior results for unmodified bitumen and sulphur modified bitumen as well as short dissection of test information. The skeleton of testing covers was chosen keeping in mind the end goal to research that impact of sulfur in the bitumen properties subjected to distinctive loading parameters. The rheological properties of the different binders were portrayed utilizing dynamic shear rheometer over wide ranges of temperatures and frequencies. In this study both VG 30 bitumen and its modification with sulphur was tried and a summary of all results introduced underneath in tables and graphical structure.

### 5.2 Rheological properties of unaged and aged binder test results

#### 5.2.1 SHRP test results for appropriate mixing/blending temperature for modification of bitumen by sulphur under Standard Conditions of SHRP test

Test results are analyzed on the basis of phase angle and complex shear modulus and their behavior with variations in blending temperature and are presented in diagrams[figure 5.1 (a) & (b)].

From the Figure 5.1 (a) two statement are observed one is the phase angle of sulfur modified bitumen having less esteem than the unmodified VG 30 bitumen, which indicate towards more elastic nature than conventional bitumen. Second is at 1400c of mixing/blending temperature the sulphur modified bitumen indicates least value of phase angle than other temperature because of the expansion in viscosity of sulphur powder for temperature beyond 150$^{o}$C.

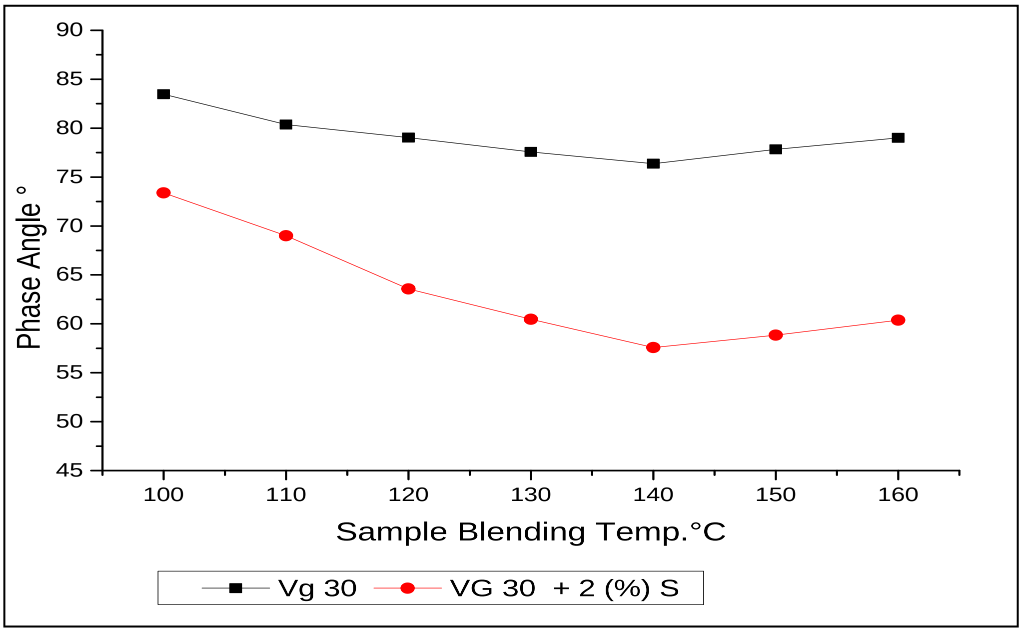



Figure 5.1 (a): Variations of phase angle with different blending temperature

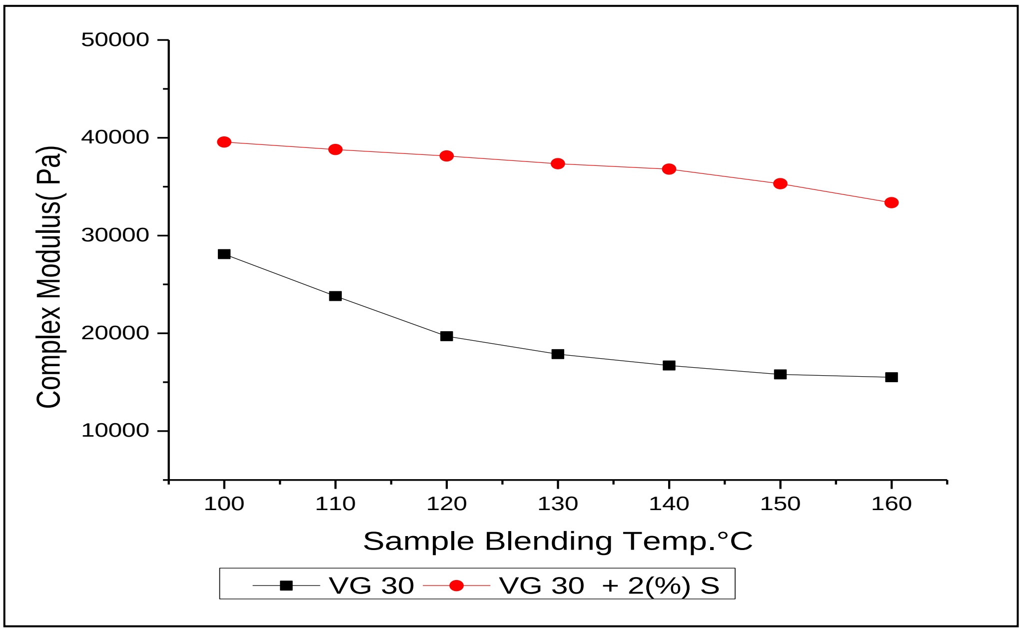



Figure 5 .1 (b): Variations of complex modulus with different blending temperatures

Then again from Figure 5.1 (b) there is an increment in complex modulus of sulfur modified bitumen than routine VG 30. Likewise the complex modulus of sulfur modified bitumen beyond 140$^{o}$C declines. Thus for legitimate mixing/blending of sulfur and bitumen, 140$^{o}$C temperature has been taken with respect to both the elastic and strength properties of sulphur modified bitumen.

### 5.2.2 SHRP test results for appropriate mixing/blending time for modification of bitumen by sulphur under Standard Conditions of SHRP test

These test results are mulled over regarding phase angle and complex modulus for proper time needed for mixing/blending of sulfur and bitumen.Their relationships with different blending temperature are presented in graphs [Figure 5.2 (a) & (b)] below.

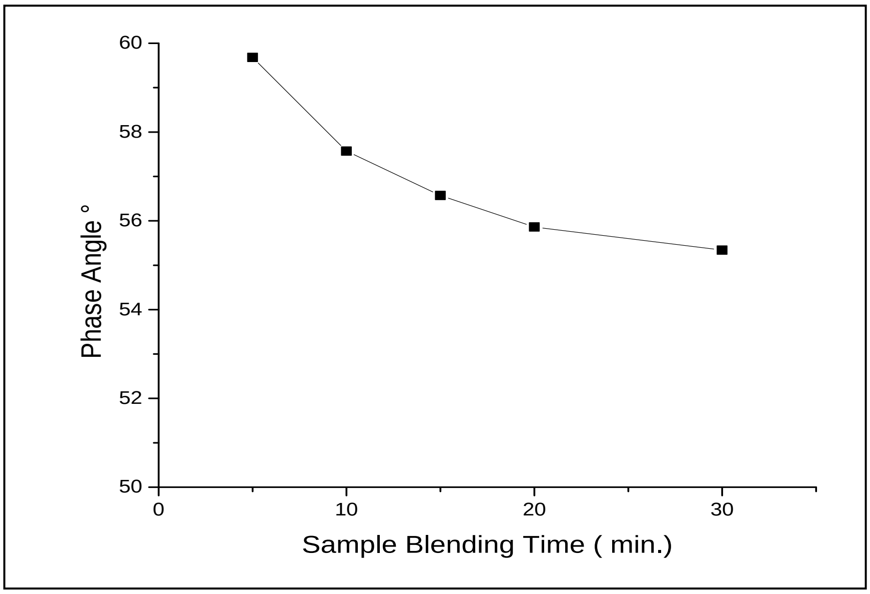



Figure 5.2 (a): Behavior of phase angle with change in blending time for 2% sulphur modified bitumen

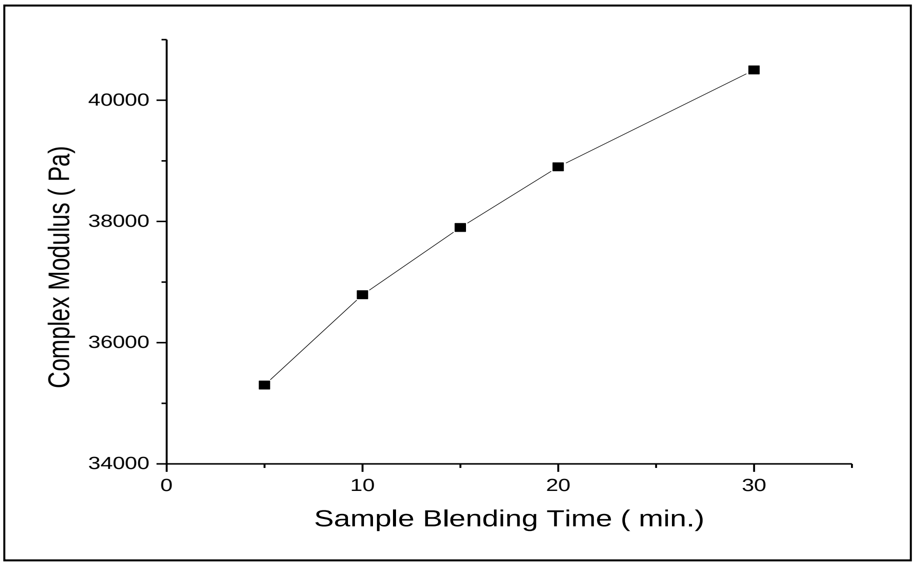



Figure 5.2 (b): Behavior of complex modulus with change in blending time for 2% sulphur modified bitumen

From the above variations of phase angle and complex modulus with variations of blending time for modification of bitumen with sulphur it can be suggest that about 30 minutes of continuous blending provides a homogeneous blending for which decrease in phase angle and increase in complex modulus occurred, as a result of which sulphur modified bitumen becomes more elastic and possess more strength.

### 5.2.3 SHRP test results for optimum sulphur content to get good modified bitumen binder

The results obtained are presented in graph [figure 5.3 (a) & (b)] to observe the variations of phase angle and complex modulus very clearly.

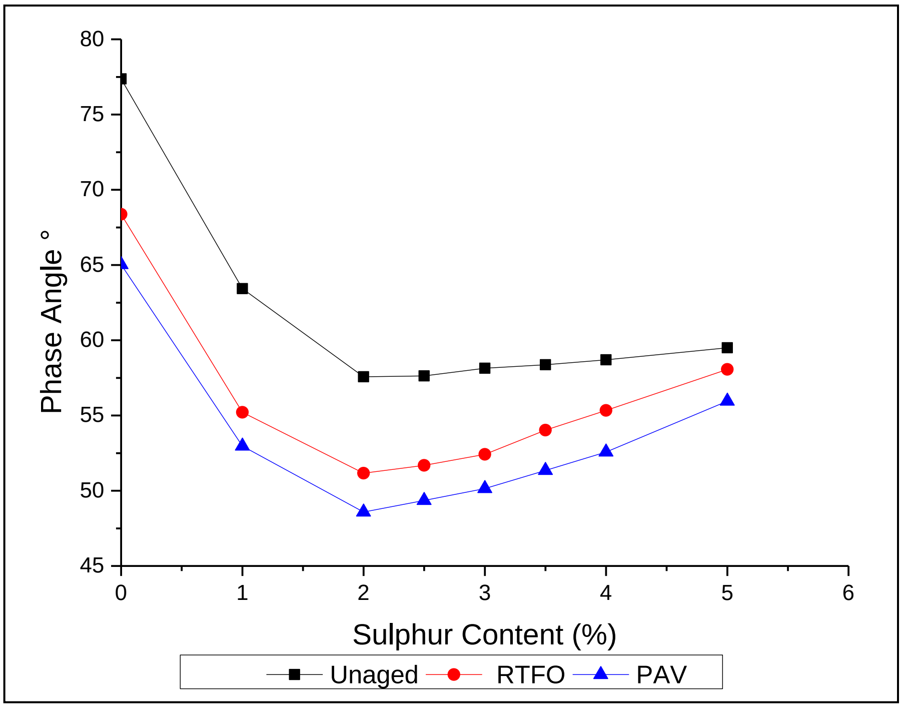



Figure 5.3 (a): Variations of phase angle with increase in sulphur content

It can be clearly seen that 2% sulphur content is the optimum modifier content from phase angle point of view due to having least phase angle value. But there is a wide variation in case of phase angle that it increases as the sulphur content increases which is due to the increase in viscous properties of bitumen with excess addition of sulphur. Also at 2% sulphur content complex modulus value is quite high from normal.

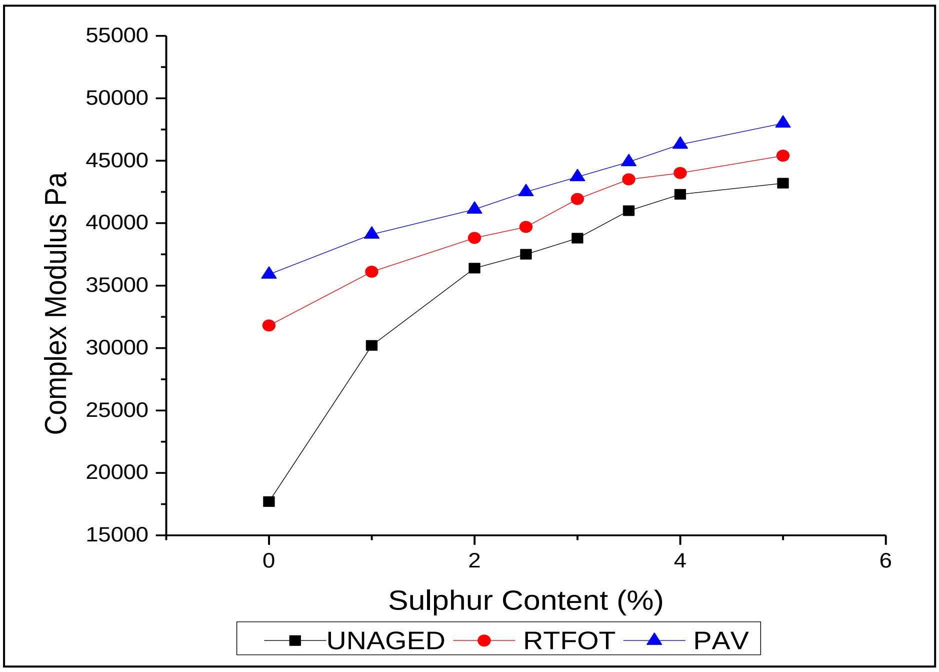



Figure 5.3 (b): Variations of complex modulus with increase in sulphur content

If we will observe the behavior of aging effect of modified binder, there variations are same as in case of unaged modified binder. Due to aging the binder becomes stiffer and possesses high complex modulus and less phase angle than unaged bitumen which is due to loss of volatile material and oxidative reaction. Hence 2 % sulphur is taken as optimum modifier content for ideal bitumen modification with sulphur.

### 5.2.4 Sulphur extended bitumen characterization through Viscosity Test results

#### 5.2.4.1 Test results for viscosity at high temperatures

This experiment has been carried out through Brookfield rotational viscometer. The tests have been carried out to observe the behavior of viscosity VG 30 bitumen after modification with sulphur with variations of temperature and shear rate. The test results are demonstrated in graphical [figure 5.4 (a), (b) & (C)] presentation underneath. From figure 5.4 (a) it is clearly shown that VG 30 bitumen and sulphur modified VG 30 bitumen showing liquid behavior with rise in testing temperature. Thus the shear resistance of bitumen to resist the pivoting of the spindle of Brookfield viscometer decreases, as a result of which the viscosity of sample decreases.

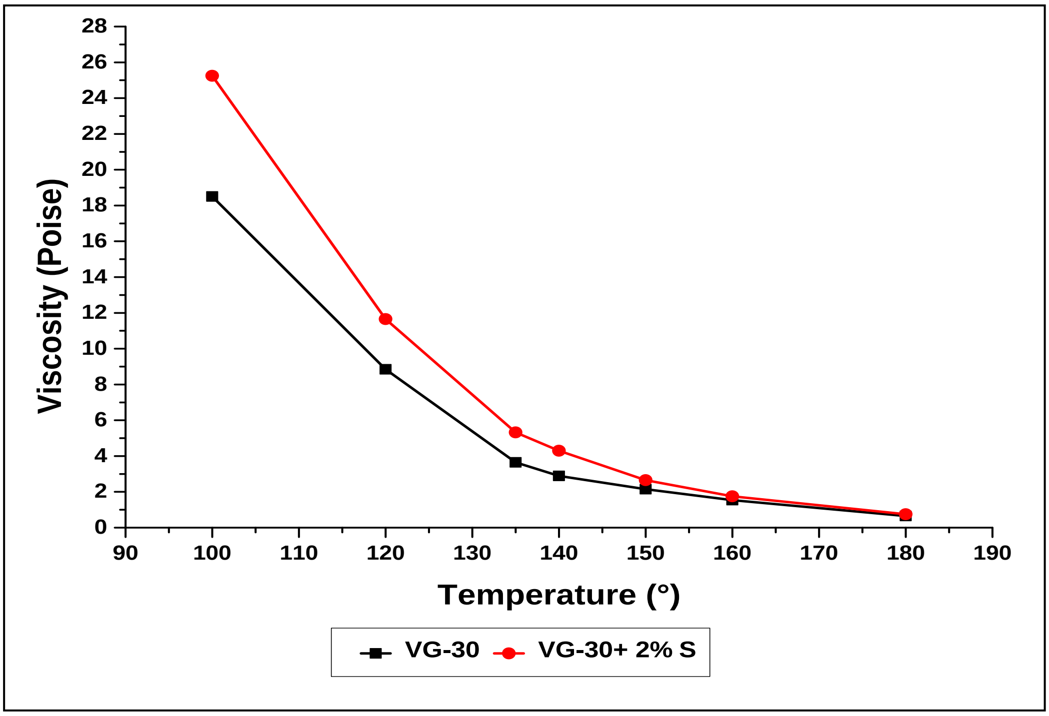



Figure 5.4 (a): Behavior of viscosity of bitumen with variation of Temperature.

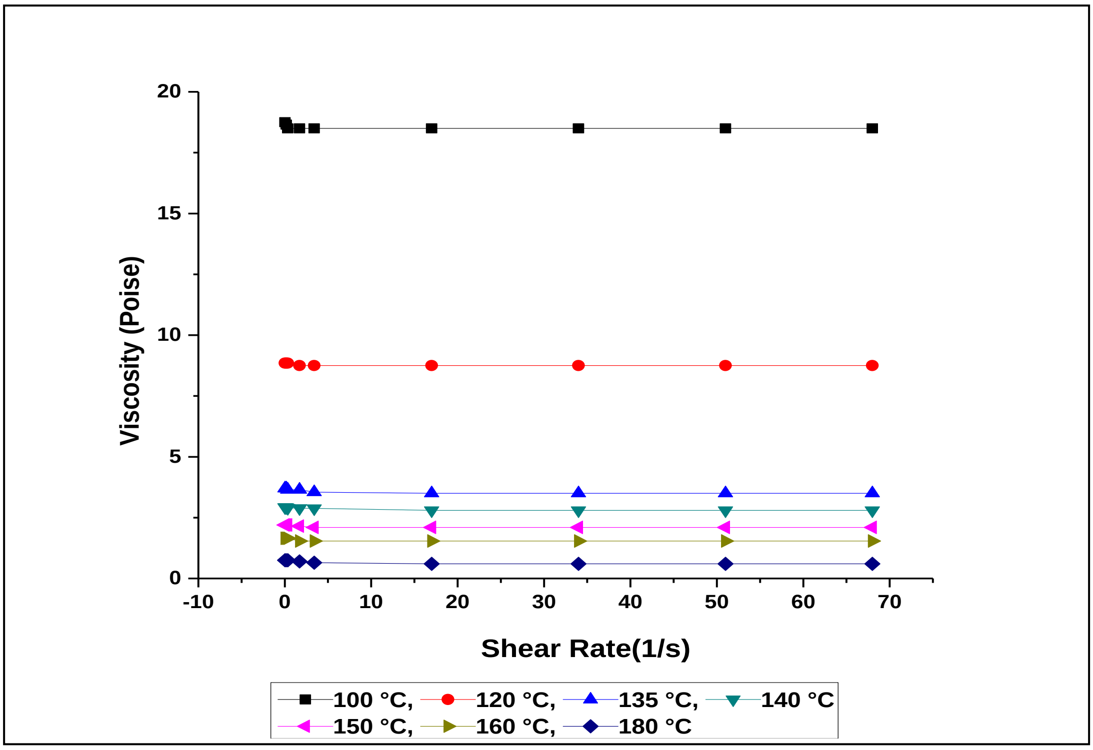



Figure 5.4(b): Variations of viscosity of VG 30 bitumen with increase in shear rate for different temperatures.

Also due to addition of sulphur the viscosity of VG 30 bitumen increases as a result of which the rutting resistance of sulphur modified bitumen increases. This test result is very helpful to decide the mixing temperature for preparation of bituminous mixes, so that required viscosity can be achieved.

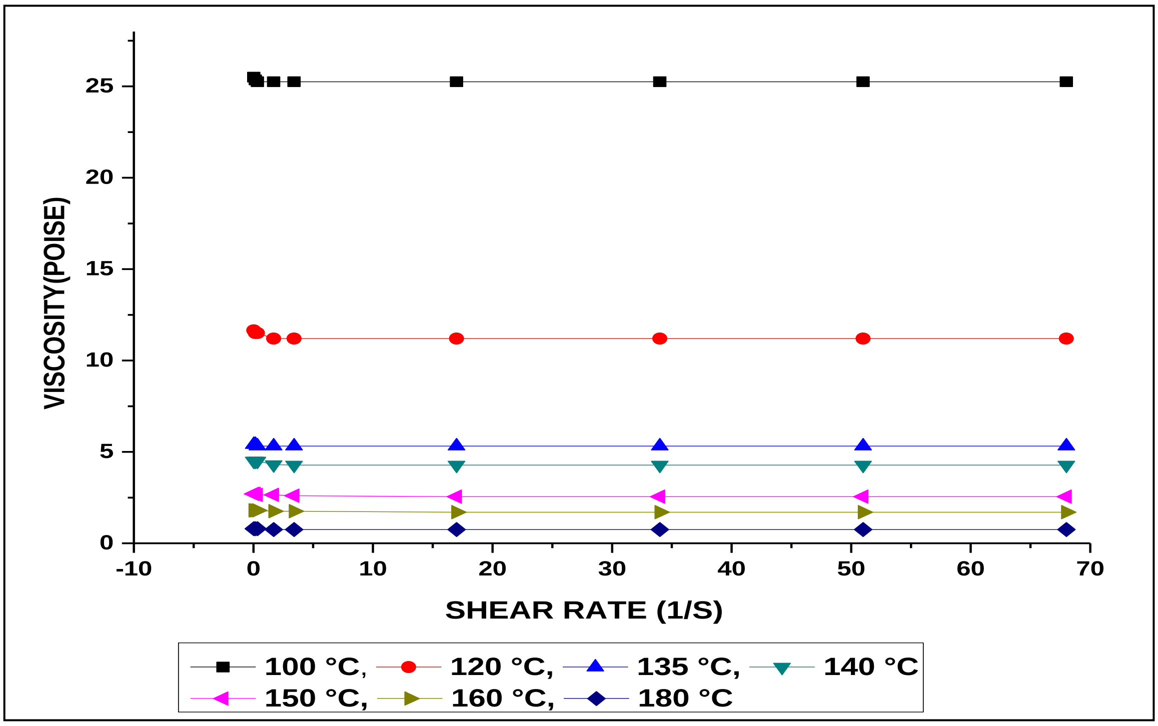



Figure 5.4(c): Variations of viscosity of 2% sulphur modified VG 30 bitumen with increase in shear rate for different temperatures.

According to Newton, for Newtonian fluid the viscosity of that fluid doesn’t change with shear rate, rather it is independent with shear rate. Here also in the figure 5.4 (a), behavior of VG 30 bitumen is just like a Newtonian fluid as there is no alteration exist in the viscosity due to change in shear rate. Similarly there is no sign of change in the viscosity of 2% sulphur modified VG 30 bitumen with rise in shear rate, which intend towards the Newtonian fluid behavior of the sulphur modified bitumen.

### 5.2.5 Sulphur extended bitumen characterization through Oscillation Tests results

#### 5.2.5.1 SHRP grade determination test results

This test result for VG 30 bitumen and 2% sulphur modified VG 30 bitumen for both un aged and aged are shown in tabular form below Table 5.1.From the result from SHRP grade determination test in tabular form indicates that the VG 30 bitumen and 2% sulphur modified VG 30 bitumen satisfy the rutting and fatigue criteria according to the specification.

Table 5.1: SHRP grade determination test results

| Sample type | Test Temp. °C | Angular Frequency rad/s | Phase Angle ° (δ) | Complex Modulus Pa (G*) | G*/Sin(δ) Pa | G*x Sin(δ) Pa | Specification pa | Remarks |
|---|---|---|---|---|---|---|---|---|
| VG 30 | 60 | 10 | 77.37 | 1.77E+04 | 1.81E+04 | | >1000 | ok |
| VG 30 - RTFOT | 60 | 10 | 68.37 | 3.18E+04 | 3.42E+04 | | >2200 | ok |
| VG30 - PAV | 60 | 10 | 65.03 | 3.59E+04 | | 3.25E+04 | < 5000 Kpa | ok |
| VG 30 + 2% S | 60 | 10 | 55.34 | 3.65E+04 | 4.44E+04 | | >2200 | ok |
| VG 30 + 2% S RTFOT | 60 | 10 | 51.17 | 3.88E+04 | 4.98E+04 | | >2200 | ok |
| VG 30 + 2% S PAV | 60 | 10 | 48.58 | 4.11E+04 | | 5.48E+04 | < 5000 Kpa | ok |

#### 5.2.5.2 Frequency Sweep Test results

Results are obtained in terms of phase angle and complex modulus with variations of frequencies and are represented in graphs are illustrated below [figure 5.5 (a) & 5.5(b)]. The graph figure 5.5(a) shows there is a decrease in behavior of phase angle for sulphur modified bitumen with increase in loading frequencies than neat bitumen, which intend towards more elastic property of sulphur modified than neat bitumen binder.

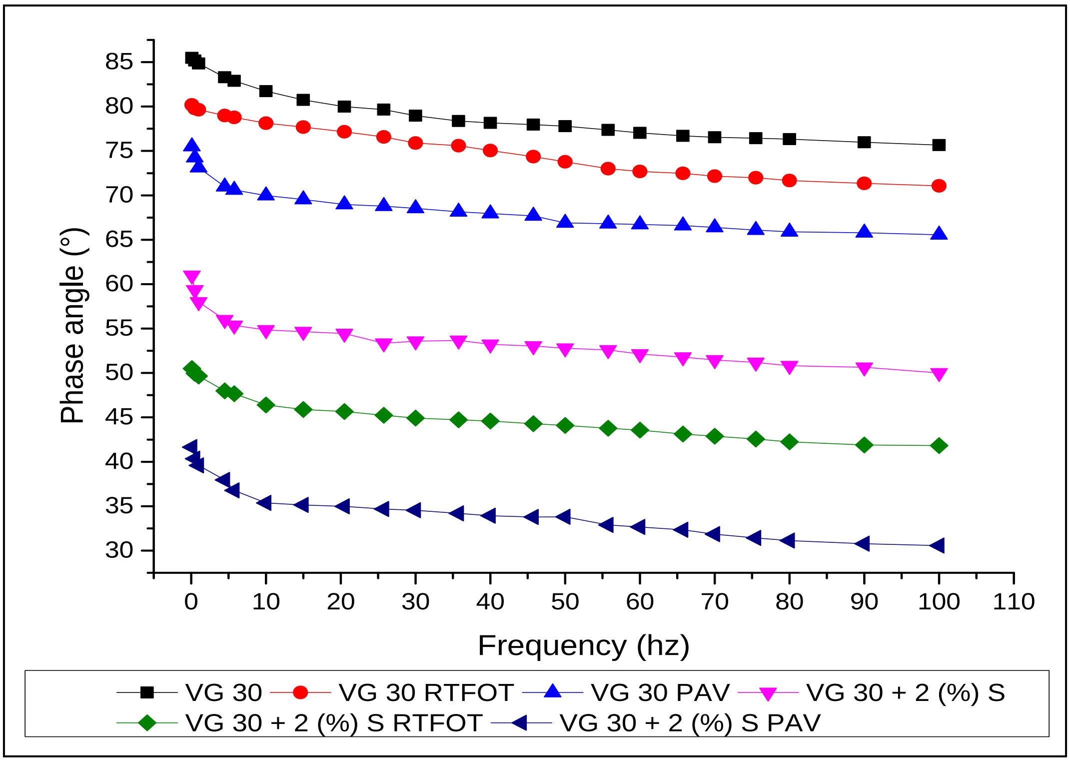



Figure 5.5 (a): Master curve: Variations of phase angle with various loading frequencies.

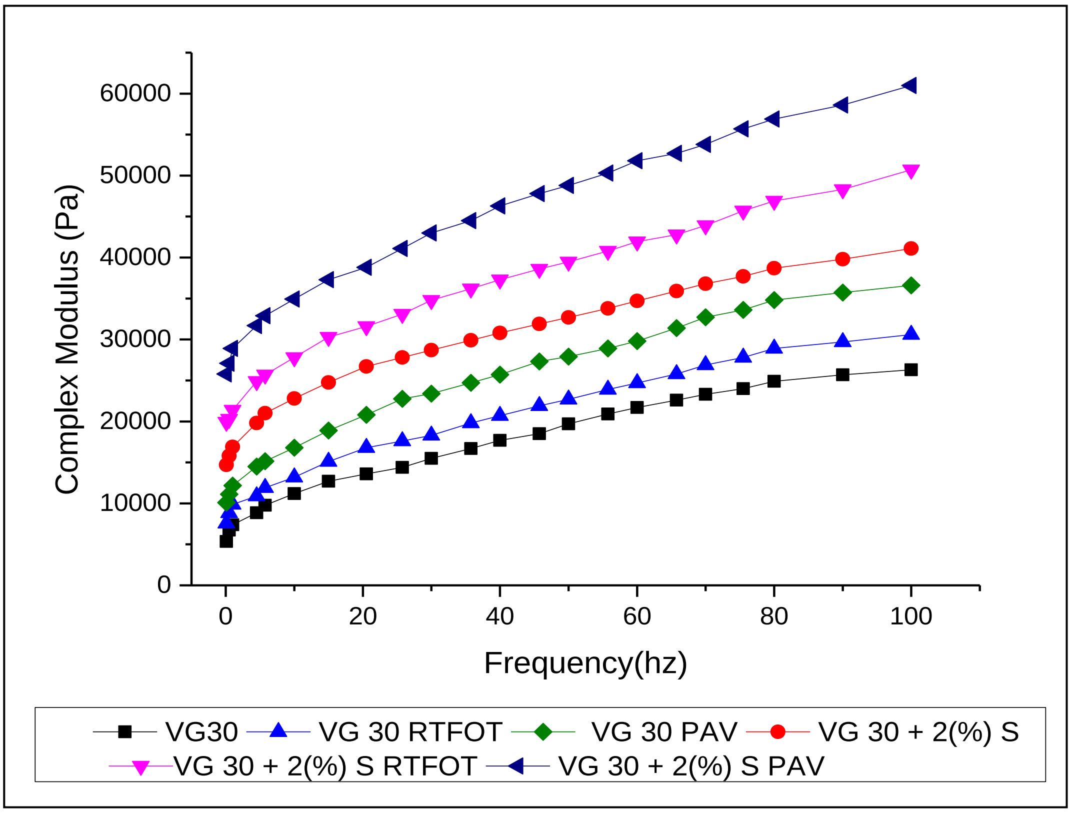



Figure 5.5 (b): Master curve: Variations of complex modulus with various loading frequencies.

In the other hand figure 5.5 (b) shows increase in complex modulus with increase in frequencies of sulphur modified bitumen than neat bitumen. This is because of the increase in both viscous and elastic modulus of the binder. This complexity in the behavior of phase angle and complex modulus for sulphur modified Vg 30 bitumen is due changes occurred with addition of sulphur in its inter molecular structure.

### 5.2.5.3 Amplitude Sweep Test results

The results of amplitude sweep of VG 30 bitumen and 2% sulphur modified VG 30 bitumen for both un aged and aged are shown in [figure 5.6 (a) & (b ] belowand are analyzed, to determine the linear viscoelastic region. Based on the viscoelastic region limits the input stress parameter for other rheological tests are chosen. It has been found that the linear viscoelastic region lies between 0.5 Kpa to 15 Kpa.

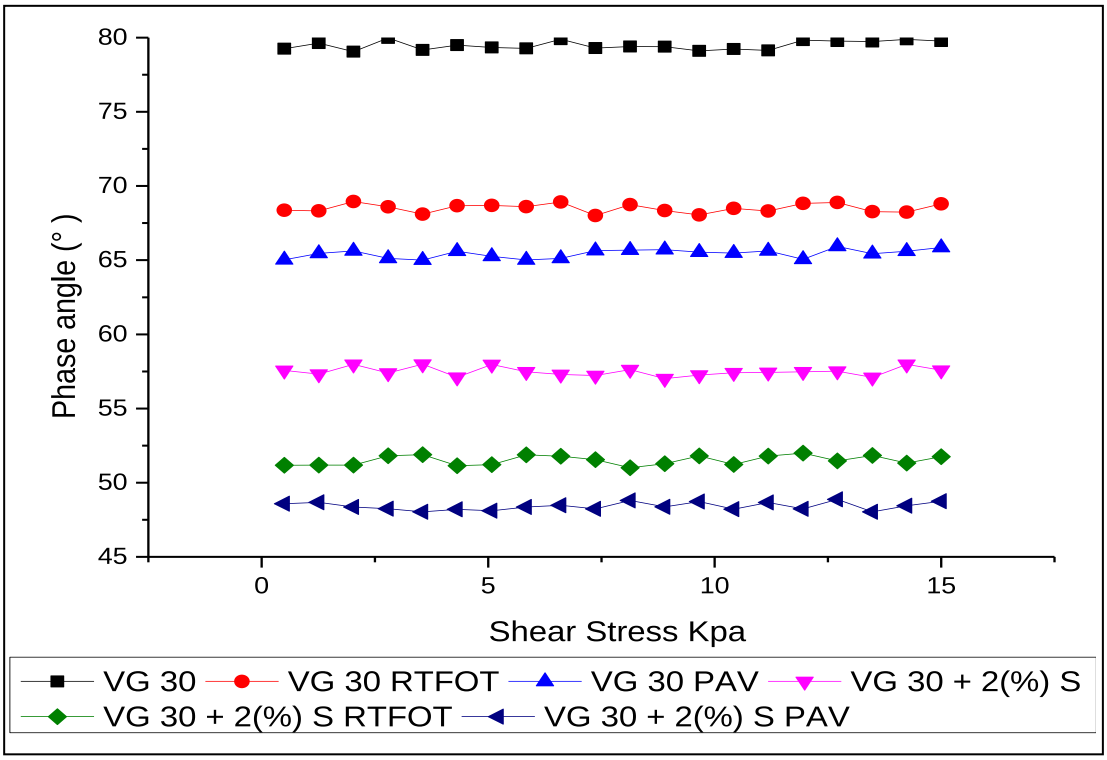



Figure 5.6(a): Variations of phase angle with shear stress

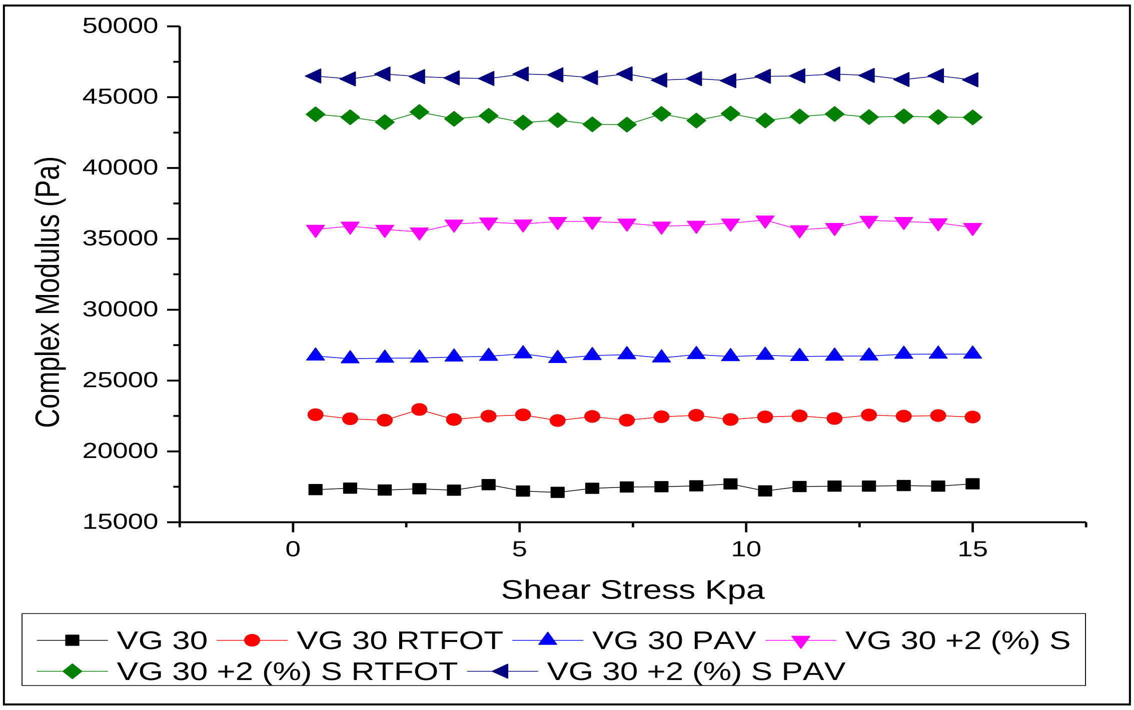



Figure 5.6(b): Variations of phase angle with shear stress

This test is intended to evaluate the ability of an asphalt binder to resist fatigue damage by applying cyclic loading at increasing amplitudes in terms of shear stress in order to accelerate damage. From the variations in both the graph for phase angle and complex modulus, it can be concluded that the 2% S modified VG30 binder will possess less damage than VG 30.

**5.3 Physical properties of modified and un modified bitumen binder test results**

The physical properties test results are presented in a tabular form Table 5.2 for unaged and aged VG 30 and 2%S modified VG 30 bitumen binder.

From the result obtained it can be concluded that 2% S modified VG 30 bitumen enhanced the physical properties more than the VG 30 bitumen. The increases in absolute viscosity of modified binder than neat binder indicate towards the more stiffness value to withstand against rutting failure. The increase in softening point of modified binder than conventional binder indicates towards increase in temperature susceptibility. Also due to less penetration value of modified binder than neat bitumen indicate the high stiffness value to resist rutting. There is an increase in elastic recovery percentage value, which results in more elasticity to resist fatigue failure. Similarly there exists a very good adhesion between modified binder and aggregate to decrease striping value more than neat bitumen binder.

Table 5.2: Physical properties of bituminous binder

| Type of Properties | VG 30 | Specification | VG 30 + 2 % S |
|---|---|---|---|
| Absolute Viscosity @ 60$^{o}$C (Poise) | 2512.15 | 2400 min. | 3208.1 |
| Kinematic Viscosity @ 135$^{o}$C (cst) | 449.72 | 350 min. | 663.75 |
| Softening Point ( $^{o}$C ) | 47 | 50 | 61 |
| Penetration Value @ 25$^{o}$C (dmm) | 57 | 50-70 | 35 |
| Ductility (cm) | >100 | >75 | 88 |
| Elastic Recovery (%) | 26 | – | 65 |
| Stripping Value<br>Boiling test (%)<br>Static Immersion test (%) | <br>2.5<br>2 | <br>< 5 | <br>2.25<br>1.5 |

**5.4 Storage stability Test results**

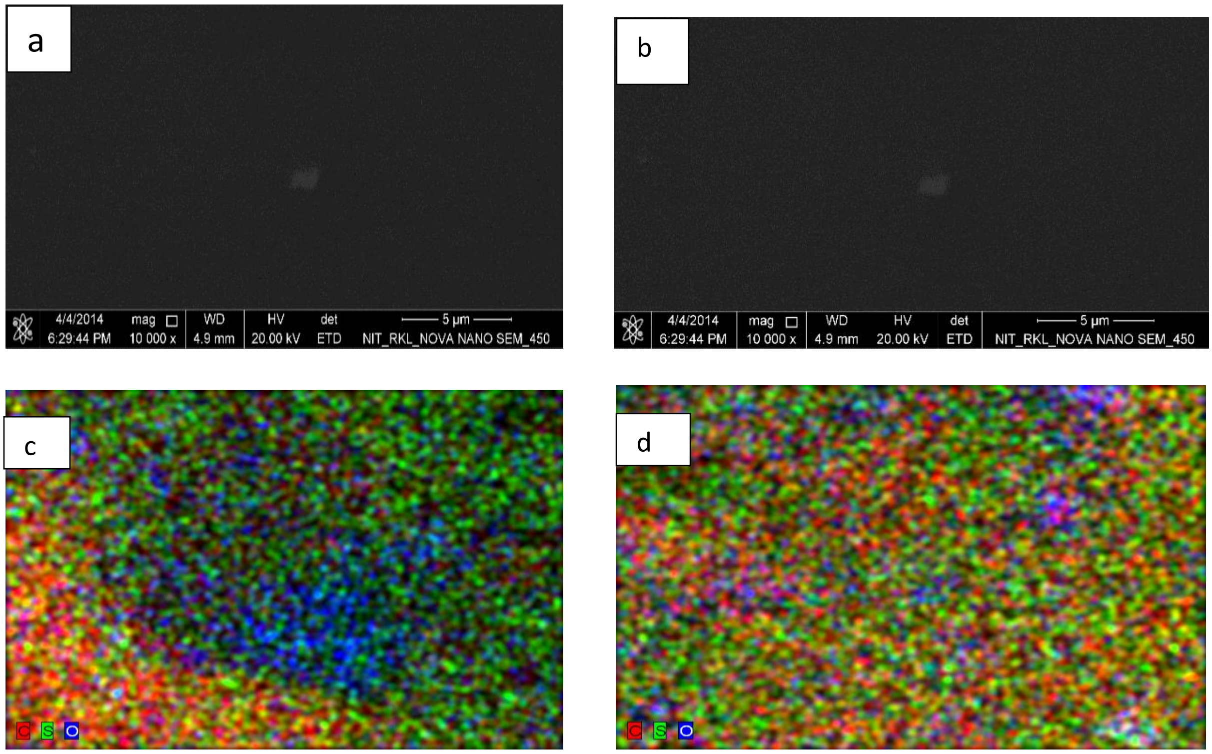
Test results are shown in tabular form in table 5.3 for VG 30 and 2% Sulphur modified VG 30 bitumen. It is clearly shown that the softening point difference for Vg 30 is 5.1$^{o}$C, while in case of modified bitumen binder the difference reduced to 2.5$^{o}$C.It can be seen that the storage stabilities of the VG 30 bitumen are improved with the addition of sulfur.

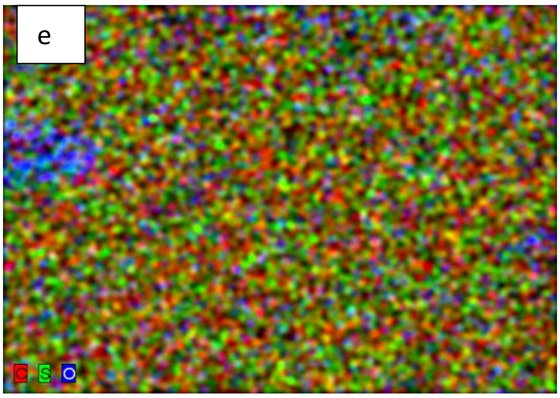
Table 5.3: Storage stability test results of bituminous binder

| Type of binder | Difference in softening point value $^{o}C$ |
|---|---|
| VG 30 | 5.1 |
| VG 30 + 2% S | 2.5 |

### 5.5 Morphology analysis test results

The compatibility between sulphur and VG 30 bitumen is critical to the properties to understand. The morphology of the Sulphur modified bitumen before and after ageing was investigated using Field emission scanning electron microscopy by characterizing the distribution and the fineness of sulphur in bitumen matrix. More focus was given towards the homogeneity in the blending of sulphur and bitumen. Some pictures have been demonstrated in figure 5.8 (a), (b), (c), (d), (e) & (f).





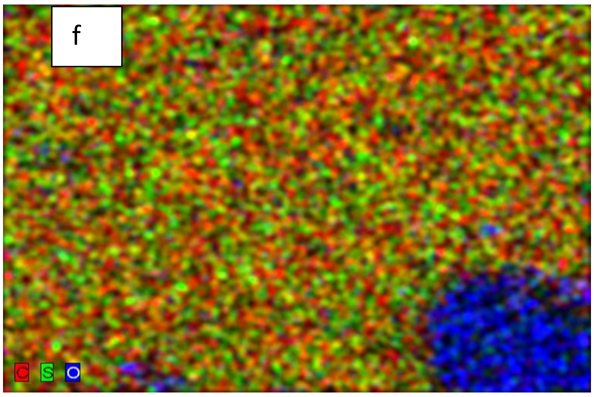




Figure 5.7: (a) morphology development for VG 30 bitumen

(b) Morphology development for VG 30 bitumen modified with 2% of sulphur

(c) EDX for VG 30 bitumen,

(d) EDX for VG 30 bitumen modified with 2% of sulphur,

(e) VG 30 bitumen modified with 2% of sulphur after RTFOT

(f) VG 30 bitumen modified with 2% of sulphur after PAV.

The EDX microanalysis technique was chosen for morphological analysis of the materials with a better understanding of the chemical composition of their samples. Due to black body of VG 30 bitumen, it is very difficult to conduct morphological analysis shown in Figure 5.7 (a) & (b). Sulphur is soluble in bitumen, as a result of which it is too much difficult to observe the homogeneity of sulphur inside the VG 30 bitumen. Hence to check the homogeneity of sulphur inside the bitumen after blending EDS technology has been taken. From EDS figure 5.7 (b) it is clearly seen the homogeneity of sulphur in green color inside bitumen after blending. Similarly there exists excess oxygen in bitumen after RTFOT and PAV in figure 5.7 (e) & (f) which is due to aging effect of VG 30 bitumen.

# CHAPTER-6

## CONCLUSIONS AND RECOMMENDATIONS

### 6.1 Conclusions

Several modifiers have been tried to improve the properties of bitumen in terms of engineering properties and performance criteria to derive the maximum benefits to withstand the wheel loads of the modern day traffic causing heavy stresses. Sulphur is one additive which is found to enhance the performance of the bitumen binder. In this research work, sulphur has been added to VG 30 bitumen maintaining at140$^{o}$C temperature through mechanical stirring for about 30 minutes to introduce a homogeneous modified binder. To ascertain the modification in quality and quantity, the temperatures for mixing/ blending, mixing/ blending time sand the sulphur concentrations in bitumen were varied from 100°C to 160°C, from 5 min to 30 min and from 0% to 5% by weight respectively. A number of rheological properties have been studied for binders under both aged and unaged conditions. The following concluding remarks have been drawn:

- Considering the criteria of complex modulus and Phase angle, addition of 2% sulphur by weight of VG 30 bitumen blended at 140$^{0}$C temperature for about 30 minutes time results in the optimum mixing/blending condition.
- In respect of unaged binder situation, the addition of sulphur to the extent of 2% to the conventional VG 30bitumen improves the viscoelastic behavior in terms of resistance to fatigue and rutting in comparison to the unmodified binder.
- The sulphur modified binder is observed to possess superior viscoelastic and other rheological characteristics in case of the aged binders also.
- The sulphur modified binder is found to satisfy the physical property requirements.
- The morphological tests show homogeneity of sulphur in the bitumen matrix.

- The storage stability test in case of modified binder does not show any non-homogeneity as observed by the conduct of the softening point test.

## 6.2 Recommendations for future work

There are some recommendations for further future research work as briefly listed below.

- It is recommended to conduct time sweep test through DSR for different models for fatigue life determination of modified binders.
- It is proposed to execute determination of fatigue life of bituminous binder with dissipated energy method.
- It is recommended for study of multiple stress creep recovery to study the performance of binder.
- The correlation of Rheological test of modified binders may be made with the mix properties such as, indirect tensile strength, resilient modulus and fatigue life.

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