# ENGINEERING CHARACTERIZATION AND EVALUTION OF SULPHUR MODIFIED BITUMINOUS BINDERS

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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*

## INTRODUCTION

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].**

**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:

1) Improving the mix design of bituminous mixtures.

2) By improving the construction methods and maintenance techniques.

Introduce modification of bitumen so as to improving the bituminous mixture.

## 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.

## 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.

## LITERATURE REVIEW

### Lesueur, (2009)

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.

### Airey,(2009)

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.

**Bahia and Davies (1994)**

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.

**A. B. Brown., et al (1957)**

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.

## METHODOLOGY

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.

### 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.

## 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.

## 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.

## Tests Performed using DSR

- Strategic Highway Research Program Grade Determination (SHRP)
- Frequency sweep
- Amplitude Sweep

## Aging of bituminous binder

Aging occurs due to exposure 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.

- Short term aging
- Long term aging

## Determination of physical properties of bitumen

- Penetration Test
- The softening point test
- Ductility Test
- Elastic Recovery
- Adhesion properties Test
- Storage Stability Test
- Morphology

## EXPERIMENTAL PROGRAM

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.

# RESULTS AND ANALYSIS

## Material

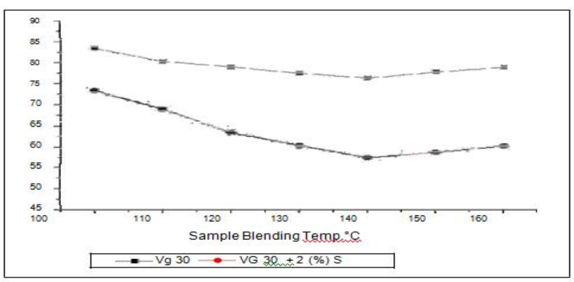
It is known from the studies that the level of modification relies on upon the neat bitumen type and modifier type

Physical properties of VG -30 bitumen

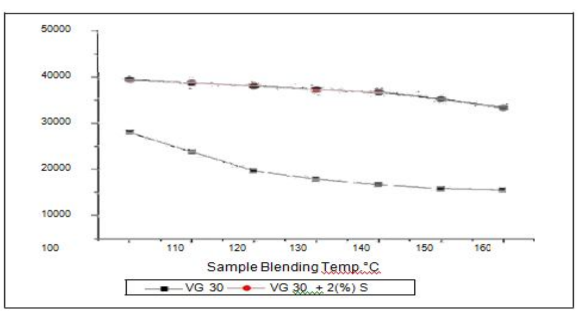
| Properties | Result |
|---|---|
| Absolute viscosity 60°C ( Cp) | 2462 |
| Kinematic Viscosity 135°C (cst) | 365 |
| Softening point °C | 47 |
| Penetration (dmm) 25 °C | 57 |
| Ductility (cm) 25°C | >100 |
| Elastic Recovery (%) | 26 |

**Rheological properties of unaged and aged binder test results**

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

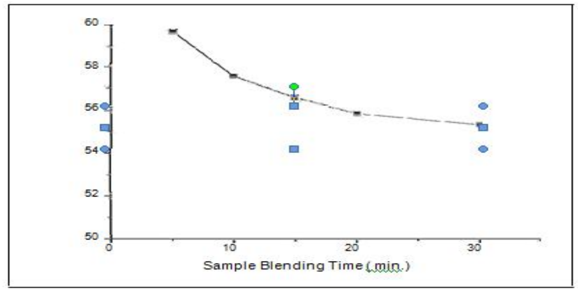



Variations of phase angle with different blending temperature

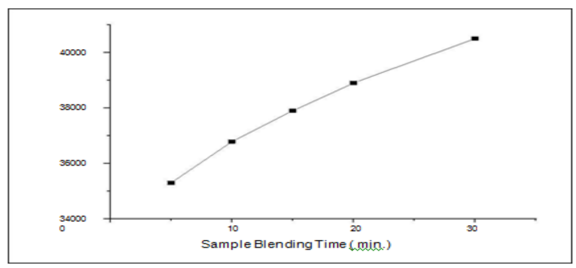



Variations of complex modulus with different blending temperatures

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

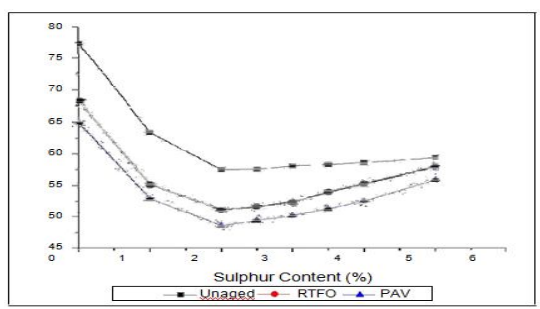



Behavior of phase angle with change in blending time for 2% sulphur modified bitumen

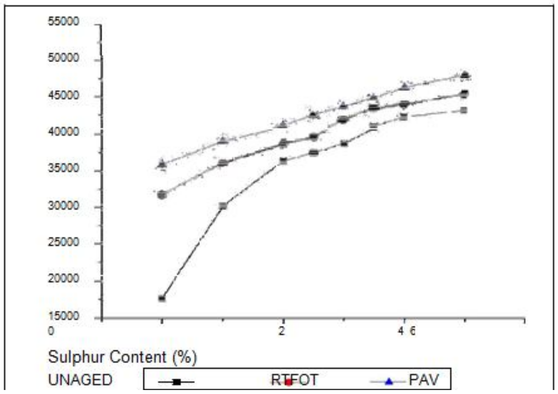



Behavior of complex modulus with change in blending time for 2% sulphur modified bitumen

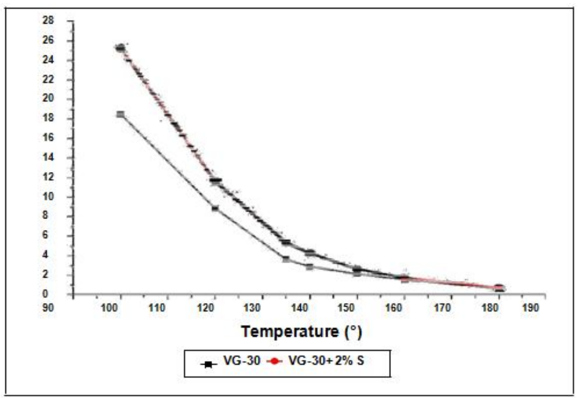
**SHRP test results for optimum sulphur content to get good modified bitumenbinder**



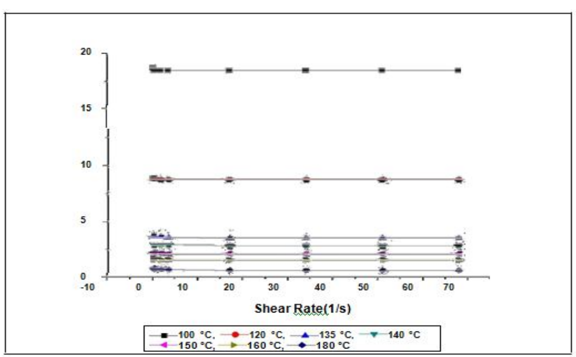

Variations of phase angle with increase in sulphur content



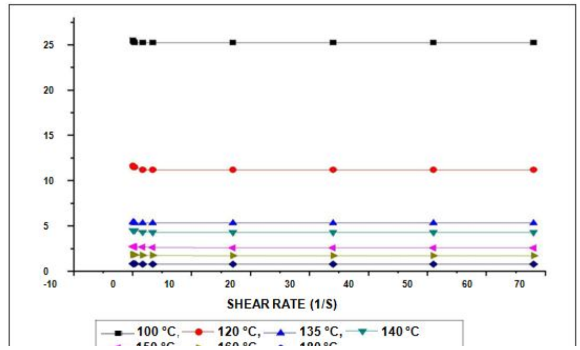

Variations of complex modulus with increase in sulphur content




Behavior of viscosity of bitumen with variation of Temperature.




Variations of viscosity of VG 30 bitumen with increase in shear rate for different temperatures



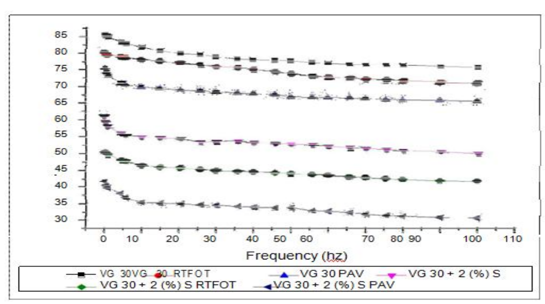

Variations of viscosity of 2% sulphur modified VG 30 bitumen with increase in shear rate for different temperatures

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

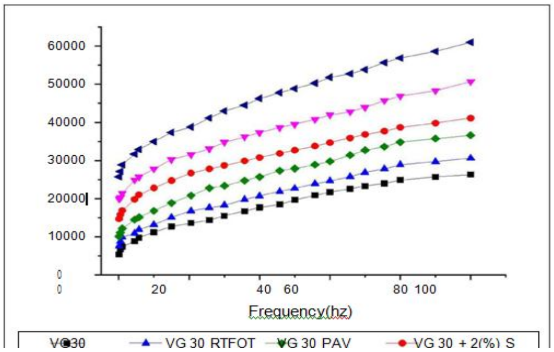
#### 5.2.5.1 SHRP grade determination test results

| 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 |

### Frequency Sweep Test results

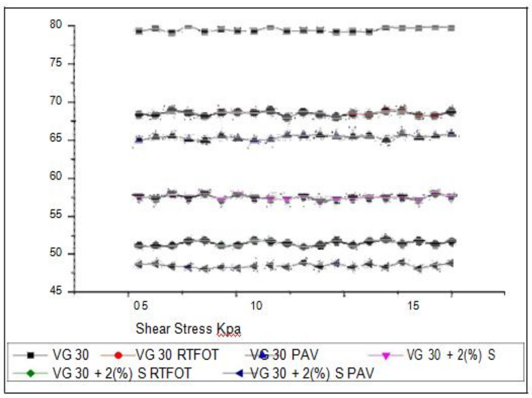



Master curve: Variations of phase angle with various loading frequencies

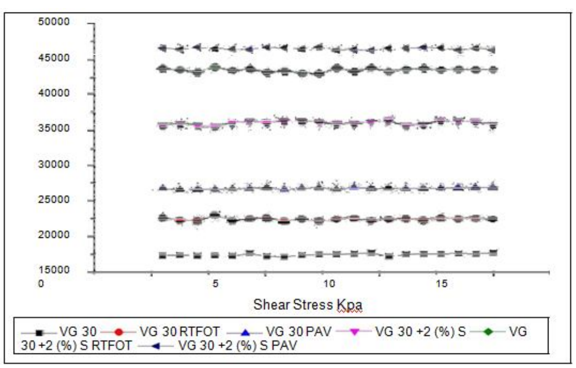



Master curve: Variations of complex modulus with various loading frequencies.

**Amplitude Sweep Test results**




Variations of phase angle with shear stress




Variations of phase angle with shear stress

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

Physical properties of bituminous binder

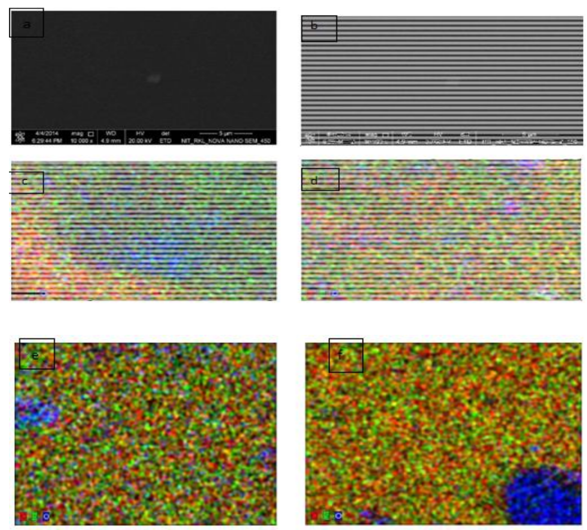
| Type of Properties | VG 30 | Specification | VG 30 + 2 % S |
|---|---|---|---|
| Absolute Viscosity @ 60°C (Poise) | 2512.15 | 2400 min. | 3208.1 |
| Kinematic Viscosity @ 135°C (cst) | 449.72 | 350 min. | 663.75 |
| Softening Point (°C) | 47 | 50 | 61 |
| Penetration Value @ 25°C (dmm) | 57 | 50-70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

**Storage stability Test results**

Storage stability test results of bituminous binder

| Type of binder | Difference in softening point value °C |
|---|---|
| VG 30 | 5.1 |
| VG 30 + 2% S | 2.5 |

**Morphology analysis test results**




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.

## 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:

1. 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.
2. 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.
3. The sulphur modified binder is observed to possess superior viscoelastic and other rheological characteristics in case of the aged binders also.
4. The sulphur modified binder is found to satisfy the physical property requirements.
5. The morphological tests show homogeneity of sulphur in the bitumen matrix.
6. 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.

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