# A Review: Use of Rheologyin Analysing the Properties of Bitumen

**Dr. Maninder Singh[1], Sharandeep Singh[*2]**

[1]Assistant Professor, Department of Civil Engineering, Punjabi University, Patiala.
[2]M. Tech Student, Department of Civil Engineering, Punjabi University, Patiala.

**Abstract:** *During the past few decades, rheology is being used enormously for the purpose of classification and evaluation of bituminous binders in relation to its performance properties. By learning about rheological properties of bitumen we get to know about bitumen behaviour better under distinguish thermal and mechanical conditions. This study aims at the use of rheological properties in analysing and evaluating properties of bitumen such as fatigue resistance, rutting resistance, ageing etc. The fatigue and rutting behaviour, which are the two pavement distress modes, are analysed using output parameters of Oscillatory Rheology using DSR Testing and the limitations of these parameters are also studied. Ageing of bitumen is using rheological properties is such as complex shear modulus and phase angle, as ageing causes certain modifications in the colloidal structure of bitumen and also in asphaltenes and maltenes fractions. The difference in rheological properties of neat bitumen and PMB bitumen due to ageing is also studied. Also comparisons are made between complex shear modulus and physical test results (penetration and softening point) of Bitumen claiming lower the penetration, and higher the complex shear modulus, harder the bitumen.*

***Keywords:*** **Rheology, Dynamic shear rheometer, fatigue, rutting, ageing**

## 1. INTRODUCTION

Bitumen is treated as a liquid which is thermoplastic and viscoelastic as it acts in elastic manner and it depicts linear behaviour like a solid glass at low temperatures and/or during short loading times and it acts like viscous fluid which can also be termed as Newtonian fluid and depicting linear behaviour at high temperatures and/or during long loading times. In simple terms, for ideal solid materials when the load is removed their deformation can be retrieved back so they are said to be behaving in an elastic manner, but in case of liquids, when the load is removed, a permanent deformation of the material is achieved yet the deformation occurring cannot be recovered so they are said to be behaving in a viscous manner. Now to further clarify the concept of rheology it is being said that materials can show a behaviour termed as viscoelastic in between these two ideal states as mentioned above, as the response to stress being partially elastic and partially viscous between these two ideal states. Thus rheology is a concept of deformation and flow which can be defined by its stress/strain/time /temperature response [1-4].

In earlier times, bitumen was predominantly characterised using empirical tests such as penetration test. With the development in road construction and the use of bitumen binders, it also leads to the development of new test methods that describe the properties in a better way. Now a days focussing is done the study of visco-elastic behaviour of bitumen for prediction of binder's properties in asphalt mixtures, especially in critical conditions of failure such as rutting, low temperature cracking and fatigue cracking in asphalt pavement roads also for evaluation of ageing. One of the famous method in evaluating visco-elastic properties of bitumen is testing by Dynamic shear rheometer (DSR) [1].

All the papers reported to date shows that by analysing rheological experiments and graphs some connection between rheological properties and properties such as ageing, rutting resistance, fatigue resistance, Softening point and penetration can be established. This paper aims to solidify such connection and co-relation between the mentioned above properties.

## 2. Composition of Bitumen

Bitumen can be separated into the four groups: saturates, aromatics, resins and asphaltenes (also known by the name SARA) [1]. The main point of difference due to which bitumen fractions can be distinguished as asphaltenes and maltenes, which comprises of saturates, aromatics and resins, is due to their solubility in n-heptane. Further the maltene fractions can be distinguished from each other on the basis of their difference in polarities [13].

Bitumen acts as a colloidal system which comprises of asphaltene micelles of high atomic weight dispersed or dissolved in maltenes, an oily medium of lower atomic weight. Bitumen can be categorised as sol and gel type on the basis of proportions of saturates, aromatics, resins and asphaltenes fractions present in it [1-4].

Other property which is quite significant in categorising sol type and gel type is their solvation capacity. The sol type allows free movements of the micelles because its solvation capacity is good enough. However in the case of the gel type, the asphaltenes micelles associate or get combined in order to produce irregular structures of connected micelles, the reason behind this is that solvation capacity of the gel type is not sufficient enough. Due to formation of these irregular structures in gel type bitumen, it is considered to have more elastic nature than sol type bitumen and less ductility [1,13].

## 3. Dynamic Shear Rheometer

In DSR testing, which stands for dynamic shear rheometer test, sinusoidal strains are enforced, in the form of oscillatory shear, on bitumen sample which is kept in between the parallel plates of the rheometer as shown in fig 1(a). The strains thus imposed should be kept small during the duration of test with as sole reason of staying of the test in the linear viscoelastic region. So generally the strains are kept less than0.5% during low temperatures, but they can be increased to higher value at higher temperatures [1-3].

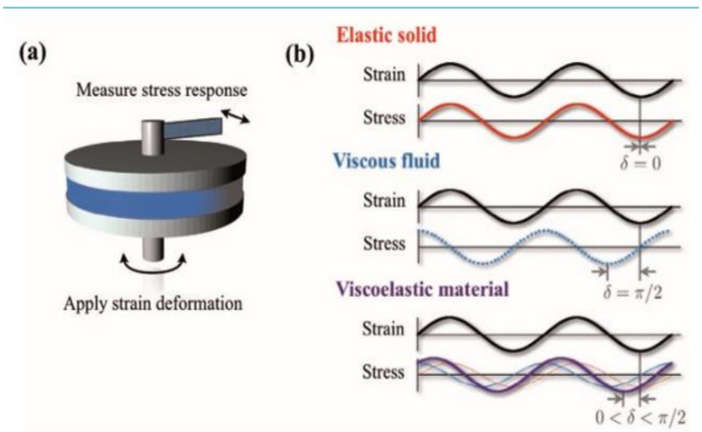


**Figure 1.Weitz (2007) et al. displayed (a)rheometry setup with samples placed between plates (b) Stress response to sinusoidal strain in elastic, viscous and visco-elastic material.**

The principal measurements from DSR testing are Torque transmitted (T) and Angular frequency (w). Other properties can be calculated from these measurements [1-3].

In response to the applied strain the responding stress can be measured by determination of torque which is transmitted in the sample.Complex shear modulus “$G^*$” called or the stiffness is defined as ratio of the resulting stress to the applied strain. The component of $G^*$ which is in phase is known as shear storage modulus ($G' = G^* \cos \delta$), also referred as the storage modulus and the component ($G'' = G^* \sin \delta$) that is out of phase is known as shear loss modulus also referred as loss modulus [1]

The developed sinusoidal stress, in DSR testing (only when small strains are applied), is out of phase with the imposed strain, this difference in phase thus generated is called phase angle (“δ”) [1-2].

If δ equals $90^o$ then the bituminous material is said to be purely viscous in nature, storage modulus dominates over loss modulus. If δ equals to $0^o$ then the bituminous material is designated to have purely elastic behaviour, loss modulus dominates over storage modulus. Between these two extremes states there occurs a stage where the material behaviour can be considered to be viscoelastic in nature with a combination of viscous and elastic responses [3].

The principle that is being used to construct master curves by co-relating the equivalency between temperature and time is called time-temperature superposition principle. For this purpose first step is to gather the dynamic data for different range of temperatures and frequencies, then in the second step selection of the standard reference temperature is done carefully generally between 0 and $25^o$ C and then in the third step data gathered at different temperatures is then shifted with reference to time until the plot converges into a one smooth function [1].

The reason for DSR testing of bitumen to become famous and worldwide is the United states bitumen research programme conducted by Strategic Highway Research Program (SHRP). A large number of tests studying rheology and failure of bitumen were developed and conducted and conducted by SHRP, and properties like complex modulus ($G^*$) and the phase angle (δ) were measured along various range of frequencies and temperatures, that co-related with the fundamental properties of bitumen and analysis of properties such as ageing, fatigue resistance, rutting resistance, ageing, softening point etc [1-5].

## 4. Literature Review

### 4.1. Analysis of Fatigue and Rutting using Rheology of Bitumen

Fatigue which occurs due to repetition of load or due to dynamic load which is variable and is lower than the ultimate static load and reduces the serviceability of flexible pavements. In order to determine fatigue cracking accurately, the pavement should be tested after long duration from its construction, because fatigue cracking takes place late in life so the bitumen is required to be tested after long term ageing [1,16].

Accumulation of permanent deformation or consolidation in the pavement along the wheel path due to high summer temperature, improper mixing etc. called rutting [16].

**Ferry (1971)** stated that ($G^* \sin \delta$) can be used as parameter to evaluate fatigue.

**Subhy (2017)** stated that SHRP Parameter ($G^* \sin \delta$) is used to determine the fatigue properties of bitumen within a intermediate range of temperatures. As the dissipated energy during each loading cycle is reduced it is required that the value of SHRP Parameter ($G^* \sin \delta$) should be small. Smaller values of complex modulus ($G^*$) will be helpful in dissipating the work energy without generating any large stresses, while smaller values of phase angle (δ), making it more elastic, will be helpful in retrieving the initial actual shape of bitumen with minimal dissipated energy

**Petersen (1994) et al.** chose 0.1 sec as loading time which is remarked as a truck tyre travelling at a speed of 80 km/hr to be regarded as the loading time in the pavement.

**Anderson (1993) et al.** applied load which had a magnitude that varies just like a sine curve at a frequency of 9.99 rad/s is equivalent to this 0.1 s loading time. The specification criterion, which is numerically equal to, $G^*/\sin\delta$ (inverse of loss compliance 1/J") can be used to evaluate rutting behaviour.

**Anderson (1993) et al.; Petersen (1994). et al.** stated that for an aged asphalt binder, rutting parameter ($G^*/\sin\delta$) shall be > 1 kPa and asphalt binder whose ageing is done using RTFOT ageing instrument the values should be > 2.2 kPa and for good fatigue resisting properties ($G^* \sin\delta$) values shall be < 5000 kPa. SHRP also approved these specifications in 1994.

**Joshi (2013) et al**. dealt with the study of rheology of the bitumen binder with a view of studying fatigue and rutting properties. The pavement stretch from which the bitumen binder was taken was four years old. Tests were conducted using DSR on the extracted bitumen binder and values of complex modulus (G*) and the phase angle (δ) were determined. On the basis of test results it was concluded that the range of complex modulus (G*) values can vary from about 0.5 kPa to 6 kPa, while the range of phase angle (δ) values can vary from about $50^o$ to $90^o$. Bitumen will turn out to be more viscous with the increase in values of the phase angle. The values including values of fatigue and rutting resistance parameter for all the sections were within permissible limits.

**Ziaria (2006) et al**. dealt with a phenomenon called fatigue which occurs due to repetition of load or due to dynamic load which is variable and is lower than the ultimate static load. Samples were prepared using carbon nanotubes as modifier in bitumen such that the samples that were prepared contained carbon nanotubes in the percentage varying from

(0 - 1.5) %. Then experiments were conducted on these samples to determine rheological and fundamental properties. With the addition of carbon nanotubes, the properties such as softening point, penetration etc. and DSR properties such as complex modulus, phase angle and shear loss modulus, ($G^* \sin\delta$) showed improvement and enhancement as compared to standard bitumen. The fatigue resistance parameter ($G^* \sin\delta$) which is also known as shear loss modulus is very helpful in determining the behaviour of bitumen under severe loading and consequently, asphalt fatigue. Hence by addition of CNT to bitumen, this parameter the fatigue resistance property of bitumen improves and also it increases long term efficiency of pavement. Since the phenomenon of fatigue generally occurs at lower values of temperatures in pavement therefore with the use of rheology behaviour of bitumen can also be studied at lower values of temperature. With addition of this nanoparticle to the standard bitumen, performance of bitumen at lower values of temperature (performance at high frequencies) significantly improves [9].

**Subhy (2017)** suggested that the current SHRP parameters does not necessarily contain the true binder contribution related to mixture or pavement performance due to following reasons:

(1) The fact that the bitumen binder used in the pavements will be subjected to subjected to very high ranges of stresses and strains however the binder that is being tested using DSR is subjected to small strains having magnitude very less in the form of sine waves so that it remains in linear viscoelastic region. Now it can be said that the values of complex modulusand phase angle from DSR testing does not fully account for the strains or stresses that takes places in bitumen used in pavements.

(2) Bitumen that is being used in the mixture to be laid on the pavement can witness a large variety of strain distribution, such that if it is to be compared with bulk strain of

mixture it can be up to 100 times, however it mainly depends upon the volume of mixture and the properties of constituent material. But in DSR testing in evaluation of the SHRP Parameter the bitumen witnesses very few loading cycles and with very low strain generally about 1%.

(3)The theoretical derivation which is used to derive SHRP parameter is not clearly understood.

(4) The yielding or bending of bitumen layer and various other damaging situation in the pavement are totally neglected by the SHRP parameter.

The use of the SHRP parameters (G*/sin δ) rutting resistance and (G* sin δ) fatigue resistance which corelate the properties of bitumen and the data measurements from DSR testing has some limitations in tracing performance of bitumen. This limitation is more of significance in case of polymer modified bitumen. The main reason attributed to this was the complex rheological nature and properties of PMB, so larger quantities of measurements and data is required for their complete analysis and evaluation. Due to these limitations DSRmethod was substituted to unconventional and uncommon methods like the MSCR test [1].

**Subhy (2017)** suggested of using Multiple stress creep recovery (MSCR) test using DSR can be used as a alternative test for evaluation of fatigue behavior based on the delayed elastic response.

**Shenoy (2001)** proposed a Shenoy rutting parameter

(G*/ (1– (1/tan δ sin δ)) which can be used as a refinement of SHRP parameter for evaluation of rutting behaviour of bitumen. This rutting parameter is more sensitive to phase angle (δ) as compared to SHRP parameter. Therefore it was said that this parameter would explain the change in elastic properties better in case of polymer modified bitumen.

**Wang (2014) et al**. identified the relationship between the two DSR parameters also known as rutting parameters ($G^*$/sin δ), the non-recoverable stiffness parameter (G*/ (1–(1/tan δ sin δ)) and the MSCR-based parameter nonrecoverable compliance (Jnr). And it was found that the nonrecoverable stiffness parameter shows a greater connection and link with the Nonrecoverable Compliance (Jnr) than the rutting parameter. Hence the nonrecoverable stiffness parameter (G*/ (1– (1/tan δ sin δ)) should be a used to substitute rutting parameter ($G^*$/sin δ) when using DSR to evaluate rutting properties of asphalt binder

**4.2. Analysis of Ageing of Bitumen using Rheology**

Ageing is considered as one of the major cause for pavement ravelling and cracking of pavement. When the bituminous pavements are exposed to oxygen, UV radiation and temperature change oxidation occurs and there is loss of volatile components this change in chemical, physical and rheological properties is called ageing. It is very complex process which makes the pavement hardened and brittle resulting in deterioration both during application and in service [4]. Dynamic rheological analysis is done by using DSR and ageing of the bitumen binder can be analysed and assessed using rheological characteristics such as complex modulus ($G^*$) and phase angle (δ). [5]

**Mastrofini, (1999) et al**. dealt with the process of evaluation of the properties of three vacuum residues before and after ageing concluded that the phenomenon of ageing is majorly dependant upon temperature. An observation about the significant increase in complex modulus at lower values of frequencies or at medium high temperatures, in case of the aged bitumen binders in contrast to the standard ones. the main reason attributed behind this increase of complex modulus was the increasing values of both the shear loss modulus (G’’) and shear storage modulus (G’) components. Further ageing does not hinders much with complex modulus at values of higher frequencies or lower temperatures values. A small decrement in loss tangent (tan δ) was noticed in graph of loss tangent and Temperature ($^{o}$C) (the original and aged vacuum residues) and main reason behind it was suggested as the conversion of sol like structure into gel like structure [3]. The slope of the linear part of the tan δ and Temperature ($^{o}$C) curve can be regarded as a significant guide and clue for the thermal susceptibility of a material. [5].

**Airey (2003) et al.** conducted a research on SBS polymer modified bitumen and ageing using DSR testing. The methods that were used for the purpose of ageing were Rolling Thin Film Oven Test (RTFOT) for short period ageing and pressure ageing vessel (PAV) for long period ageing of the SBS polymer modified bitumen.

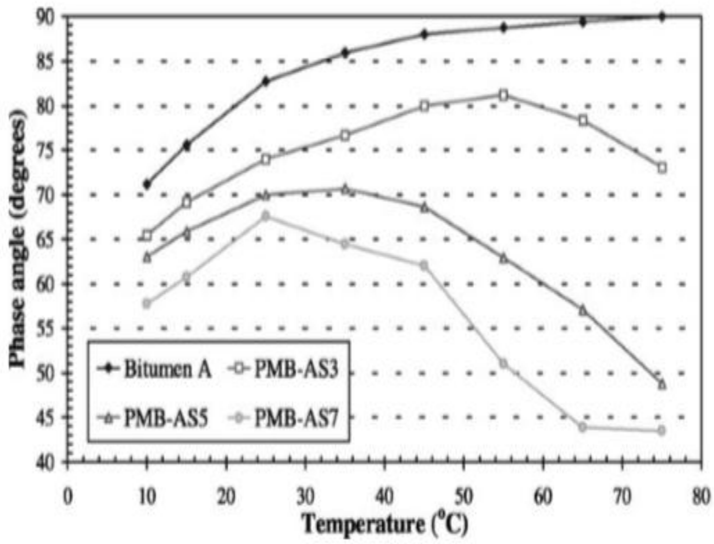


**Figure 2. Airey (2003) et al. plotted this graph of phase angle at 0.02 Hz versus temperaturefor polymer modified bitumen [SBS].**

The phase angle versus temperature plots, as illustrated in fig 2, shows increased elastic response as the values of phase angles reduces more in case of the PMB with respect to base bitumen. While the phase angles of the two base bitumen approach 90$^{o}$ and therefore they have predominantly viscous behaviour with increasing temperatures. Thus it can be said that with the use of SBS polymer the elastic deformation behaviour or which can be said as elastic response of bitumen increases considerably. The credit for this increment at high temperatures can be ascribed to the viscosity of the base bitumen. The mechanical properties of the modified bitumen gets affected easily by the elastic system of the polymer since the viscosity of standard bitumen is low and improves elastic response.

**Weigl (2017) et al.** highlighted the importance of asphaltenes content in the unaged bitumen in the view of ageing of bitumen by making comparisons of chemical characteristics of bitumen with the rheological and ageing characteristics. When ageing of bitumen takes place the bitumen becomes more stiff, this is due to increasing nature of the binder into gel like as the content of asphaltenes in the bitumen gets increased. This conversion from sol to gel is explained in changes in complex modulus readings before and after ageing. Secondly the phase angle (δ) captures an increasing elastic deformation behaviour. With the ageing of bitumen the values of phase angle starts decreasing. So a inversely proportional relationship between the phase angle and the asphaltene content is identified.

**Bachir (2016) et al**. dealt with the rheological properties to compare the Styrene-Ethylene-Butadiene-Styrene (SEBS) polymer modified bitumen (PMB) and 35/50 penetration grade bitumen, before and after ageing [6].

Lower slope of complex modulus (G*), as shown in figure 3, means that the asphalt is softer nature, also higher complex modulus has advantage that it reduces rutting problems in the asphalt. The results obtained indicate that the aged SEBS modified bitumen has better rheological properties than the corresponding base bitumen so it should have better durability.

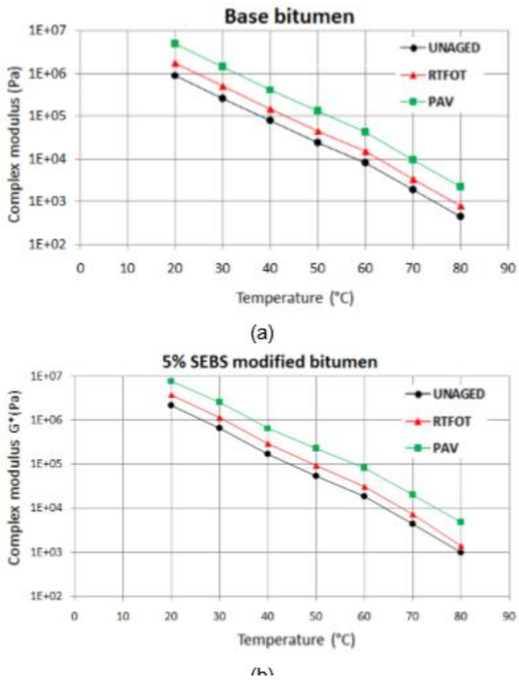


**Figure 3. Bachir (2016) et al. plotting an increase in complex modulus for the aged PMB as compared to the original one**

**Wu (2008) et al.**dealt with the rheological properties of standard bitumen binder (AH70) and styrene butadiene styrene polymer modified bitumen binder (PG70 and PG76) before and after aging. It was noticed that there was increment in values of complex shear modulus (G*) of SBS modified binders than that of standard bitumen at higher temperature values while there was slight decrement in values than that of standard bitumen at lower values of temperatures. Thus this proves that the with the use of styrene butadiene styrene polymer, it can improve the bitumen’s temperature susceptibility. With the expansion of aging time (0–2000 h), the values of complex shear modulus (G*) of bitumen gets increased, and three G*-temperature curves of base bitumen and its SBS modified binders tended to become closer, as shown in figure 4, indicates a somewhat same qualities and properties after a long-term aging.

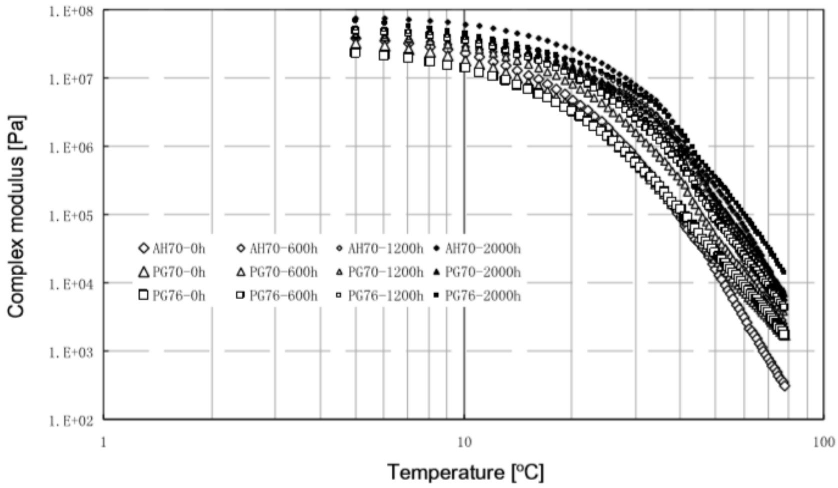


**Figure 4. Wu (2008) et al. plotted this graph of complex modulus versus temperature for standard and SBS modified bitumen at various aging times.**

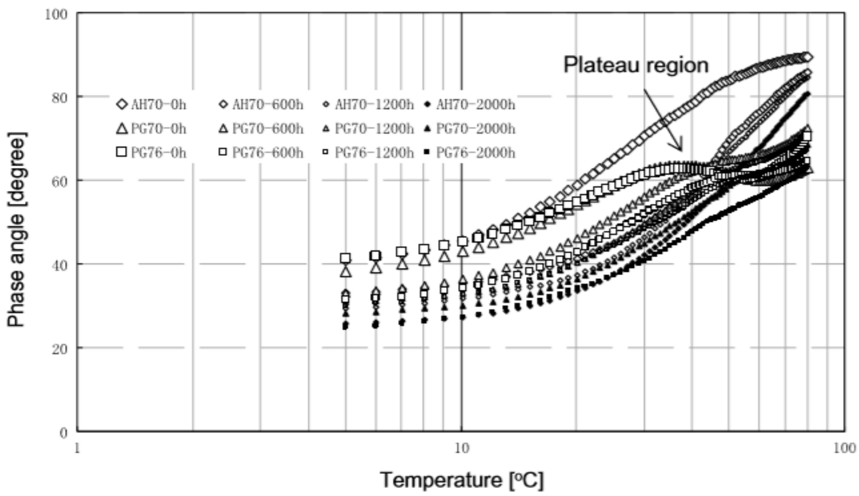


**Figure 5. Wu (2008) et al. plotted a graph phase angle versus temperature for standard and SBS modified bitumen at various aging times.**

From the plot as illustrated in figure 5 for the standardbitumen and polymer modified bitumen (SBS) shows that at higher values of temperature, the addition of modifier the values of the phase angle of the polymer modified bitumen faces decrement. A phenomenon of formation of plateau regions in the curves is visible in case of polymer modified bitumen. But as the ageing period elongates, those plateau regions appears to be vanishing finally. The plateau regions serve the purpose of indicating the damage ageing could cause to system of networks of polymers in bitumen.

**Kaya (2019) et al.** conducted research on the manner in which rheology of bitumen gets affected by on short term ageing and long term ageing. Varying shear rate and mixing time were the methods adopted to attain short term ageing and PAV and RTFOT were the methods adopted for long term ageing. DSR testing was performed on samples of SBS modified bitumen. The main effect of the short term ageing was oxidative ageing which in turn was the result of larger values of shear rate and longer values of blending times which further in increasing the values of the activation energy. As the temperature goes on increasing, samples of PMB samples illustrated a larger value of phase angle as compared with the value of standard bitumen in regarding to the same complex modulus behaviour. The main reason attributed for this was the significant increment in increasing viscosity at increasing values of temperatures.

### 4.3 Analysis of Physical Properties of Bitumen using Rheology

**Remišová (2019) et al**. dealt with the investigation of the properties of rheology for non-modified and polymer modified bitumen binders using dynamic shear rheometer by evaluating complex shear modulus and phase angle. Greater elasticity for PMB was considered as the reason for high complex modulus and low phase angle. Comparison of complex shear modulus values at 25 °C with penetration results showed that the harder the bitumen is, the lesser will be the penetration value and higher will be the complex shear modulus further comparison of phase angle at 50 $^{o}$C with softening point and frequency 1.5 Hz showed that lower the softening point higher will be the phase angle and less elastic will be the bitumen.

**Gershkoff (1995) et al.** in his experiment concluded that the softening point temperature and tan δ which is also known as loss tangent establish a linear correlation with each other.

**Mastrofini (2000) et al.** applied rheology victoriously to specify and analyse properties of bitumen using the linear correlation between loss tangent and the softening point temperature and using correlation between penetration value and the complex modulus.

## 5. Conclusion

This paper discussed the co-relation between rheological properties and properties such as ageing, rutting resistance, fatigue resistance, Softening point and penetration. Following conclusions can be drawn from this study:

(1) Shear loss modulus ($G^{*}$ sin δ) can be used as parameter to study the fatigue behaviour of bitumen while the parameter (G*/Sin δ) tells about rutting behaviour of bitumen.

(2) The strains of small magnitude are recommended to be applied on bitumen sample during the DSR testing which do not seems to fully represent the pavement conditions, and due to this there is rise of alternative approaches such as the MSCR test based upon delayed elastic response for calculation of fatigue resistance, and Shenoy rutting parameter (G*/ (1– (1/tan δ sin δ))for calculation of rutting resistance as more sensitive to phase angle (δ).

(3) For Ageing, at medium-high temperatures, there is an increase in complex modulus and ageing does not influence much complex modulusat low temperatures, and decrement in (δ)was due to conversion of sol like structure into gel like structure

(4) The complex shear modulus (G*) values of Polymer modified binders (such as SBS) were much greater in magnitude than as compared to standard neat bitumen at higher magnitudes of temperatures while they were marginally lesser at lesser magnitudes of temperatures. As the aging period continues to extend (G*) values of standard neat bitumen and SBS polymer modified bitumen tended to become closer, indicating somewhat same performance and properties after long term ageing.

(5) there is reduction in phase angles of the modified binders compared to base bitumen which is a indicator of improved elastic response

(6) Further the that the softening point temperature and tan δ which is also known as loss tangent establish a linear correlation with each other and while complex shear modulus and penetration value are inversely proportional.

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