EXPERIMENTAL STUDIES ON BIO-BITUMEN PRODUCED USING CHARCOAL FROM COCONUT SHELL WASTE

A Thesis submitted in fulfillment of the requirement for the award of the degree of

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IN

CIVIL INFRASTRUCTURE ENGINEERING

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DECLARATION

I hereby declare that this work which is being presented in the thesis entitled "**EXPERIMENTAL STUDIES ON BIO-BITUMEN PRODUCED USING CHARCOAL FROM COCONUT SHELL WASTE**" in partial fulfilment of the requirement for the award of degree of **Master of Engineering** in the field of **Civil Engineering** with specialization in **Infrastructure Engineering** submitted at **Thapar Institute of Engineering & Technology (Patiala)** is an authentic record of my own work carried out during the period from 13.8.2018 to 15.7.2019 under the guidance of Dr. Tanuj Chopra and Dr. Anush K C.

The matter embodied in this thesis has not submitted by me for the award of any other degree or diploma.

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This is to certify that the above declaration made by the student concerned is correct according to the best of our knowledge and belief.

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Dedicated to

My Parents

And

My Guides

Dr. Tanuj Chopra   Dr. Anush K C

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ABSTRACT

Road network plays important role in transportation to carry passengers and goods from one place to another. It is the basic need for development of infrastructure, helps in increasing socio-economic standards and economic growth of country. Pavement should be designed such that it can be capable to bear heavy loads in extreme environment conditions, undergo less wear and tear, good riding quality and drainage to prevent from moisture damage. Most of the Indian roads are of flexible pavements and the main distresses occur in the pavements are rutting and fatigue cracking which affects the serviceability and durability of the pavement. Although bitumen is only 5-6% by weight of mixture, it has 40% effect on the performance during service life. With the increase in percentage of over-loaded commercial vehicles, premature rutting in the bituminous mixtures has been observed very often on pavements and which leads to increase the maintenance cost of road network. So modification of binder is necessary now days.

In order to stiffen the bitumen, several methodologies have been adopted in the past and still research is being carried out in various other aspects to produce bitumen with superior permanent deformation resistance i.e. modified bitumen. On the other hand, tropical countries such as Indonesia, India, Philippines, and Srilanka being one of the largest producers of coconut in the world also produce burnt coconut shell (CS) waste, which creates environmental concern regarding disposal due to large percentage of carbon.

The main objective of this study was to reduce the environmental waste and use the charcoal powder from the CS waste as additive to VG30 bitumen at 0, 1, 2, 3 and 4% (by wt. of bitumen) and investigate its permanent deformation characteristics. The size of the MCP is less than 75 µ. High shear mixture at 1720 rpm at 150°C for 60 min was used to mix the MCP into bitumen. The basic properties of bitumen such as penetration value, softening point, penetration index were determined to understand the consistency of the bitumen. Further, dynamic shear rheometer (DSR) was used to determine rutting parameter ($|G^*|/\sin\delta$), complex modulus (G^*) phase angle (δ), loss factor ($\tan\delta$) and more other parameters. The permanent deformation resistance of bitumen was studied using multiple stress creep recovery (MSCR) test to quantify creep compliance, %recovery, and stress sensitivity. Frequency and temperature sweep test was also done to see the loading time. Moreover SPSS software was used for statistical analysis and scanning electron microscope test was conducted at MCP and modified binder to see the dispersion and homogenous mix of modified bitumen. The results showed that addition of MCP into neat binder has significantly increased the properties of control binder. Modified binder increases the rutting performance, stiffness of binder, creep recovery, elastic properties and makes the binder less sensitive at 2% addition of MCP.

Keywords: Modified bio-bitumen, Microcharcoal Particles, Coconut Shell Waste, Rheology and Rutting Parameter.

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LIST OF ABBREVATIONS

AASHTO- American Association of State Highways and Transportation Officials

ANOVA- Analysis of Variance

ASTM- American Society for Testing and Materials

C.B- Control Binder

CS -Coconut Shell

CSA- Coconut Shell Ash

CV- Coefficient of Variable

DBM- Dense Bituminous Macadam

DSR- Dynamic Shear Rheometer

FHWA- Federal Highways Administration

IRC- Indian Road Congress

IS- Indian Standards

LVE- Linear Viscoelastic Range

M.B-Modified Binder

MCP- Micro Charcoal Powder

MSCR- Multiple Stress Creep Recovery

PG-Performance Grade

PI- Penetration Index

POS-Palm Oil Shell

PMB-Polymer Modified Binder

PKS-Palm kernel shell

SEM-Scanning Electron Microscopy

SHRP- Strategic Highway Research Program

SPSS- Statistics Package for Social Science

VG- Viscosity Grade

LIST OF SYMBOLS

G^* - Complex Shear Modulus, kPa

δ - Phase Shift Angle, degree (°)

J_{nr} - Non-recoverable Creep Compliance, kPa^{-1}

f- Frequency, Hz

T- Temperature, °C

μ- Micron

η- Viscosity, P

ω- Angular Frequency, rad/s

Υ- Shear Strain, %

τ- Shear Stress, N/m^2

$\mathcal{E}_r$- Percentage Recovery, %R

G'- Storage Modulus, kPa

G''- Loss Modulus, kPa

$tan\delta$- Damping Factor

$G^*/sin\delta$- Rutting Parameter, kPa

Υ_L-Limiting Strain, %

CV- Coefficient of Variable, %

CHAPTER 1

1.1 BACKGROUND

Development of the country is based on the good connectivity of different places with adequate highway network. Road network plays significant role in transportation to carry passengers and goods from one place to another. It is the basic need for development of infrastructure, helps in increasing socio-economic standards and economic growth of country. Providing good quality of road network has direct impact on the growth of country and it is the assets for any developing or developed countries. Engineered and good quality road network starts with planning. Pavement should be designed such that it can be capable to bear heavy loads in extreme environment conditions, undergo less wear and tear, good riding quality and drainage to prevent from moisture damage. Tropical countries like India have large quantities of natural resources, which has been an important source for construction activities. The natural resources, which are mainly used in construction products, include jute fibres, coir, rubber, coconut shell waste, etc. These natural materials have decreased the usage of non-renewable resources in construction significantly and have been found to improve several properties. Pavement infrastructure being an important portion of construction activity provides a broad scope for the usage of natural products in pavement materials. Conventionally, pavements can be categorized into flexible pavements and rigid pavements. The bituminous pavements are class of flexible pavements, which consist of bituminous mixture layer in the surface to provide an impermeable layer, which prevents the entry of moisture in sub-base, base and subgrade, thus protecting the pavement structure. The rigid pavements consist of concrete slab laid on uniform foundation. The vehicular load is transferred by the slab action to lower layers. The typical cross-section of flexible and rigid pavement is shown in Figure 1.1 (a) and (b), respectively.

Among the bituminous and concrete pavements, the bituminous pavements constitute major portion of roads due to ease of construction, degree of quality control required and ease in repair and maintenance compared to concrete pavements. However, owing to increased overloading of commercial vehicles, rutting in bituminous pavements has become a major distress affecting the performance.

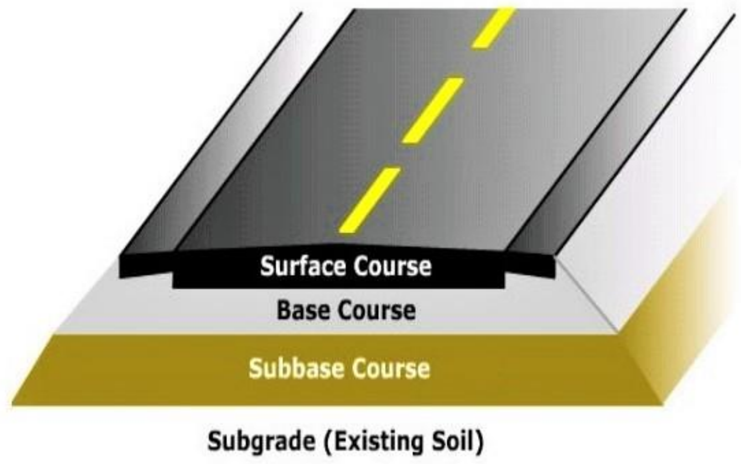


Figure 1.1 Typical Cross-Sections of Flexible Pavement

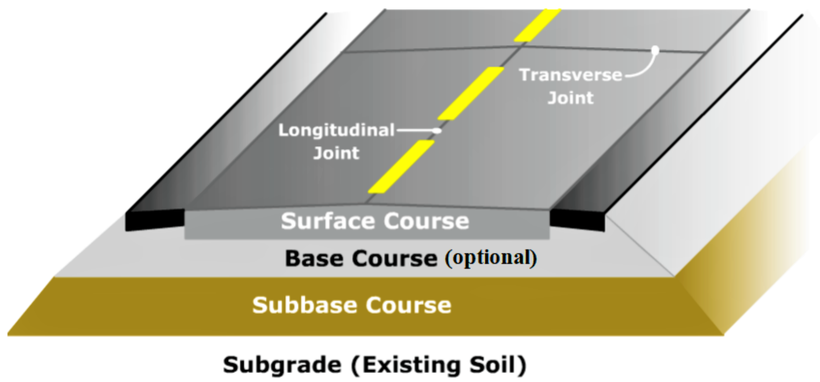


Figure 1.2 Typical Cross-Sections of Rigid Pavement

Road network in India is 59,03,293 km as per 31 January 2019. It is the 2nd leading country in the world. The quantitative density of road network in India is higher than Japan, U.S and Russia. Over 1, 01,011 km road network of national and expressways and 1, 76,166 km of state highways as per March 2016. Most of the Indian roads are of bituminous layers (flexible pavements) and the main materials for construction of bituminous layers are asphalt mixtures. It is the composite material which consists of coarse and fine aggregates with bitumen. Bitumen is also the main component and plays an important parameter in asphalt mixture to strengthen and resist the permanent deformation. Bitumen is the viscoelastic material, whose behaviour depends on time (loading conditions) and temperature. Rutting and fatigue are the major distresses occurring on asphalt pavement. At high temperature, bitumen shows viscous behaviour and at this temperature condition asphalt material pavement shows permanent deformation, which is referred to as rutting as shown in Figure 1.3. Rutting distress is the distortion of surface along the wheel path. It occurs because of inappropriate asphalt mix design or permanent deformation of subgrade and it occurs after few years laying of the asphalt layer, due to overloading and its repetitions, tire pressure at high temperature. The rutting performance depends on the angularity of aggregates, its gradation, nominal size of aggregates, stiffness of bitumen and contact between bitumen and aggregates in asphalt mixture. At intermediate temperature (10°C-35°C), pavement undergo fatigue failure due to repeated application of vehicular loads as shown in Figure 1.4. Vehicle loadings, environmental conditions and pavement structure are the three parameters which affect the fatigue cracking. It is difficult to prevent distresses in asphalt pavement due to seasonal variation in climate and increased overloading in commercial vehicles.

Figure 1.3 Rutting

Figure 1.4 Fatigue

In order to reduce the rutting, several methodologies have been developed, where usage of modified bitumen is one the most accepted methods. The modification of bitumen has been achieved using different compatible materials such as polymers, waste tire rubber, natural rubber, waste plastic, etc. These materials being polymeric in nature have been shown to improve the performance of the bitumen after modification. On the other hand, modification of bitumen using bio-materials such as soya bean oil, swine manure, etc. has been gaining attention of the asphalt technologists. In this research study, coconut charcoal powder, a class of bio-material is used to modify bitumen to investigate the changes in the performance characteristics of bitumen.

1.2 INTRODUCTION

Coconut is the member of palm tree family. The word "coco" is derived from Portuguese and Spanish in 16th century and its meaning is head or skull. The dried coconut named as copra. The production of coconut is on large scale in tropical countries like India, Malaysia, Sri Lanka, Philippines and Indonesia. India is the leading country in production and productivity of coconut from the last few years have significantly increased. The productivity of nuts per hectare is increased to11481 in 2016-2017 as compared to 10615 in 2012-2013. In 2017-2018, 13117 hectares new land was brought for new plantation of coconuts as compared to previous years. According to 2016-2017 for production of coconuts out of 15 states, top five states are Kerala (7448.65 million nuts), Karnataka (6773.05 million nuts), Tamil Nadu (6570.63 million nuts), Andhra Pradesh (1377.53 million nuts) and West Bengal (374.56 million nuts). Due to increase in productivity and production of coconuts, India is exporting dry coconut to the U.S and other European countries on large scale. Since April 2017 India has exporting coconut oil to Malaysia, Sri Lanka and Indonesia. Total coconut worth is Rs 1602.38 crore. India is ranked 3rd position with production of more than 21500 million tonnes coconut, with Indonesia in 1st position, Philippines in second and Sri Lanka in 4th position various coconut based products in the market like coconut water, coconut oil, milk powder and coconut sweets. The fibrous layer and shell part have no use to industries and they have no economic value and have no commercial returns therefore available abundantly in large quantities as agricultural waste from industries in tropical countries. Around 3.18 million tonnes of CS as solid waste formed annually which contributes global pollution and annual global production of coconut fiber or coir fiber is 350,000 metric tonnes but 90% production from India and Sri Lanka. This waste is present in huge quantity due to increment in production and demand and effecting on environment and landfills so it is easy to reuse as coconut shell (CS) charcoal, activated carbon from CS, ropes, brooms, doormats, rugs, mattresses and many other products. Coconut biomass has huge potential to generate electricity and heat. Activated charcoal from CS helps to remove the impurities from water. Due to high calorific value of 20.8 MJ/kg, it can be used to generate steam, bio-oil and bio-char. Before CS was burnt for land disposal as solid waste which emits carbon dioxide and methane but now the Nigeria is using CS as a source of fuel for electricity generation and for boilers and the reside used as modification of binder and replacement as filler in concrete. The parts of the coconut are outer layer, fibrous layer or coconut husk, shell (strongest part of coconut), coconut meat and coconut water as shown in the Figure 1.5.

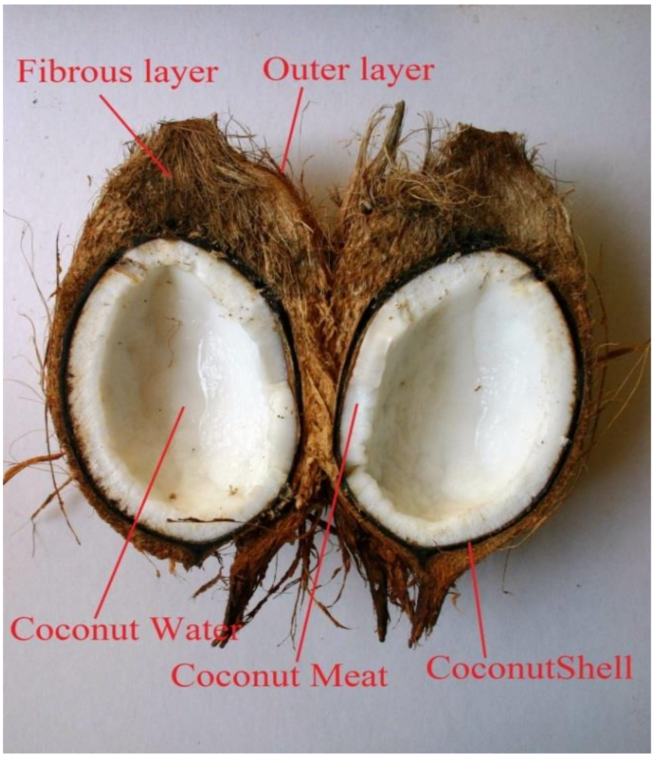


Figure 1.5 Description of Coconut

1.3 PROBLEM STATEMENT

The characteristics of neat bitumen without any modifications have been found to be not satisfactory considering the present scenario of vehicular traffic. Most of the flexible pavements have undergone permanent deformation due to lack of stiffness and overloading of traffic on the roads which is further exaggerated by high temperature. Although, several additives such as polymers, crumb rubber have been used to improve rheological properties of bitumen, such bitumen has been found to be highly expensive due to raw materials and production process. On the other hand, environmental wastes like coconut shell charcoal, palm oil shell ash and rice husk ash are creating disposal issues. Due to their fine nature, these environmental wastes can be used in the bitumen as fillers in order to bring about improvements in rheological properties, which may prove to be economical.

1.4 RESEARCH OBJECTIVE AND SCOPE

The major objective of research is to evaluate the physical and a rheological property of the bitumen modified using coconut microcharcoal powder and understands the permanent deformation characteristics using oscillation and multiple stresses creep recovery (MSCR) test. The scope of research work included:

- Literature review to understand the current situation of research work in modification of control binder and role of rheological properties moreover identification of gap in research
- Selection of coconut shell microcharcoal powder (MCP) and VG-30 bitumen grade for modification at various percentages
- Investigation of basic and rheological properties of C.B and M.B
- Determination of Linear Viscoelastic Range (LVE)
- Examination Complex Shear Modulus (G*) and Phase Angle (δ)
- Determination of Frequency and Temperature Sweep test
- understand Percentage Recovery (%R) and Non Recoverable Creep Compliance (J_{nr}) of control and modified bitumen
- Understanding stress sensitivity of control and modified bitumen
- Scanning electron microscopy (SEM) analysis of coconut shell charcoal and modified binders
- Statistical Analysis of MSCR test using SPSS and Coefficient of variable (CV %)

The basic outline of the research study shown in Figure 1.6

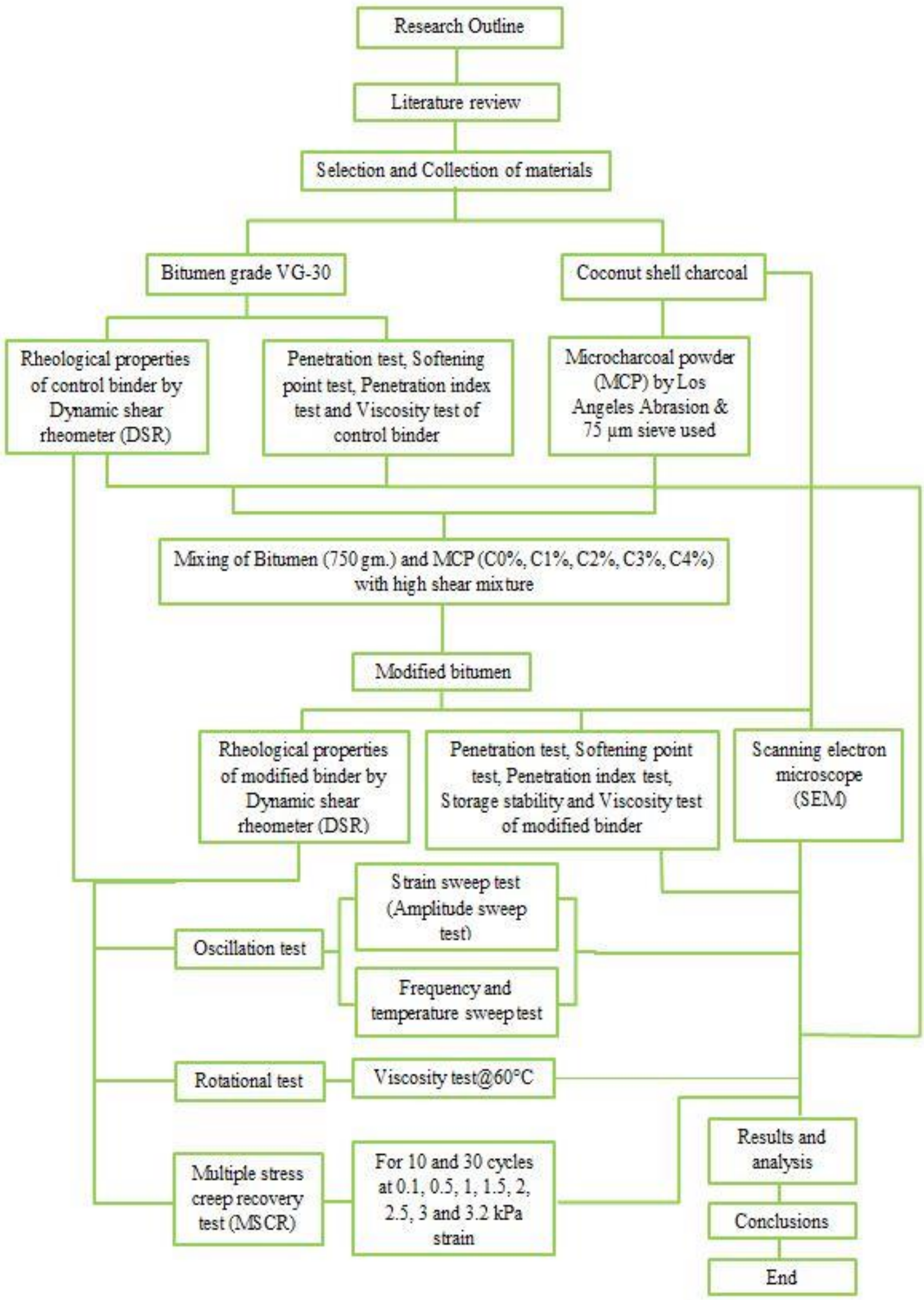


Figure 1.6 Research Outline

1.5 RESEARCH CONTRIBUTION

Evaluation of the neat and modified binder properties by dynamic shear rheometer (DSR) is the new technique. The major contributions of this research are as follow:

- Environmental waste i.e. coconut shell waste as micro particle was used in this study which helps to reduce the pollution in environment and also it is cost effective and reduces the cost of modification and also reduces the maintenance cost during service life of pavement.
- Basic properties of the C.B and M.B were investigated after addition of MCP and checked the feasibility of coconut charcoal as bitumen additive.
- From rheological properties the major parameters such as rutting performance, G* and δ were calculated at 65°C using DSR and analyzed the values because it was directly connected to the viscoelastic properties of binder, which helps to understand importance of modification. Reason behind chosen of 65°C was maximum temperature in India.
- MSCR test was conducted at 30 cycles also because 10 cycles was not simulating the actual loading conditions and this test was also conducted at different creep stress levels to see the stress sensitivity of binder at high temperature.
- Frequency and temperature sweep test was conducted to get the behavior of C.B and M.B with varies frequencies and temperatures because variation in frequency is related to loading time of vehicles which helps to identify the elastic behavior of bitumen.
- Statistical analysis was also conducted to check the repeatability in the trials and see the effect of independent variables on dependent variables.
- Scanning electron microscopy (SEM) helped to know the homogeneous dispersion of MCP into conventional binder.

1.6 THESIS ORGANIZATION

This report is divided into sixth chapters and is listed below:

- Chapter 1 introduces the problems related to flexible pavements and research objective and scope.
- Chapter 2 discussed the literature review on different research studies on rheological properties of binder and discussed regarding the gap in study.
- Chapter 3 is related with different materials and methods that were used to perform the testing.
- Chapter 4 investigates the physical and rheological properties of binder
- Chapter 5 conducts statistical analysis of MSCR test for 10 and 30 cycles.
- Chapter 6 summarizers the conclusions of the each research study and provide the scope for future research. At the end references are provided regarding this research.

CHAPTER-2

LITERATURE REVIEW

2.1. GENERAL

Literature review section introduces properties of coconut shell, coconut shell charcoal and other bio waste materials such as rice husk, palm oil fruit ash etc. in construction materials such as cement concrete and bitumen. As the application of coconut shell charcoal powder has seen limited studies as bitumen additive, the available literature in this area has been discussed. In addition, the physical and chemical properties of coconut shell charcoal were discussed to understand the characteristics, which have the potential to improve the rheological properties of bitumen.

2.2. EFFECTS OF BIO WASTE USED IN CONCRETE MODIFICATION

E.A. Olanipekun et al. (2005) compared cost analysis and compressive strength of concrete was evaluated with the help of crushed aggregates of coconut shell and palm kernel shell with replacement of coarse aggregates with 0%, 25%, 50%, 75% and 100%. Total 320 cubes were casted to check the strength and cost analysis. Physical and mechanical properties were analyzed. It can be seen that compressive strength of addition of coconut shell crushed aggregates is more as compared to palm kernel shell crushed aggregates but cubes strength decreased as percentage of coconut shell increased and cost of production of concrete from the coconut shell reduced by 30% and 42% with the addition of palm kernel shell.. Therefore, it can be concluded that production of concrete with help of coconut shell is economical and strength is also more as compared to palm kernel shell.

P.B Madakson et al. (2012) studied the inclusion of coconut shell ash in MMC as reinforcement for automobile parts similar to other materials such as fly ash and rice husk ash. From the result of particle size analysis, coconut shell ash has grain fineness number of 75.08 which means ash was finest and density of ash was 2.05 g/cm^3 indicating it to be light in weight. From X-ray fluorescence (XRF), it was found that the major components in ash are SiO_2, Al_2O_3, MgO and Fe_2O_3. The chemical composition of coconut shell indicated that it can be used as reinforcement in metal matrix composite (MMC). SEM showed ash particles are solid in nature and irregular in size, with particles being spherical in nature. Further, as the CSA withstands temperature up to 1500°C, it is good in thermal resistance and can be used in MMC component.

Pravin Khandve et al. (2014) replaced 10-20% of coarse aggregates was replaced with CS by doing replacement it decreases density of concrete. The compressive strength and split tensile strength of CS modified concrete was lower than the unmodified concrete. Also found that permeable voids and absorption were high for CS modified concrete than normal concrete. Further, crushed granite was replaced with coconut shell by 20%, 30%, 40%, 50% and 100%by volume to produce the concrete. Cubes were cast to find the compressive strength at 7, 14, 21 and 28 days. It can be seen that it reinforced the concrete construction by replacement of 18.5 % of crushed granite with CS and the use of CS save the environment and decrease the cost of construction. 3.) The PKS was used as lightweight concrete of grade 35. Concrete having 10% of silica fume, cementitious materials and 5% of fly ash from result found that with the increase of sand content affects the mechanical properties of the concrete i.e. increase the density of concrete and compressive strength.

Vignesh Kumar N. et al. (2014) states that CSA reduce the environmental waste and CO_2 while production of cement. The partial replacement of cement in concrete was 0% and 5% with CSA. CSA contains high quantity of lignin content which gives strength and makes the materials weather resistance. For making of activated carbon from coconut shell first of all clean and dry the shells of mature coconut then put these shells into muffle furnace at temperature of 600°C to 800°C. It showed that as increase of CSA in cement the initial and final setting also increases from 1 hour 5 minutes to 3 hour 26 minutes and 1 hour 26 minutes to 4 hour 22 minutes respectively. It also effects on compressive strength, it reduce with increase in content (30%) of CSA in concrete.

M.Nivedhitha et al. (2017) replaced the coconut shell and egg shell powder with cement in M30 grade of concrete at 0%, 2.5%, 5% and 15% ratio. The objective of this paper was to utilize the naturally occurring waste materials and reduce environmental pollution and also check the strength and stability of concrete with these materials replacement. 7 and 28 days were curing time after casting of samples. Then these samples were tested to find compressive strength, flexural strength and split tensile strength. Results showed that an average compressive strength was achieved after partial replacement of cement and we get the cheaper and light weight structure. Flexural strength also increased up to 15% addition of egg shell powder.

Dr.R.Umamaheswari et al. (2018) this study evaluates the strength of cement after modification with CSA and silica fume. Partial replacement of cement with CS was 5%, 10% and 15% and with silica fume was 5% and 10% by weight of cement at w/c ratio of 0.35. OPC of 43 grade and size of coconut shell ash was less than 75 µm. Compressive strength, flexural strength, tensile strength, durability test were conducted to check strength of modified concrete. From results it can be seen that compressive strength was increased by 30%, flexural strength by 52.5% and tensile strength by 60% at replacement of cement with 10% of ash and 10% of silica fume in concrete mix compared to conventional mix.

2.3. EFFECT OF BIO WASTE USED IN ASPHALT BINDER ON PHYSICAL AND RHEOLOGICAL PROPERTIES

Ramez A. Al-Mansob et al. (2013) used palm oil and coconut shells in asphalt mix with size of 4.75 mm with 0%, 5%, 10%, 15% and 20% total weight of 4.75 mm size aggregates to compare the mix performance of POS and CS with control mix through the resilient modulus and creep tests. From the indirect tensile strength test, it was found that the resilient modulus reduces with the increase in POS but in case CS the resilient modulus increased. However, for 5% and 10% addition of CS, resilient modulus was lower than that of POS addition at same percentage. In static creep test, static load was applied on specimen and deformation is measured after removal of load. For additives, first strain increases and then became steady. It was found that 5% addition of CS showed credible performance. Also in dynamic creep test 5% addition of CS has improved the mix. In both the cases more than 5% of POS and CS lower the creep resistance because of loss of cohesive and plastic properties. Overall CS showed good results as compared to POS.

Meizhu Chen et al. (2014) take vegetable oil waste in three different drags of aged binder and in each binder at different content of waste vegetable oil was added such as 0%, 3%, 4%, 5%, 6% and 7% then analysis the physical, chemical and rheological properties of binder. Results evaluates that waste oil improved penetration, softening point, ductility, penetration index, penetration ratio of the modified binders at optimum dosage. Some modified binders have similar effects as of control binder at different contents. Complex modulus, phase angle and rutting performance also improved of modified binders and make the binder fatigue resistance. It improves the creep response of binder. Addition of waste oil reduces the asphaltenes content of aged binders. In short it improved the both properties.

Jan Krol et al. (2015) showed the microstructure of PMB with the help of physical properties, MSCR test and fluorescent microscopy and also analysis its viscoelastic properties. From results it can be seen that according to MCSR test, half of the tests showed polymer content in modified bitumen because variation in structure of polymer at dispersed phase and its elastic recovery depends on the microstructure of PMB. With the help of fluorescent microscopy they were able to find optimum dosage of elastomer used for modification of asphalt binder.

Ramadhansyah Putra Jaya et al. (2015) used the CS and coconut fiber (CF) to construct road. CF helps to enhance strength, skid resistance and CS helps to improve tensile strength and creep strength of modified binder and also have capacity to resist weather action, impact and crushing as compared to crushed aggregates. CF reinforced the binder and helps to control the bleeding of asphalt pavement. In this review numerous properties were studied like shell content, size and shape, length of fibers. Different tests like resilient modulus creep test, skid resistance tests were analysis by using CS and CF. Results showed that both agricultural wastes improves the performance of road.

Aswathy A et al. (2016) Stone mastic asphalt was used first time in European countries. It is gap graded contains course and fine aggregates, stabilizers and bitumen. In this paper the samples contains stone dust and coconut shell charcoal as a filler, cellulose fibers for stabilizers and to check flow stability and air voids, Marshall Test was performed to compare the result. Results showed Marshall Stability value is 1037.4 kg when stone dust was used as a filler and 975.8 kg when coconut shell charcoal was used. The difference in stability was less than 3.61%. So coconut shell charcoal might be used as filler. The flow value is maximum when coconut shell charcoal was used as filler. The air voids started decreases maximum when stone dust was used and in case of coconut the decrease in air voids is steady.

Joelle De Visscher et al. (2016) MSCR test was done to do improvement in test procedure by the help of 7 different binders which includes paving grade binders and

PMB and 25 participants. The aim of this test is to simulate actual loading conditions and to reduce the rutting. The range of application of stress level is 0.1 to 3.2 kPa but in this study 6.4 kPa stress also applied to analysis the stress sensitivity of binders. At every binder 3 trials were done and check the repeatability and reproducibility of trials. During MSCR test soft binder show negative values of %R. Test was done on two temperatures i.e. 60°C and 70°C. Results showed that 60°C is reliable for binders but 70°C is too high for soft binders. Finding showed that 6.4 kPa stress level is less reliable to determination of J_{nr} and %R.

Tay Lay Ting et al. (2016) the production of coconuts is at large scale, more than 93 countries grown the coconut and from this coconut shell waste production as a solid waste is 3.18 million tonne annually. The ash from coconut shell waste and ash from rich husk have low density, cheaper as solid waste. As a waste it is new source of energy bio-fuel. Mature coconut shells has 33.61% cellulose, 36.51% lignin, 29.27% pentosans and 0.61% ash. Water absorption of CS is high as compared to usual aggregates. Coconut shell is also converted into charcoal or ash which is used as replacement of aggregates with charcoal and as additive into the binder to enhance the properties of the concrete and bitumen. Various tests are conducted to analysis structure of the coconut shell at micro and nano scale tests like SEM, XRD, BET, EDX, and FTIR. From Scanning electron microscopy (SEM) results discrete cells, micro pores and continuous cells sizes are measured which showed that coconut shell can resist heavy load and it has high crushing and abrasion resistance value. And result from coconut shell ash it was observed that structure of CSA is solid in nature and irregular in size. X-Ray Fluorescence (XRD) test was used to analysis elemental of materials, results showed that highest component of SiO2 was lower in coconut shell as compared to rice husk still CS is hard because of it contains large amount percentage of alumina, silicon oxide and iron oxide which makes the CS strongest material among the other two. X-Ray Diffractometer (XRD) test is used to evaluate the chemical composition; it uses the Cu Ka radiation at scan speed of 30/min. the results showed that CSA contains SiO2 component and other elements are c, Mg, O, Al, Fe. Result also showed that it did not contain radioactive materials like Dy, Xe, Pr and Eu. Fourier Transform Infra-Red (FTIR) is used to test its functional groups of the coconut shell ash.

Siti Nur Amiera Jeffry et al. (2017) In Malaysia 95% construction of roads are of asphalt concrete and mostly asphalt pavement fails in rutting and fatigue due to increase in overloading of traffic and weather conditions so to construct the strong structure we need to modify the asphalt binder. In this study for modification waste coconut shell was used to produce nano charcoal ash as bitumen additive. PEN 60/70 grade of bitumen was used and for nano charcoal ash production first of all burnt the coconut shell at 450°C for 5 min to prevent the excessive burning and to produce high content of carbon. To analysis the morphology and thermal properties of the CS and CSC microstructure test were performed i.e. X-ray fluorescence (XRF), Field emission scanning electron microscopy (FESEM), Particle size analysis (PSA) and Thermogravimetric and Derivative Thermogravimetric analysis (TGA/DTA) from temperature 28°C to 900°C. Also Penetration, softening point and dynamic shear rheometer tests were performed on control and modified binder. Results showed that 490°C is the optimum temperature to produce the charcoal. From the FESEM test result showed that the surface of CS was rough, irregular in shape and with elongated layers and for coconut shell charcoal the surface was fine, smooth but coconut shell charcoal showed more carbon content as compared to dry coconut shell from EDX test. From the particle size analysis test result showed that as the grinding time increasing, the particle size also increasing. Agglomeration might be the reason for this, when optimum time exceeded. Penetration test result indicates that if the penetration vale lies between 20 to 30 dmm, cracking might be occur due to high stiffness of binder due to cracking value should be more than 30 dmm. Rutting performance was evaluated by using DSR, it showed that modified binder have high value of rutting performance.

Mohd Ezree Abdullah et al. (2017) added waste plastic at different content into bitumen to analysis the physical and rheological properties of C.B and M.B. Plastic increase performance of C.B and also helps to decrease the waste and protect environment. Plastic content was 1.5%, 3%, 4.5%, and 6% by weight of bitumen and its size was 2 mm. High shear mixture was to mix plastic into bitumen. The speed to high shear mixture was 1200-1500 rpm for 1 hour at 170°C. Results showed that as the increase in plastic waste content penetration value decreases and softening point value increases of modified binder. Viscosity also increases as addition of plastic waste. At 135°C showed more viscosity as compared to 165°C which means rutting will occurs. Rutting performance parameter also improved by adding of plastic at high temperature. 6% of plastic waste was found the optimum dose in bitumen to modify.

Mohd Ezree A. et al. (2017) added the coconut shell charcoal (CSC) at different fineness content i.e. <75, 75-150 and 150-300 µm to enhance the physical properties of the bituen. The percentage added of CSC was 0, 10, 15and 20 by weight of bitumen. PEN 60/70 grade of bitumen was used. The mixing time of CSC was 60 min at 1500 rpm to ensure uniform distribution. Penetration, softening point and PI test were analysis. From results it was concluded that particle size less than 75 µm given good results as compared to other size charcoal particles. At 20 % of CSC to control binder effects significantly on penetration and softening point test. Penetration index value increase with increase of CSC and high penetration index value shows high resistance to low temperature and reduce the permanent deformation. So modified binder improves the performance of roads.

Raissa Romastarika et al. (2017) Waste black rice husk ash (BRHA) was used to modify the binder. To form the fine ash grinding ball mill was used after this ash was sieved through 75 µm sieve. Content of BRHA was 0%, 2%, 4% and 6% of total weight of bitumen (60/70 PEN). High shear mixture was used to blend BRHA at 800 rpm for time period of 60 min. Choose the time to ensure uniform distribution. Physical and rheological properties were analysis. Significant effects were seen on penetration and softening point test for both aged and unaged bitumen. The penetration value decrease and softening value increase. Retained penetration is the ratio of penetration of aged samples to penetration of unaged samples and result showed that hardness of samples decrease as increase in retained penetration. At 6% addition of BRHA is given the PI value of 0.25 which showed the high resistance to low temperature susceptibility. From rheological result rutting performance increased of modified binder at higher temperature. 2% was the optimum dose of BRHA.

Ali Behnood, Jan Olek (2017) MSCR test was done at various PMB to find out the stress dependent behaviour, J_{nr}, %R and its average was found. MSCR test was done on DSR with non-standards. Finding shows that PMB increase the rutting performance of binder and also increase the %R but they are depends on the type and amount of polymer. Second finding was that at high stress levels binder shows non linearity. The average %R is decreasing as the stress was increasing expect SBS modified binder. SBS modified binder first increase by increasing stress and number of cycles but then decrease in its %R. PPA modified binder and control binder reach faster in its steady state as compared to SBS modified binder. Because it is require more loading cycles to reach. Overall SBS modified binder is best among all to resist the distresses due to traffic loading.

H. Yaacob et al. (2017) CS is agricultural waste and that can be reduce by using as additive or replacement in asphalt concrete. Study concluded that CS was used as replacement in asphalt mixture. Quantity of CS was 0, 10, 20, 30 and 40% by weight volume. To measure performance of asphalt concrete resilience modulus, dynamic creep and cumulative strain with respect to time of loading tests were evaluated. The size of CS was 5 mm and grade of bitumen was 60/70 PEN. Results showed that 10-20 % of CS gives good improvement in conventional asphalt mix.

Ayad Subhy (2017) discussed the various tests and helps to increase the binder's fundamental and mechanical properties because these properties have direct effects on rutting and fatigue cracking. So modification is the solution to reduce the distresses due to overloading of traffic and extreme climate conditions and enhance the service life of flexible pavements. For analysis the distresses tests like DSR to measure response of the binder at different time and temperature after loading the asphalt binder which helps to relate fatigue and rutting distresses, ZSV, MSCR is the improvement of oscillation test's results because oscillation test is unable to response under higher strain level and that is possible for MSCR test, double edge notched tension, these tests helps to understand the viscoelastic properties of binder.

Monketh Mohammed et al. (2018) investigated the effect of cellulose and glass fibers on rheological properties of asphalt binder. Physical properties were analysed of modified binder and also done the DSR test and made the master curve. Results presented that fibers enhanced the physical properties and long glass fibers raise the viscosity, softening point value and reduce penetration which helps to reduce the permanent deformations. Complex modulus value increased at low frequencies and high temperature which helps to increase the stiffness of binder. Addition of glass fibers were decrease the δ at low and high frequencies and improve the stiffness. Rutting performance also increased and improves the toughness of binder.

Junfeng Gao et al. (2018) Bio-bitumen is the binder made from biomass and petroleum asphalt. In this study, bio-oil used and extracted from the sawdust having brown colour and show plasticity state at room temperature. The motive of this study was to assess the rheological properties of the bio-binder with help of (DSR). Test included G* and δ based on time and frequency sweep. RFTO test was done to simulate the condition of short term aging of the bitumen. 50 penetration grade control binder and SBS modified asphalt binder was used having 1% of SBS by weight of control binder. 5%, 10%, 15%, and 20% of bio-oil was used by weight of SBS modified binder. Mixing of SBS and control binder was done with high shear mixture of 3000rpm at 180°C for 15min and bio-oil with modified SBS binder mixed at 3000rpm for 20 min at 140°C. The results showed that after heating of bio-oil at 163°C alcohol, phenol, ether and other substances were decreased and a firm amount of acetal and ketal substances were produced so these changes affects the properties of the bitumen. The G* increases which are desirable for rutting prevention asphalt mixture at same frequency condition after RFTO. The viscosity of bitumen with 15% bio-oil was weaker than other asphalt after RFTO. With increase in percentage of bio-oil loss modulus and complex modulus increases which showed that anti deformation capacity of asphalt after RFTO. From the master curve, it showed that G*/sinδ of bio-asphalt increases with rise in frequency range before and after RFTO. Rutting factor increased after adding of bio-oil into SBS modified binder at each loading frequency.

Du Yinfei et al. (2018) Permanent deformation due to rutting is the major distress in bituminous road. To make pavement distresses free we need to modify the conventional binder or strengthen the asphalt mixture by providing good interlocking which makes the road safe and give long life. For that cool asphalt pavement, hot mix and warm mix pavement was reviewed and analysis the rutting performance of binder by MSCR test was done and for asphalt pavement wheel tracking test was done. For modification of bitumen different materials are used like nanoclay, RAP, steel slag, crumb rubber, SBS. Results showed that to prevent the rutting in warm mix asphalt (WMA) Asphamin and Evotherm are used as additive. Cold mix asphalt is better than WMA because rutting in CMA is 0.9 mm with 4.4% of cement as compared to emulsified bitumen. G*/sinδ parameter is used to investigate the rutting performance of binder but MSCR test is better to analysis the rutting parameter from DSR.

Rui Xiong et al. (2018) replaced mineral filler in asphalt mixture with activated coal gangue (ACG) and the properties of ACG are compared with limestone mineral filler (LMF). Coal gangue is produced by processing of coal mining and washing. To reduce solid waste and decrease pollution, it is used as filler. Bitumen used in this research is VG-10. The filler and binder ratio is 1:1. Both type fillers are used which was passed through 0.075mm sieve and then added with different % into binder to make the asphalt mastic. Cone penetration test was done to check the shear strength of asphalt mastic. It shows that ACG has improved the shear strength of binder than LMF and improve the viscosity and stiffness of bitumen. DSR was conducted to find out rutting resistance of prepared sample at 30°C, 40°C, 50°C, 60°C, 70°C and under strain sweep test, G^* and δ were obtained at different temperature. By doing this test was concluded that by adding ACG, it increased the rutting performance than LMF under load frequency range. Brookfield viscosity test (BVT) used to investigate the mixing and compaction temperature of asphalt at 135°C, 150°C, 165°C, and 180°C. at high temperature ACG has better effect on sensitivity property of binder than LMF. Then dynamic mechanical thermal analysis was done to check response of the material under alternating stress or strain. The relationship between (modulus, internal friction), temperature and frequency can be achieved. At high temperature the G′ decreases gradually then finally mild. First loss modulus (G″) increases then decreases. So ACG is used in cold weather area, at low temperature performance of mixture take care.

Siti Nur A.J. et al (2018) used the nano coconut shell ash (NCA) modified bitumen in bituminous mixture to evaluate mechanical properties. To modification bitumen 0%, 1.5%, 6%, and 7.5% by weight of bitumen, NCA was used. The size of the particle is 57.5 nm. Different tests were performed to analysis the asphalt mixture like Marshall Stability flow test, ITS, resilient modulus, dynamic creep test, AFM and FESEM. Finding shows that NCA improve the properties of mixture compared to C.B. Optimum content of NCA was 6%. High tensile strength achieved was 1288 kPa. AFM showed good adhesion properties between binder and aggregates and FESEM indicates the flat and dense mixture.

Zhanyong Yao et al. (2018) used the waste tyres and plastic film to modification and enhancing the properties of bitumen. Physical and rheological properties were determined at low and high temperature. Results showed that plastic waste is dominant for improvement of bitumen stiffness and when they used both, the softening point was similar to SBS modified binder. FM showed the continuous phase of dispersed polymers when adding the waste plastic after adding the rubber waste they formed the twisted phase. G* increases and δ decrease by adding wastes and increases the elastic behavior. MSCR test revels that creep strain decreases and increase the rutting resistance performance. BBR test at low temperature decrease the stiffness and increase the m-value with the help of waste crumb rubber.

R N A Raja Zulkefli et al. (2018) POFA was added in asphalt binder and finds the physical and rheological properties of the M.B POFA mixed at different content i.e. 0%, 5%, and 7% and with different grinding time i.e. 1 and 4 hour. The size of particle was 0.075 mm passing. If we grind for 1 hour we get 3 to 7 µm particle and for 4 hour grinding we get 500 nm to 3 µm size particle. Binder having 80/100 penetration was used and 400 gm of bitumen was taken to mix. Mix the POFA into bitumen at temperature 160°C for 60 min and at speed of 800 rpm. After preparation modified binder test the physical properties of M.B. Results showed storage stability of M.B is good. The penetration value decreased for both unaged and aged binder. But optimum result obtained at 7% addition of POFA and 4 hour grinding time. Same result showed softening point test. Result from penetration index showed that modified bitumen was less temperature susceptible.

Siti Nur Amiera Jeffry et al. (2018) In Malaysia country where rutting and fatigue are frequently occurring distress in pavement which decreases serviceability, efficiency, and safety of asphalt pavement. The study focused on nano-sized particles (1-100nm). For this coconut shell ash powder having size 57.7nm was used as additive in bitumen (PEN 60/70) with different proportions i.e. 1.5%, 3%, 4.5%, 6%, and 7.5%. Penetration point, softening point, viscosity test, PI, storage stability test, ductility test and DSR tests were completed to analysis and effects of nano charcoal ash powder on physical and rheological properties of bitumen. RTFO and PAV tests were ended to simulate the aging properties of the bitumen and 6% is optimum content which retard aging of bitumen. The test results showed the modified bitumen has improved penetration compared to control bitumen. 6% is the optimum nano charcoal coconut shell ash which displays a higher percentage of improvement for RTFO aged sample than unaged sample. Softening point increased as the content of nano charcoal ash (NCA) increased, for that 6% is optimum content. NCA showed the higher rutting performance and improvement for the unaged binder at 6% content. This percentage resists the rutting till 70°C and failure at 76°C. Thus addition of nano charcoal shell ash improves the performance of the binder in rutting and fatigue cracking.

Yong Lei et al. (2018) is used the CRM asphalt with and without bio-oil. The size of crumb rubber is 20 mesh (841µ) and 80 mesh (177µ) was used and MSCR, temperature sweep and rotational viscosity test was conducted. Results showed that without bio-oil with CRM asphalt, viscosity is more when 20 mesh crumb rubber was used as compare to 80 mesh asphalt binder. At 10% addition of bio-oil, composite modified binder gives maximum viscosity. The size of crumb rubber also affects performance of bitumen at high temperature. Larger size of particle exhibit more elastic properties. Composite modified binder also resists the fatigue cracking. Short term aging increase the mixing process with bio-oil and increase elastic property. From MSCR result %R increase with 20 mesh size particle of crumb rubber than 80 mesh particle. In this study adding of bio-oil has not much significant effect.

Yu Chen et al. (2018) in this study, ten combinations of asphalt binder and crumb rubber performances were tested by DSR and MSCR. Crumb rubber usage in bio-asphalt resists rutting at high temperature and at low temperature cracking. Materials used in research were bio-oil extracted from wood-chips, the color of bio-oil has dark drown, control binder having penetration 60-80 and crumb rubber. For mixing of bio-oil and binder high shear mixture was used having 5000 rpm for 10min at 140°C and for crumb rubber modified bio-asphalt heat the control binder at 180°C and then add the crumb rubber after manual mixing for some time high shear mixture was used for 1hour having 5000rpm at 180°C. For fully swell of crumb rubber let the crumb rubber modifier binder in over for 1hour at 180°C then add bio-oil. From the result it was concluded that bio-oil first soften the asphalt binder before RFTO and gives lower the G* and higher δ after RFTO it gives opposite results. Adding of crumb rubber exhibited the %R than the bio-oil C.B and M.B. Rutting performance from DSR and MSCR test have same ranking for unrecovered creep compliance.

T.L. Ting et al. (2018) in double layer porous asphalt (DLPA) pavement CS and coconut fibers were used. DLPA is combination of noise decline properties of mix at top layer (20 mm) of pavement and drainage of water at bottom layer (50 mm) pavement. Size of coconut shell is 5 mm was used as aggregate at 5%, 10% and 15%

by weight of bitumen and coconut fibers were added into the bitumen at 0.3% and 0.5% by weight. Before adding both the materials these were treated with 5% weight of NaOH for 1 hr. at room temperature to reduce water absorption. The objective of research was to evaluate resilient modulus of unaged and aged asphalt mix. From resilience modulus pavement mix response can be find while traffic loading by ITS test. The results showed that for unaged bitumen 0.3% CF and 10 CS has highest resilience but if we compare both replacement of coconut shell and additives of coconut fibers, the coconut shell was affected most on resilience. For long term aging of asphalt mix results showed that 10% coconut shell replacement has highest resilience modulus but after aging resilience modulus for coconut fibers decreasing.

A H Norhidayah et al (2019) coconut shell (CS) was added as course aggregates with size of 5 mm and coconut fibers (CF) were added in pours asphalt mixture. Before addition, sock the materials into sodium hydroxide (NaOH). The quantity of CS was 0%, 5%, 10% and 15% and CF quantity was 0%, 0.3% and 0.5%. Permeability test was carried to evaluate the permeability of bituminous mix. This test was conducted at 25°C and specimen sealed all the sides then permeability coefficient was analysis. The results showed that 5% replacement of CS was the optimum content for permeability of water and addition of CF reduce the permeability of water as increase of content, this might be of air voids decreases.

Y Haryati et al. (2019) Stability and rutting performance were examined by using coconut shell (CS) and coconut fibers (CF). Before use of CS and CF soak the materials into sodium hydroxide for 1 hour to reduce the water absorption. The specific gravity of CS was 1.45 g/cm^3. Conduct the tests related to aggregates like impact value, flakiness and elongation index test. Results showed that stability was more of samples treated with sodium hydroxide as compared to untreated samples. Stability increases as content of CS increases but at certain extend. At 15% content marshal stability was less as compared to 10% CS content. CF also helped to increase the stability and gives very good results when we add 0.3% CF. To analysis the rutting performance of asphalt concrete four samples were made. Out of four, three samples have combination. The rutting depth with combination 10% CS and 0.3% of CF gives less rutting depth as compared to other combination. Asphalt having 15% of CS and 0.5% of CF also gives less rutting depth but as increase in content in asphalt mix, it decrease the interconnection between aggregates.

2.4. OBSERVATION FROM LITERATURE REVIEW

The literature review indicated that modification of bitumen is one of the major means of enhancing the rheological properties of bitumen to suit different traffic and environmental conditions. The modification of bitumen can be achieved in various ways with the addition of polymers, fillers, nanomaterials, etc. such that the modifiers should be compatible with the bitumen. The modifiers used are expensive and usually results in high cost of materials. The recent trend in asphalt research is to incorporate bio wastes in the bitumen to bring about changes in the rheological properties. In line with current research trends in the asphalt, in this research, coconut shell charcoal was used in modifying bitumen. Coconut shell waste has been extensively used in the cement concrete as a replacement for aggregates and its benefits have been well studied.

2.5 GAP IN STUDY

Coconut shell charcoal due to its fine nature has the potential to be one of the air polluting agents. Due to its high carbon content and skeletal microstructure, it can be one of the modifiers in bitumen to enhance the rheological properties. However, such studies, which prove the effectiveness of coconut shell charcoal on bitumen, are largely discounted and require a thorough experimental and statistical study, which forms the main objective of this research study.

CHAPTER-3

MATERIALS AND METHODS

3.1. MATERIALS

3.1.1. Bitumen

The bitumen used in this study included viscosity grade 30 (VG30), whose physical properties are given in IS: 73-2013 and recommended in IRC: 37 for use in dense bituminous macadam (DBM) layer. The binder was procured from construction of flexible pavement site near Barnala city, Punjab.

3.1.2. Charcoal

The charcoal in this study was obtained from burning the coconut shell.

3.1.3. Preparation of Microcharcoal Powder (MCP):

The coconut shell charcoal as shown in Figure 3.1 was taken and crushed into Los-Angeles abrasion machine to generate the finer size particles. Crushed charcoal material was sieved through 75 µm sieve to obtain the micro size particles and weighted at different contents for modification of conventional binder as shown in Figure 3.2. The CS microcharcoal powder (MCP) added at various percentage such as 0%, 1%, 2%, 3% and 4% by weight of bitumen. CS charcoal modified binder was named as C0%, C1%, C2%, C3%, C4%. This 1% gap was taken to check the sensitivity of charcoal powder content on neat bitumen. The coconut shell charcoal was procured from new Punjab foundry store, Ludhiana.

Figure 3.1 Coconut Shell Charcoal

Figure 3.2 Coconut Shell Micro Charcoal Powder

3.2. METHODS

3.2.1. Physical Properties of Bitumen

3.2.1.1. Mixing of MCP with Bitumen

The blending of micro charcoal powder (MCP) at various percentages such as C0%, C1%, C2%, C3%, C4% with by weight control binder (750 gm.) was done with high shear mixture as shown in Figure 3.3. Mixing of MCP with bitumen was done at 1750 rpm at constant temperature of 150°C for 60 min to get the modified bitumen. The full setup of blending machine also known as high shear mixture, bitumen sample and heater are as shown in Figure 3.4. The mixing time, temperature and a speed was chosen for uniform and homogeneous dispersion of coconut shell charcoal powder in the control binder (CB). Total number of samples was five which included four blended samples i.e. modified bitumen are shown Figure 3.5 and one was control bitumen sample. The maximum speed of shear mixture is 2640 rpm.

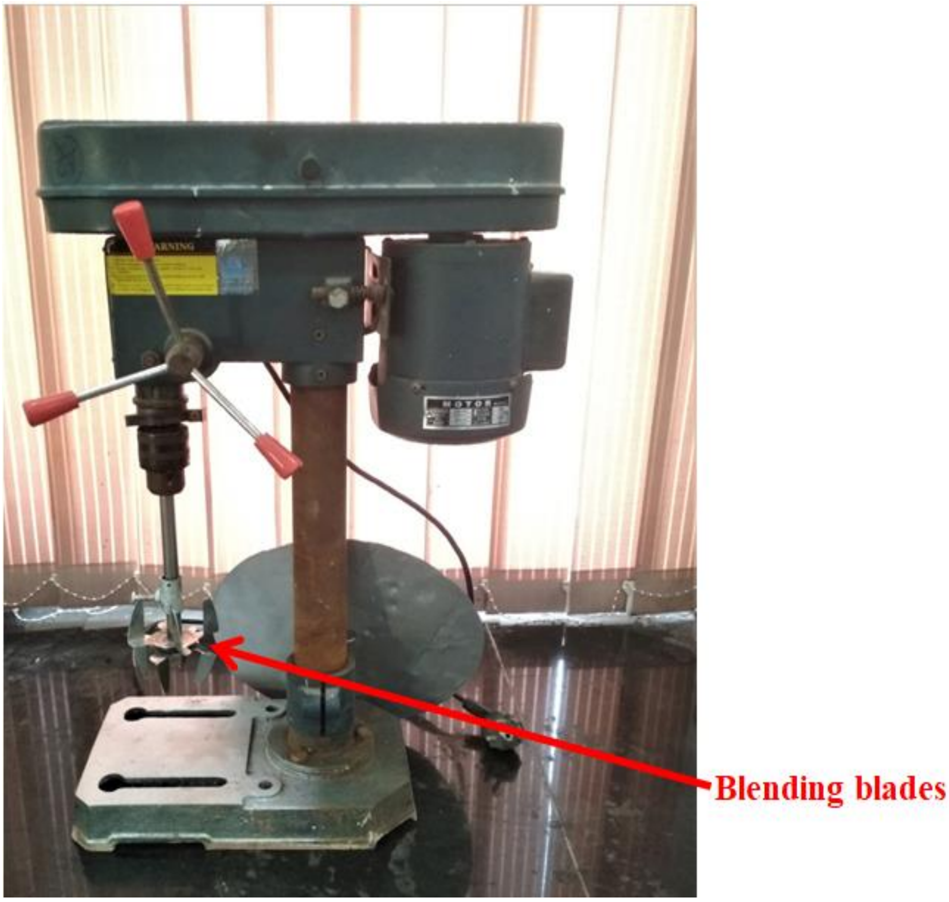


Figure 3.3 High Shear Mixture

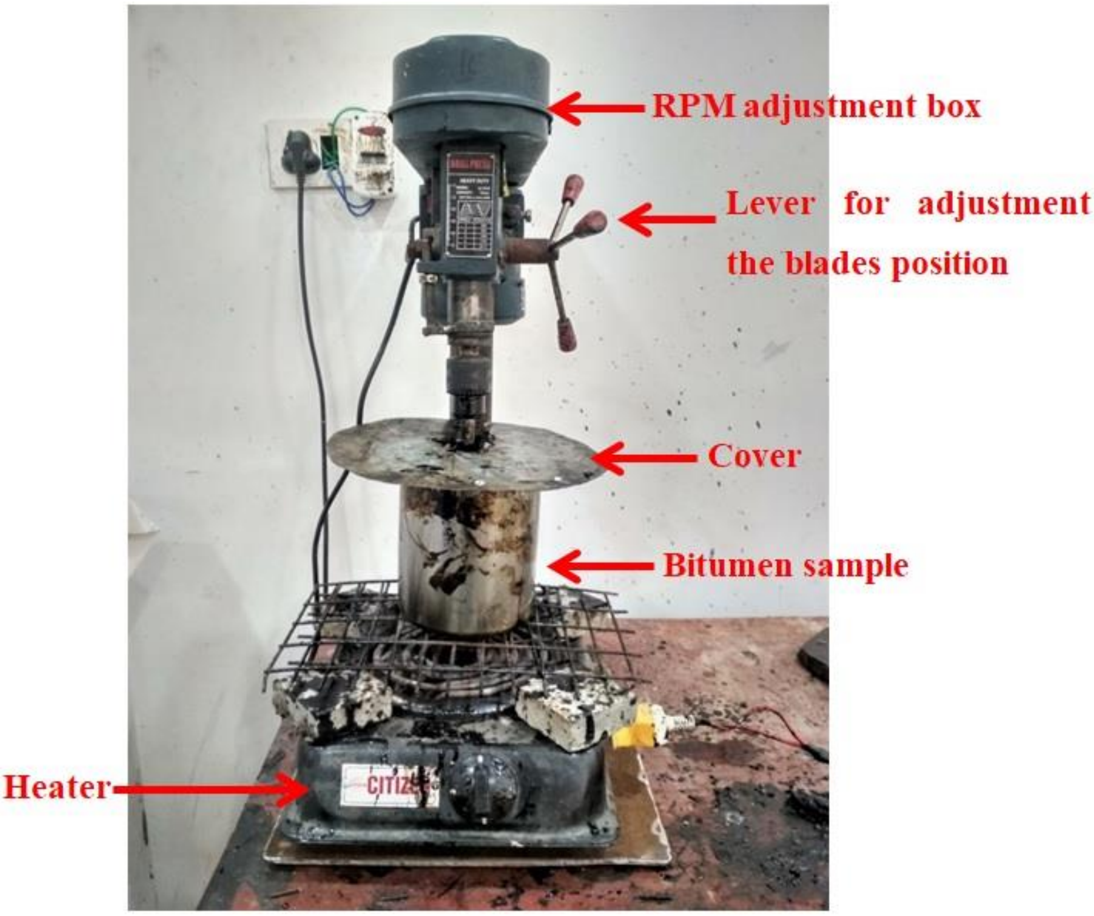


Figure 3.4 Setup for Blending

Figure 3.5 Modified Samples

3.2.1.2. Determination of Penetration Test

Penetration test is the simplest and indirect test to find out the constancy of bitumen. It is also used for grading the binder. The test was conducted according to IS-1203. Penetrometer is the instrument which is used to find the penetration values as shown in Figure 3.6. To find out the value first of all heat the binder about 75°C to 100°C and stirred well to make it homogenous and free from air bubbles and also from water then pour the binder into container having depth 35cm and diameter is 55 mm then cool down in atmosphere about 90 min. After cool down, sample is placed in water bath and maintained at 25°C for a period of 90min. At end penetrometer is used with 100g total load for 5 sec at 25°C to find out the value of penetration value. The depth of penetration is recorded in $1/10^{th}$ of millimeter. While performing the test, temperature should not more than 25°C and weight of load should be 100 grams. High penetration value means binder is soft, this type of binder is used in cold regions and low value indicates hardness of binder, used in hot regions. This test is not performed for cutback or tar bitumen because these are soft binders for this orifice viscometer is used.

In this research, at each samples (control and modified bitumen) three trials were done and in each trial minimum three values were recorded at 10 mm apart. The difference between initial and final reading is the penetration value. The mean value of penetration reading should not differ than the value specified below:

Table 3.1 Range and Maximum Difference of Penetration Value

Range of penetration value	**Maximum difference**
0-49	2
50-149	4
150-249	6

The limitation of this test is that it is empirical test and no fundamental relation to bitumen. Test is conducted at 25°C but in actual service temperature on pavement is about 60°C or more depends on the location. To care of these limitations we shifted from penetration grade to viscosity and performance grade.

Figure 3.6 Penetrometer Instrument with Sample

3.2.1.3. Determination of softening point

The softening point is an important test to determine the temperature at which bitumen changes phase from solid to flow-able phase. The test was conducted as per IS: 1205 using brass ring and ball apparatus. Bitumen was heated between 100°C and 110^{o}C or above until it is completely fluid and poured into rings and allowed to cool down for 30 min in room temperature as shown in Figure 3.7. These rings are put on glass plate which is coated with glycerine so that base to rinds does not stick. The excess bitumen is trimmed with hot knife and placed on the metallic support. After that trimmed sample were dipped in the water bath at 5°C for 15 min and two steel balls having weight 2.5 gm. with centering guide placed on each ring in the liquid and heated. The temperature at which bitumen touches base plate placed at distance of 25 mm below rings was recorded as softening point shown in Figure 3.7. Harder the bitumen grade higher will be softening point and vice versa. The material having softening point more than 80°C, in this case glycerine is used instead of water as heating medium and staring temperature will be 35°C instead of 5°C.

In this test, at each samples (control and modified bitumen) three trials were done. Softening point gives an idea about the temperature at which it gains certain viscosity.

The mean value of softening reading should not differ than individual observations by more than the value specified below:

Table 3.2 Repeatability and Reproducibility Value of Softening Point

Softening point (°C)	Repeatability (°C)	Reproducibility (°C)
40 to 60	1.0	5.5
61 to 80	1.5	5.5
81 to 100	2.0	5.5

Figure 3.7 Final Result of Softening Point Test

3.2.1.4. Determination of penetration index (PI) or Temperature Susceptibility:

The penetration Index (PI) value for conventional and modified binder was calculated from Equation 3.1. It gives penetration and softening point values relation. PI indicates susceptibility to temperature of binder. The suitable value of PI for construction of road is +1.0 to -1.0. It means if the value of PI is approaching to negative which is -1, it means the bitumen is highly susceptible to low temperature and asphalt binder leads to cracking or permanent deformation. On the other side if the value of PI approaching to positive value which is +1.0 it means binder is low susceptibility at high temperature and can resist the rutting cracking or permanent deformation.

$$PI = \frac{(1951.4 - 500 log P - 20 S.P)}{(50 log P - S.P - 120.14)} \qquad (3.1)$$

Where

P = Penetration Value

S.P = Softening Point Value

3.2.1.5. Determination of Storage stability using separation test

The addition of modifiers in bitumen tends to settle at the bottom when stored for long duration. In addition, large differences in densities between bitumen and modifier may lead to issues in stability for storage. To determine the storage stability of M.B with coconut charcoal powder, storage stability test was performed as per IS-15642 and ASTM D5976. About 50 to 55 grams of modified bitumen was poured into the steel pipes having 1 mm thickness, diameter of pipe was 25.4 mm (1 inch) and height was 150 mm. The sealed steel pipes with modified bitumen were conditioned in vertical position in oven for 24 hours at 163°C as shown in Figure 3.8. Following this, leave heated modified bitumen tubes were placed in straight position in deep freezer for 4 hrs. to solidify as shown Figure 3.9. The steel pipes with frozen modified bitumen put on flat surface and were cut into three identical parts. Neglect middle portion of tube and softening points of top and bottom portion was tested to determine softening point temperature as shown in Figure 3.10. Difference in softening point value of top and bottom should be less than 2.2°C.

Figure 3.8 Prepared Sample for Storage Stability Test

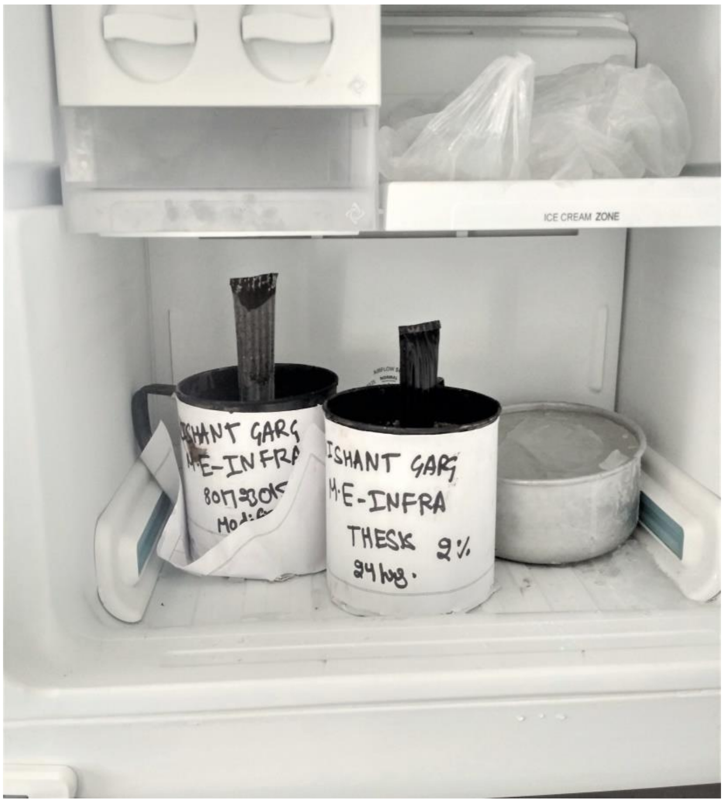


Figure 3.9 Frozen Samples for Storage Stability Test

Figure 3.10 Softening Point Test Samples

3.2.2. Rheological properties of bitumen using Dynamic Shear Rheometer (DSR)

3.2.2.1 Rheology

Rheolog**y** is the science which deals with the material's deformation and flow behaviour. It depends on the three factors such as inner structure of material (arrangement of molecules), stressing the outside forces and ambient conditions (temperature) of the stressed material. We have different types of materials like highly viscous materials. These are ideally viscous or Newtonian liquids, at constant ambient temperature ideally viscous liquids always shows constant viscosity such as water no matters how the materials are stressed. Materials which have elastic behaviour have some stiffness and shows complex flow behavior such as rubber.

Hooke's law is also applied on the viscoelastic materials with help of two plate model. Solid is firmly tight between the two parallel plates so that it cannot slip or slide. Now move the upper plate as the plate moves solid feels certain amount of force parallel to surface as shown in Figure 3.11.This force is known as shear force. This concept will help us to understand the strength of viscoelastic materials.

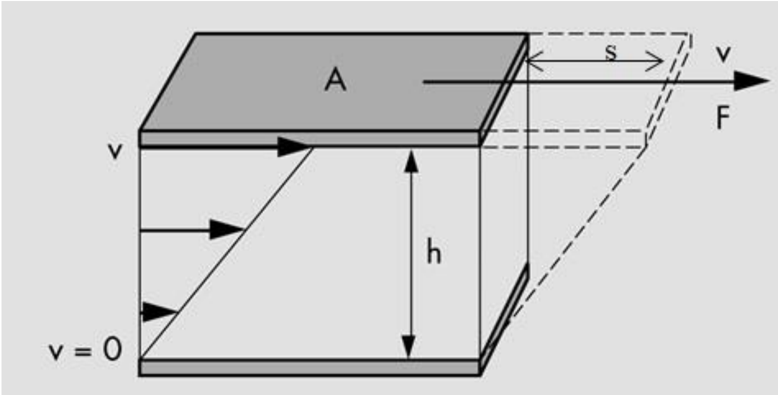


Figure 3.11 Two Plate Model

From this model an Equation (3.2) is defined which measure the force stressing the solid. Unit of this equation is N/m^2= Pa

$$\tau = \frac{F}{A} \qquad (3.2)$$

Where

τ = shear stress

F= shear force

A= area of upper plate

The 2nd parameter is shear deformation or shear strain (ϒ) as shown in Equation (3.3). It is in percentage.

$$\Upsilon = \frac{s}{h} \qquad (3.3)$$

Where

ϒ= Shear deformation

s= deflection path

h= distance between plates

3.2.2.2 Basics of rheometry on Oscillation test

To analysis the rheology we perform the various tests, one of them is oscillation test with the help of DSR as shown in Figure 3.12. Oscillation is the harmonic, periodic and sinusoidal motion as shown in Figure 3.13, which follows the patterns of sine waves. An example of oscillation is musical tone, human voice, tidal streams, swing of pendulum and alternating electric current. In this test sample is shears by oscillation between the two parallel plates without destroying the inner structure of the sample within linear viscoelastic range (LVE).

Figure 3.12 Dynamic Shear Rheometer (DSR)

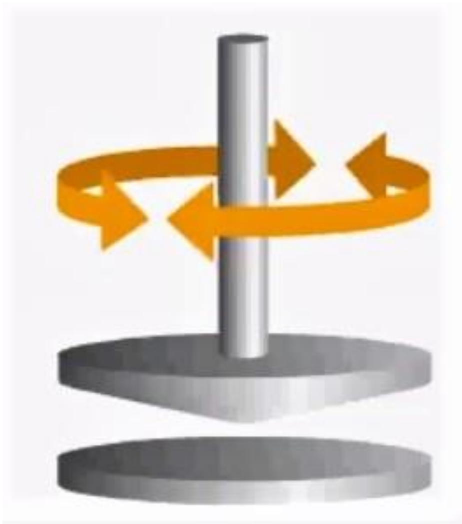


Figure 3.13 Movement of Oscillation Test in DSR

Two types of tests are performed by using oscillation (a) Strain sweep amplitude test and (b) Frequency sweep test.

3.2.2.3 Strain Sweep Amplitude Test

The bitumen being viscoelastic in nature has a characteristic region where applied oscillating shear stress is proportional to shear strain rate, which is referred to as linear viscoelastic region. To determine the LVE region of bitumen without destroying inner structure of materials, strain sweep test was conducted at different shear strain levels of 2,4,6,8,10,12,14 and 16% called as strain amplitude sweep test. To find out the particular strain value that which bitumen is in LVE range. In this test only strain will vary and frequency will be constant as demonstrated in Figure 3.14. The results of amplitude test is depends on the frequency chosen. The units of amplitude (ω) and frequency (f) are rad/s and Hz respectively.

To start the test, first of all heat the bitumen until it is completely fluid and poured into silicone mould as shown in Figure 3.15. In the meantime start the DSR and attach the accessories such as parallel plate geometry as shown in Figure 3.16. 8mm parallel plate geometry is used for aged binder in RTFOT or PAV and thickness between 8 mm parallel plate geometry is 2 mm. Next step is set the required temperature. After reaching the temperature place the bitumen sample from silicon mould to between the parallel plates then wait for 10 minutes so that bitumen sample also reach at that temperature as shown in Figure 3.17, extra bitumen is trimmed with the help of spatula. Then set the measurement values related to test and start the analysis.

The linear viscoelastic (LVE) range was determined such that the reduction in complex shear modulus (G*) shall not be less than 0.95 times the G* corresponding to 2% strain as show in Figure 3.18. Further, the oscillation test was carried out to determine the complex shear modulus (G*), phase angle (δ), rutting parameter (|G*|/sinδ) and other parameters such as loss modulus (G″), storage modulus (G′), and damping factor (tanδ) of bitumen within LVE range. For control and each modified bitumen samples three trials were done to check repeatability. The test was conducted as per AASHTO T-315 on a 25 mm diameter sample and 1 mm thickness at a temperature of 65^{o}C; representing pavement surface temperature during peak summer and loading frequency was 1.57 Hz (1.57 cycles per sec) and 4% strain was found to ensure the measurement in linear viscoelastic region is known as limiting value (Υ_L).

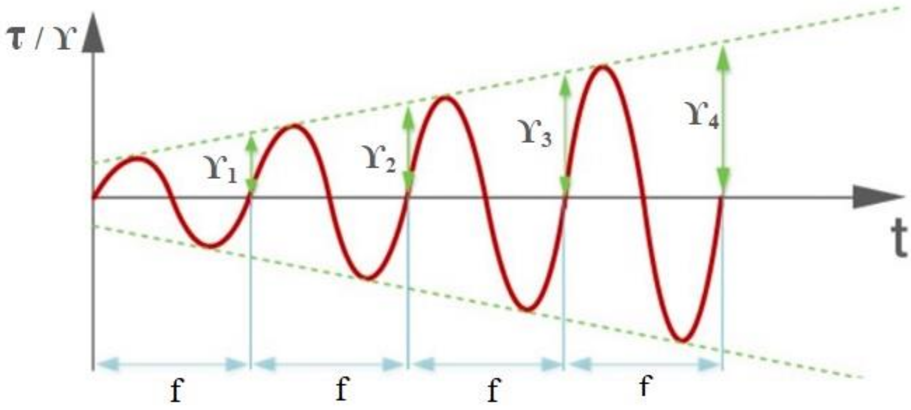


Figure 3.14 Variations of Strains with Constant Frequency

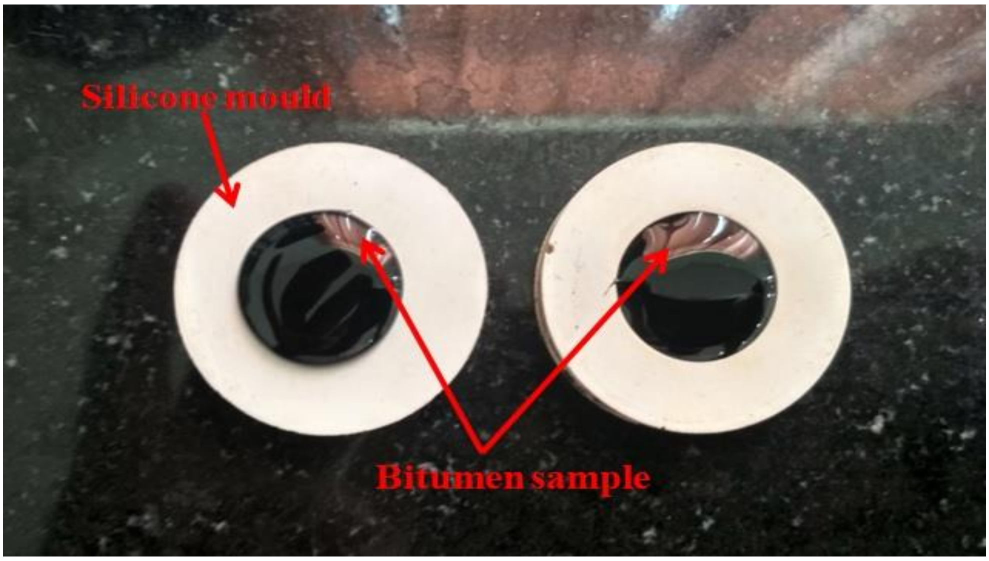


Figure 3.15 Bitumen Samples on Silicone Mould

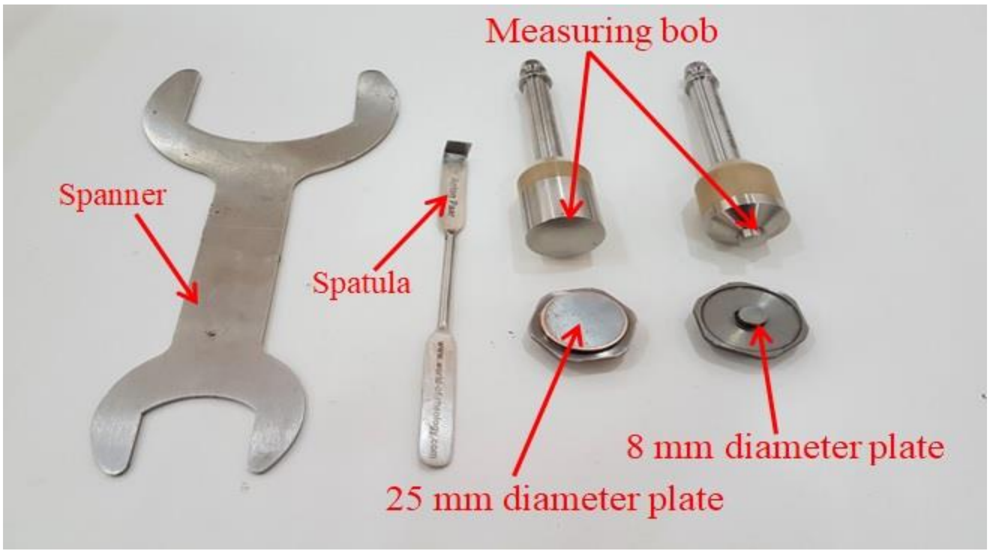


Figure 3.16 Accessories of DSR

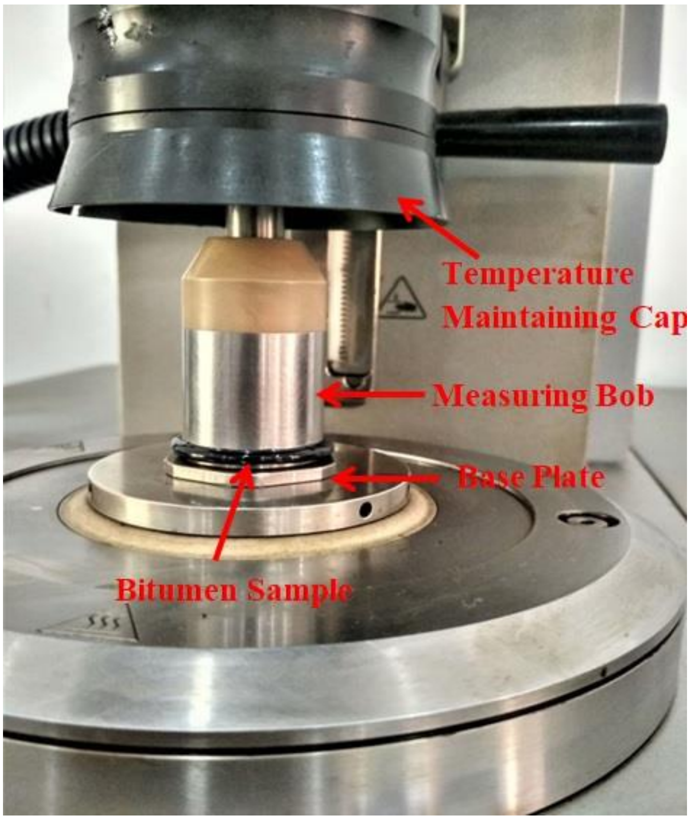


Figure 3.17 Placement of Bitumen Sample between Parallel Plates

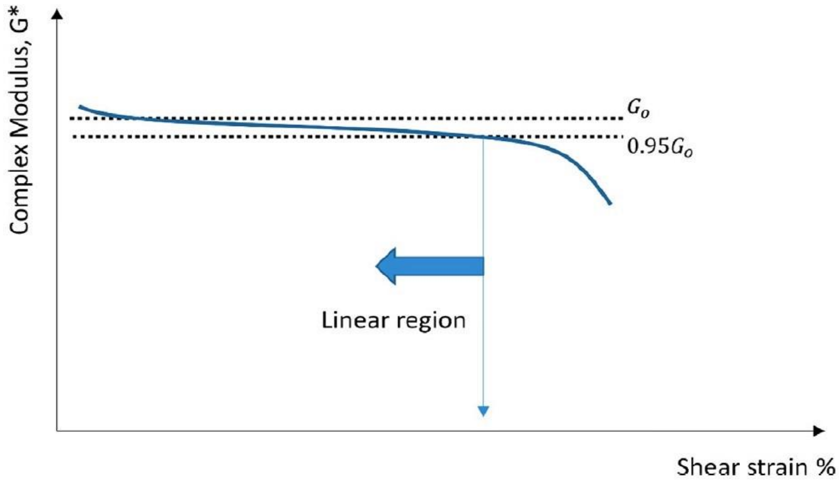


Figure 3.18 Strain Sweep to Find Linear Range

From the oscillation test following parameters are derived:

(a) Complex Shear Modulus (G)*

It is ratio of shear stress (τ) to shear deformation (ϒ) is known as shear modulus (G). Shear modulus measure the materials stiffness and strength. The unit of shear modulus is kPa. Variation in time and temperature affects shear modulus. To analysis shear modulus measurements there is test called as oscillatory test which is performed on DSR and results gives the complex shear modulus (G*). The ratio of stress amplitude to strain amplitude gives the Complex shear modulus. Complex term is added because oscillation is harmonic periodic function in sinusoidal process. Range of G* is mentioned below:

When 0.1 MPa < G*< 30 MPa then use 8 mm with 2 mm gap parallel plate geometry

When 1 kPa < G*< 100 kPa then use 25 mm with 1 mm gap parallel plate geometry

When the value of G*< 1 kPa then 50 mm parallel plate geometry

Complex shear modulus gives the two more parameters known as G prime (G′) and G double prime (G″). G′ is storage modulus (measure elastic behaviour) and G″ is loss modulus (measure viscous behaviour).

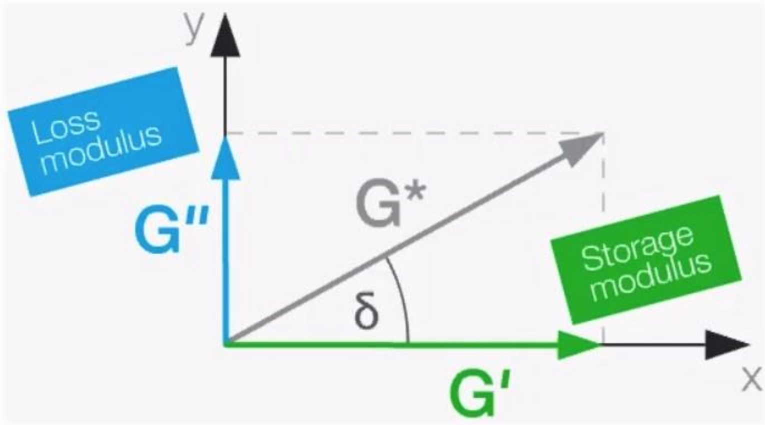


Figure 3.19 Analysis of Complex Shear Modulus

From the Figure 3.20 following equations are developed:

$G^* = G' + i\,G''$

$G' = |G^*|\cos\delta$

$G'' = |G^*|\sin\delta$

(b) Loss or Damping Factor (tan δ)

To express the G′ and G″, we can use a single name called as loss or damping factor (tan δ). It is the ratio of G double prime (G″) to G prime (G′) as shown in Equation 3.4. If the material is half viscous and half elastic then loss modulus (G″) equal to storage modulus (G′) and its value is tan δ is one.

$$\tan\delta = \frac{G''}{G'} \qquad (3.4)$$

If the value of damping factor is less than one it means material is in elastic state and if value is more than one, material is in viscous state. When G″ is equal to G′, this point is known as sol-gel transition point. At this state materials passes from one state to another that is call as phase transition.

(c) Phase Shift Angle (δ)

Phase shift angle (δ) is the time lag between the applied shear strain and resultant shear stress of asphalt binder. It characterizes the viscous and elastic behaviour of binder at medium and high temperature. Figure 3.20 shows the measurement of full period of oscillation and follows the sine curve. The amplitude of sine curve shows that sample is strained maximum and is known as amplitude of shear strain or strain amplitude (Υ_A). Carves shows the water is purely viscos in nature. Ideally viscos liquid shows 90° phase shift angle.

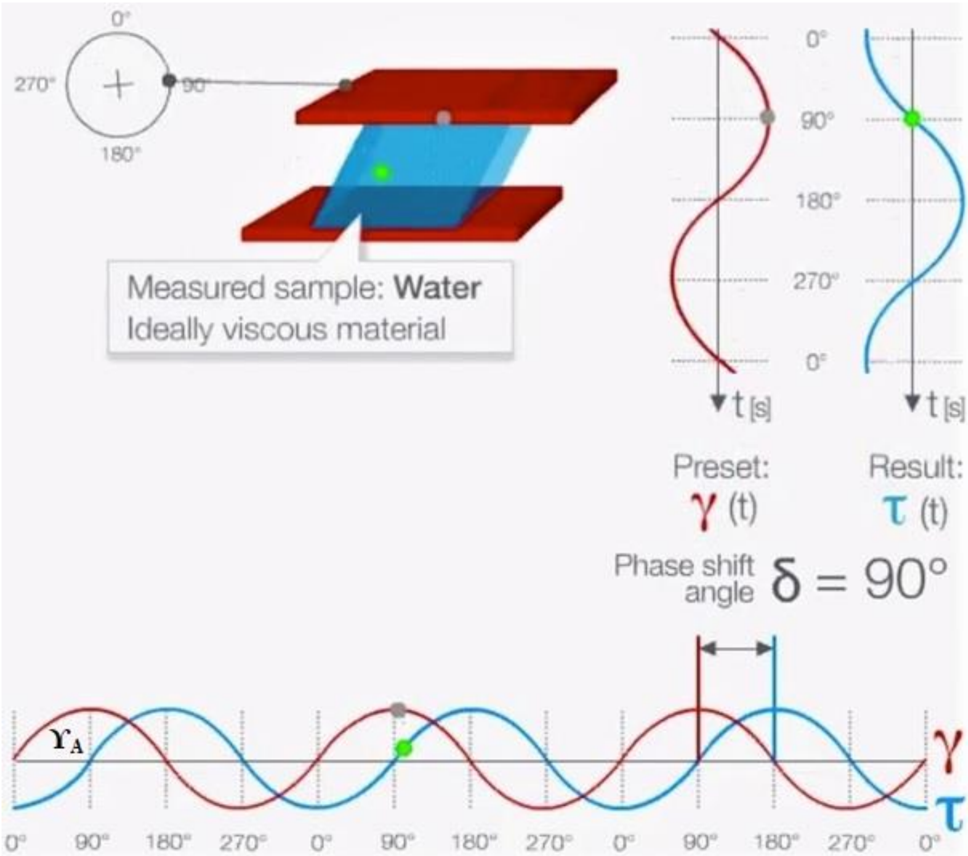


Figure 3.20 Two Plate Model with Ideally Viscous Sample

From the above results showed that for ideally viscos materials has 90° phase shift angle, for ideally elastic materials has 0° phase shift angle and for viscoelastic materials phase angle is varies between 0° to 90°.

(d) Rutting Parameter (|G|/sinδ)*

|G*|/sinδ is the another parameter which is derived from the oscillation test. It also gives the stiffness of binder. If the value of rutting parameter is more, means binder is able to resist the rutting or vice versa.

Rutting parameters have following values:

G*/ sinδ for control binder at 10 rad/s should greater or equal to 1 kPa.

G*/ sinδ for aged binder by RTFO at 10 rad/s should greater or equal to 2.2 kPa.

G*/ sinδ for aged binder by PAV at 10 rad/s should less or equal to 5 MPa.

3.2.2.4 Frequency sweep test

Frequency sweep test is performed at constant amplitude and temperature with variable frequencies (change the speed) as shown in Figure 3.21. But frequency and temperature sweep test is performed at constant amplitude value for make the master curve. In the research, Frequency and temperature sweep tests was done through oscillation test which is conducted at different frequencies and temperature range within linear viscoelastic range at shear strain value of 4% and C0%, C1%, C2%, C3% and C4% were tested at 99 Hz to 0.1 Hz range of frequency at 25° to 85 °C range of temperature with increase of 10°C by 25 mm parallel plate with 1 mm thickness of gap using DSR. Test was conducted within linear range based on the result from amplitude sweep test with value of 4% strain. The test produces a number of parameters such as G′, G″, tanδ, G* and δ. Low frequencies indicate the high temperature it means loss modulus is more as compare to storage modulus which shows viscous behaviour of binder and at high frequencies indicate the low temperature which means storage modulus is more than loss modulus and at high frequency binder behaves like elastic means it will be stiff and resist the cracking and permanent deformation. This is known as time dependent of asphalt binder.

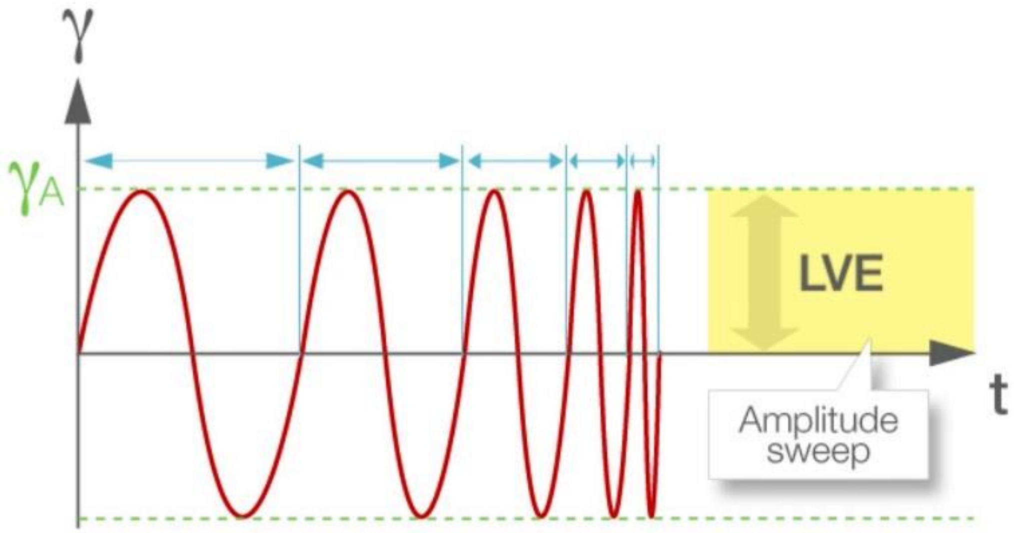


Figure 3.21 Frequency Sweep Test

3.2.2.5 Basics of Rheometry on Rotational viscosity test

In this research, Absolute viscosity (η) was determined by rotational strain rate sweep test at 60°C. The test was conducted at dynamic shear rheometer (DSR). Strain rate levels are 1, 3, 5, 7, 9, 11, 13 and 15 (1/s) and viscosity at different stain rate was determined to find out the range of viscosity. Viscosity is the property of liquids that hinders its flow due to internal friction. Viscosity measure the resistance of liquids. The fluidity of binder depends on the temperature, which enhances the capability of binder to spread and enter in to the voids and make bond with aggregates. For every grade of bitumen there is suitable temperature range at which they give appropriate viscosity for mixing, transportation and effective compaction of road construction. The unit of absolute viscosity is Pa-s or N-s/m^2 in IS units and in CGS terms g/cm-s and P.

At low viscosity, bitumen lubricates the aggregates and make good bond. At high viscosity, binder is not able to coat the whole surface of aggregates and give high resistance during compaction by reducing the movement or flow rate of bitumen. Different types of test methods are available for determination of viscosity. Some tests are an empirical method which gives indirect viscosity like orifice viscometer and some of them give direct viscosity that is absolute viscosity.

Viscosity (η) is equal to shear stress (τ) divided by shear rate ($\dot{\Upsilon}$) as shown in Equation 5. This is the Newtonian liquid's shear viscosity.

$$\eta = \frac{\tau}{\dot{\Upsilon}} \qquad (3.5)$$

Two parameters that influences are time and temperature, so when you are analyzing the viscosity of Non-Newtonian liquids, you must consider time and temperature otherwise you won't get accurate values.

Materials like Newtonian liquids have constant viscosity at different shear rate called as ideally flow behaviour. If the viscosity decreases as the shear rate increases called as shear thinning or pseudoplastic materials like ketchup or mayonnaise. If the viscosity increases as the shear rate increases called as shear thickening or dilatant materials.

3.2.2.6 Multiple Stress Creep Recovery (MSCR) of Bitumen

MSCR is the newest test for improvement to asphalt binder specifications. It gives the high temperature performance of bitumen through which we can analysis the rutting parameter of unmodified and modified binder. This test is also used for aged binder. More advantage of this test is that it abolishes the elastic recovery, toughness and ductility tests. In this test higher value of stress and strain are applied to simulate actual pavements conditions. By using higher stress and strain values it doesn't give only the stiffness of binder but it also give the elastic delayed effects.

Rutting Criteria

Federal highway administration (FHWA) constructed a test section with multiple C.B and M.B such as air blown binder, SBS modified binder, and crumb rubber modified binders. Test of MSCR parameter J_{nr} and actual rutting parameter was evaluated. The pavement was heated at 64°C and trafficked with single tire load of 10,000 lbs (44.5 KN) on neat binder test roads and modified roads. Then rutting performance was determined and compared to both MSCR parameter and superpave performance grade (PG) binder for both the road sample, unmodified and modified asphalt pavements. From the results it was concluded that MSCR results showed great performance over Standard superpave performance graded (PG) binder criteria, |G*|/sinδ as shown in Figure 3.22.

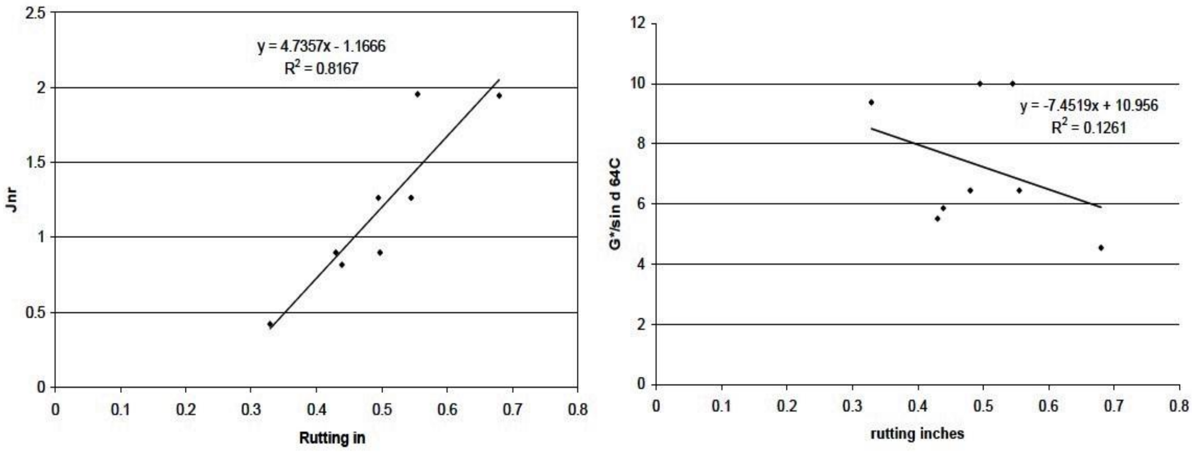


Figure 3.22 Comparisons between MSCR Test and Superpave PG Binder Test Results

MSCR test was conducted to evaluate stress sensitivity and recovery behavior at 65°C, which gives an indication of resistance to permanent deformation. The test was conducted as per AASHTO TP70 which uses %R as shown in Equation 3.6 and J_{nr} as shown in Equation 3.7 to describe the elastic and plastic state of the asphalt binder using dynamic shear rheometer (DSR). The sample was placed between the parallel plate assembly with 25 mm diameter and 1 mm gap between the plates. 10 and 30 creep-recovery cycles was run on each different stress levels of 0.1 kPa (lowest), 0.5 kPa, 1 kPa, 1.5 kPa, 2 kPa, 2.5 kPa and 3.2 kPa (highest) to understand the stress sensitivity of control and modified binders and determine the elastic response presence in the binder under load. One cycle in test consists of 1 sec of creep loading followed by 9 sec of recovery. Two parameters, which includes: percentage recovery (%R) indicating the amount of recovery achieved in binder during recovery period which indicates the permanent deformation under repeated loading and J_{nr} indicating the amount of residual strain left in the binder were calculated from the creep-recovery data. During each cycle the binder reaches a peak value of % strain and then recovers before the next load cycle is applied as shown in Figure 3.23.

% Recovery equation:

$$\varepsilon_r = \frac{(\varepsilon_1 - \varepsilon_{10})}{\varepsilon_1} \times 100 \qquad (3.6)$$

Where

ε_r= Percentage recovery

ε_1= Strain value at end of creep portion

ε_{10}= Stain value at end of recovery portion

Non-recoverable creep compliance equation (J_{nr}) kPa^{-1}:

$$J_{nr} = \frac{Non\text{-}recovered\ strain}{Stress\ (0.1,1,2,3.2kPa)} \qquad (3.7)$$

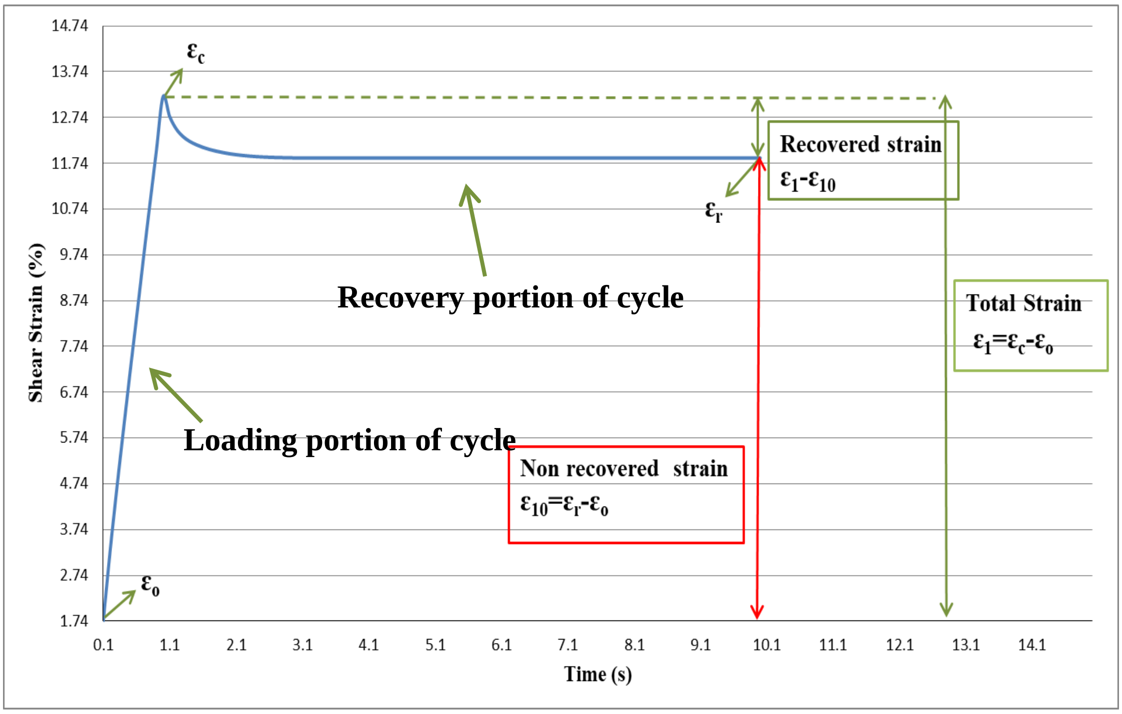


Figure 3.23 First Creep Strain and Recovery Cycle with Parameters Derived For 10-Cycle

3.2.3 Morphology of microcharcoal powder and modified binder

(a)Briefing of scanning electron microscope (SEM)

It is the category of electron microscope that captures the image of sample's surface with a beam of focused electron. It is used to analysis the solid materials in details. When the beam of electrons (known as primary electron) from the source of electron gun with high voltage (20kV) moves from top to down and passes through electromagnetic lenses and focused beam interacts with atoms of sample, it excites the electrons of the specimen and releases with energetic form these electrons are detector are known as secondary electrons then various signals send to monitor to produce the image. Captured image contains information regarding surface topography such as size, shape and texture. It also gives the information of morphological and composition of specimen. Before doing the SEM solid samples are placed in vacuum and if the sample in non-conducting then gold coating is done to make it conducting. It produces the black and white images of samples.

The microstructure of coconut shell micro charcoal powder (MCP) and modified binder (C1%, C2%, C3%) was analysis using JEOL JSM-6510LV SEM equipment as shown in Figure 3.24. The MCP and modified binder sample was placed on sample holder as shown in Figure 3.25 and at various magnifications images were captured. The magnification power of SEM is 30,000 x to 40,000 x and accelerating voltage is 0.5 kV to 30kV. This test is operated at 15 kV. Due to viscoelastic behaviour of bitumen first modified binder are dipped into liquid nitrogen as shown in Figure 3.26 to get solid bitumen otherwise it catches fire due to inside heat of SEM and then modified samples were coating with gold as shown in Figure 3.27 to avoid the charge effect and get clear images and our samples also non conducting of electricity.

The reason of doing SEM is that to check dispersion of MCP into bitumen. Because SEM gives the image of micro scale so we can easily see the clear image of dispersion. If the MCP is homogeneously and well dispersed into bitumen then MCP is compatible to bitumen otherwise not.

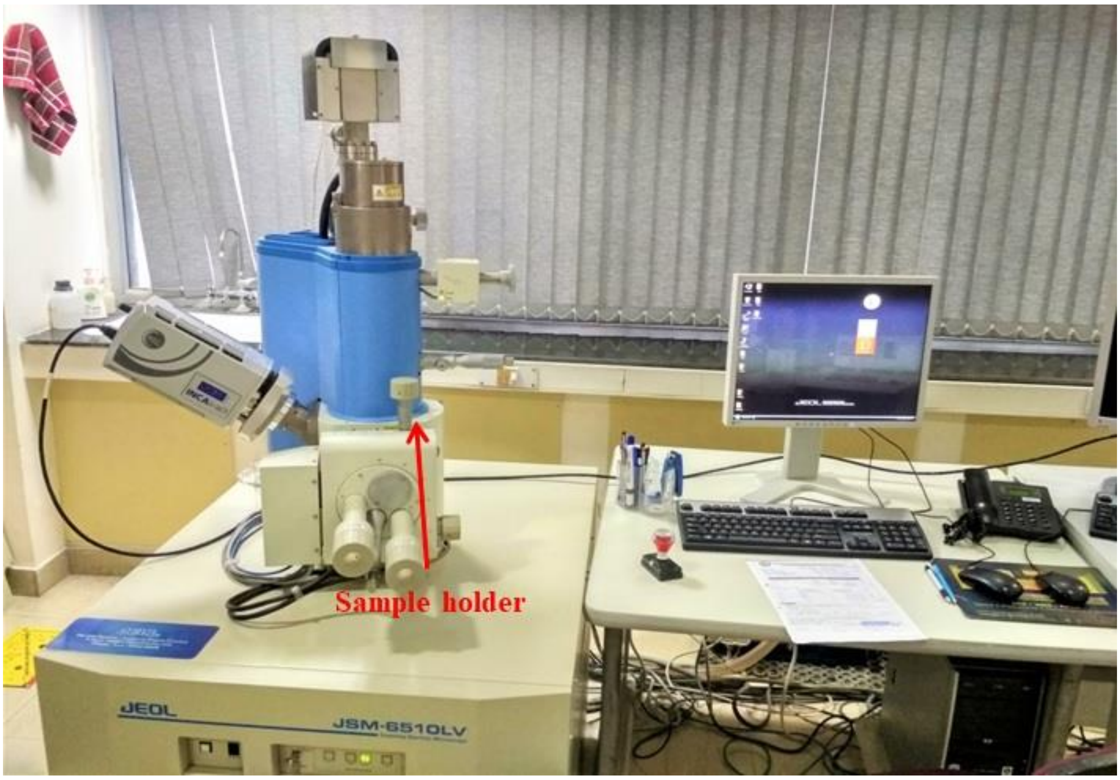


Figure 3.24 Scanning Electron Microscopy (SEM)

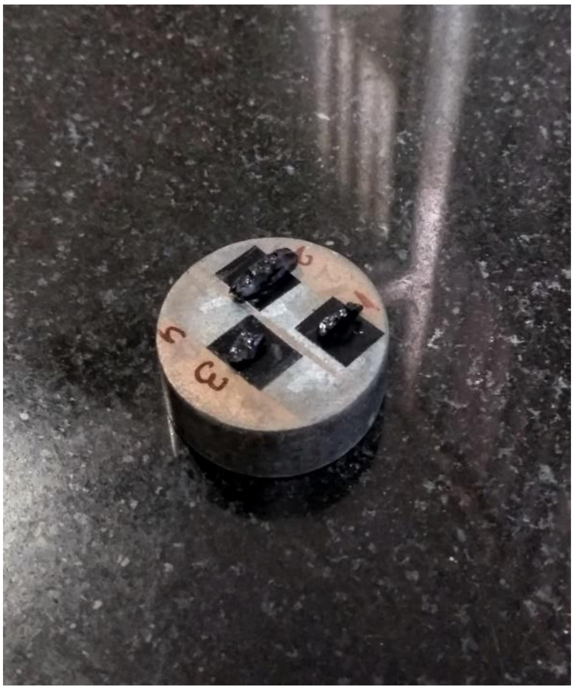


Figure 3.25 SEM Samples

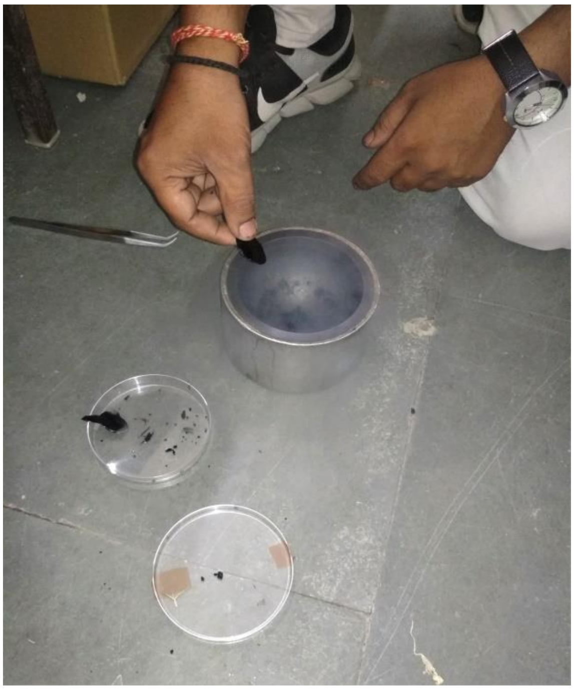


Figure 3.26 Liquid Nitrogen

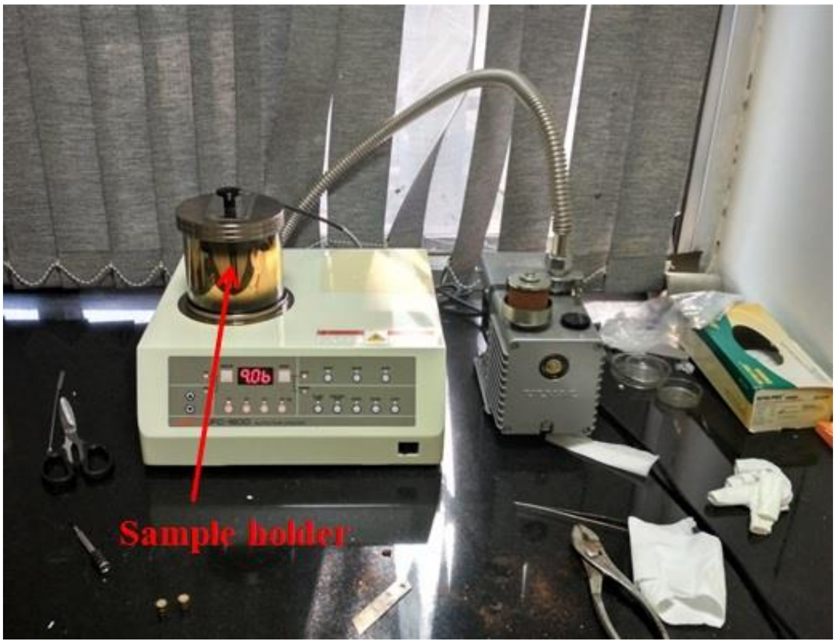


Figure3.27 Golden coating equipment

CHAPTER-4

4.1 GENERAL

This section includes the investigation of physical and rheological properties of control binder (C.B) and modified binders (M.B). The basic tests included penetration, softening test, PI or Temperature Susceptibility test and also the storage stability of MCP modified binders. In order to determine rheological properties, oscillation test which includes strain sweep test, frequency sweep and temperature sweep tests were done. In this test different parameters were evaluated to understand the flow and deformation behaviour of C.B and M.B, these parameters are G*, δ and |G*|/sinδ, tanδ under LVE range. Frequency and temperature was done on different frequency range and different temperatures to see the effect of both on binder behaviour. MSCR test was carried out to understand elastic, deformation and the creep recovery behaviour of control and modified binders using %R and J_{nr}. In MSCR, test was run at 10 and 30 cycles to simulate the actual loading cycles during service of pavement. In addition, the stress sensitivity of both binders was investigated by conducting MSCR test at various stress levels. The results of MSCR test gives large number of %R and J_{nr} data points so to understand the data, Analysis of Variance (ANOVA), non-parametric and coefficient of variance (CV) were conducted. Rotational viscosity test was also done on DSR to determine the viscosity at 60°C at different shear strain rate levels. For each test three trails were done to check the repeatability of test results. Moreover Scanning electron microscope test was also done for MCP and modified binder on different magnifications to visualize the dispersion and homogeneous of MCP with different contents in bitumen.

4.2 RESULTS AND ANALYSIS

4.2.1 Penetration Value Test

The Figure 4.1 shows that penetration value of C.B and M.B. The penetration value decreases at certain limit as the addition of MCP content increases. The reductions in penetration value of M.B are 19.07%, 24.52%, 17.17%, and 14.99% as compared with C.B for C1%, C2%, C3% and C4% addition of MCP, respectively. The maximum reduction was 24.52% for C2% addition of MCP. Low prnetration value indicates the M.B is stiff and hard. Due to high value of stiffness, modified binder is low susceptibility at high temperature. A very stiff binder is more elastic and leads to cracking under repeatable loading if the value of penetration lies between 20 dmm to 30 dmm. When the value of penetration is greater than 30 dmm, binder has highly capable to resist the cracking with proper mix design and compaction.

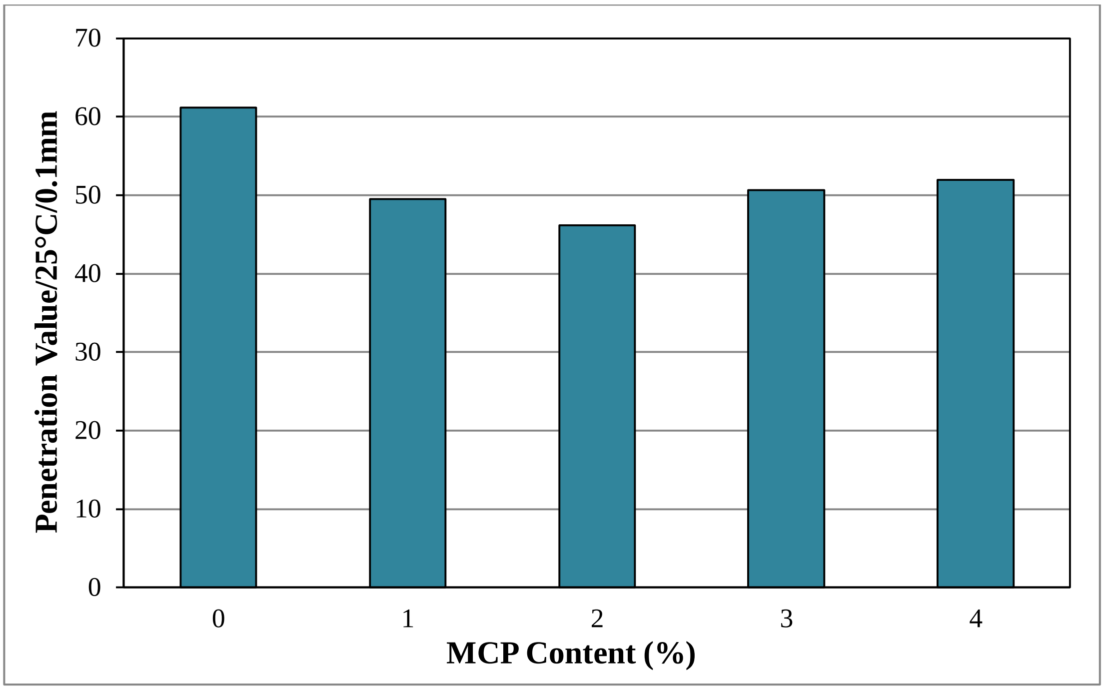


Figure 4.1 Penetration Test Values

4.2.2 Softening Point Test

The softening point test results indicated that the softening point temperature initially showed an increasing trend up to 2% as shown in Figure 4.2. The softening point of C.B was 47.5^{o}C whereas the softening point temperature at C2% addition was 58^{o}C indicating a 10^{o}C increase in softening point. After 2%, any further addition did not show significant increase in the softening point temperature. The increase in softening point value of M.B are 15.789%, 21.052%, 22.105%, and 22.105% as compared with control binder (47.5°C) for C1%, C2%, C3% and C4% addition of MCP respectively. The increase in softening point value shows us that binder can resist some level of high temperature and deformation. This also shows that between bitumen and MCP has good compatibility and less susceptible to high temperature. The phase change for modified binder from solid to liquid is less as compared to control binder. The consistency of the neat and modified binder was determined by physical properties. The temperature at which consistency of the bitumen reaches is calculated by softening point value and the consistency determined at same temperature i.e. 25°C is calculated by penetration value so the compatibility of binder is calculated by penetration index (PI).

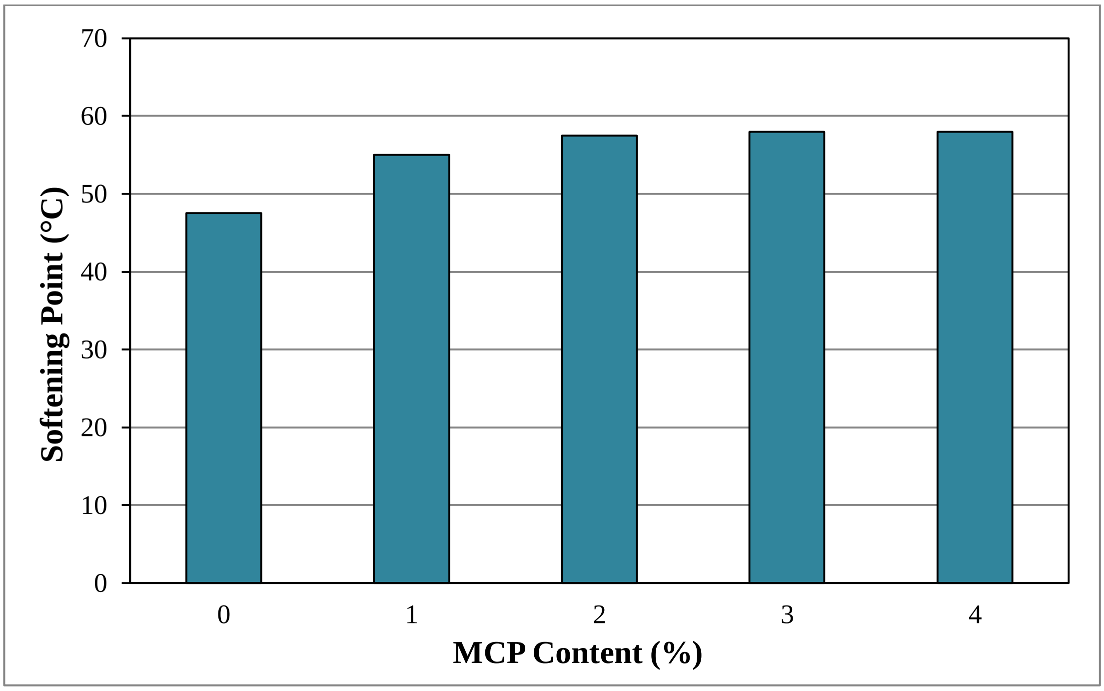


Figure.4.2 Softening Point Values

4.2.3 Penetration Index (PI) or Temperature Susceptibility Test

The PI value for control and modified binder was calculated from following Equation 4.1. Penetration index value gives the behaviour of the binder with temperature change. Basically it gives the thermal sensitivity of binder. From the Table 4.1 the PI value of control binder is less than -1 indicating that it is highly susceptibility to low temperature, it means cracks and deformation occurs in early stage of road construction. However for modified binders, the value of PI is approaching to +1 and more than control binder indicating that addition of MCP, the modified binder has low susceptibility to high temperature and resist the rutting and cracking in permanent deformation.

Table 4.1 PI of C.B and Modified Binder

MCP (%)	Penetration Test, 1/10th mm	Softening Point Test, ^{o}C	PI
C0	61	47.5	-1.380
C1	50	55.0	-0.045
C2	46	57.5	0.325
C3	51	58.0	0.656
C4	52	58.0	0.721

$$PI = \frac{(1951.4 - 500 logP - 20 S.P)}{(50 logP - S.P - 120.14)} \qquad (4.1)$$

4.2.4 Storage Stability Test

The Table 4.2 shows the results of storage stability test. It can be seen that the differences in the top and bottom portion of the specimen is within the prescribed limit i.e. less than 2.2°C. It means added MCP is well homogenously dispersed in bitumen at high temperature, it also showed that adopted method for blending MCP with bitumen is adequate. Thus the addition of charcoal powder in bitumen does not lead to any storage stability concerns.

Table 4.2 Storage Stability Test

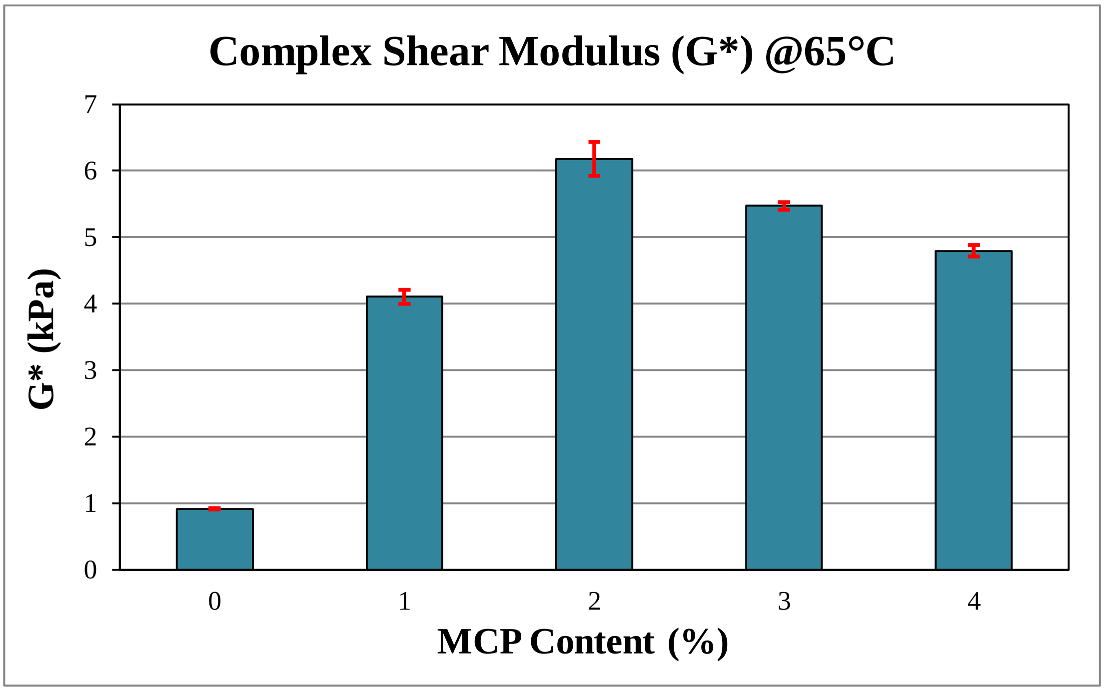
Microcharcoal Powder (MCP)%	**Difference between top and bottom parts specimen (°C)**	**Specification <2.2°C**
C1%	1.2	ok
C2%	0.9	ok
C3%	1.4	ok
C4%	1.0	ok

4.2.5 Oscillation Test

Oscillation test was performed after finding the strain value with the help of stain sweep test (amplitude sweep test). To conduct oscillation test 4% shear stain is obtained.

4.2.5.1 Complex Shear Modulus (G)*

Complex shear modulus is obtained from oscillation sweep test and it gives the total stiffness and resistance of binder to deformation and also characterizes the elastic and viscous behavior of the binder at medium to high temperature. Higher G* gives the better resistance to flow deformation and improves the rutting performance of the binder. The Figure 4.3 shows the G* at 65°C and at different content of MCP and it was observed that G* of control binder is very less even it is unable to reach the require value of G* i.e. 1kPa. The value of G* for control binder is 0.9118 kPa. It means C.B is highly viscous and couldn't resist the cracking and permanent deformation under loading process. With the increase in content of MCP, G* value is also increases. Addition of C1% of MCP into conventional binder, G* value is increases However, after C2% addition, the G* starts to decrease. At C2% addition, value of G* is 6.17 kPa and the percentage increase in M.B is 577.33%. The probable reason for initial increase in G* could be due to the fact that the charcoal powder are well dispersed up to certain addition level providing enhanced filler effect. However, increased addition may cause flocculation leading to improper dispersion of modifier resulting in lack of synergistic effect. It implies that excessive addition of MCP reduce the performance of binder and decrease resistance to flow deformation. Sample containing C2% MCP gives the higher value of G* and shows high stiffness and behaves like elastic material which helps to resist permanent deformation of modified binder as compared with control.

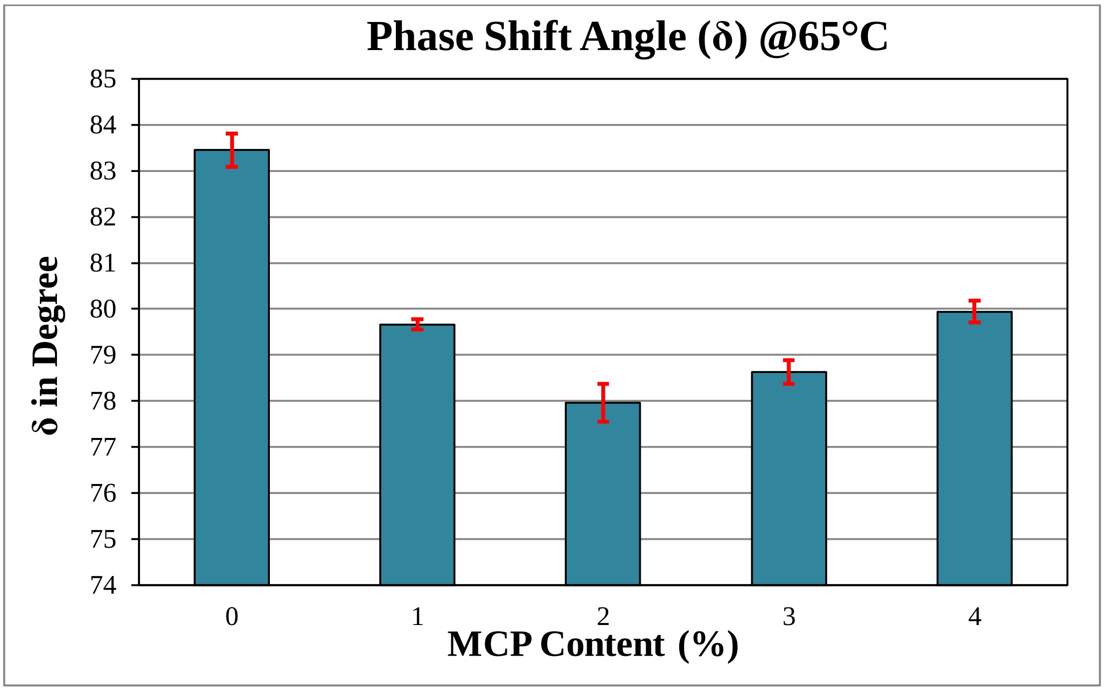


**error bar indicates one standard deviation*

Figure 4.3Complex Shear Modulus at Different MCP Content

4.2.5.2 Phase Shift Angle (δ)

Along with G*, phase angle is also the parameter which defines the viscous and elastic components of binder. Generally, binder having phase angle 0^{o} to 45° shows elastic nature and 45° to 90° shows viscous nature. The Figure 4.4 shows that for C.B the time lag between shear strain and shear stress is more i.e. value of phase angle is 83.44°, indicating highly viscous property, which means binder is less stiff and unable to resist permanent deformation but with the addition of MCP, the lag starts to decrease indicating better elastic property. First addition of C1%, the phase shift angle decrease. However the minimum time lag is obtained at C2% of MCP i.e. 77.95° and the percentage decrease in phase angle as compare to control binder is 6.58% which helps to retard rutting parameter and shows improved elastic recovery performance and after excessive addition of MCP the phase angle starts increasing again. The increase in phase angle beyond C2% addition may be due the fact the applied shear stress is shared more by the bitumen due to lack of proper dispersion of charcoal powder particles. It shows that phase shift angle is highly depends on the content of MCP. So C2% addition of MCP is the optimum dose.

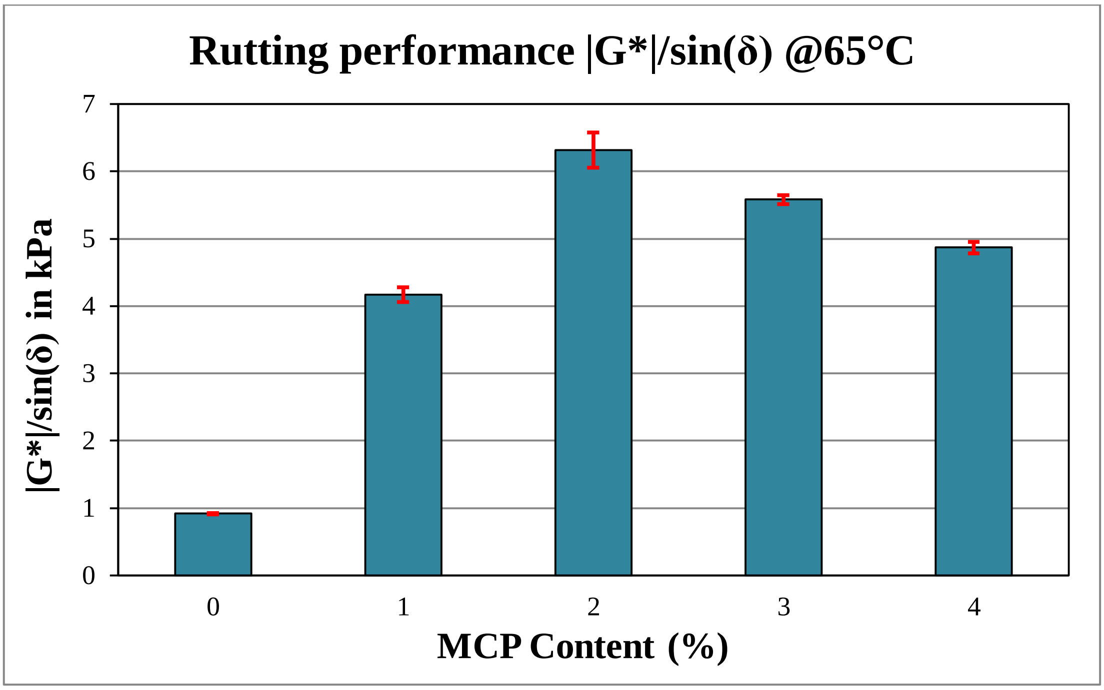


**error bar indicates one standard deviation*

Figure 4.4 Phase Angle at Different MCP Content

4.2.5.3 Rutting Performance Indicator (|G|/sinδ)*

Rutting performance indicator is considered as an important parameter to evaluate the rutting resistance and elastic property of the binder at 65°C and 10 rad/s frequency. If the value of rutting parameter is equal or greater than 1 kPa, that bitumen is considered as rut resistance. From Figure 4.5, addition of MCP causes the increase in rutting parameter as compared to the C.B. The rutting resistance capacity of control binder is not satisfied. The highest value of (|G*|/sin δ) was achieved at C2% of MCP content which means maximum resistance to permanent deformation of asphalt binder. This finding indicates that modification makes the binder stiff and enhances the rutting parameter with MCP however after C2% content of MCP i.e. at C3% addition, rutting parameter start decreasing. The analysis shows that it makes the binder stiff than the control binder by reinforcing the bitumen and makes the strong and cohesion bond between MCP and asphalt binder.



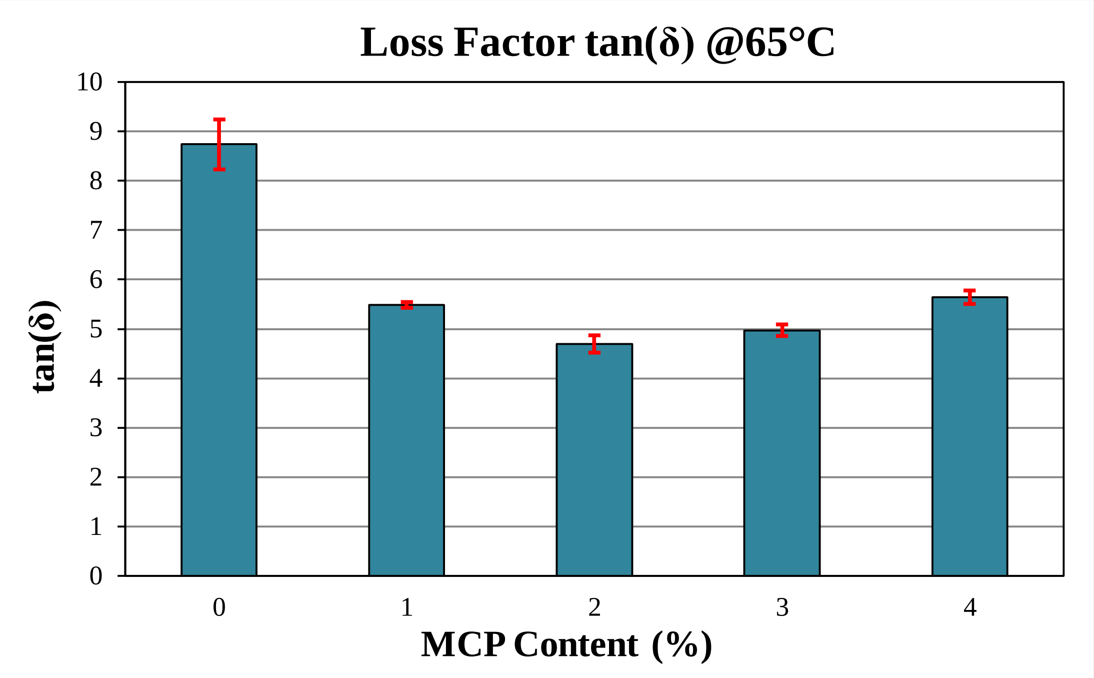
**error bar indicates one standard deviation*

Figure4.5. Rutting Performance at Different MCP Content

4.2.5.4 Loss or Damping Factor (tanδ):

Loss or damping factor is related to storage moduli (G′) and loss moduli (G″) and defined as the ratio of viscous component of complex modulus (G″) and elastic component of complex modulus (G′) as shown in Equation 4.2. It describes the average energy dissipation in continuous steady oscillation dynamic test. Figure 4.6 shows that the loss factor for control binder is higher due to high viscous state i.e. loss of energy is more in control binder than the modified binders. But with the addition of MCP, loss factor starts decreasing resulting in reduction of the loss factor showing elastic behavior i.e. storage of energy is more in modified binder. In this case we can say that tanδ for modified binder is approaching to less than one. Lower loss factor can easily correlate the binder with more rutting resistance performance and improve the elastic properties and vice versa. The minimum loss factor is achieved at 2% addition of MCP content.

$$tan\delta = \frac{G''}{G'} \qquad (4.2)$$



**error bar indicates one standard deviation*

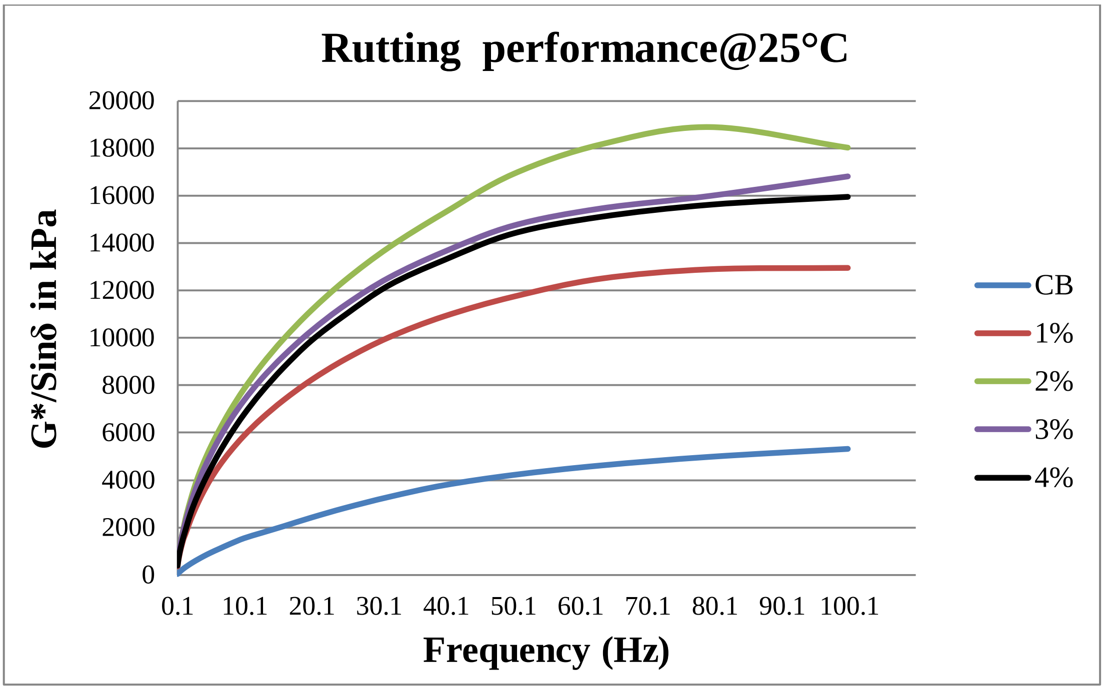
Figure 4.6 Loss Factor at Different MCP Content

4.2.6 Frequency and Temperature Sweep Test

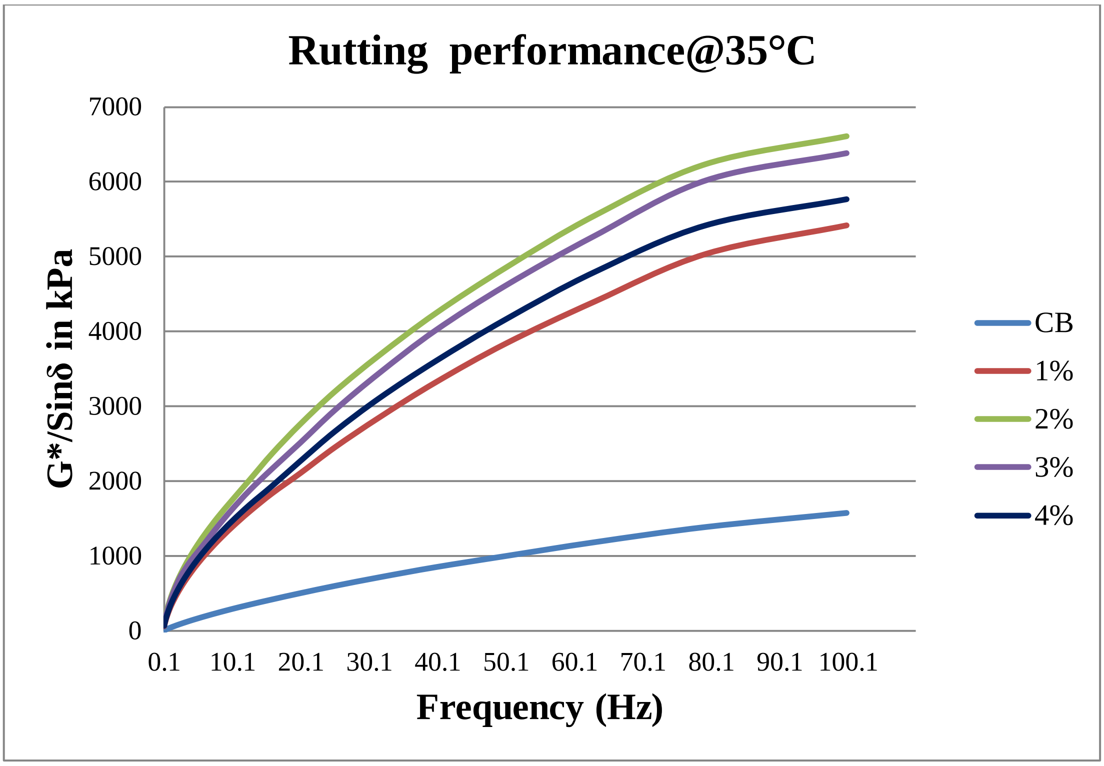
4.2.6.1 Effects of MCP dosages on Rutting performance (|G|/sinδ)*

Frequency and temperature sweep tests were conducted at different frequencies range (99.9 Hz to 0.1 Hz) and at different temperatures (25°C to 85°C) by oscillated at 4% of shear strain level with 25 mm parallel plate geometry and gap of 1mm. In this research, C.B and M.B were tested and |G*|/sinδ, G* and δ were evaluated. Figure 4.7 (a-g) shows the rutting performance of C.B and M.B at frequency range of 99.9 Hz to 0.1 Hz and at different temperature i.e. 25°C-85°C. In Figure 4.7 (a) shows that rutting performance of C.B is less as compared to M.B but as the addition of MCP, |G*|/sinδ is drastically increase and the percentage increase in |G*|/sinδ of C1%, C2%, C3% and C4% MCP modified binder at 50.1 Hz frequency is 178.37%, 301.16%, 249.53% and 241.63%, respectively. It is also observed that as the increase in frequency the asphalt binder shows elastic properties because of shorter duration of vehicle loading and the value of rutting parameter also increases which means better will be the performance of binder but as the frequency decreases, the |G*|/sinδ of binder also decreases. Maximum elastic and rutting resistance properties are shown by C2% addition of MCP in conventional binder (C.B). However after C2% addition of MCP; the rutting performance of binder M.B reduces.

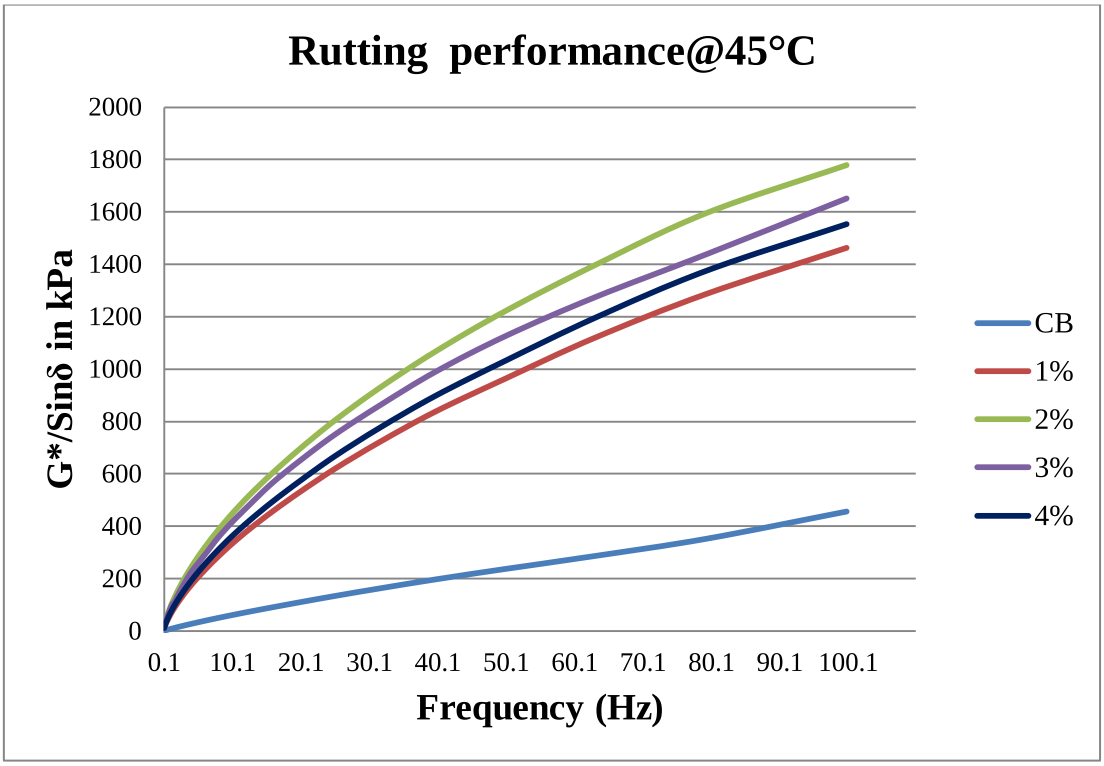
As the increment in temperature the performance of C.B and M.B decreases as shown in Figure 4.7 (b-g). From 65°C temperature the values of the rutting parameter of modified binders are overlapping that means at higher temperature binder become more viscous but still rutting performance of modified binder is very good as compared to control binder as shown in Figure 4.7 (e-g).



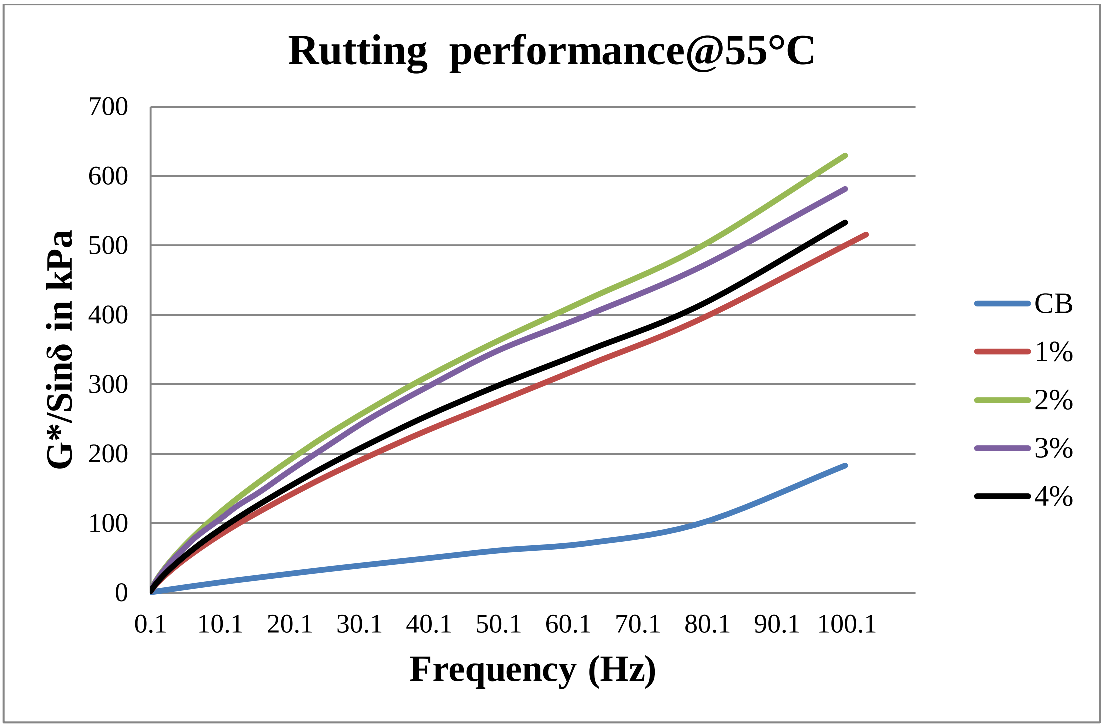
(a)


Rutting performance@35°C
7000
6000
5000
4000
3000
2000
1000
0
G*/Sinδ in kPa
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
CB
1%
2%
3%
4%

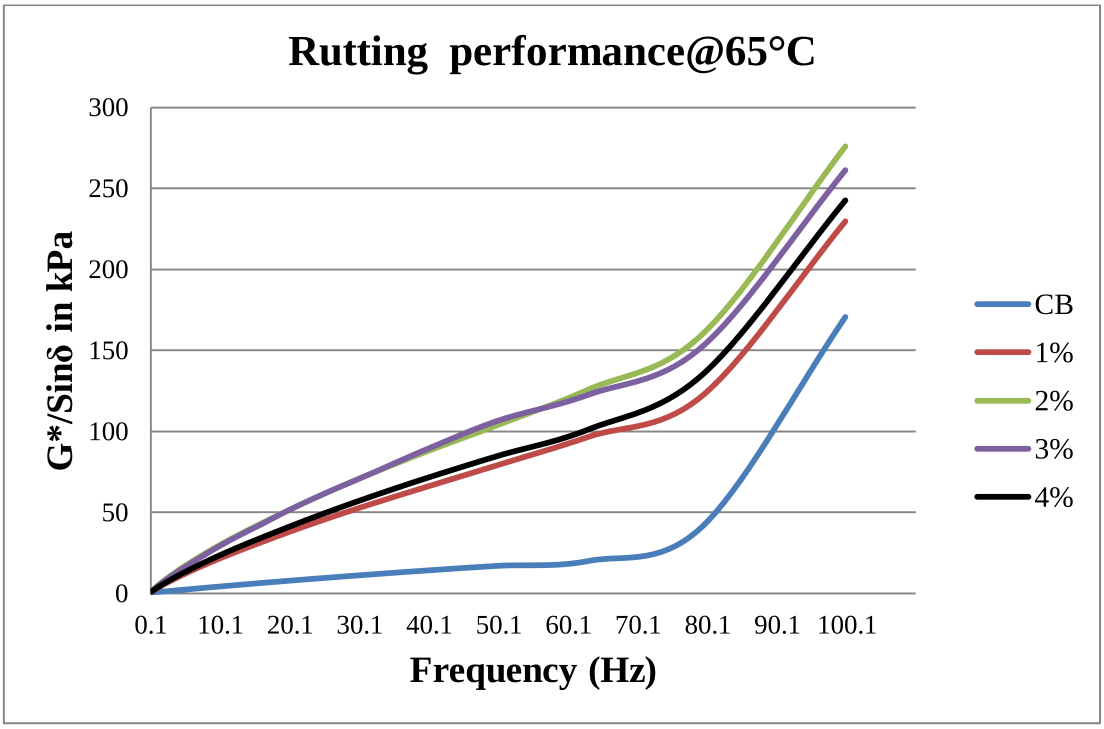
(b)


Rutting performance@45°C
2000
1800
1600
1400
1200
1000
800
600
400
200
0
G*/Sinδ in kPa
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
CB
1%
2%
3%
4%

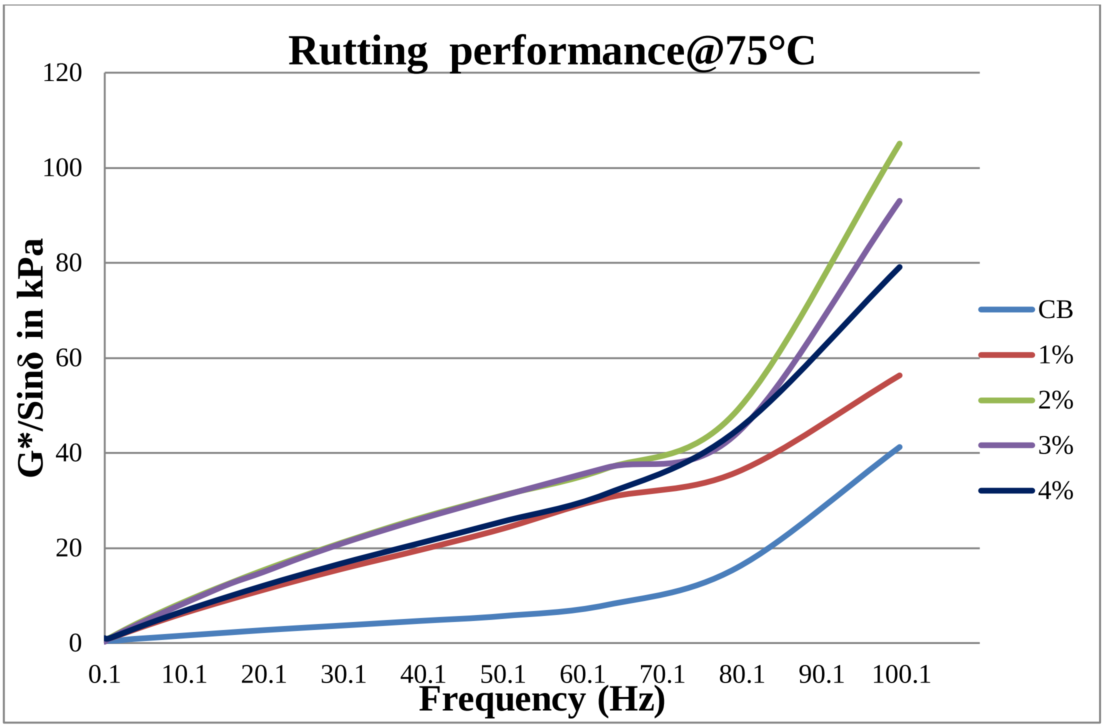
(c)


Rutting performance@55°C
G*/Sinδ in kPa
700
600
500
400
300
200
100
0
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
CB
1%
2%
3%
4%

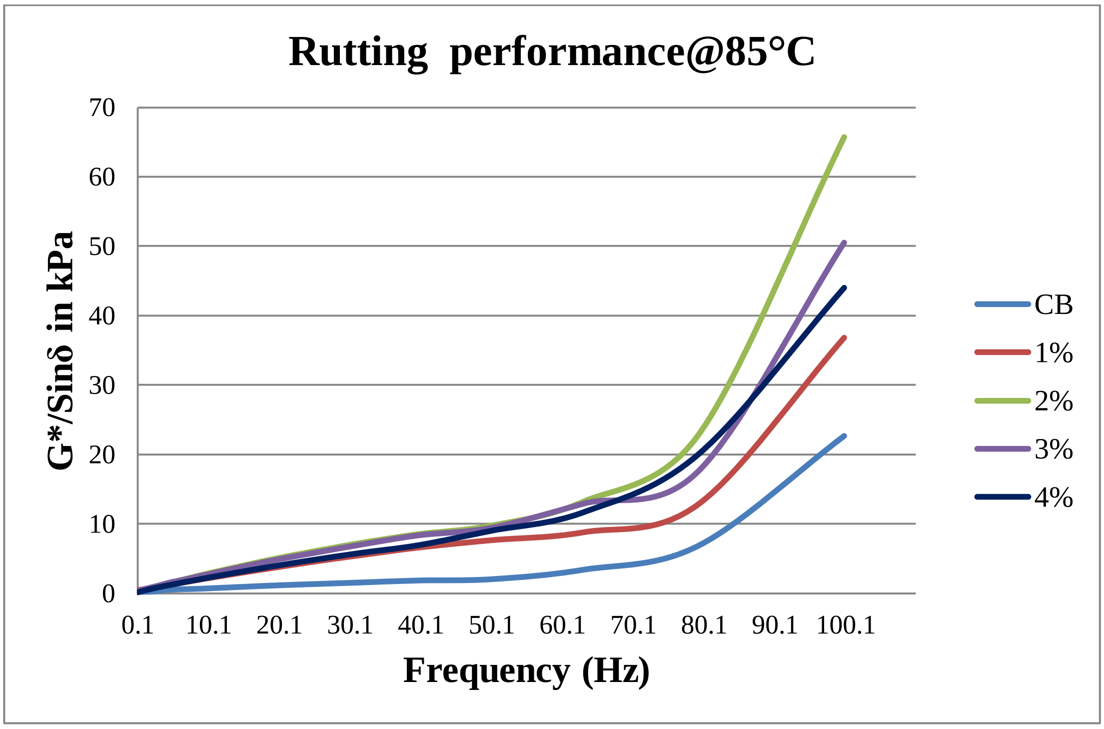
(d)


Rutting performance@65°C
G*/Sinδ in kPa
300
250
200
150
100
50
0
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
CB
1%
2%
3%
4%

(e)



(f)



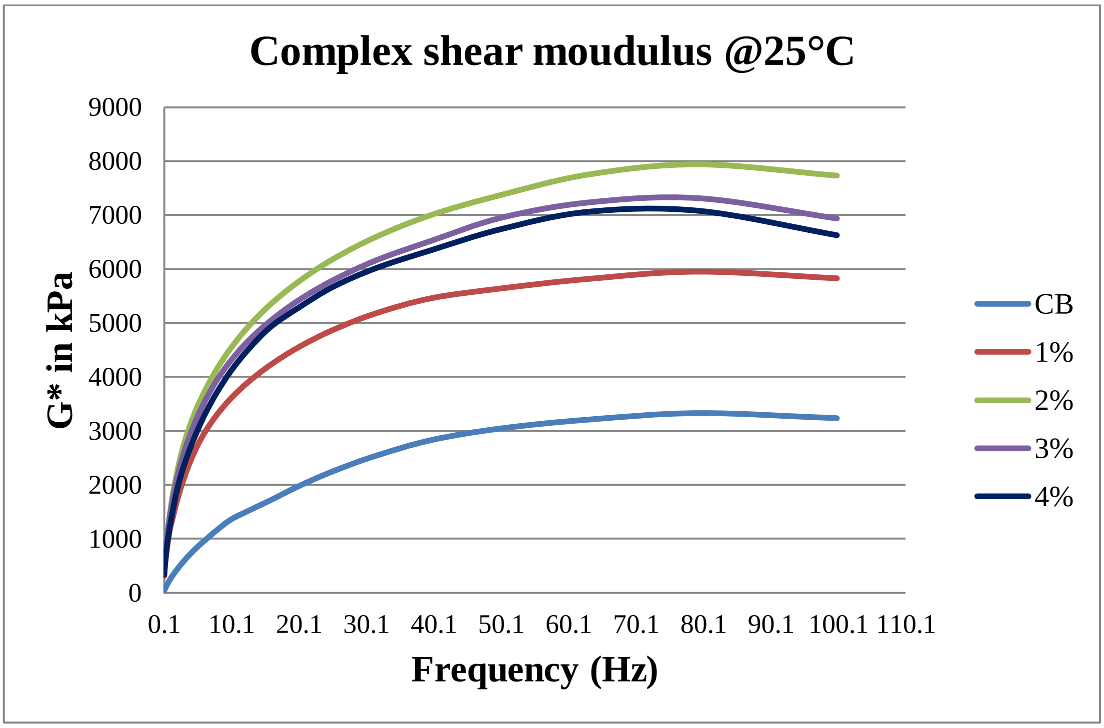
(g)

Figure4.7 Effects of MCP Dosages on |G*|/sinδ

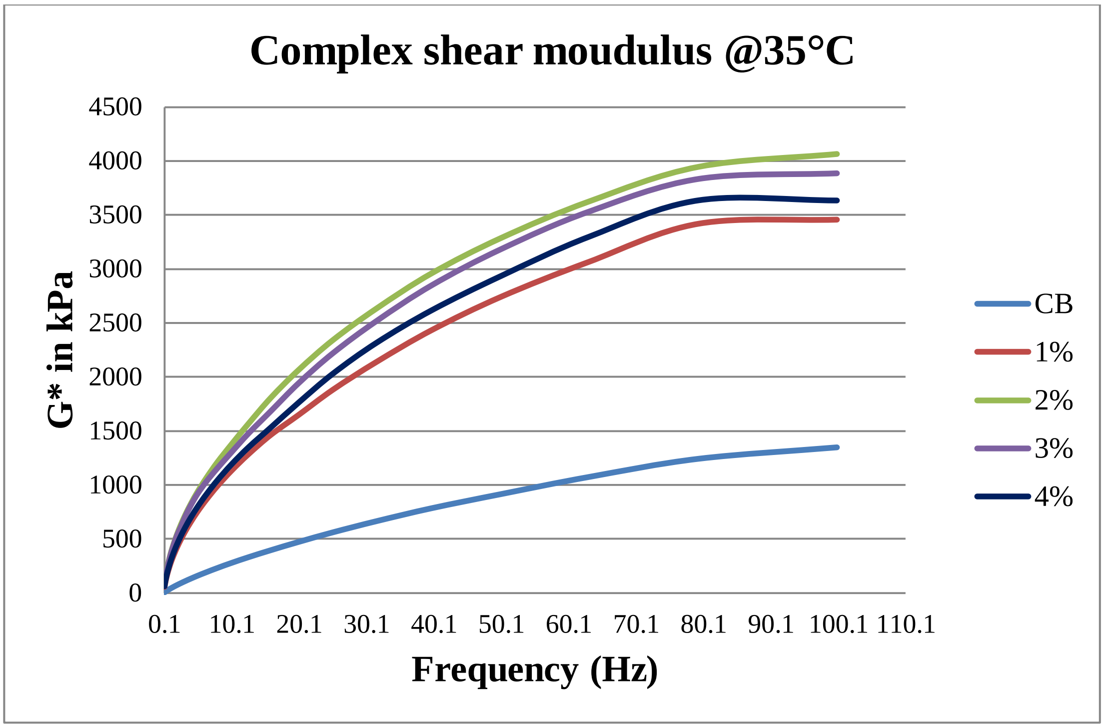
(a) at 25°C, (b) at 35°C, (c) at 45°C, (d) at 55°C, (e) at 65°C, (f) at 75°C, (g) at 85°C

4.2.6.2 Effects of MCP Dosages on Complex Shear Modulus (G)*

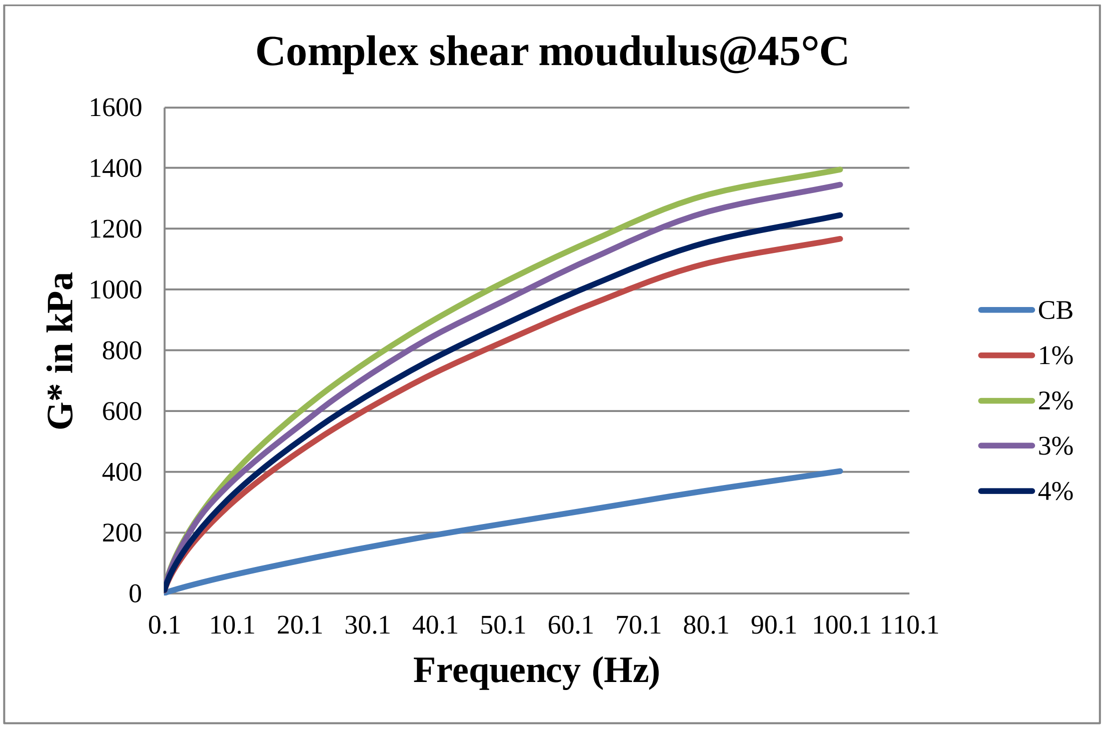
G* indicates the binder stiffness. From Figure 4.8 (a-g) it can be seen that G* also show the same trend as in rutting parameter. The G* of modified binder is more as compared to control binder. As the frequencies increases, G* also increase as loading time is less and vice versa. The binder stiffness also depends on the temperature. From Figure 4.8 (a-g) showed that due to increase in temperature the stiffness of binder decreases i.e. G* is decreasing and the point will come on which G*'s values overlapping each other as shown in Figure 4.8 (e-g). It is observed that at higher temperature modified binder gives almost same results irrespective of modifier content but still better than C.B. It can be noticed that from all the modified binder 2% addition of MCP gives very good results.



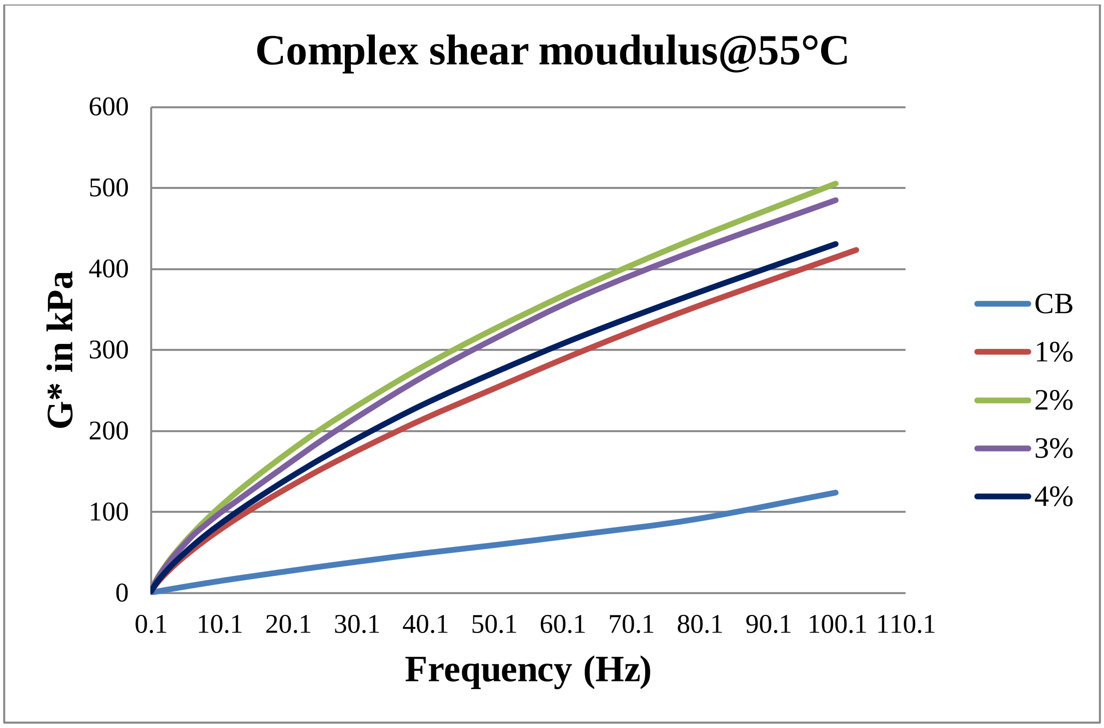
(a)


Complex shear moudulus @35°C
G* in kPa
4500
4000
3500
3000
2500
2000
1500
1000
500
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
CB
1%
2%
3%
4%

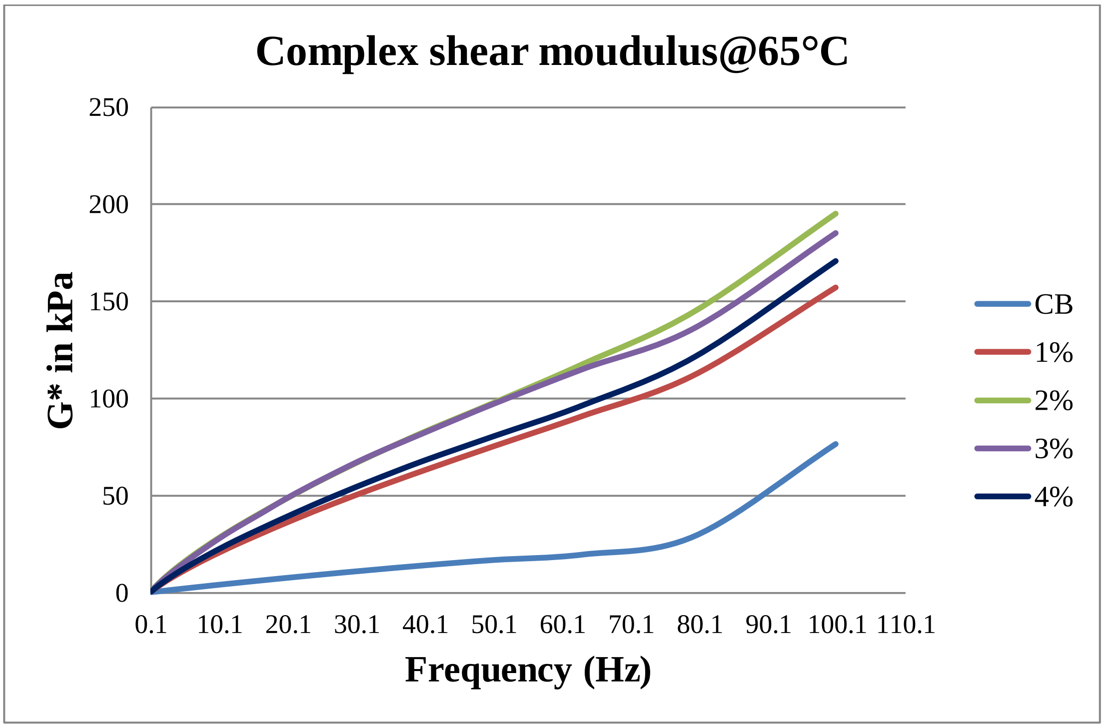
(b)


Complex shear moudulus@45°C
G* in kPa
1600
1400
1200
1000
800
600
400
200
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
CB
1%
2%
3%
4%

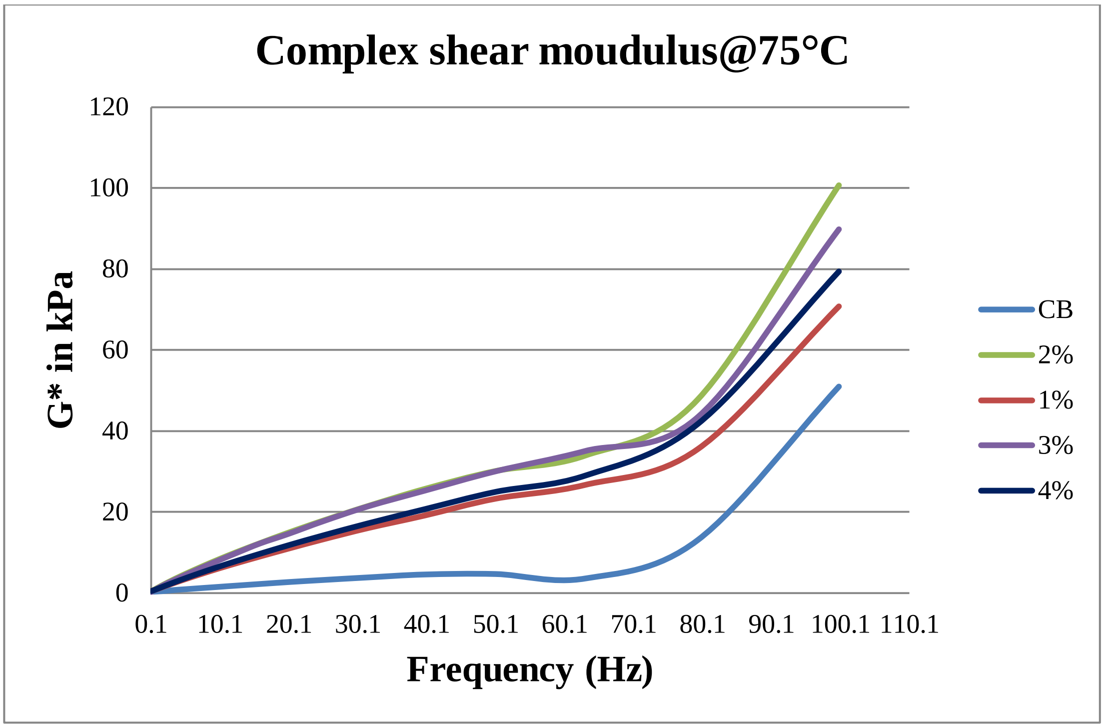
(c)


Complex shear moudulus@55°C
600
500
400
300
200
100
0
G* in kPa
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
CB
1%
2%
3%
4%

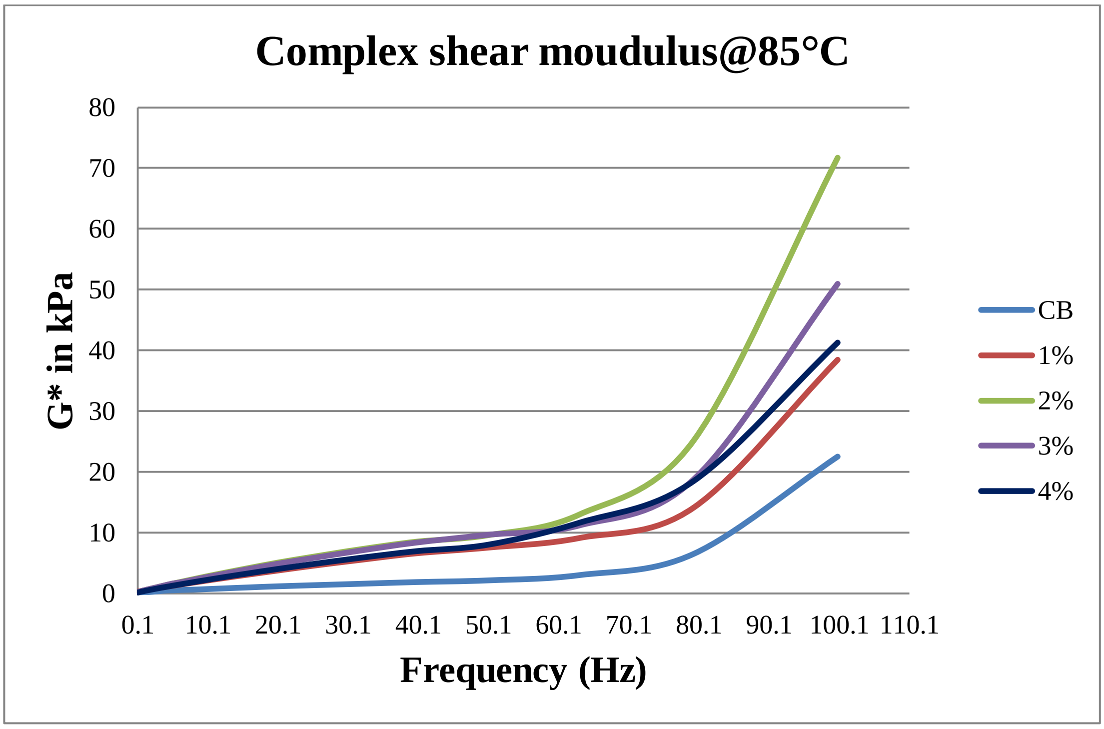
(d)


Complex shear moudulus@65°C
250
200
150
100
50
0
G* in kPa
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
CB
1%
2%
3%
4%

(e)



(f)



(g)

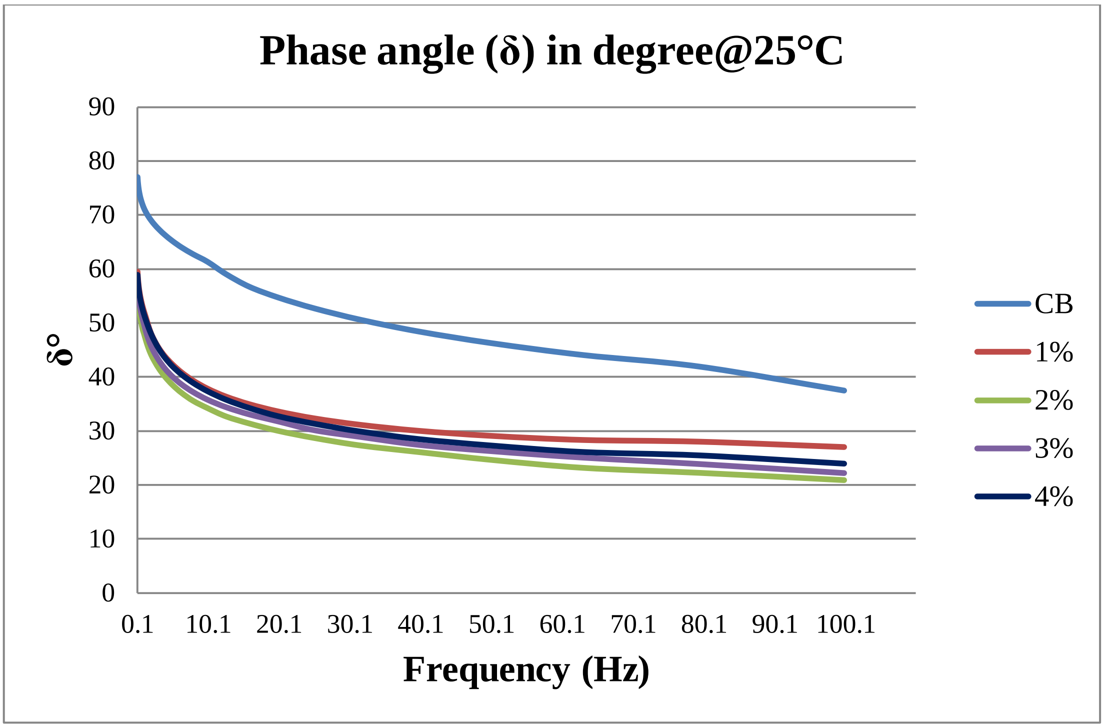
Figure 4.8 Effects of MCP Dosages on G*

(a) at 25°C, (b) at 35°C, (c) at 45°C, (d) at 55°C, (e) at 65°C, (f) at 75°C, (g) at 85°C

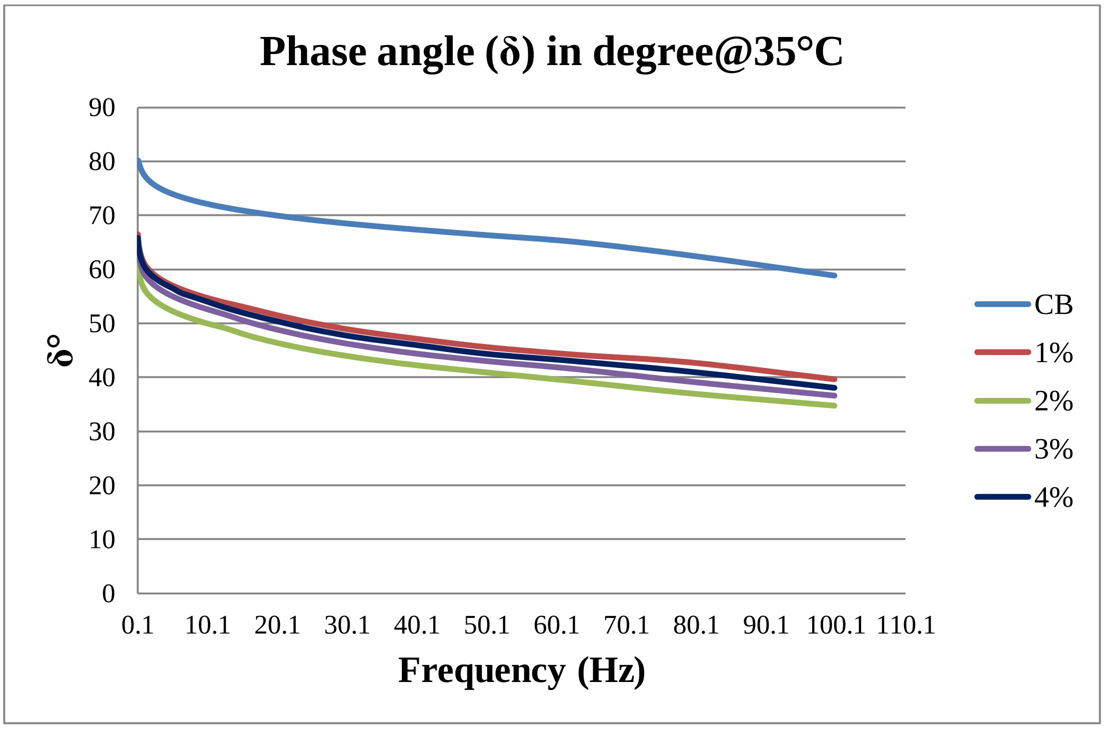
4.2.6.3 Effects of MCP Dosages on Phase Shift Angle (δ)

δ also defines stiffness of binder in terms of elastic and viscous behaviour. From Figure 4.9 (a-g) shows that the phase angle is more for control binder at low temperature, gives viscous behaviour and at high frequency phase angle is less, shows elastic behaviour. As the addition of MCP in control binder phase angle decreases significantly as compared to control binder as shown in Figure 4.9 (a-g). It can be observed that at low frequencies modified binder exhibited shear thinning behaviour and phase angle values are more, at high frequencies phase angle is less, become stiff and shows shear thickening behaviours means elastic behaviour. Its viscosity decreases at high frequencies and increases at low frequencies. Figure 4.9 (f, g) it can be observed that the change in δ is not much for modified binders, they are almost overlap to each other. It can be seen that at high temperature, modified binders gives same values of phase angle. But still modified gives better results as compare to control binders. At high temperature on when the frequency is low i.e.at 0.1 Hz control binder touches 90° in that condition binder is purely viscous material as shown in Figure 4.9 (e, f). As increase in temperature as shown in Figure 4.9 (g) shows that even modified binder also close to 90°. From this it is concluded that at high temperature and at low frequencies modified binders fail to show elastic behaviour.

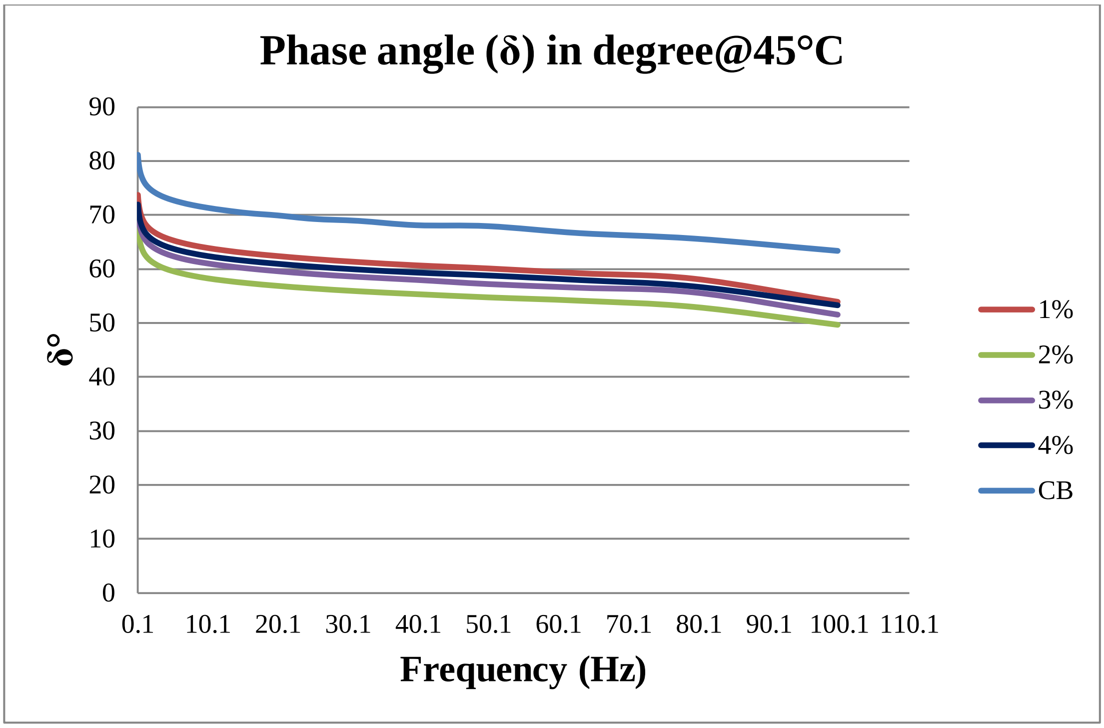
From all the results such as rutting performance (G*/sinδ), complex shear modulus (G*) and phase angle (δ) of control and modified binders, it was concluded that at every temperature 2% modified binder gives better results.


Phase angle (δ) in degree@25°C
δ°
90
80
70
60
50
40
30
20
10
0
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
CB
1%
2%
3%
4%

(a)

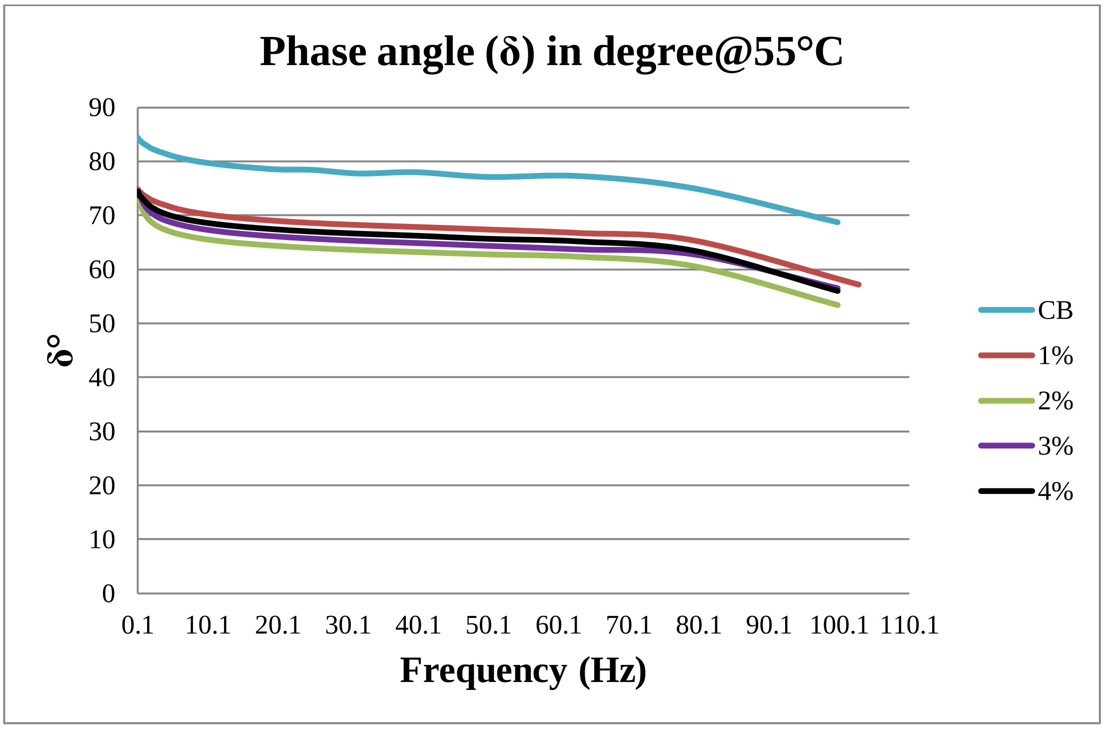

Phase angle (δ) in degree@35°C
δ°
90
80
70
60
50
40
30
20
10
0
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
CB
1%
2%
3%
4%

(b)



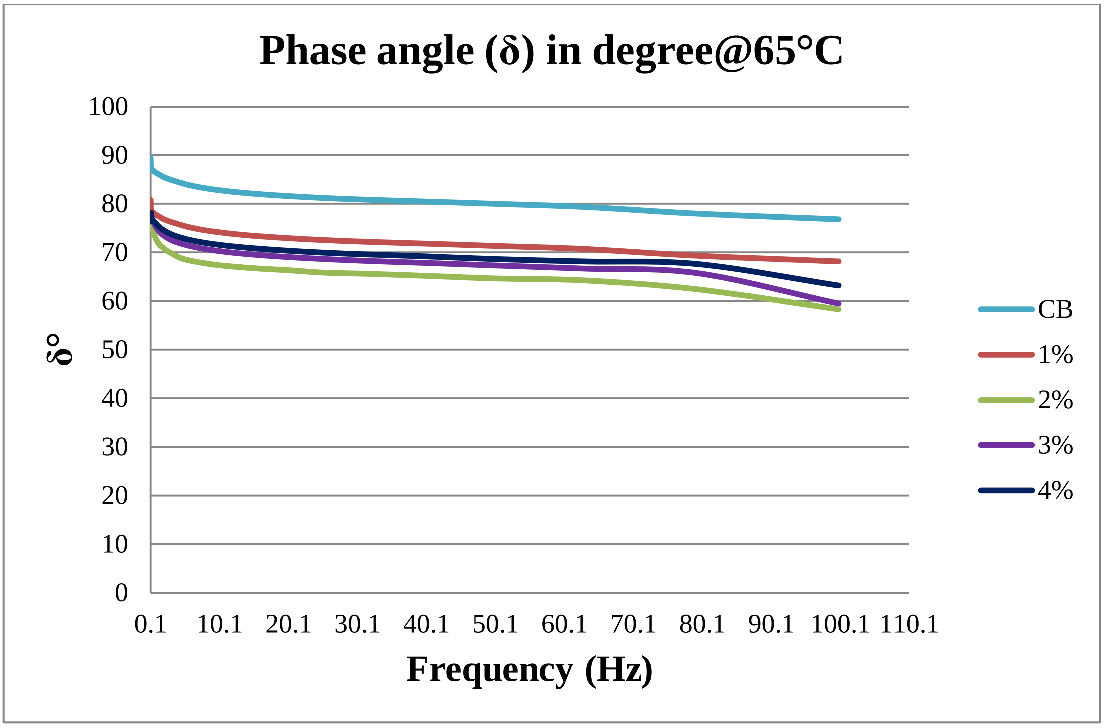
Phase angle (δ) in degree@45°C
90
80
70
60
50
40
30
20
10
0
δ°
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
1%
2%
3%
4%
CB

(c)

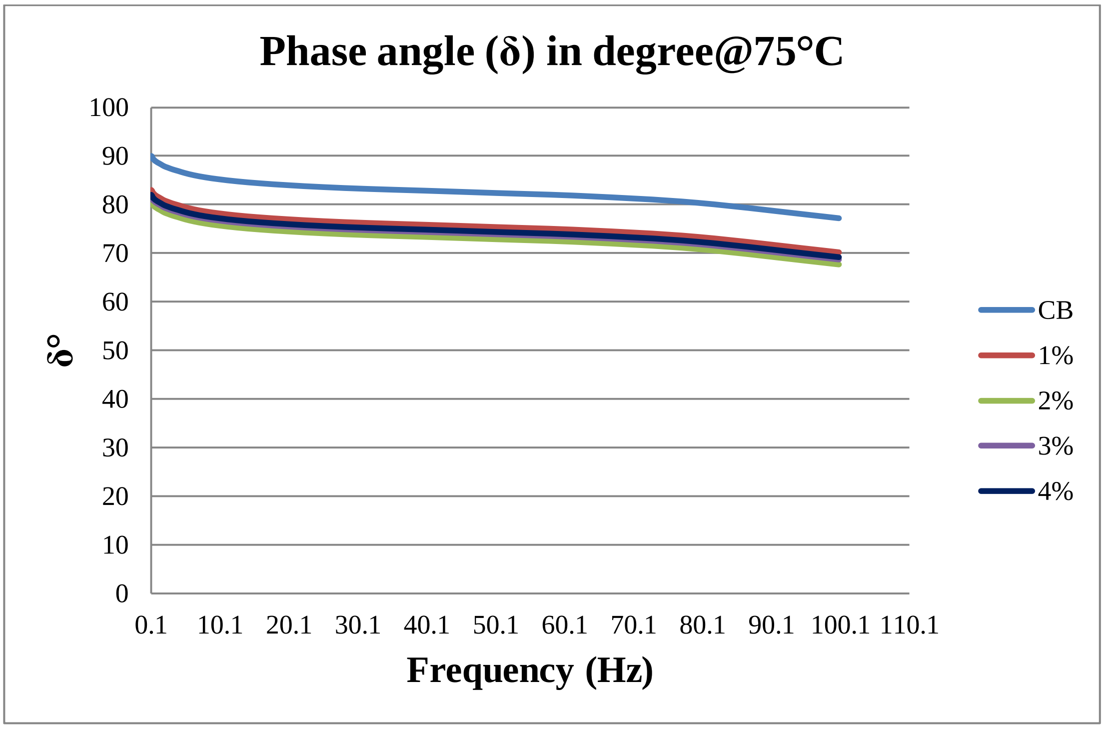


Phase angle (δ) in degree@55°C
90
80
70
60
50
40
30
20
10
0
δ°
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
CB
1%
2%
3%
4%

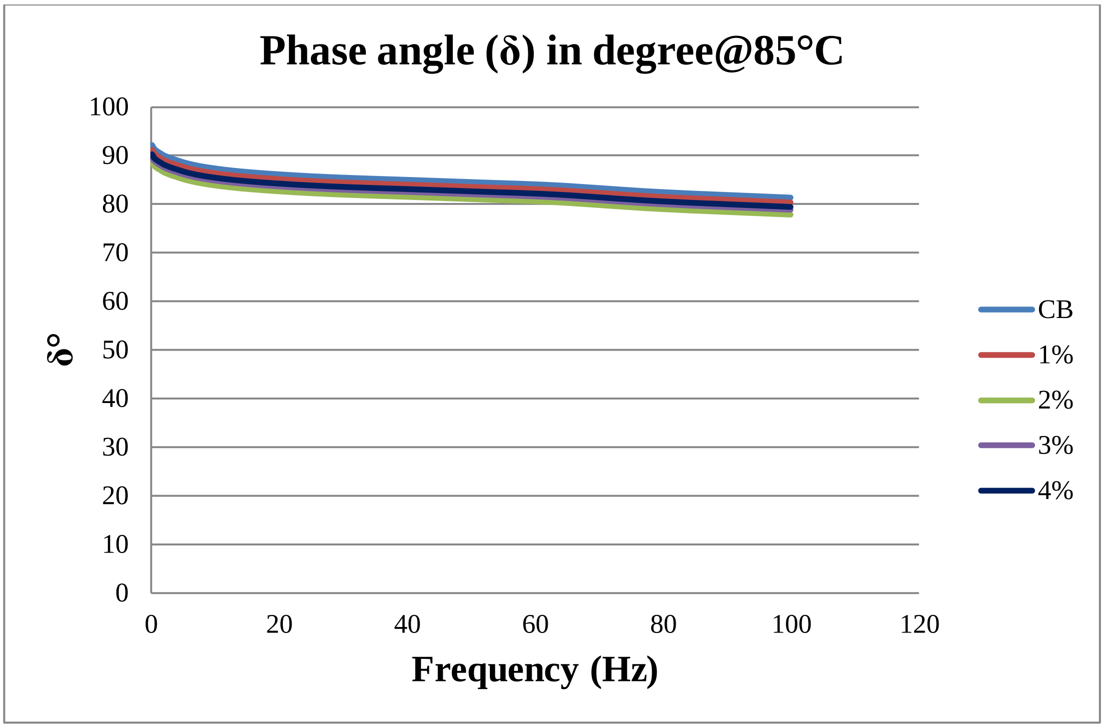
(d)


Phase angle (δ) in degree@65°C
100
90
80
70
60
50
40
30
20
10
0
δ°
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
CB
1%
2%
3%
4%

(e)


Phase angle (δ) in degree@75°C
100
90
80
70
60
50
40
30
20
10
0
δ°
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
CB
1%
2%
3%
4%

(f)



(g)

Figure 4.9 Effects of MCP Dosages on δ (°)

(a) at 25°C, (b) at 35°C, (c) at 45°C, (d) at 55°C, (e) at 65°C, (f) at 75°C, (g) at 85°C

4.2.6.4 Effect of Temperatures on Rutting Performance (|G|/sinδ)*

Rutting performance of C.B and M.B were evaluated at different frequencies and with varying temperature from low to high as shown in Figure 4.10 (a-e). Rutting performance of control binder is as shown in Figure 4.10 (a). Take 65°C is the reference temperature because to simulate the field conditions. It can be seen that as we reduce the temperature, the rutting performance of binder increases as asphalt binder is going to be stiff at low temperature and its elastic behavior will increase. The values increase in rutting performance at 50.1 Hz at 55°C, 45°C, 35°C and 25°C with respect to 65°C (17.008 kPa) are 60.83 kPa, 237.47 kPa, 1000.5 kPa and 4216.2 kPa. But as we increase the temperature, the performance of binder is decreasing. At 75°C the value is 5.67 kPa and at 85°C is 2.03 kPa. This significantly reductions due to asphalt binder get soften and give viscous behavior. It is noticed that at higher temperature binder starts to flow, means phase is changed from solid to liquid. It is also noticed that as the frequency increases the rutting performance also increases.

This is due to shorter duration of traffic loading time and better will be the resistance performance but as the frequency decreases the resistance to deformation also decreases; this is due to longer duration of traffic loading which makes the binder viscous and soft.

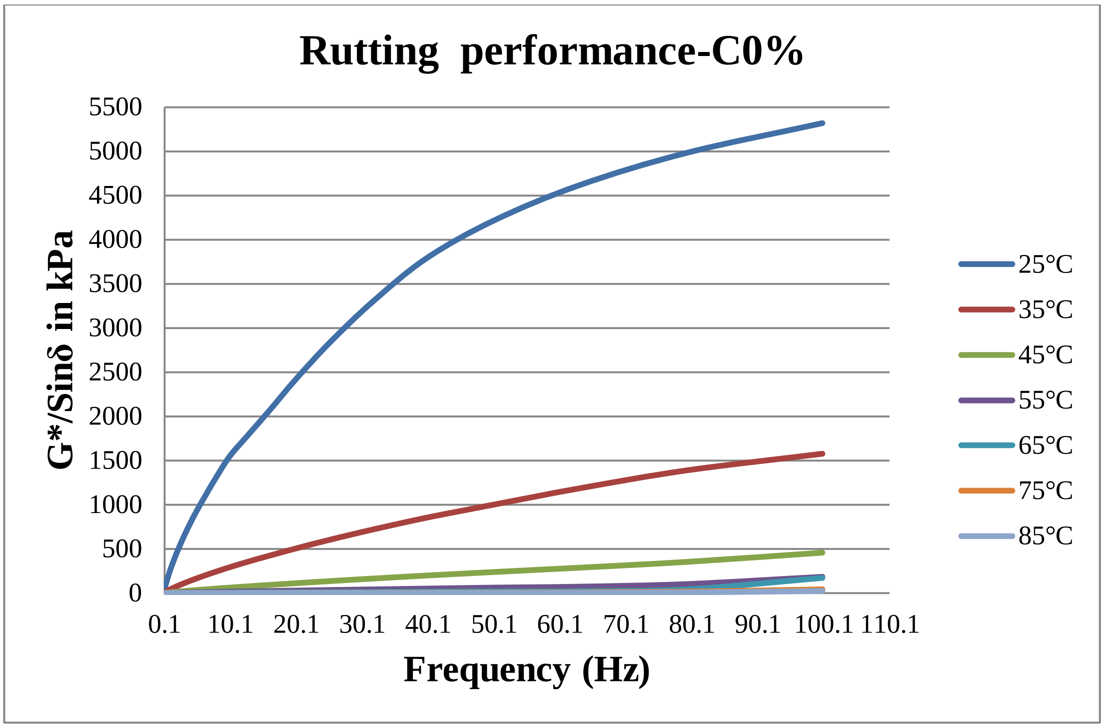
Figure 4.10 (b) shows the 1% addition of MCP modified binder. It can be seen that modified bitumen gives improved results and enhance the performance of rutting parameter as compared to control binder. The value at 50.1 Hz and at 65°C for control binder was 17.08 kPa now after modification the value is 79.367 kPa. The percentage increase is 364.63. In this case also as the increase in temperature the rutting performance is decreasing and vice versa. At 25°C, 35°C, 45°C, 55°C, 75°C and 85°C the value are 11737 kPa, 3840.9 kPa, 964.86 kPa, 275.48 kPa, 24.07 kPa and 7.65 kPa. It is concluded that MCP increase the stiffness and elastic behavior of binder. Modified binder is more capable to resist the rutting and permanent deformation.

Figure 4.10 (c) also shows the rise in rutting performance as the addition of MCP at 2%. Now the value is 104.18 kPa as compare to 1% addition of modified binder. The percentage increase is 31.26. At 25°C, 35°C, 45°C, 55°C, 75°C and 85°C the value are 16914 kPa, 4885.2 kPa, 1223.1 kPa, 363.21 kPa, 31.08 kPa and 9.76 kPa.

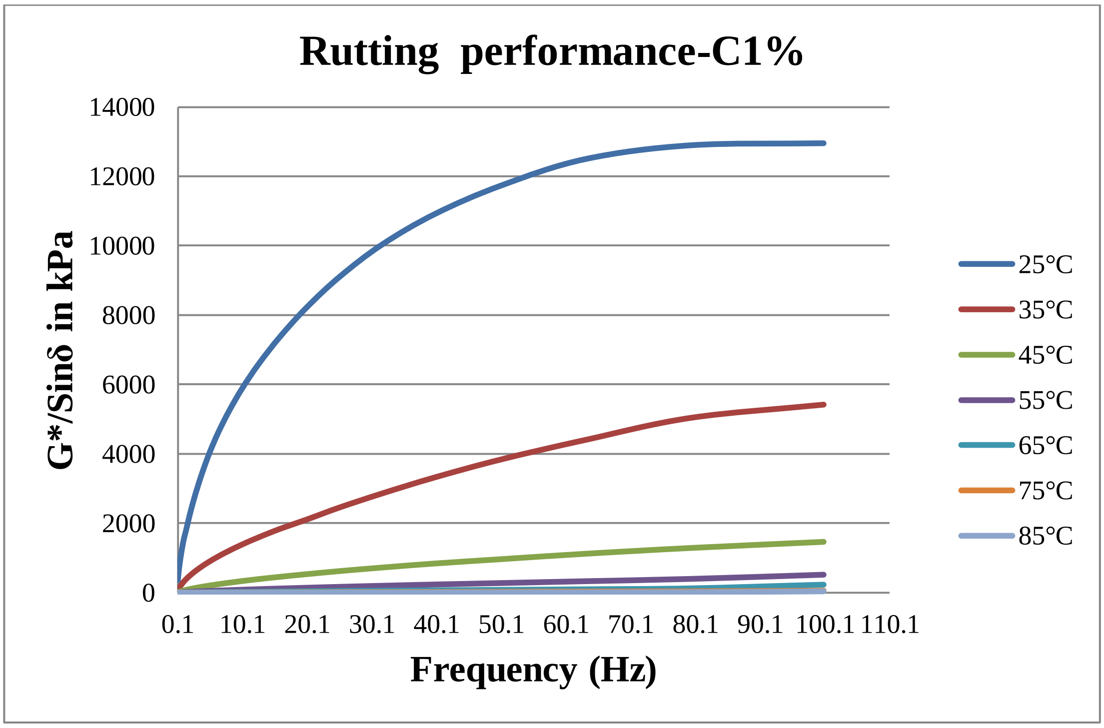
Figure 4.10 (d) shows that as the addition of 3% of MCP rutting performance increased by percentage of 2.44 at 65°C. From the other tests it was observed that at addition of 3% MCP the properties are getting decreasing but in this case rutting performance was increase by 2.44%. But at other temperatures the values are less as compare to 2% addition of MCP modified binder. It is also noticed that the trend is same as in other cases; rutting parameter is decreasing at high temperature and vice versa. The values at different temperatures are at 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°C the value are 14737 kPa, 4617 kPa, 1127.1 kPa, 349.1 kPa, 106.73 kPa, 30.99 kPa and 9.45 kPa.

Figure 4.10 (e) shows that as addition of 4% MCP in neat binder also enhance the rutting performance as compare to conventional binder but at high percentage addition decreases the performance as compare to other modified binders. Values at different temperatures are at 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°C the value are 14404 kPa, 4164.7 kPa, 1033.2 kPa, 298.58 kPa, 85.00 kPa, 25.573 kPa and 9.0252 kPa.

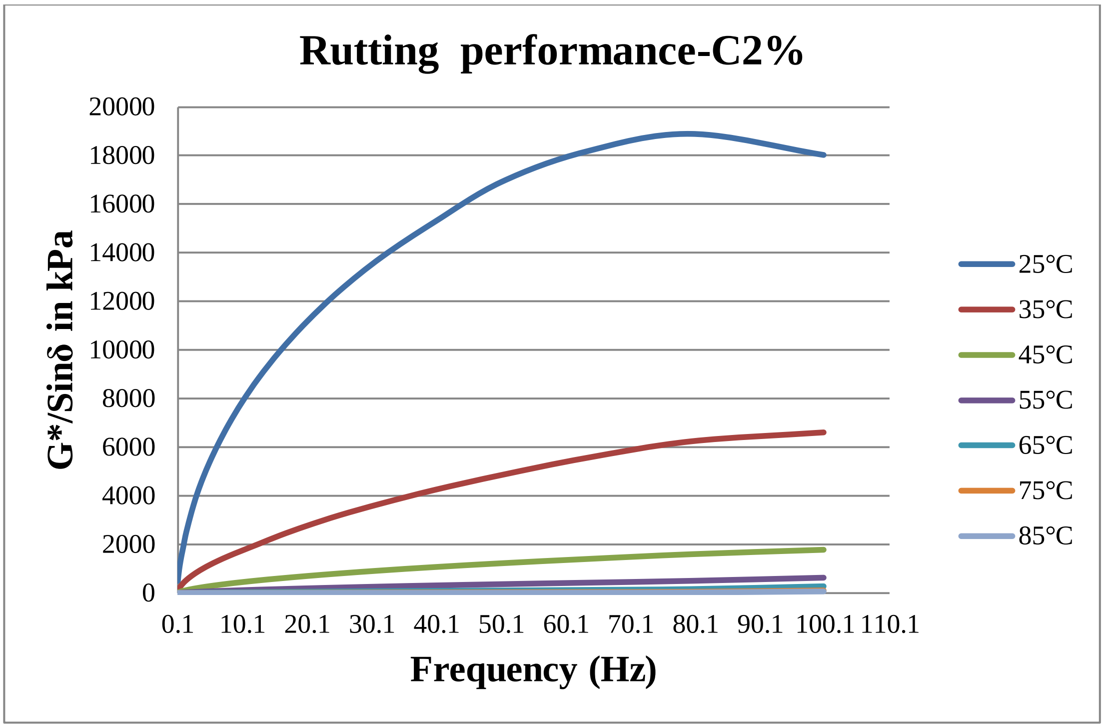
From all this it was concluded that 2% addition of MCP gives better results.


Rutting performance-C0%
G*/Sinδ in kPa
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
25°C
35°C
45°C
55°C
65°C
75°C
85°C

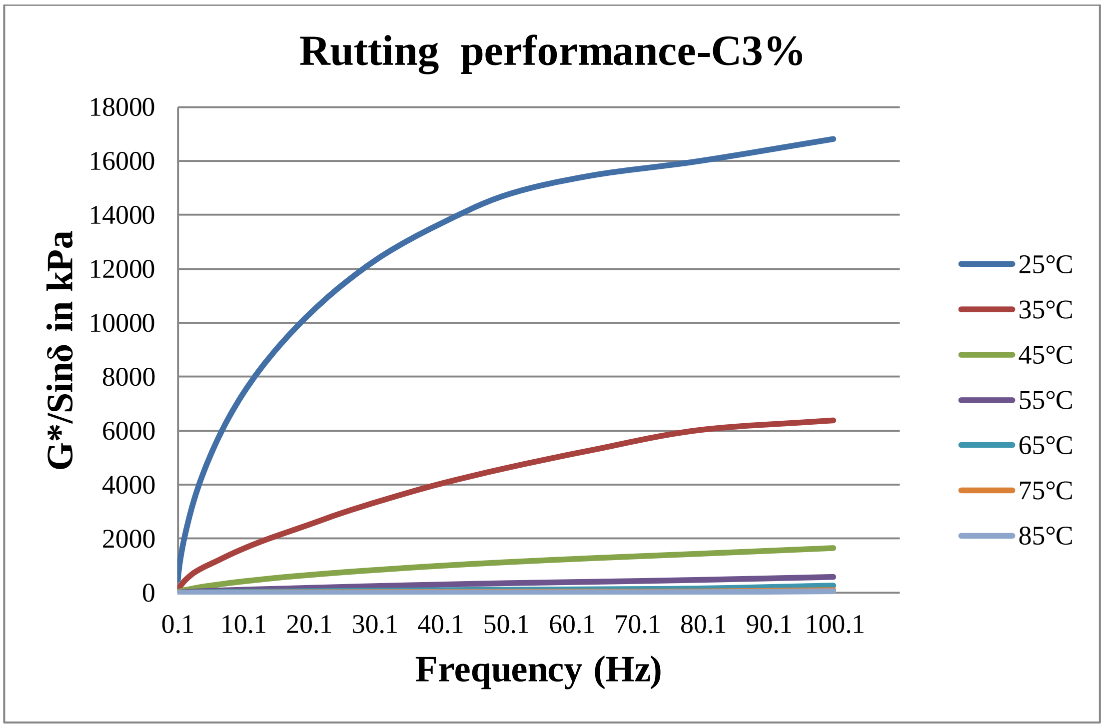
(a)


Rutting performance-C1%
G*/Sinδ in kPa
14000
12000
10000
8000
6000
4000
2000
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
25°C
35°C
45°C
55°C
65°C
75°C
85°C

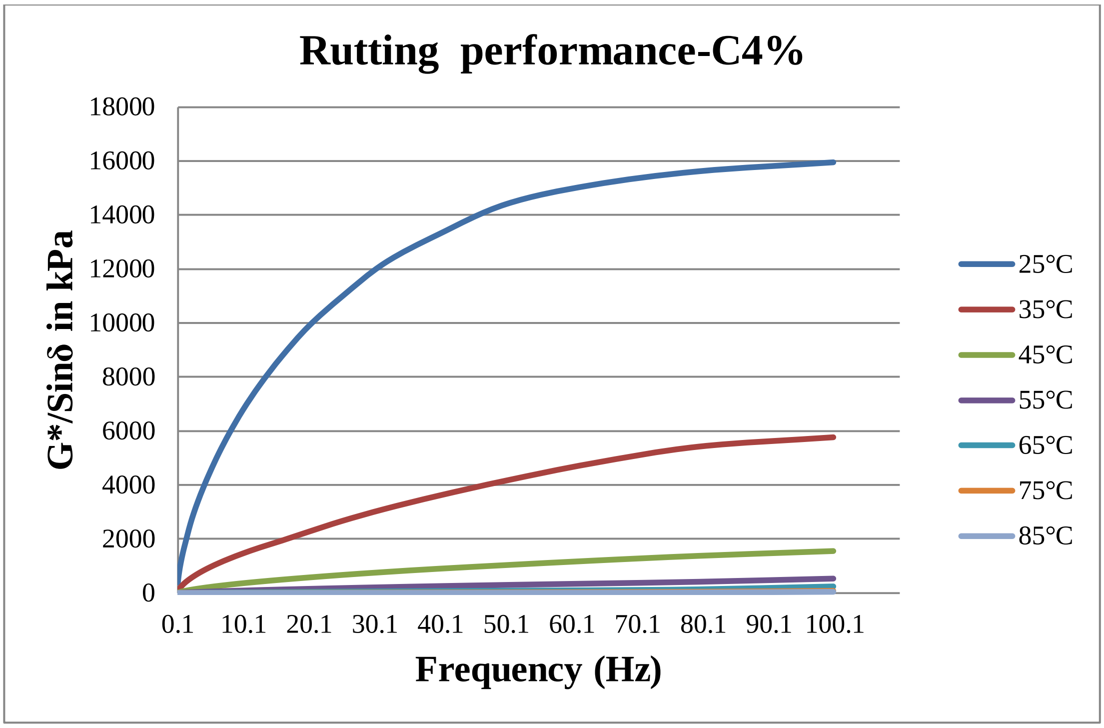
(b)


Rutting performance-C2%
G*/Sinδ in kPa
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
25°C
35°C
45°C
55°C
65°C
75°C
85°C

(c)


Rutting performance-C3%
G*/Sinδ in kPa
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1
Frequency (Hz)
25°C
35°C
45°C
55°C
65°C
75°C
85°C

(d)



(e)

Figure 4.10 Effect of Temperatures on Rutting Performance |G*|/sinδ

(a) at C0%, (b) at C1%, (c) at C2%, (d) at C3%, (e) at C4%

4.2.6.5 Effect of temperatures Complex Shear Modulus (G)*

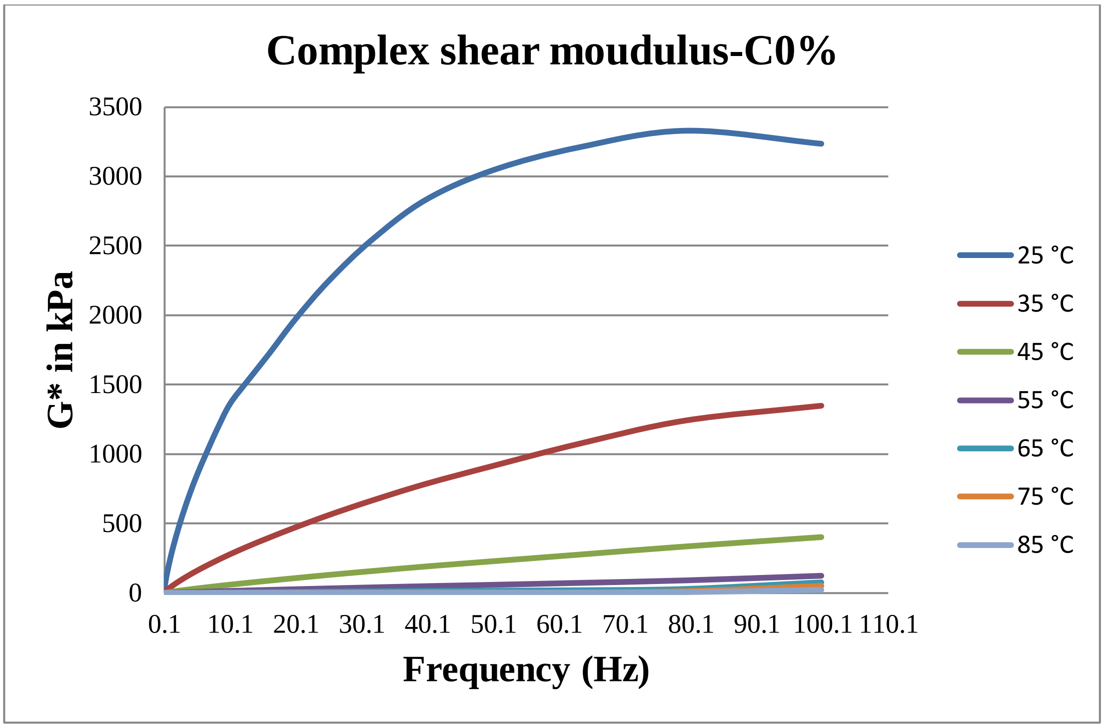
Figure 4.11 (a-e) shows the effect of temperature range from low to high on G* of C.B and M.B. G* gives the stiffness and elastic behavior of asphalt binder. In Figure 4.11 (a) shows that the value of G* of C.B at 65°C is 16.937 kPa at 50.1 Hz. Take this temperature as reference, if we decrease the temperature, the G* value increases and the highest value of G* is achieved is 3046.1 kPa at 25°C which indicates the stiffness of the binder and improves the elastic behavior of binder. As we increase the temperature beyond 65°C, the G* values will reduce. The values at 75°C and 85°C are 4.675 and 2.1371 kPa. This shows that the binder is converted into fluid and gives viscous behavior, also reduction in viscosity.

Figure 4.11 (b) shows the results of M.B at 1% addition of MCP. It can be seen that M.B shows better results as compared to control binder. Now the value of G* at 65°C is 75.54 kPa. This increment makes the binder stiff at higher temperature and gives better recovery results as compare to control binder. At 25°C the G*- value is 5639.5

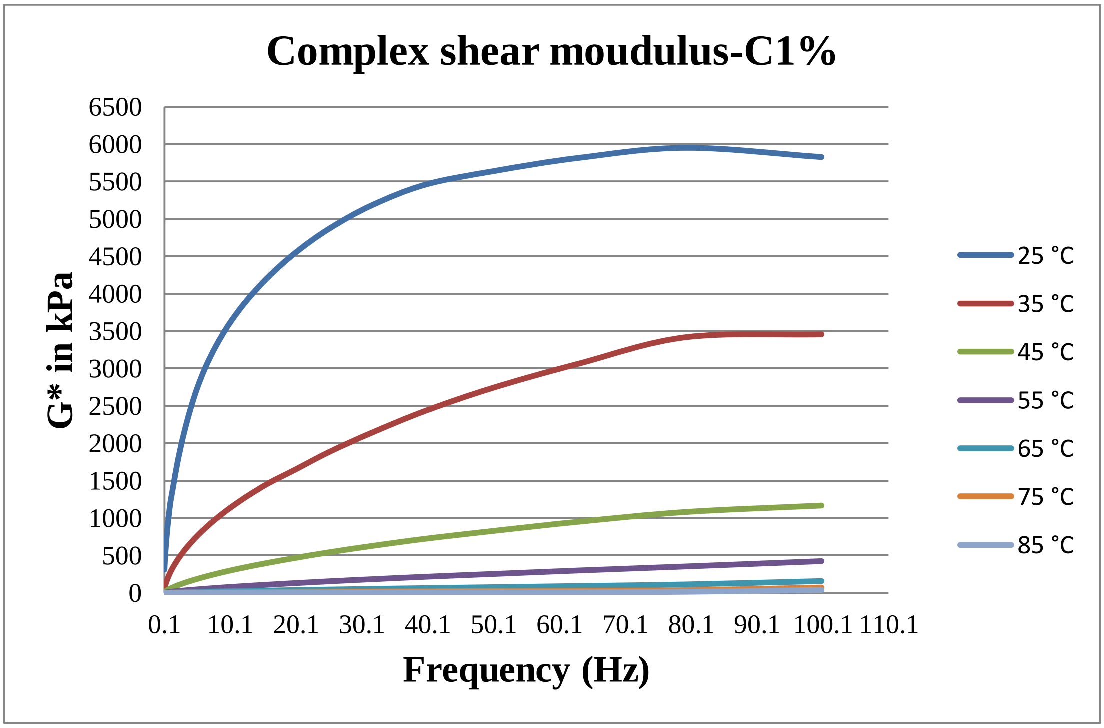
kPa which is 85.13% more than control binder. At 75°C and 85°C values are 23.296 and 7.5304 kPa, which is 398.26% and 252.36% higher than values of neat binder.

Figure 4.11 (c) shows the 2% MCP modified binder. This modified binder significantly improves the G* values as compared to control and 1% addition modified binders. The increased value of 2% addition of MCP at 65°C is 97.93 kPa which is 478.20% and 29.63% more than control and 1% modified binder respectively. As the temperature decrease, G* values increases and vice versa. The change in frequencies also effect the G* values as the frequency increases the G* values increases and vice versa. High frequency means low temperature and low frequency means high temperature. The G* values at different temperatures 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°Care 7372.3 kPa, 3288.4 kPa, 1022.8 kPa, 325.8 kPa, 97.93 kPa, 30.121 kPa and 9.58 kPa respectively.

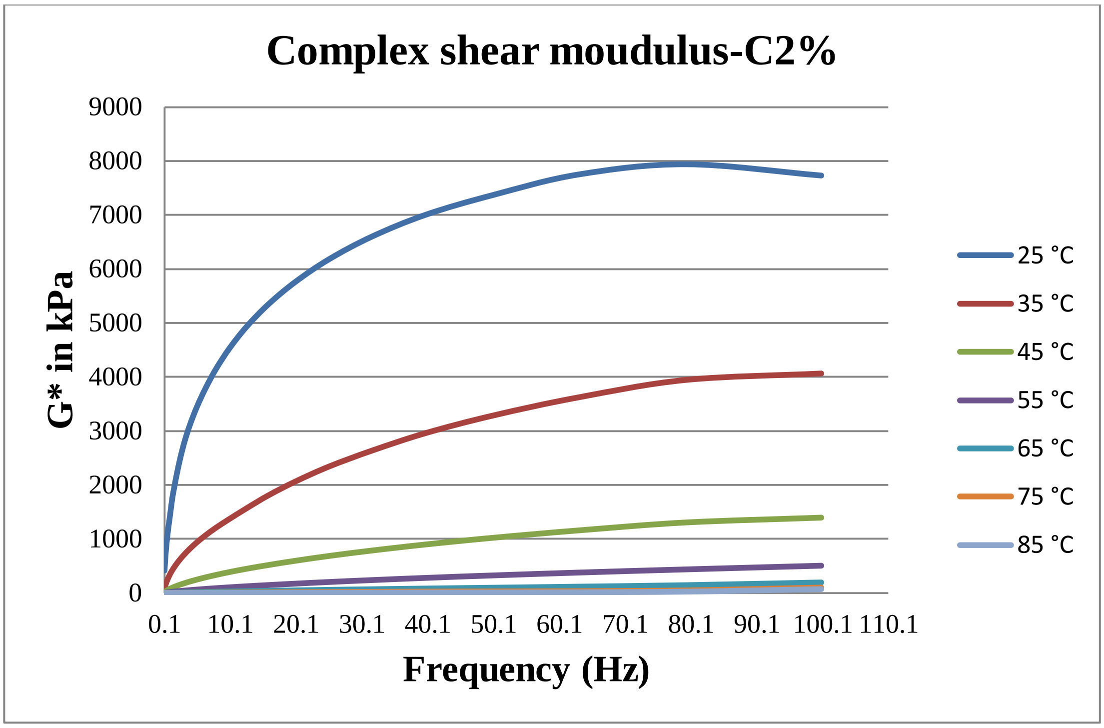
Figure 4.11 (d-e) shows that excessive addition of MCP in asphalt binder decrease the G* values. The trend of increase and decrease are same but the G* values for 3% and 4% addition of MCP in neat binder is less as compared to 2% addition of MCP. The values at 50.1 Hz for 3% modified binders at different temperatures 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°C are 6949.5 kPa, 3185.2 kPa, 961.3 kPa, 313.69 kPa, 97.39 kPa, 30.053 kPa, 9.6432 kPa and for 4% modified binder are 6739.1 kPa, 2936.7 kPa, 883.64 kPa, 272.02 kPa, 80.697 kPa, 24.97 kPa and 8.0167 kPa.


Complex shear moudulus-C0%
G* in kPa
3500
3000
2500
2000
1500
1000
500
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

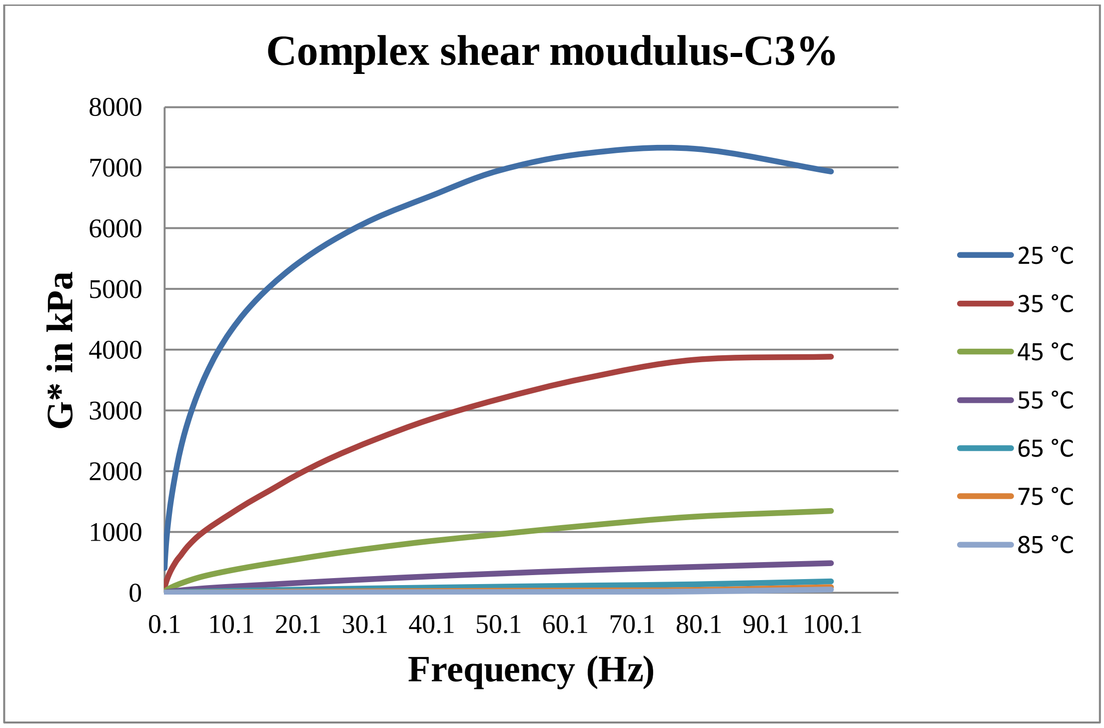
(a)


Complex shear moudulus-C1%
G* in kPa
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
0.1 10.1 20.1 30.1 40.1 50.1 60.1 70.1 80.1 90.1 100.1 110.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

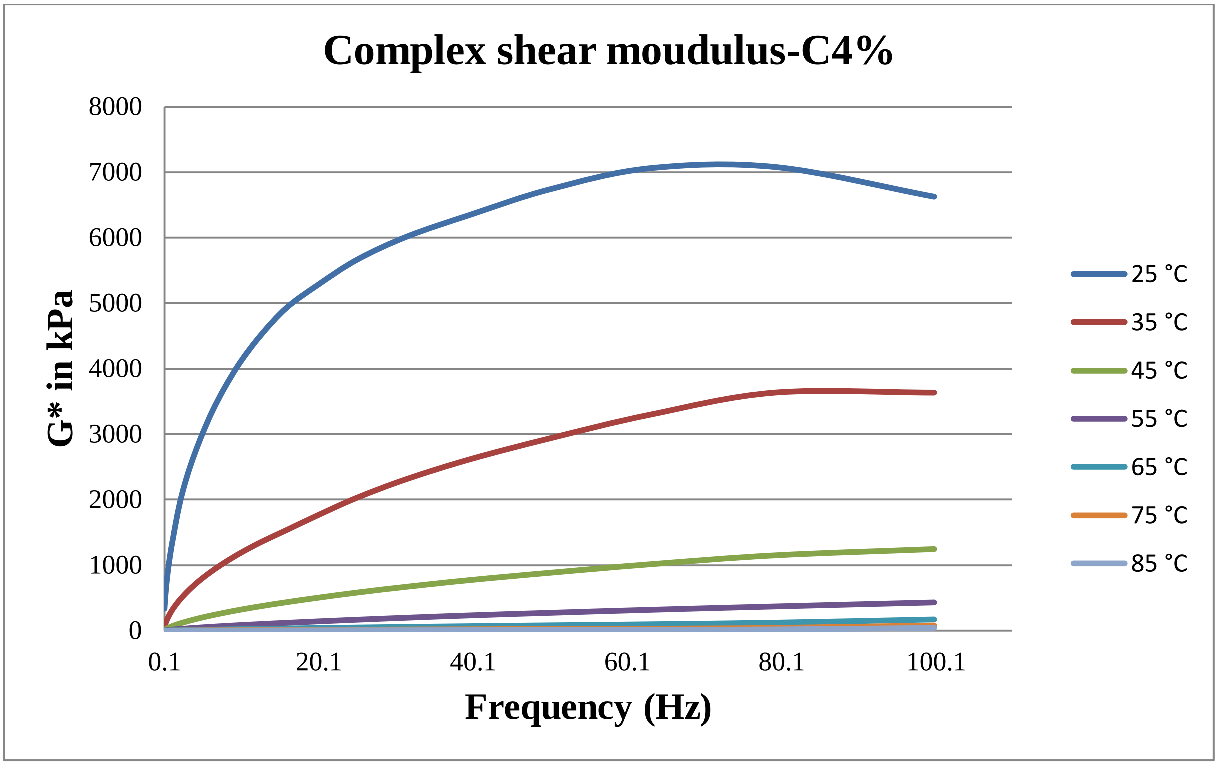
(b)


Complex shear moudulus-C2%
G* in kPa
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

(c)


Complex shear moudulus-C3%
G* in kPa
8000
7000
6000
5000
4000
3000
2000
1000
0
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

(d)



(e)

Figure 4.11 Effects of Temperatures on Complex Shear Modulus (G*)

(a) at C0%, (b) at C1%, (c) at C2%, (d) at C3%, (e) at C4%

4.2.6.6 Effect of temperatures Phase Shift Angle (δ)

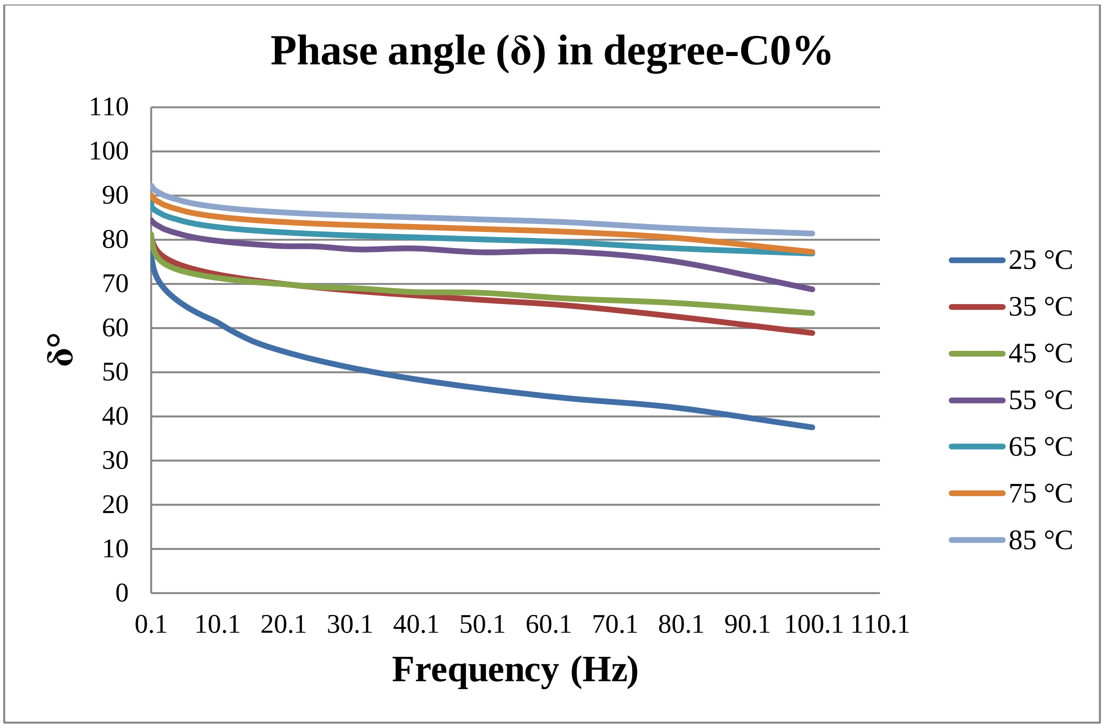
Figure 4.12 (a-e) shows the phase shit angle of control and modified binders. It gives the binder's elastic and viscous behavior. Figure 4.12 (a) shows that phase angle is less at low temperature i.e. at 25°C and higher at high temperature i.e. 85°C and one more thing was noticed that at high frequencies phase angle is less as compare to low frequencies. This is because of phase angle is directly related to temperature and frequency range. After 55°C temperature binder reaches the 90° which shows highly viscous binder it means control binder is unable to recover at higher temperature and also shows permanent deformation. It was also noticed that after 55°C the values of phase angle are almost near to each other and at 35°C and 45°C values are overlapping at low frequency range. Maximum value of phase angle is obtained at 25°C as compared to other temperature. The values of phase angle at different temperatures 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°C are 46.26°, 66.33°, 67.92°, 77.10°, 80.01°, and 84.53°.

Figure 4.12 (b) shows the 1% addition of MCP modified binder. The values in phase angle for modified binder are reduced. This is the due to uniform dispersion of MCP in asphalt binder. The reduction in δ values is 29.09° (37.11%), 45.59° (31.26%), 60.09° (11.52%), 67.36°(12.63%), 71.36° (10.82%), 75.36° (8.51%), and 83.54° (7.17%) for 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°C respectively. This means modified binder helps to increase the elastic and make the asphalt binder rutting resistance.

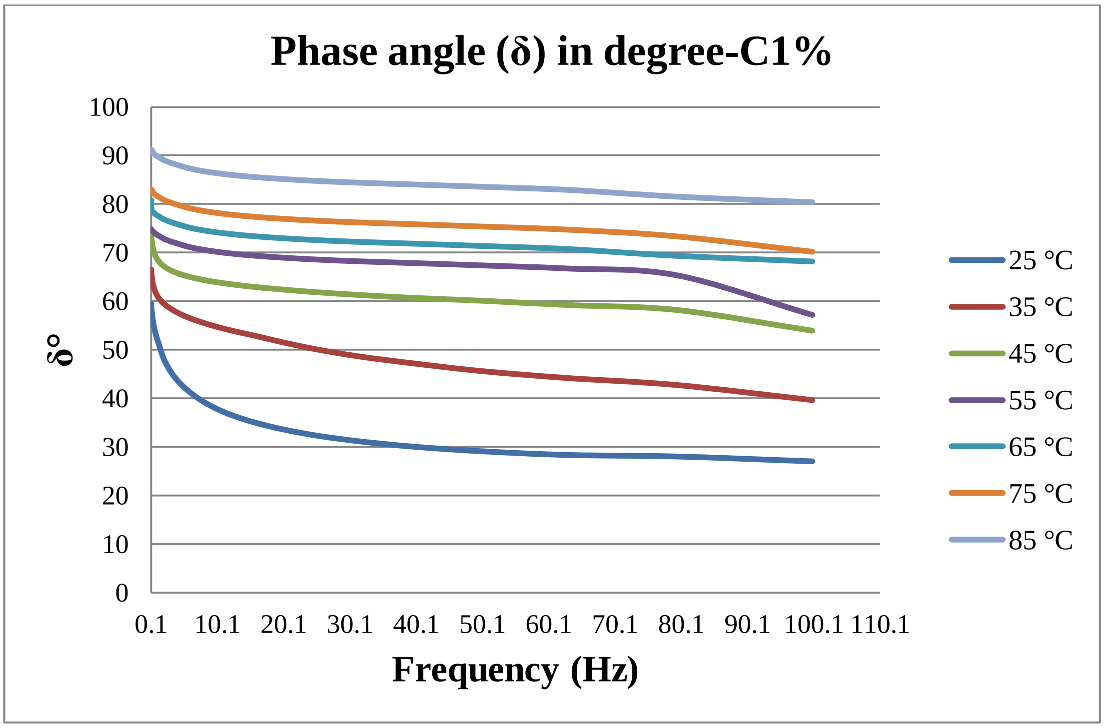
Figure 4.12 (c) shows that 2% addition modified binder gives best results as compared to control and other modified binders because maximum reduction in phase angle is due to 2% MCP in neat binder. The phase angle at 55°C and 65° is almost near due to difference in phase angle on both the temperature is 1.9°. The reduction in phase angle values at 50.1 Hz are as compared to control binder are 24.64° (46.73%). 40.9° (38.33%), 54.74° (19.40%), 62.77° (18.58%), 64.67° (19.18%), 72.86° (11.54%) and 81.035° (4.12%) at different temperatures 25°C, 35°C, 45°C, 55°C, 65°C, 75°C and 85°C respectively.

Figure 4.12 (d-e) shows the reduction in phase angle as compared to other modified binders. The values for 3% modified binder are 26.26°, 43° 57.2°, 64.34°, 67.34°, 73.88° and 82.04°. For 4% modified binder is 27.3°, 44.34°, 58.79°, 65.65°, 68.65°, 74.36° and 82.63.

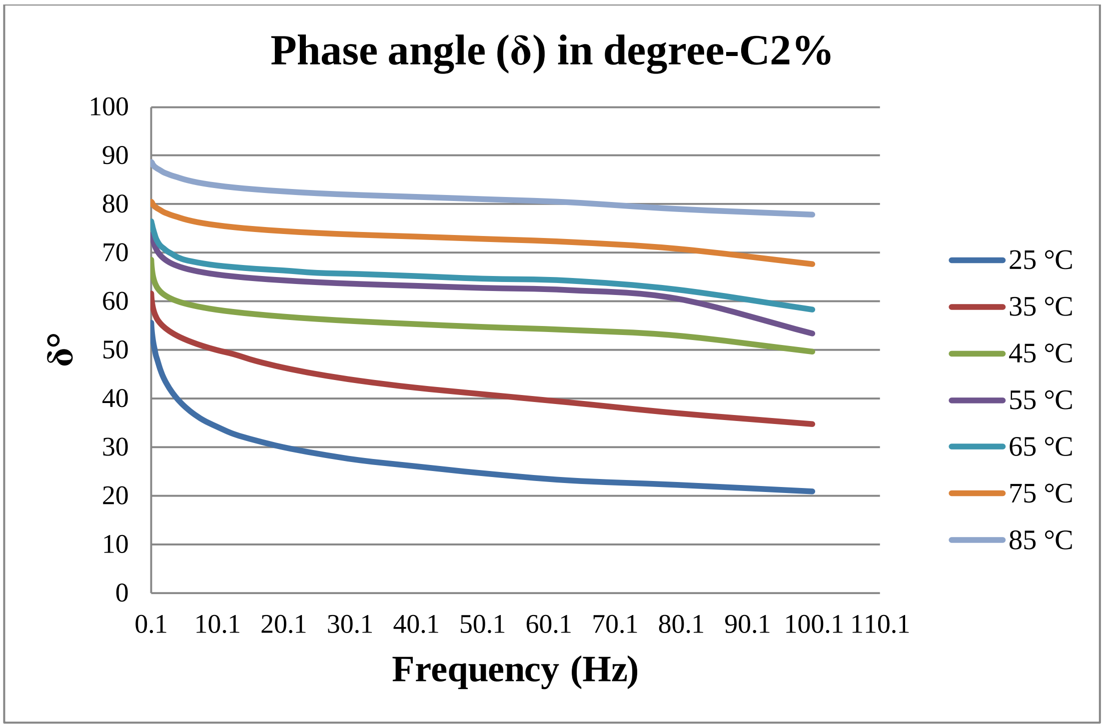
From the data, it is concluded that 2% addition of MCP modified binder gives best results i.e. maximum reduction in phase angle.


Phase angle (δ) in degree-C0%
110
100
90
80
70
60
50
40
30
20
10
0
δ°
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

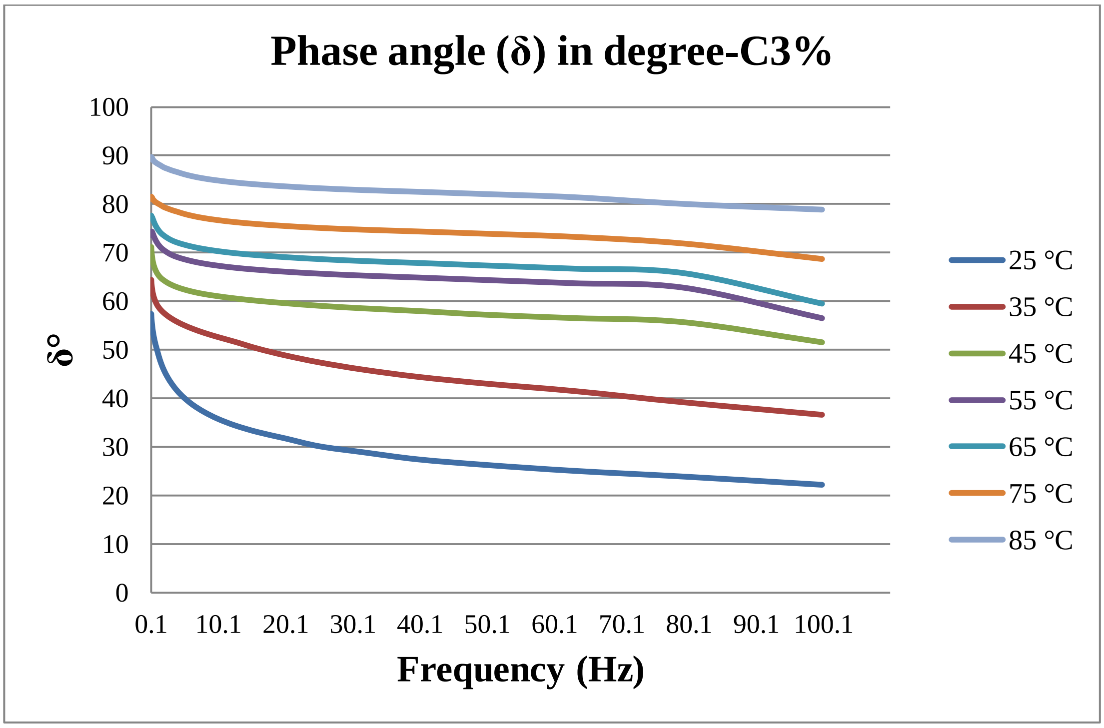
(a)


Phase angle (δ) in degree-C1%
100
90
80
70
60
50
40
30
20
10
0
δ°
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

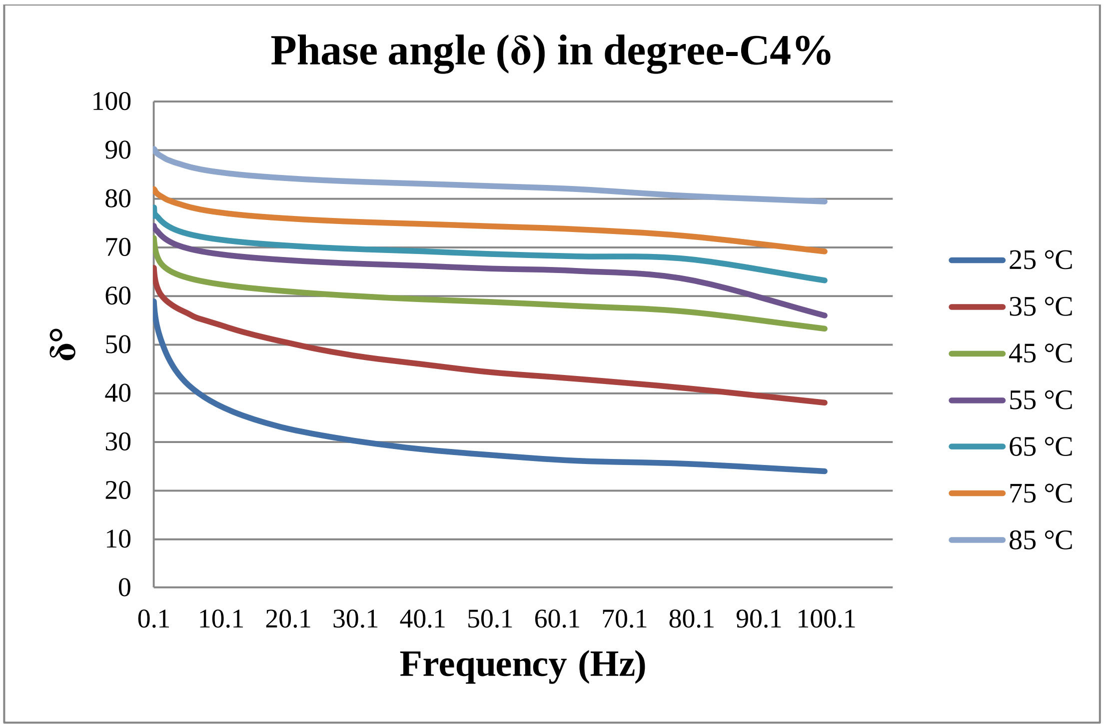
(b)


Phase angle (δ) in degree-C2%
100
90
80
70
60
50
40
30
20
10
0
δ°
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
110.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

(c)


Phase angle (δ) in degree-C3%
100
90
80
70
60
50
40
30
20
10
0
δ°
0.1
10.1
20.1
30.1
40.1
50.1
60.1
70.1
80.1
90.1
100.1
Frequency (Hz)
25 °C
35 °C
45 °C
55 °C
65 °C
75 °C
85 °C

(d)



(e)

Figure 4.12 Effects of Temperatures on Phase Shift Angle (δ)

(a) at C0%, (b) at C1%, (c) at C2%, (d) at C3%, (e) at C4%

4.2.7 Multiple Stress Creep Recovery Test (MSCR)

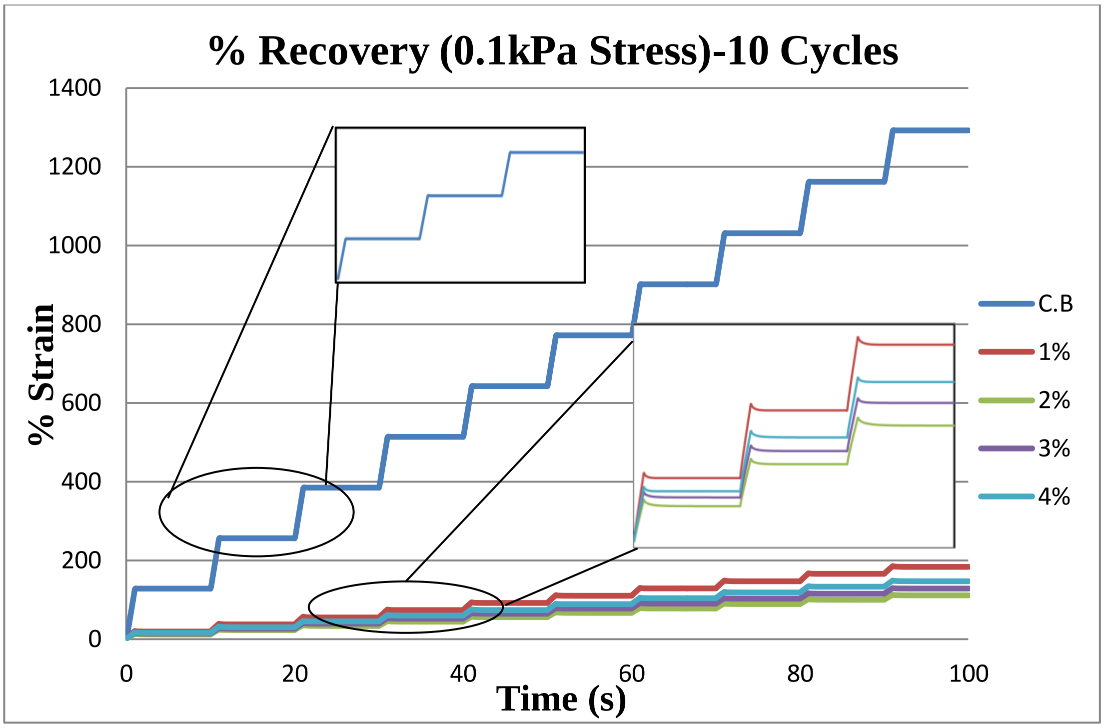
4.2.7.1 Effect of different Creep Stresses on C.B and M.B under 10 cycles loading

Oscillation sweep test works with small change in strain value within LVE range and rutting resistance parameter from oscillation test does not show actual resistance of rutting of binder. Furthermore oscillation test applies reversible shear cyclic loading, which is unable to simulate the irreversible deformation of rutting.

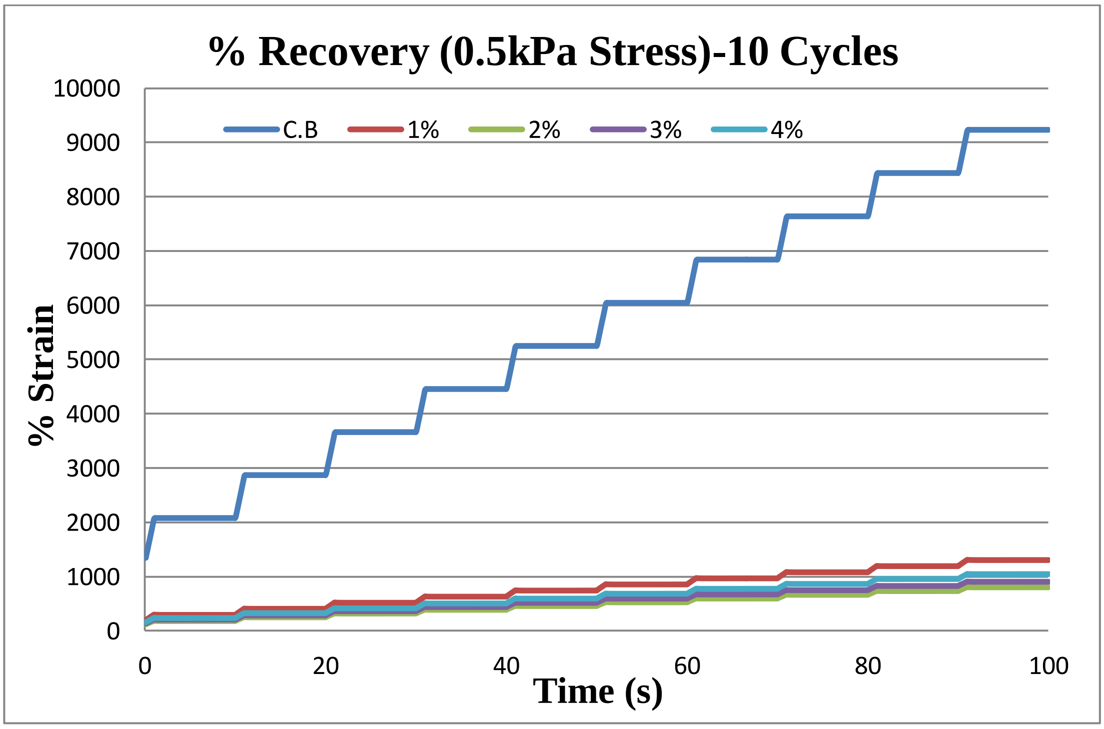
MSCR is a test in performance grading of binder performed according to AASHTO TP70 which uses the high temperature (65°C) to evaluate the rutting resistance performance of the control and modified binders under 10 and 30 cycles for each all eight loading stresses such as 0.1 kPa, 0.5 kPa, 1 kPa, 1.5 kPa, 2 kPa, 2.5 kPa, 3 kPa, 3.2 kPa. All the stresses were applied on the same sample. This test analyses the potential of binder to permanent deformation and delayed in elastic response under creep loading and recovery, which are simulative of field loading and also check the stress dependent behaviour of binders. Percentage accumulated strain verses loading and unloading time graphs were plotted for 10 cycles as shown in Figure 4.13. it is observed that after applying the load stresses control binder doesn't show any recovery at unloading time but after the addition of coconut shell MCP the accumulated creep decreases and modified binder gives recovery which means after modification the elastic response increases. At 2% addition of MCP, modified binder gives better results as compared to other M.B as shown in Figure 4.13 (a-h) because after addition of 2% MCP i.e. at 3% addition accumulated strains starts increasing as shown in Figure 4.13 (a-h).

It is also observed that at low creep stress, control binder (CB) and modified binder show less accumulated creep strain as shown in Figure 4.13 (a) but as the creep stresses increases the accumulated strain also increases for all creep stress levels after 0.1 kPa creep stresses. The addition of MCP in the bitumen significantly improved the creep-recovery behavior of the bitumen indicating enhanced rutting performance. The %strain underwent by the modified binder for a given stress level reduced significantly when MCP was added in the bitumen. It is also observed that for all creep stress levels 2% addition of modified binder has less accumulated creep strain. This can be mainly attributed to the filler effect of coconut charcoal powder, where the applied creep load is shared between charcoal powder and bitumen enhancing the stiffness of bitumen leading to lower strain. So MCP improves the elastic behaviour of conventional binder to resistance to permanent deformation.

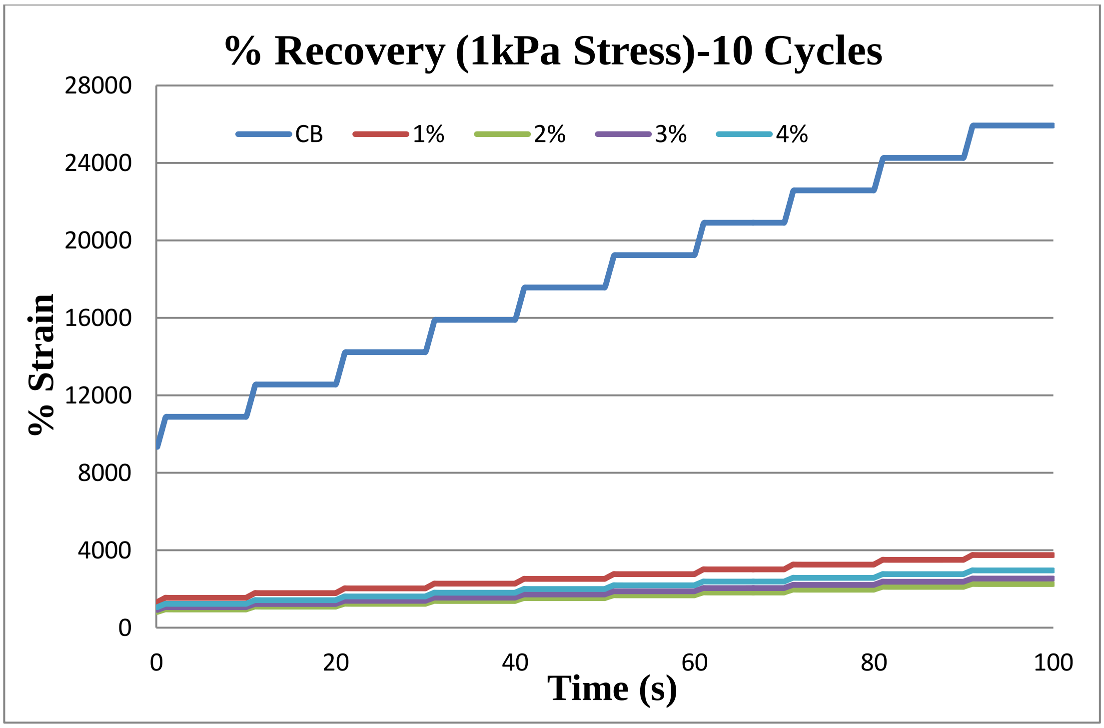
From Figure 4.13 (a-h) it is observed that at higher creep stress values the accumulated strain values are much greater than low stress values for control binder. This is due to at high stresses the binder started flowing.


% Recovery (0.1kPa Stress)-10 Cycles
1400
1200
1000
800
600
400
200
0
% Strain
0
20
40
60
80
100
Time (s)
C.B
1%
2%
3%
4%

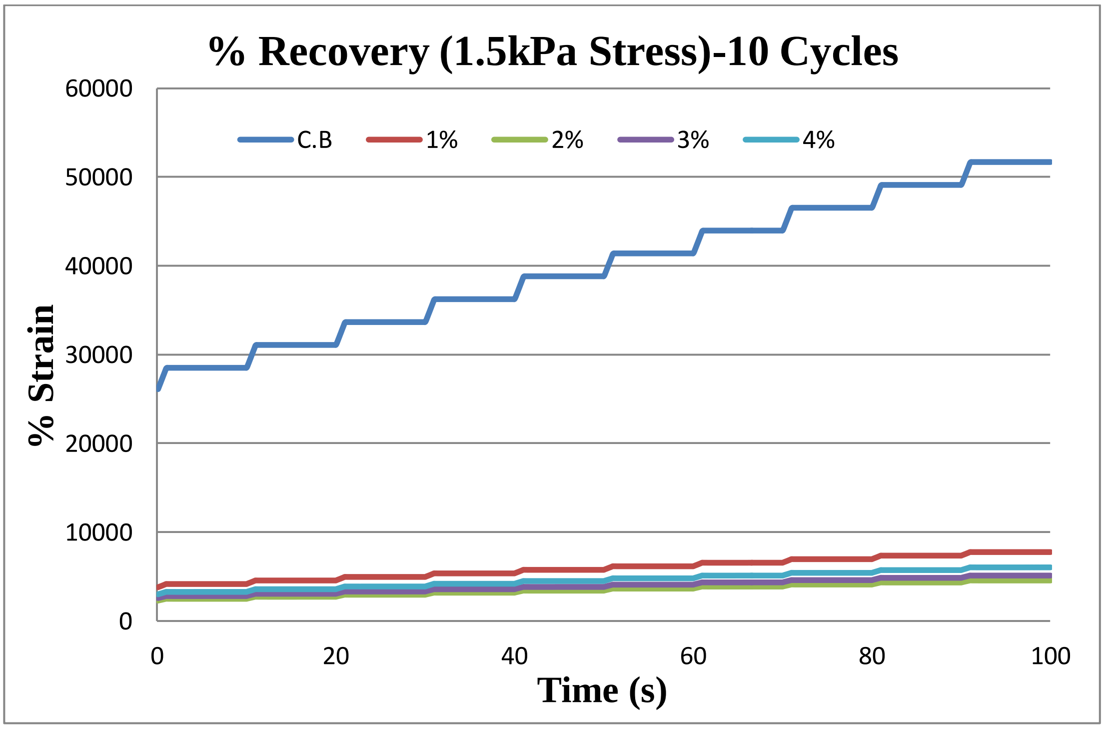
(a)


% Recovery (0.5kPa Stress)-10 Cycles
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
% Strain
C.B
1%
2%
3%
4%
0
20
40
60
80
100
Time (s)

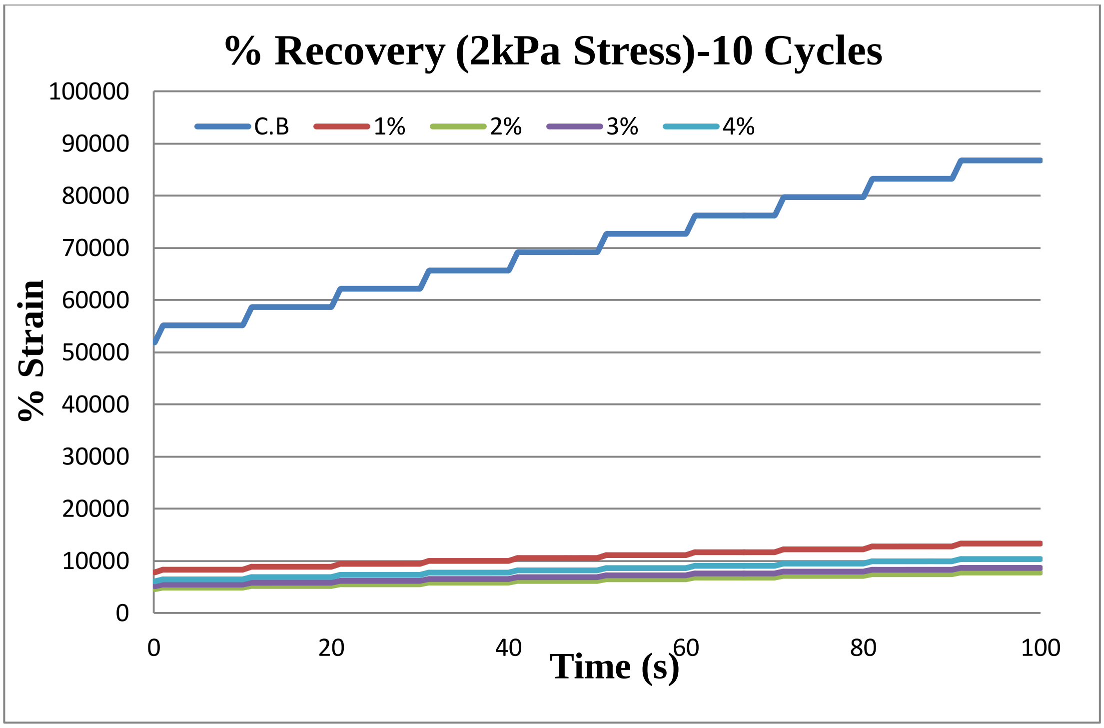
(b)


% Recovery (1kPa Stress)-10 Cycles
CB
1%
2%
3%
4%
% Strain
28000
24000
20000
16000
12000
8000
4000
0
0
20
40
60
80
100
Time (s)

(c)

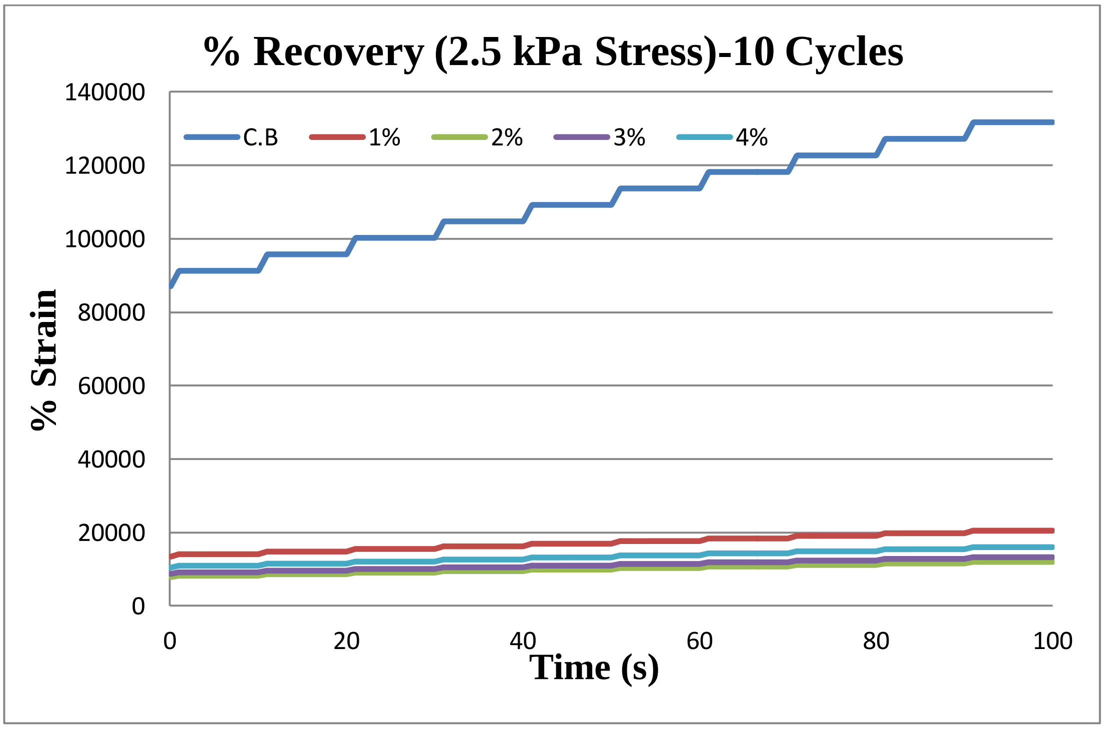

% Recovery (1.5kPa Stress)-10 Cycles
C.B
1%
2%
3%
4%
% Strain
60000
50000
40000
30000
20000
10000
0
0
20
40
60
80
100
Time (s)

(d)



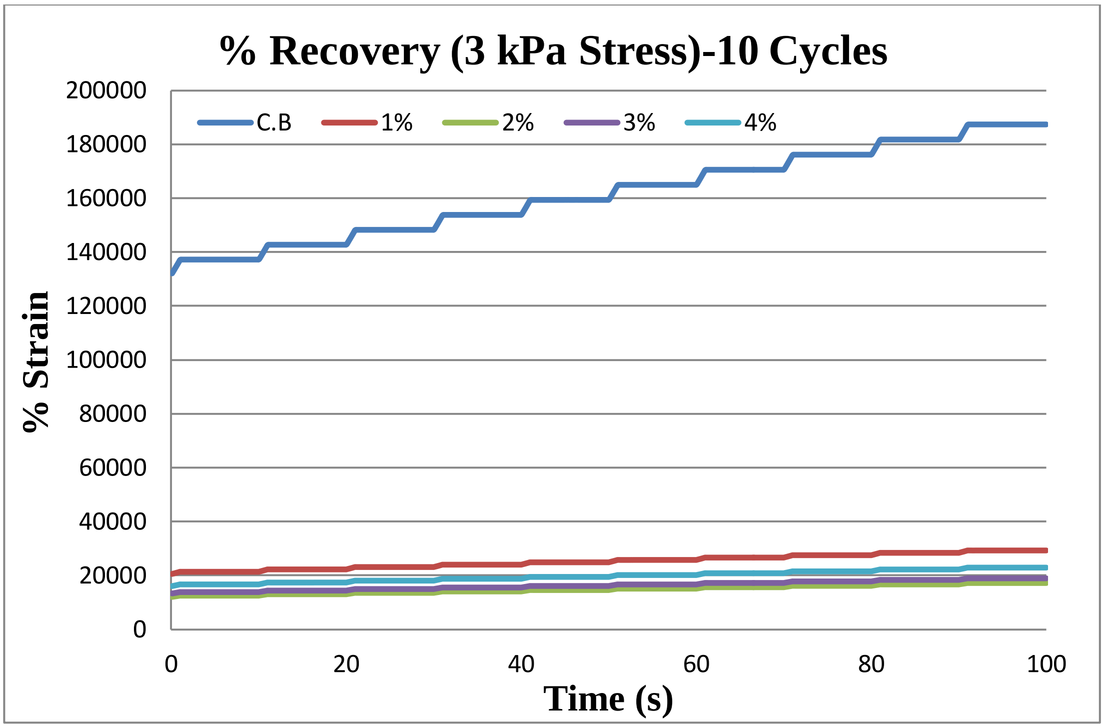
% Recovery (2kPa Stress)-10 Cycles
C.B
1%
2%
3%
4%
% Strain
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
0
20
40
60
80
100
Time (s)

(e)

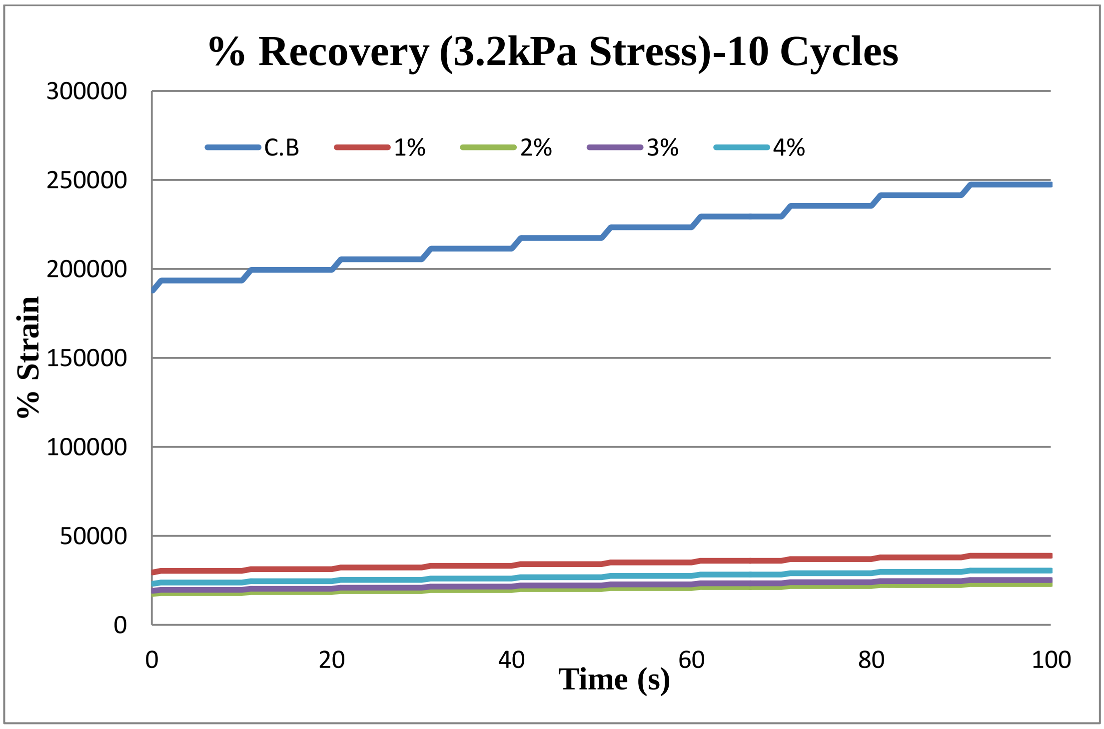


% Recovery (2.5 kPa Stress)-10 Cycles
C.B
1%
2%
3%
4%
% Strain
140000
120000
100000
80000
60000
40000
20000
0
0
20
40
60
80
100
Time (s)

(f)



(g)

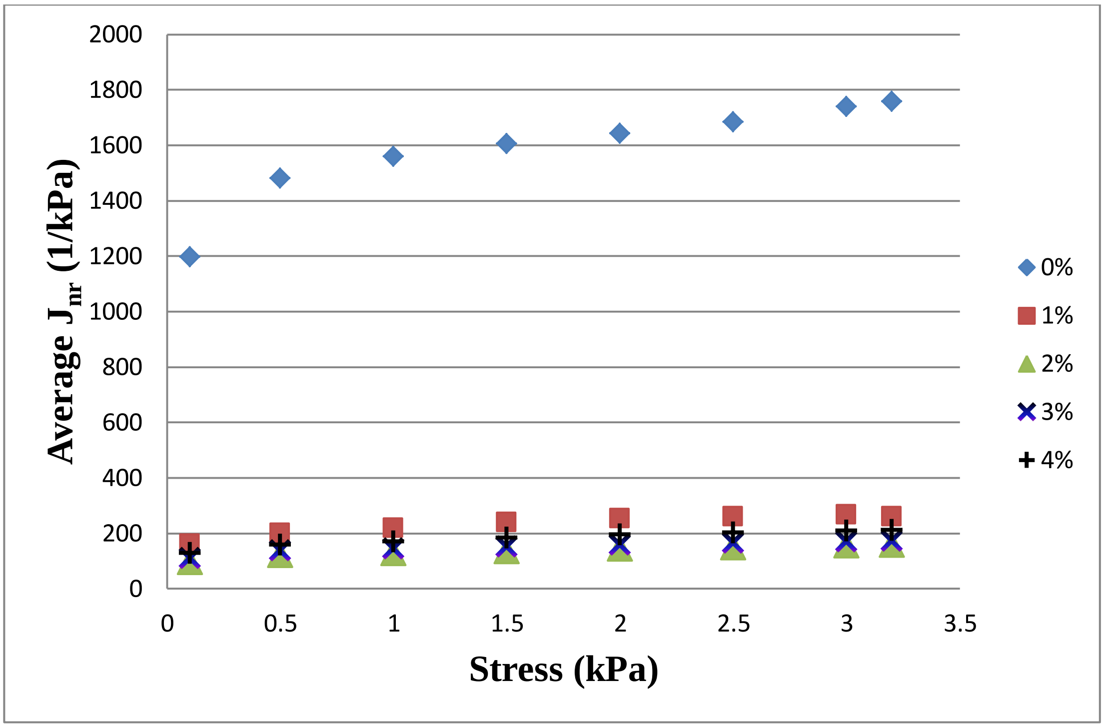


(h)

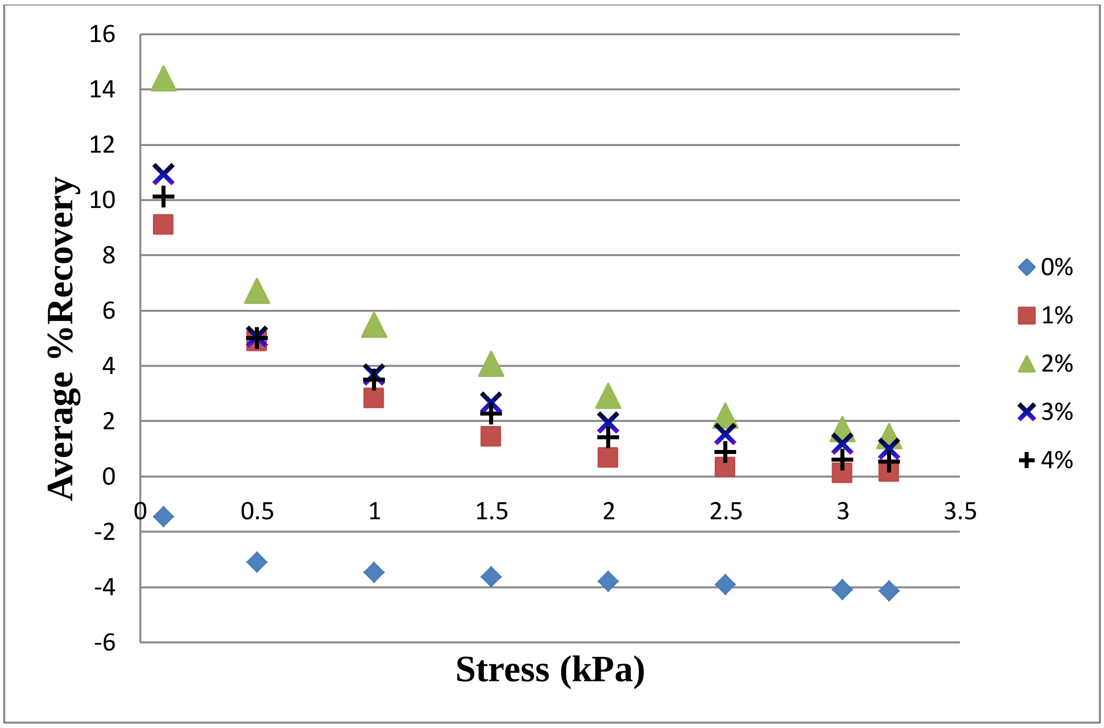
Figure 4.13 %Strain vs. Time Graphs

(a) at 0.1 kPa; (b) 0.5 kPa (c) 1 kPa; (d) 1.5 kPa; (e) 2 kPa; (f) 2.5 kPa ;(g) 3kPa and (h) 3.2 kPa Creep Stresses

Figure 4.8 (a) shows that creep stress of 0.1 kPa for control binder is less, as the 0.5 kPa stress is applied the sudden increase in J_{nr} which shows control binder lost its linear viscoelastic range and at higher stress i.e. 3 kPa the J_{nr} value subsequently increase shows more non linearity. This may be due to the breakup of the structural network and unable to resist the deformation. But modified binder does not show sudden change after modification. The viscoelastic behaviour of binders also analysed through %recovery. From Figure 4.8 (b) shows that average percentage recovery for modified binder is higher than control binder and also show viscoelastic behaviour at low creep stresses but as the increase in stresses binder moves towards viscous range when recovery starts decreasing. In this control binder shows the negative values of recovery for all the creep stresses levels because load is not removed during testing which indicates the strain increasing under zero.



(a)



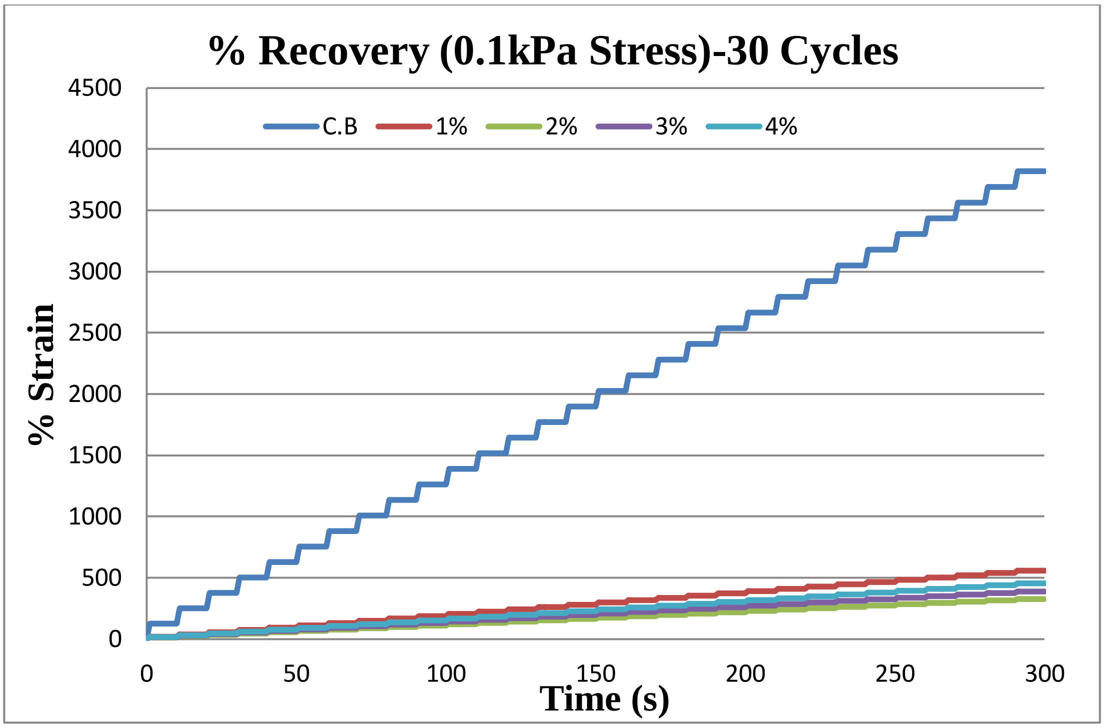
(b)

Figure 4.14 Average Cumulative (J_{nr}) and %R for C.B and M.B

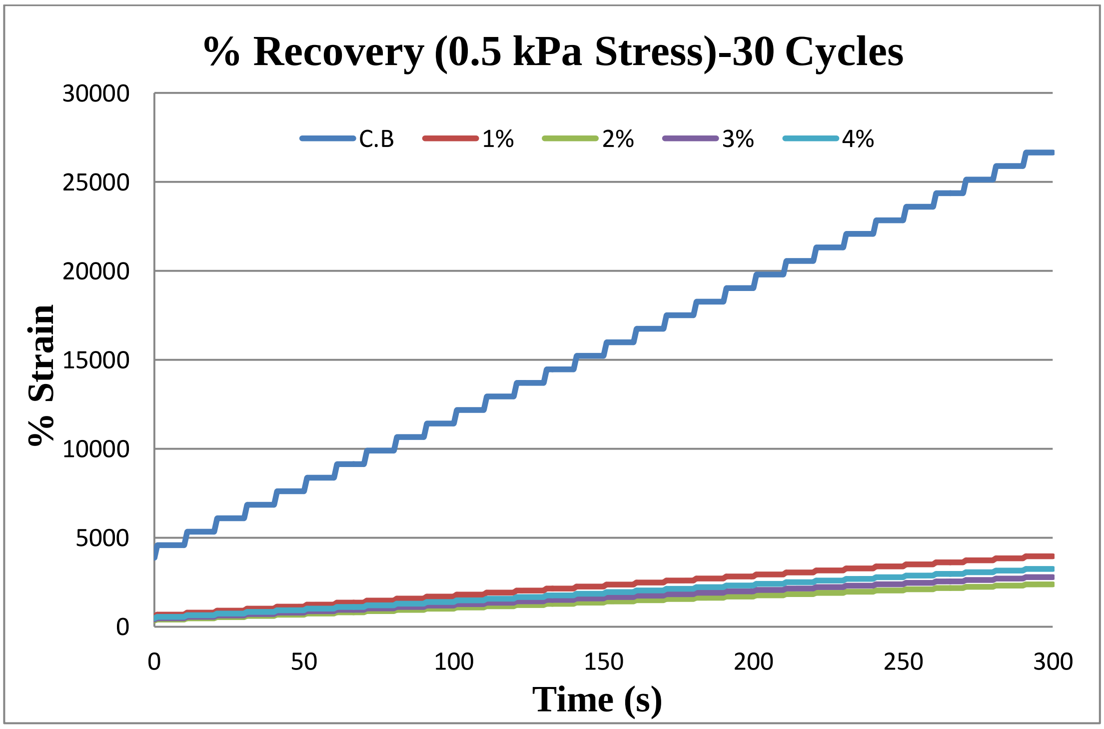
(a) Average Cumulative J_{nr} (b) Average Cumulative %R under 10 Cycle Loading

4.2.7.2 Effect of different Creep Stresses on C.B and M.B under 30 cycles loading

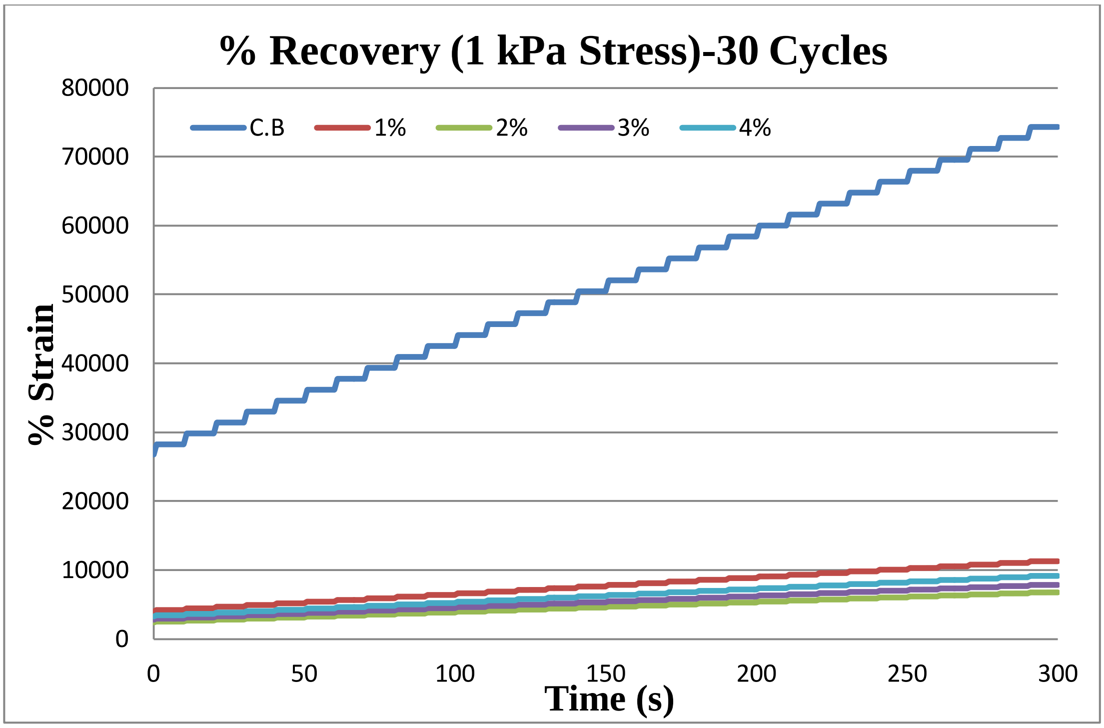
In the second MSCR test, control and modified binders are subjected to 30 cycles for each stress levels such as 0.1 kPa, 0.5 kPa, 1 kPa, 1.5 kPa, 2 kPa, 2.5 kPa, 3 kPa, 3.2 kPa. New samples are used for 30 cycles and again all the stresses are applied on same sample, total three trials are run i.e. total three samples used to analyse the stress behaviour of binders. Some studies have shown that 10 cycles may be are insufficient to stress the modified binders and hence 30 cycles of creep-recovery was used in addition to 10 cycles. Figure 4.15 (a-h) shows that at higher stress accumulated strains values are higher same as 10 cycles MSCR test.


% Recovery (0.1kPa Stress)-30 Cycles
C.B
1%
2%
3%
4%
% Strain
4500
4000
3500
3000
2500
2000
1500
1000
500
0
0
50
100
150
200
250
300
Time (s)

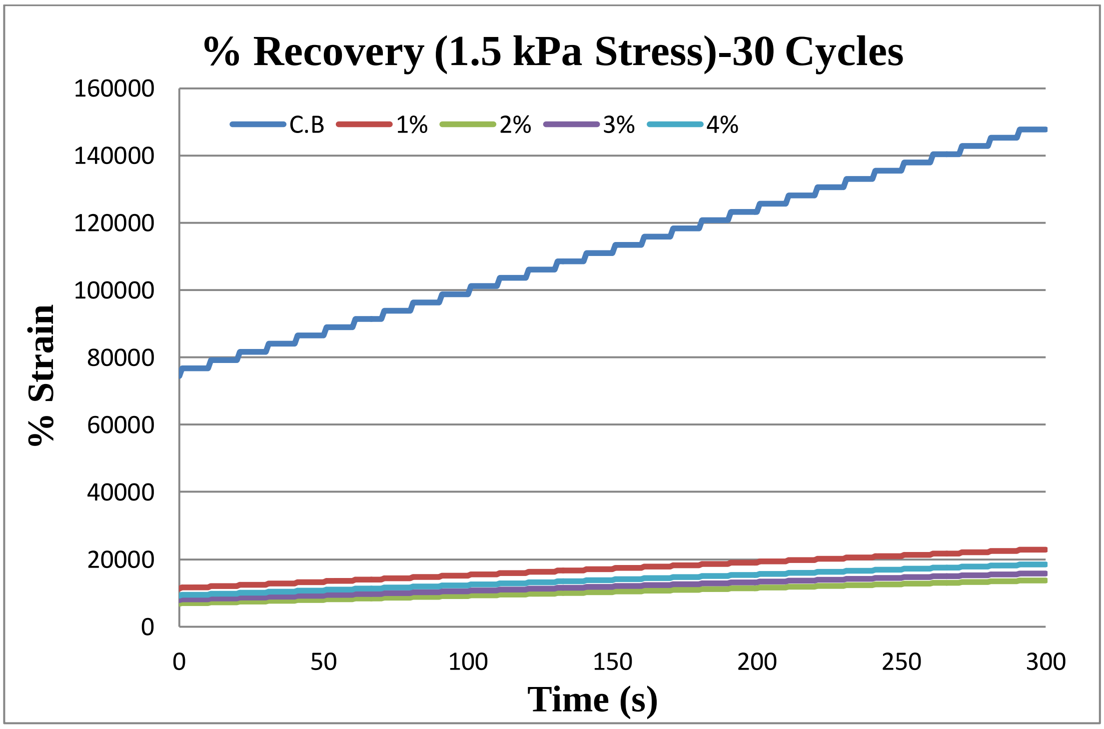
(a)


% Recovery (0.5 kPa Stress)-30 Cycles
C.B
1%
2%
3%
4%
% Strain
30000
25000
20000
15000
10000
5000
0
0
50
100
150
200
250
300
Time (s)

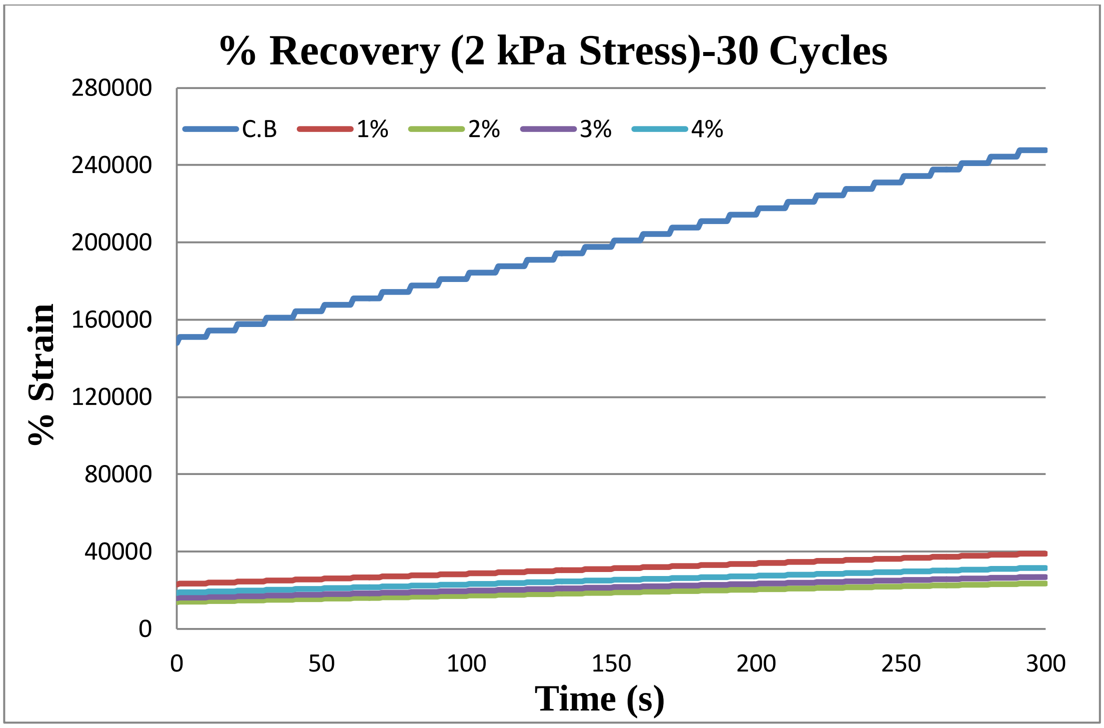
(b)


% Recovery (1 kPa Stress)-30 Cycles
C.B
1%
2%
3%
4%
% Strain
80000
70000
60000
50000
40000
30000
20000
10000
0
0
50
100
150
200
250
300
Time (s)

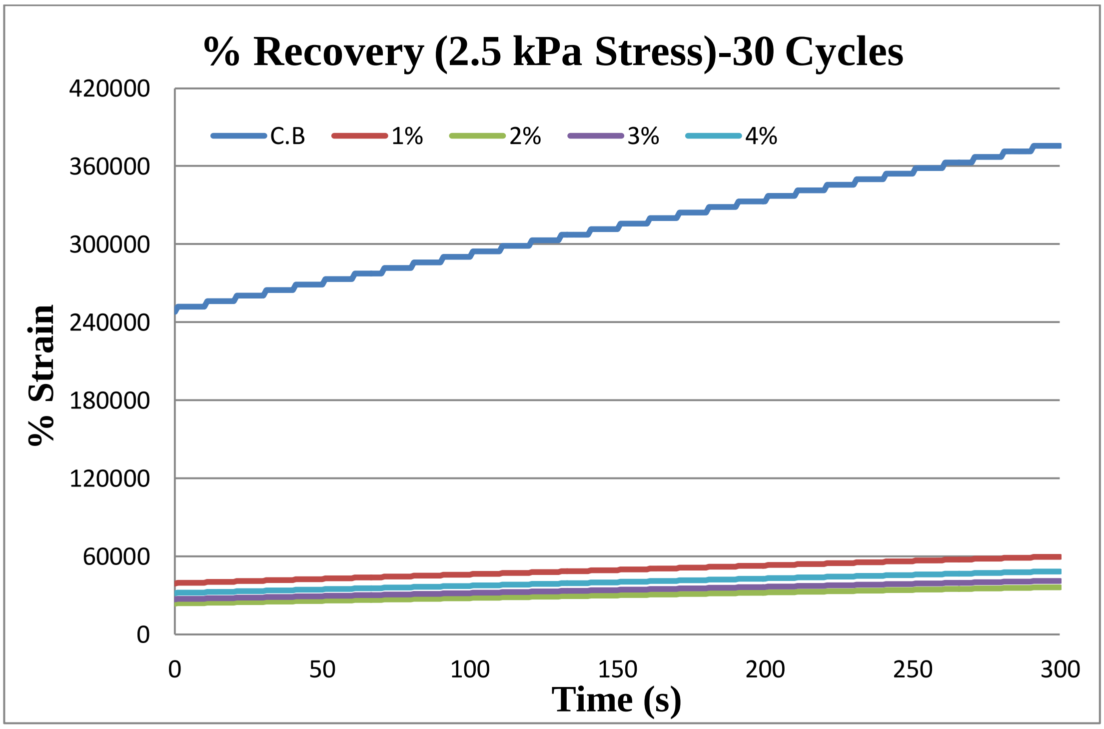
(c)


% Recovery (1.5 kPa Stress)-30 Cycles
C.B
1%
2%
3%
4%
% Strain
160000
140000
120000
100000
80000
60000
40000
20000
0
0
50
100
150
200
250
300
Time (s)

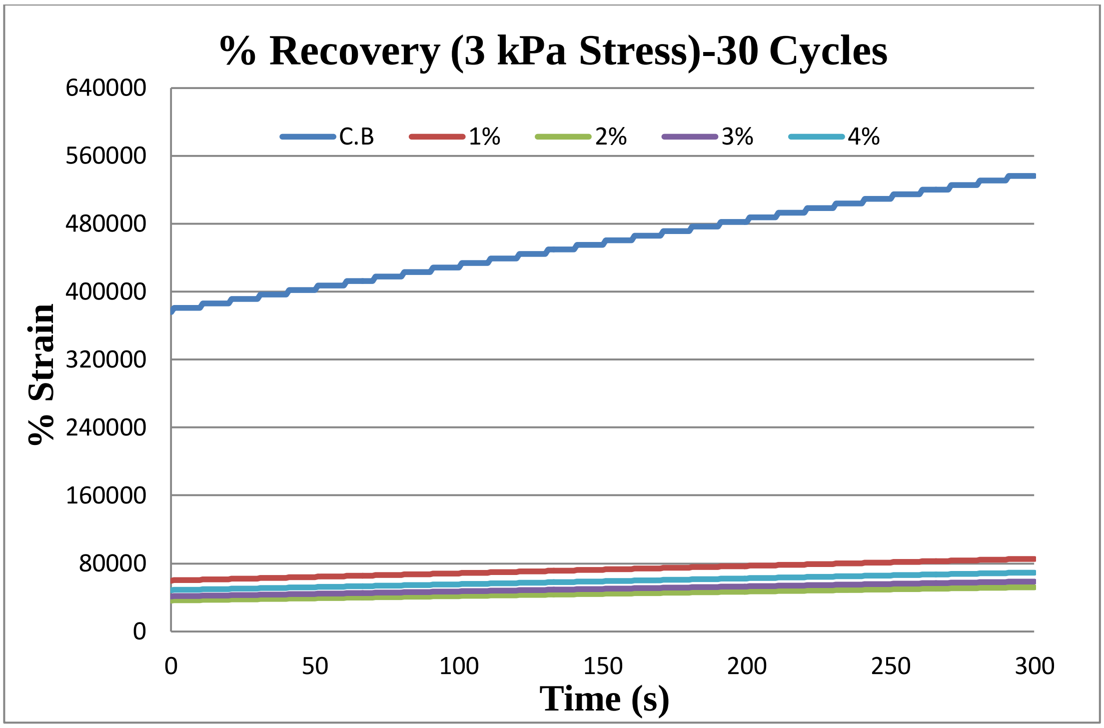
(d)


% Recovery (2 kPa Stress)-30 Cycles
280000
240000
200000
160000
120000
80000
40000
0
% Strain
C.B
1%
2%
3%
4%
0
50
100
150
200
250
300
Time (s)

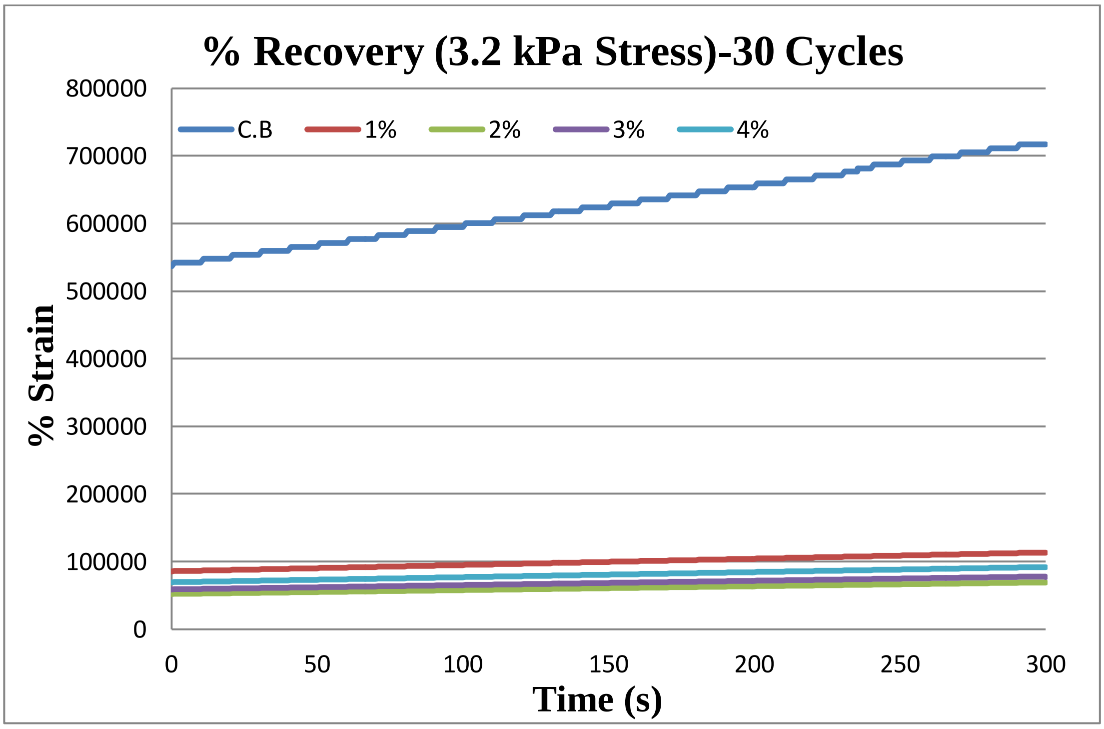
(e)


% Recovery (2.5 kPa Stress)-30 Cycles
420000
360000
300000
240000
180000
120000
60000
0
% Strain
C.B
1%
2%
3%
4%
0
50
100
150
200
250
300
Time (s)

(f)



(g)

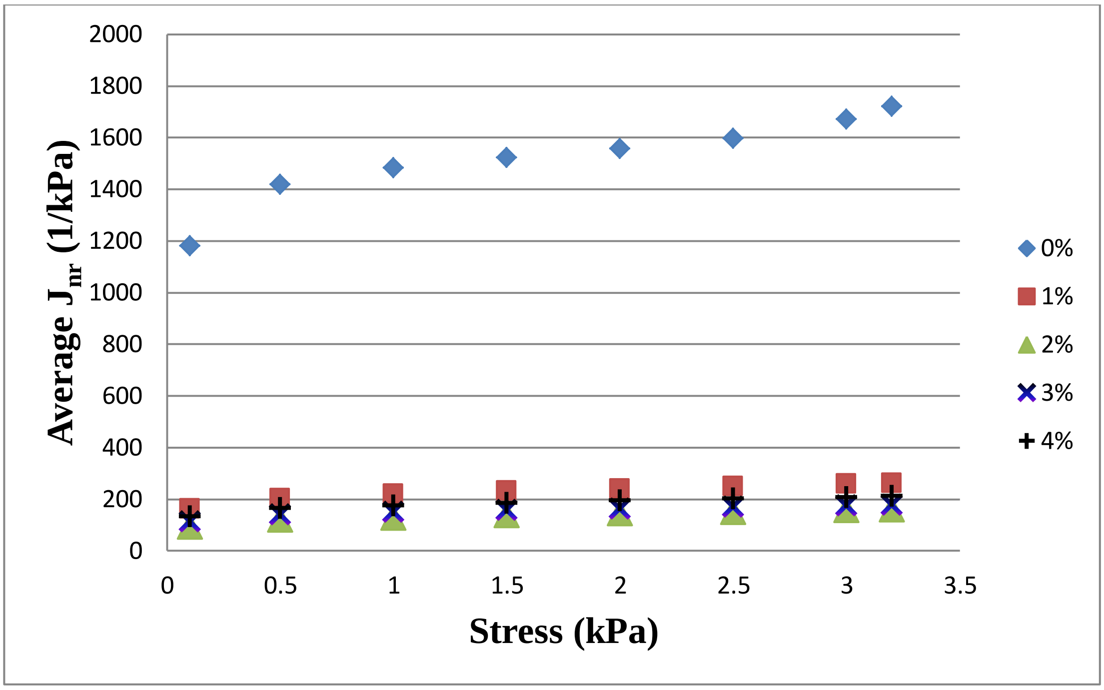


(h)

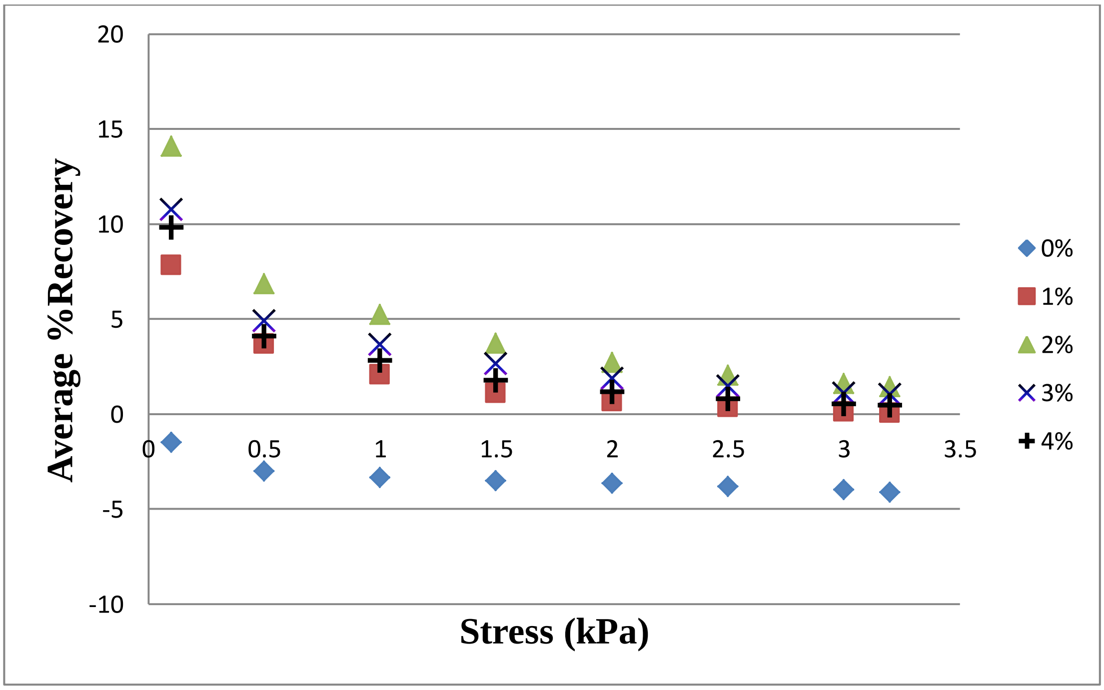
Figure 4.15 %Strain vs. Time Graphs

(a) at 0.1 kPa; (b) 0.5 kPa (c) 1 kPa; (d) 1.5 kPa; (e) 2 kPa; (f) 2.5 kPa ;(g) 3kPa and (h) 3.2 kPa Creep Stresses

Figure 4.16 (a) shows the same trend as in 10 cycles test. As the stress level vales are increasing the creep compliance values of C.B and M.B are also increasing and decrease in resistance to deformation. The creep compliance values for 30 cycles are more as compared to 10 cycles test and percentage recovery is less for 30 cycles test.



(a)



(b)

Figure 4.16 Average Cumulative (Jnr) and %R for C.B and M.B

(a) Average Cumulative J_{nr} (b) Average Cumulative %R under 30 Cycle Loading

Figure 4.17 it is observed the control binder shows negative recovery at 0.1 kPa stress indicated in circle. After loading of 1 sec time period there is small load observed for C.B as compared to M.B in the magnification of loading and unloading as shown in Figure 4.17, which means that control binder is unable to recover when strain is increasing in unloading phase.

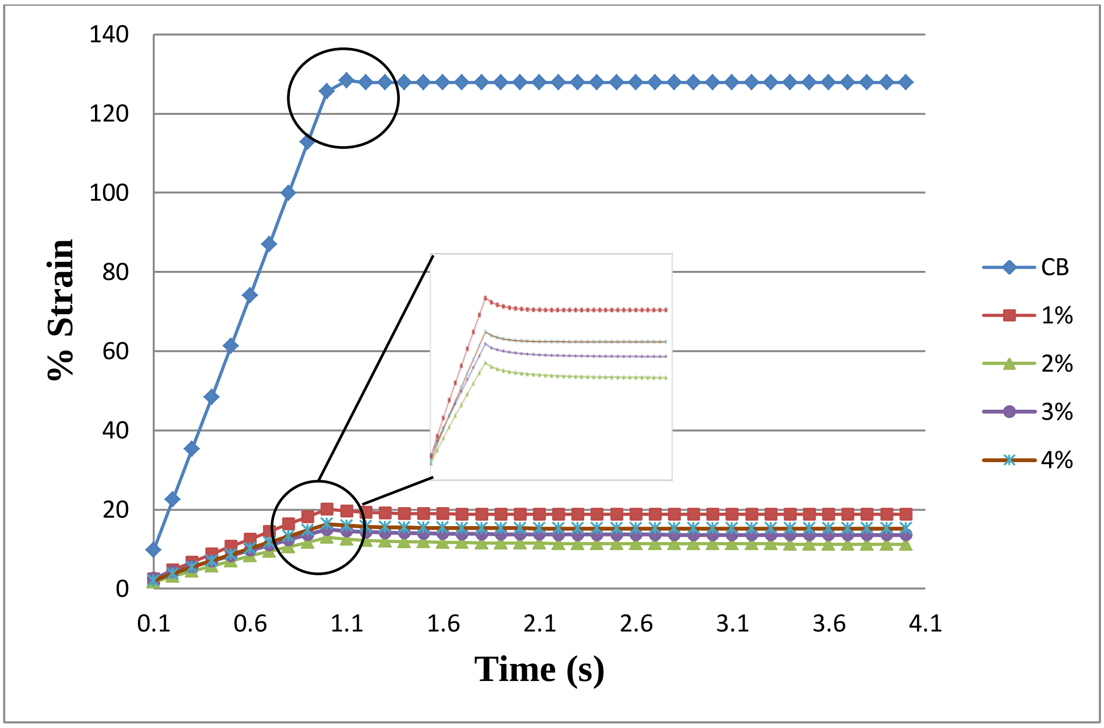
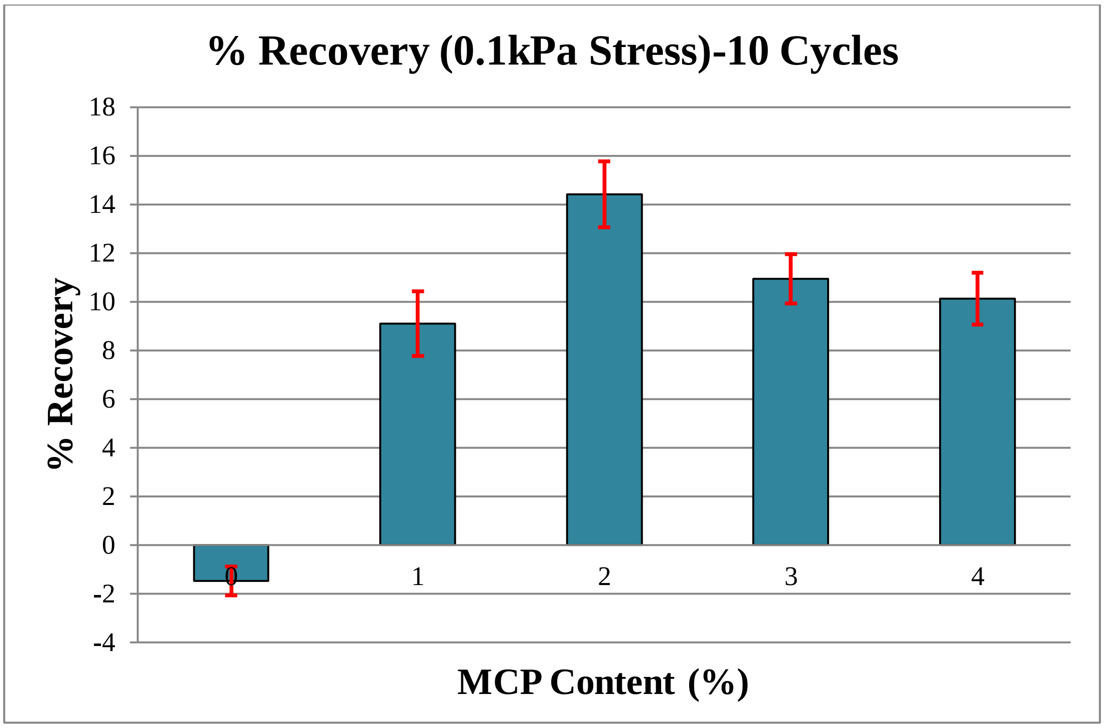


Figure 4.17 Unloading Cycle Magnification for Control and Modified Binders At 0.1 kPa Stress

4.2.7.3 Effects of Creep Stresses on %R at Different MCP Dosages under 10 cycles

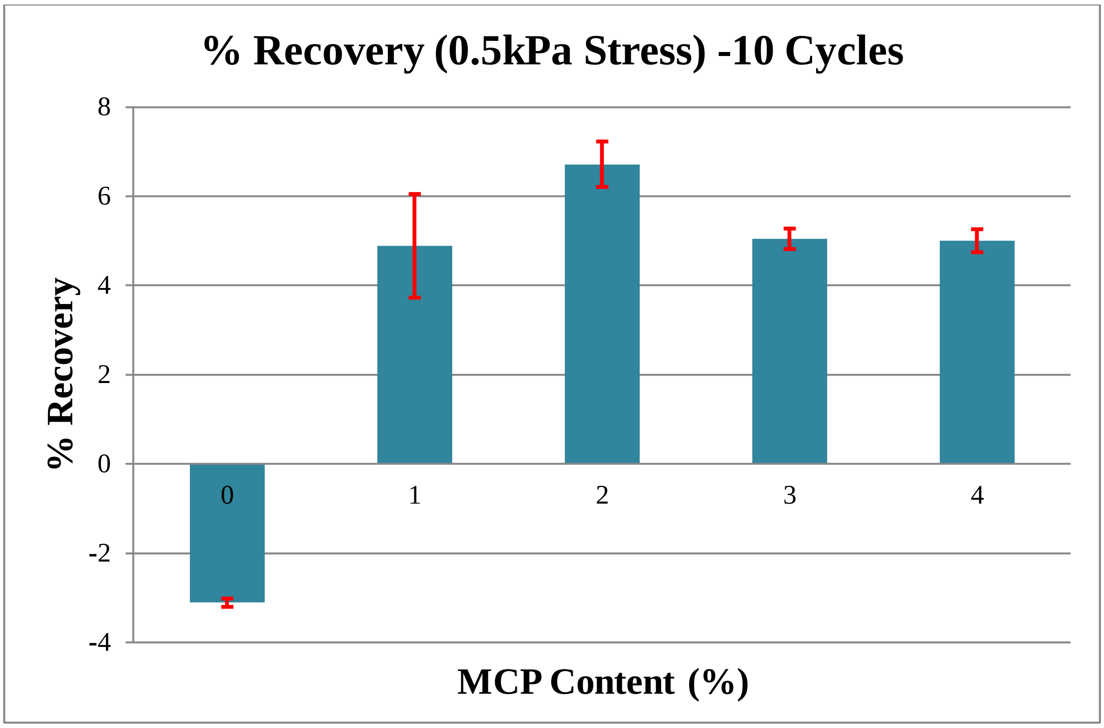
%R and J_{nr} are calculated according to the AASHTO TP70 and is shown in Figure 4.18 (a-h) and 4.19 (a-h) respectively.

It can be observed that at every creep stress levels i.e. from low to high stresses control binder does not show any recovery and keep on going in negative as the stress is increasing as illustrates in Figure 4.18 (a-h). As the addition of coconut MCP in bitumen initially increased the recovery but after 2% addition, there was a decrease in recovery and keep on decreasing when we are applying more and more stresses but one thing should be noted that at every stress level, addition of 2% MCP has more recovery than other percentage addition as shown in Figure 4.18 (a-h). Maximum percentage of recovery is 14.40% at 0.1 kPa for 10 cycles MSCR test. This behaviour can be due to fact that as the content of coconut charcoal was increased initially, there was uniform and homogeneous distribution. However, after 2% addition, there is a possibility of agglomeration of charcoal powder leading to lack of homogeneous distribution where the effect of charcoal powder was reduced. In addition, an interesting observation was that the control binder showed negative recovery even at 0.1 kPa stress level indicating the control bitumen was highly rut susceptible. The negative recovery can be either due to low quality bitumen or binder can be soft which is unable to recover after removal of load, whereas after removal of creep load, the binder still experienced creep load or the equipment was not capable of unloading immediately at the end of creep cycle. As negative recovery was not observed in modified bitumen, it can be stated that the control binder had very low rut resistance. This clearly signifies the effect of charcoal addition, which improved the creep-recovery behaviour. In all the Figures 4.18 (a-h), error bars indicate one standard deviation.

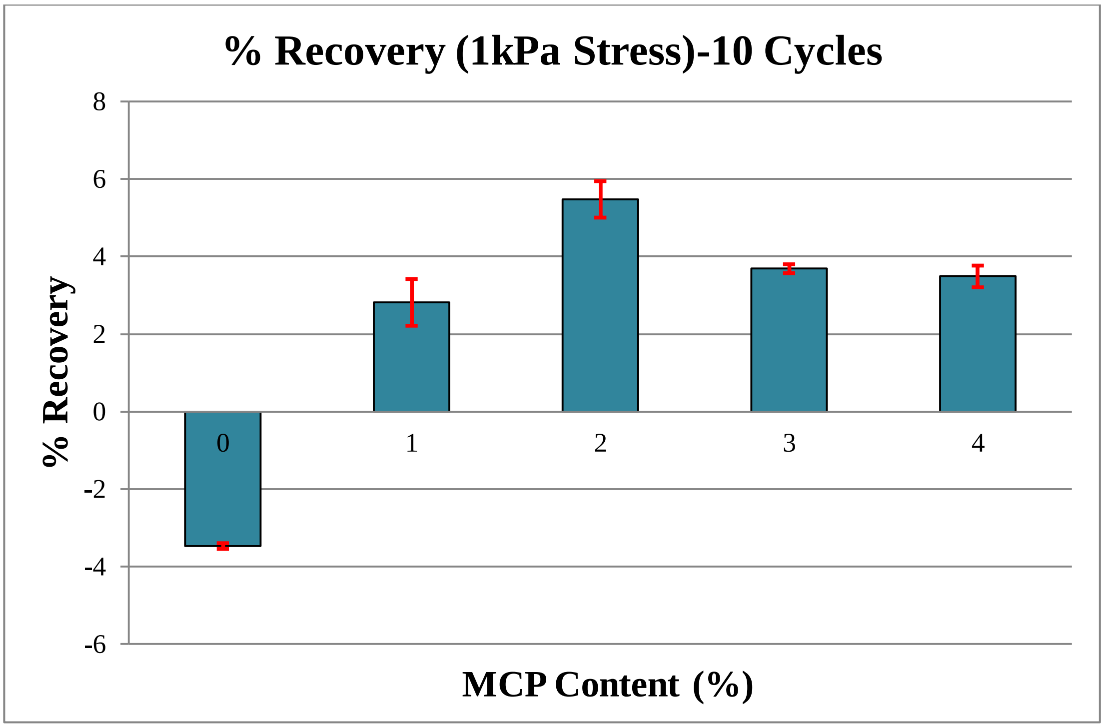


**error bar indicates one standard deviation*

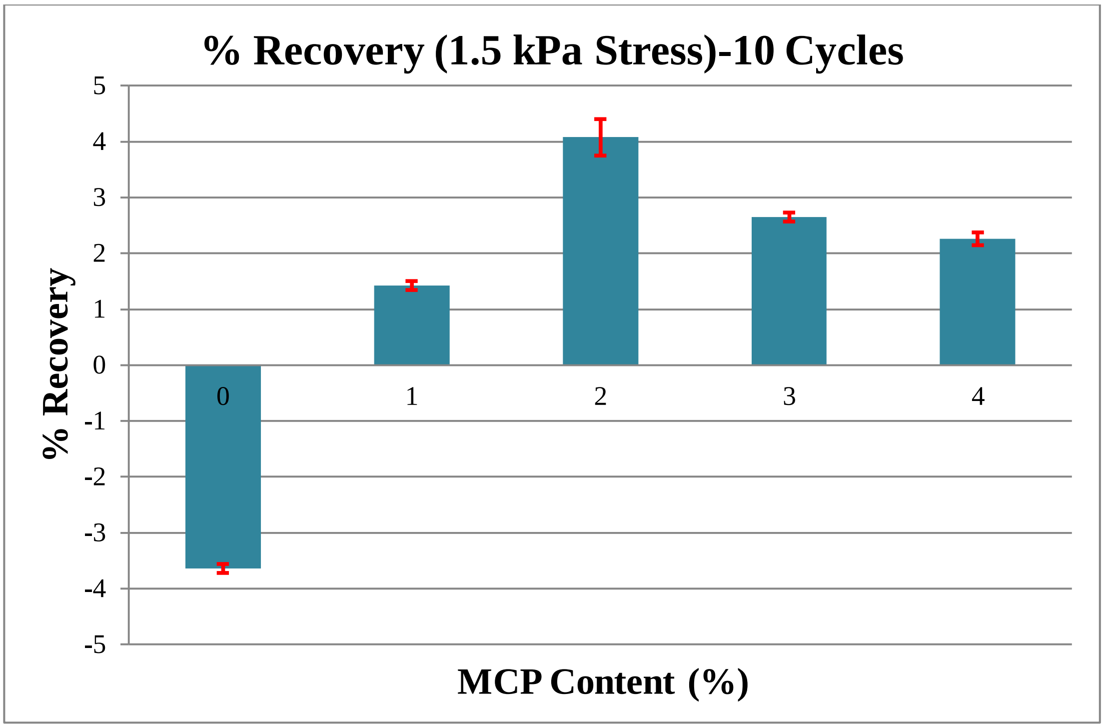
(a)


% Recovery (0.5kPa Stress) -10 Cycles
% Recovery
8
6
4
2
0
-2
-4
0
1
2
3
4
MCP Content (%)

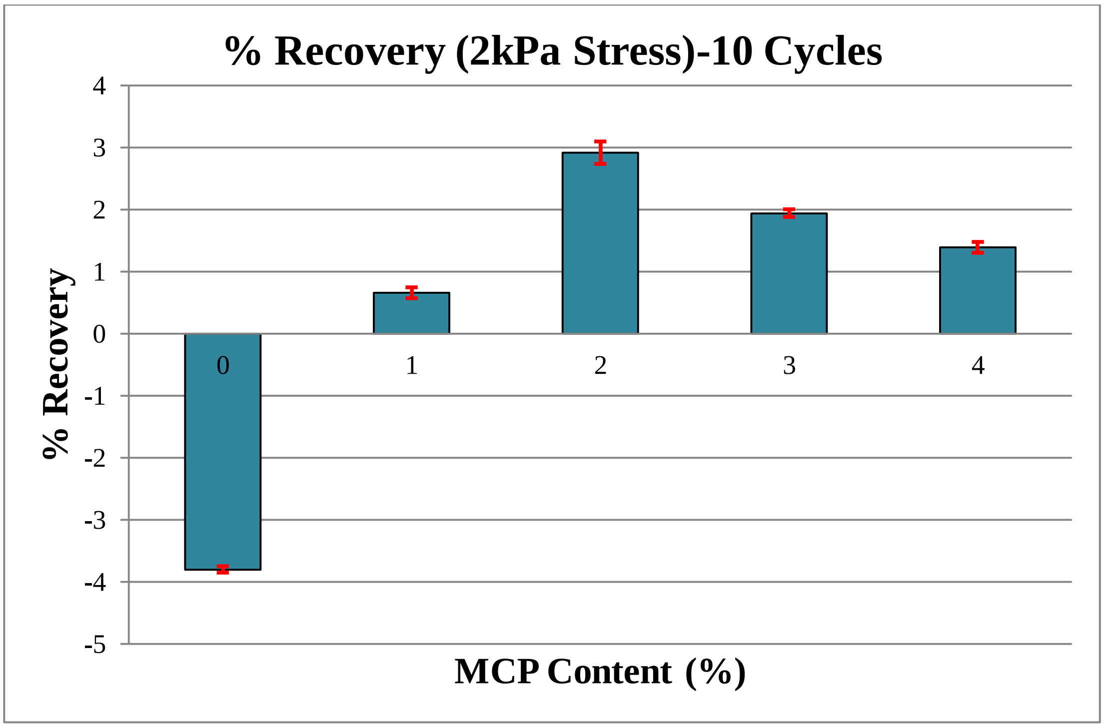
(b)


% Recovery (1kPa Stress)-10 Cycles
% Recovery
8
6
4
2
0
-2
-4
-6
0
1
2
3
4
MCP Content (%)

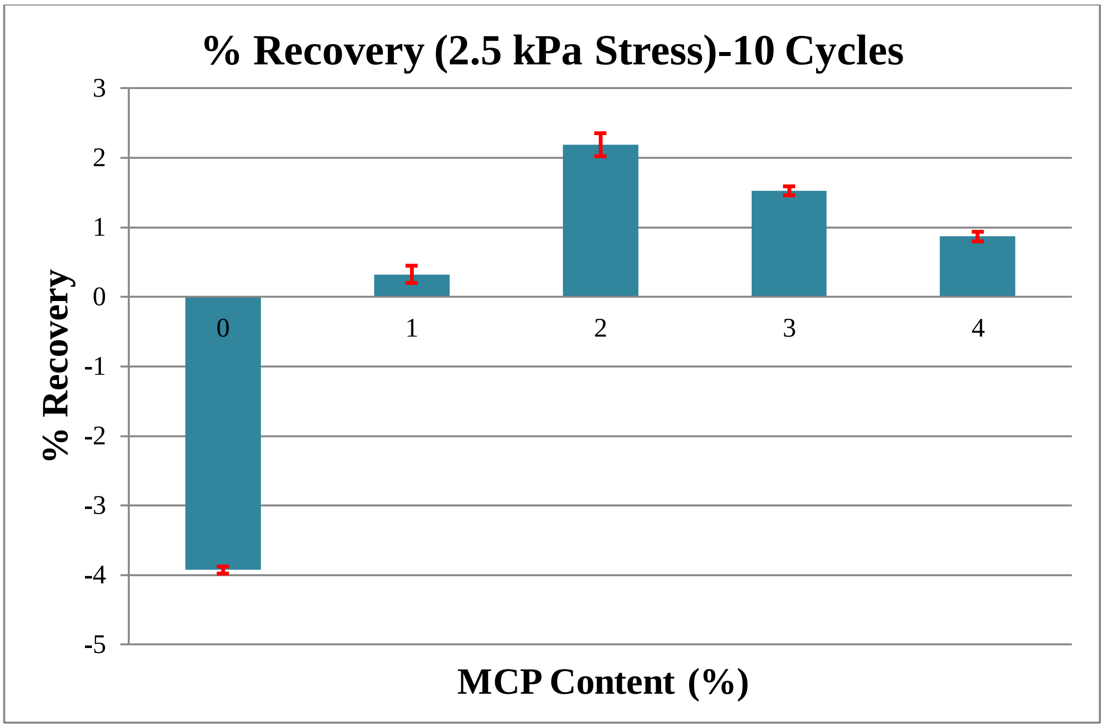
(c)


% Recovery (1.5 kPa Stress)-10 Cycles
5
4
3
2
1
0
-1
-2
-3
-4
-5
% Recovery
0
1
2
3
4
MCP Content (%)

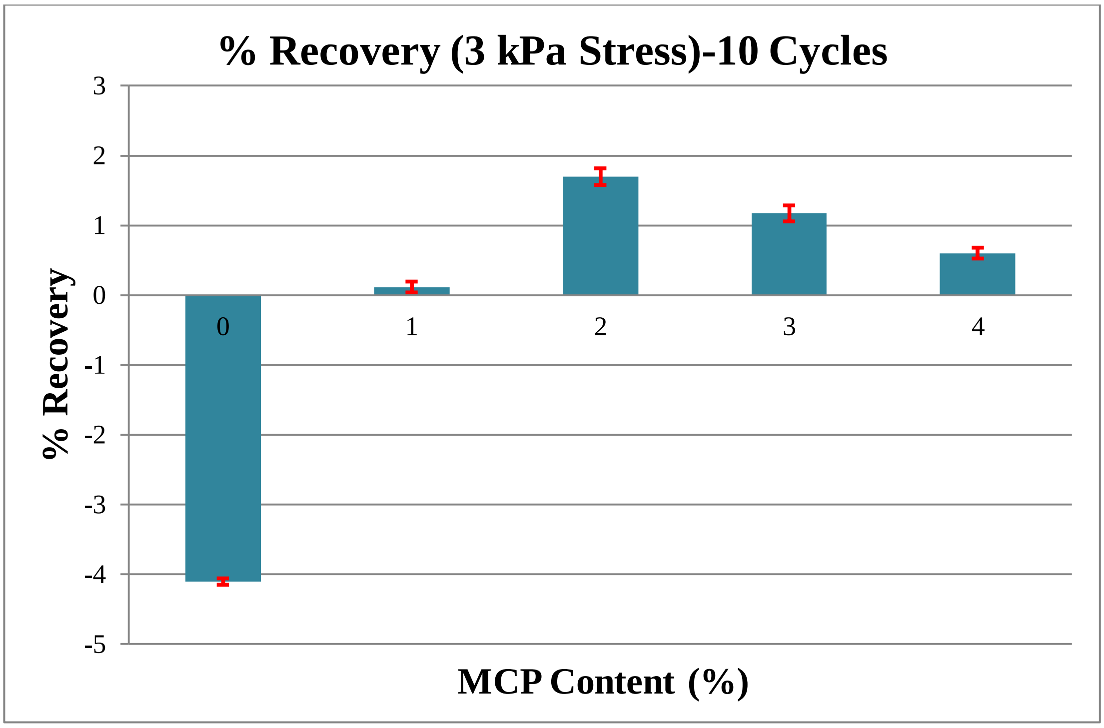
(d)


% Recovery (2kPa Stress)-10 Cycles
4
3
2
1
0
-1
-2
-3
-4
-5
% Recovery
0
1
2
3
4
MCP Content (%)

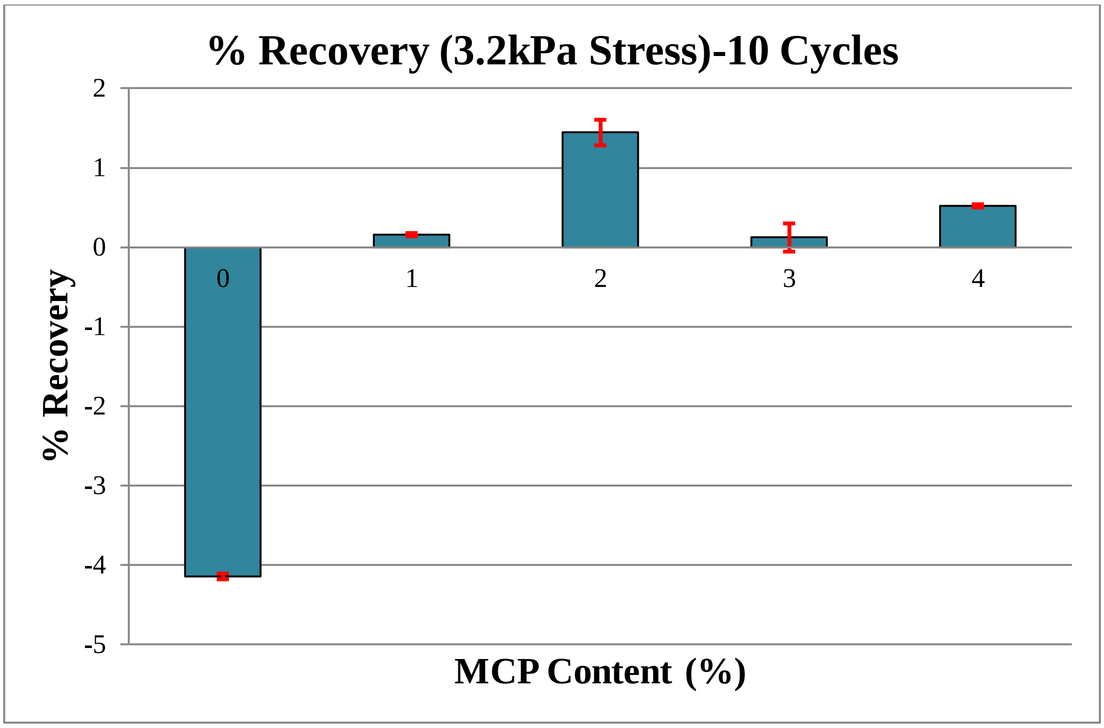
(e)


% Recovery (2.5 kPa Stress)-10 Cycles
3
2
1
0
-1
-2
-3
-4
-5
% Recovery
0
1
2
3
4
MCP Content (%)

(f)


% Recovery (3 kPa Stress)-10 Cycles
3
2
1
0
-1
-2
-3
-4
-5
% Recovery
0
1
2
3
4
MCP Content (%)

(g)



(h)

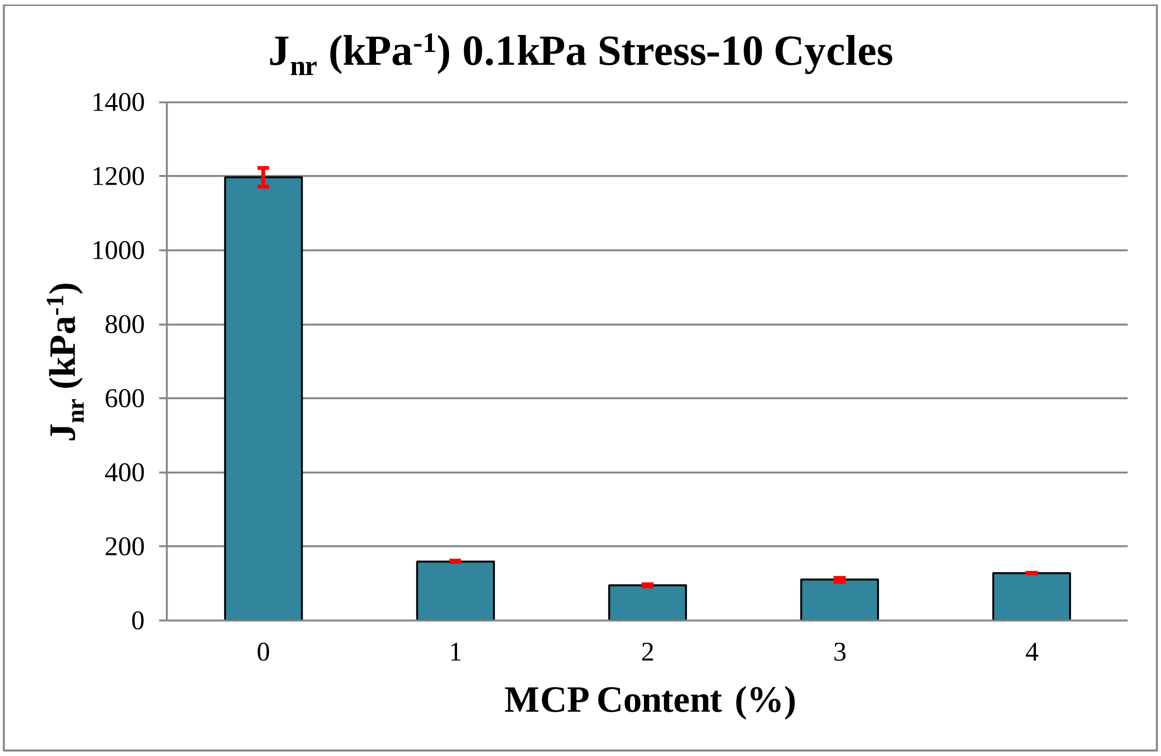
Figure 4.18 Effects of Creep Stresses on %R at Different MCP Dosages under 10 cycles

(a) at 0.1 kPa, (b) 0.5 kPa (c) 1 kPa, (d) 1.5 kPa, (e) 2 kPa, (f) 2.5 kPa,(g) 3kPa and (h) 3.2 kPa Creep Stresses

4.2.7.4 Effects of Creep Stresses on J_{nr} at Different MCP Dosages under 10 cycles

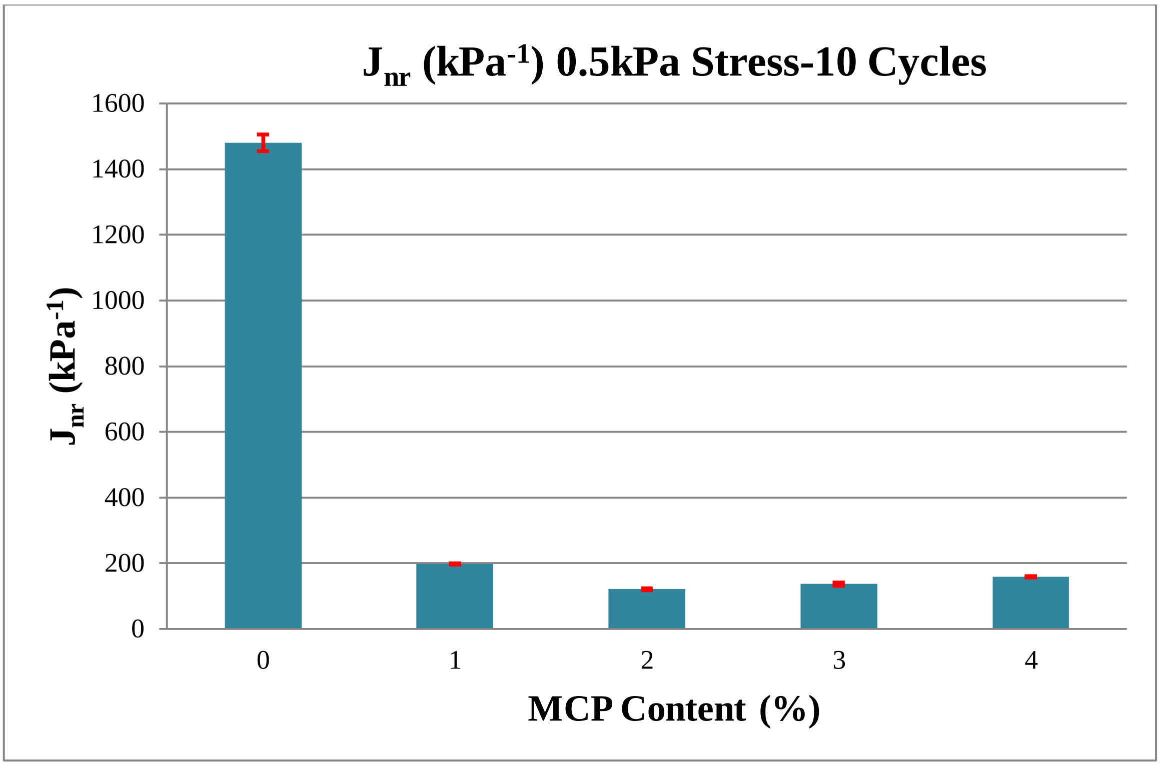
In the previous section percentage recovery at different MCP and at different creep stresses for 10 cycles was discussed. Now same proceduce was followed to on same creep stresses and at same MCP is analysed for non-recoverable creep compliance. Figure 4.19 (a-h) shows that at low creep stress i.e. 0.1 kPa control binder gives maximum value of non recoverable compliance (J_{nr}) and keep increasing as the stresses are increasing from a to h in Figure 4.19. As the addition of MCP into conventional binder, it show significant decrease in non recoverable compliance (J_{nr}) as shown in Figure 4.19 (a). Maximum decrease is obtained at 2% addition of MCP. Non recoverable creep compliance also follow same trend as in percentage recovey, as the stresses are increasing non recoverable creep compliance also increase and maximum value is obtained at 3.2 kPa creep stress. In case of modified binder J_{nr}

increases as the stresses increase but 2% addition of MCP gives less value as comapred to other modifier binders as shown in Figure 4.19 (a-h).



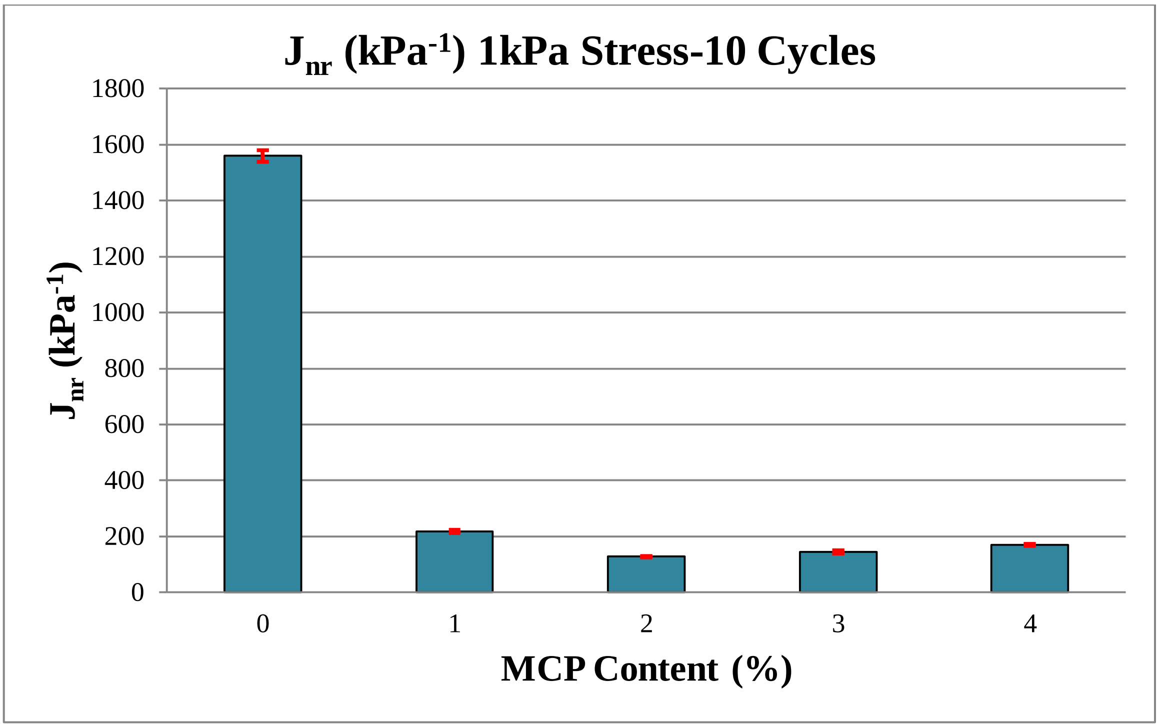
**error bar indicates one standard deviation*

(a)



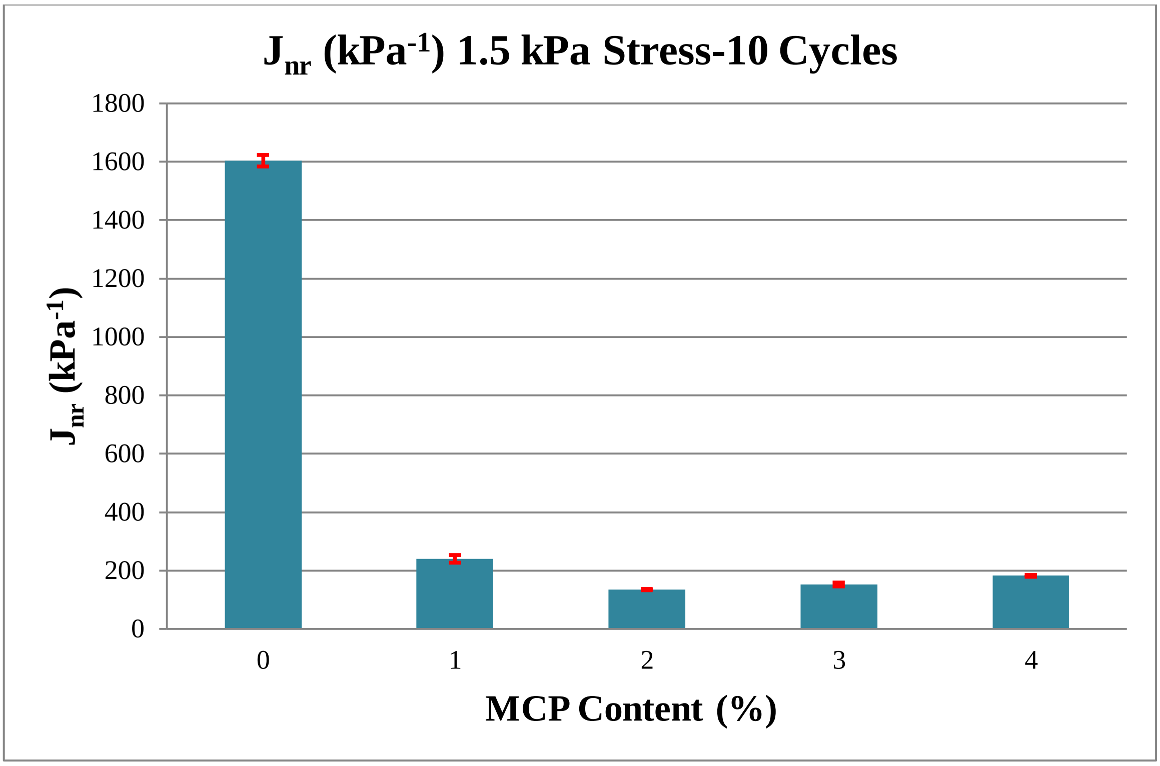
J_{nr} (kPa^{-1}) 0.5kPa Stress-10 Cycles
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0
1
2
3
4
MCP Content (%)

(b)

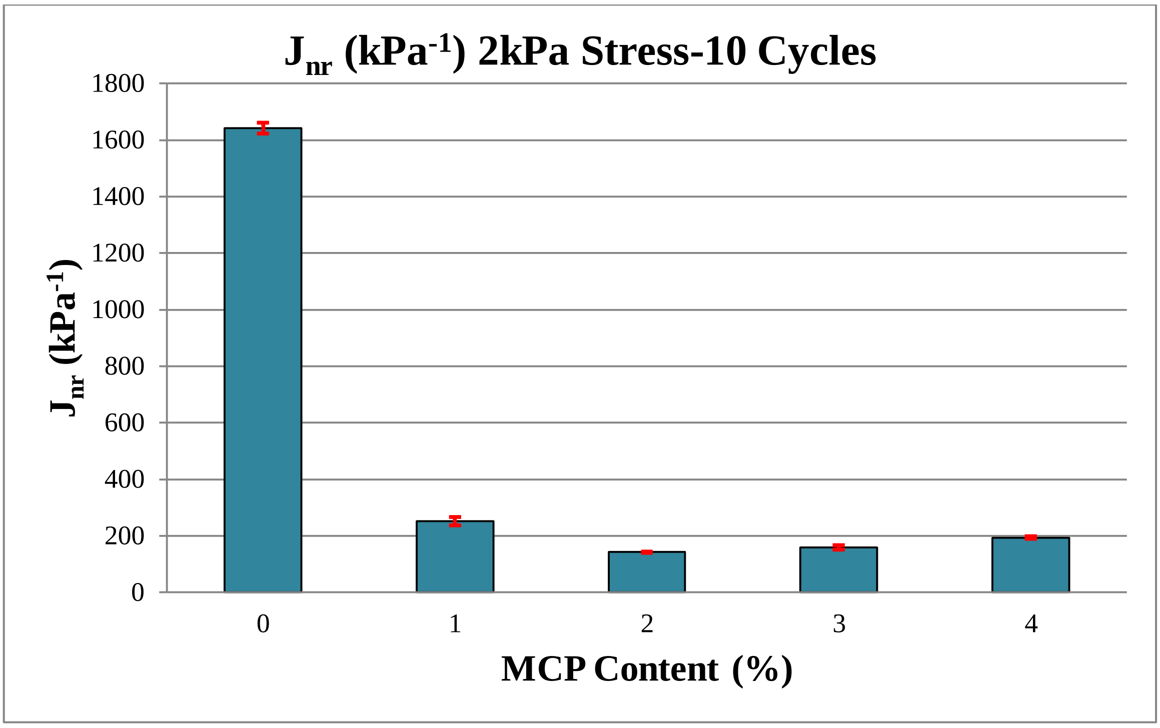


J_{nr} (kPa^{-1}) 1kPa Stress-10 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0
1
2
3
4
MCP Content (%)

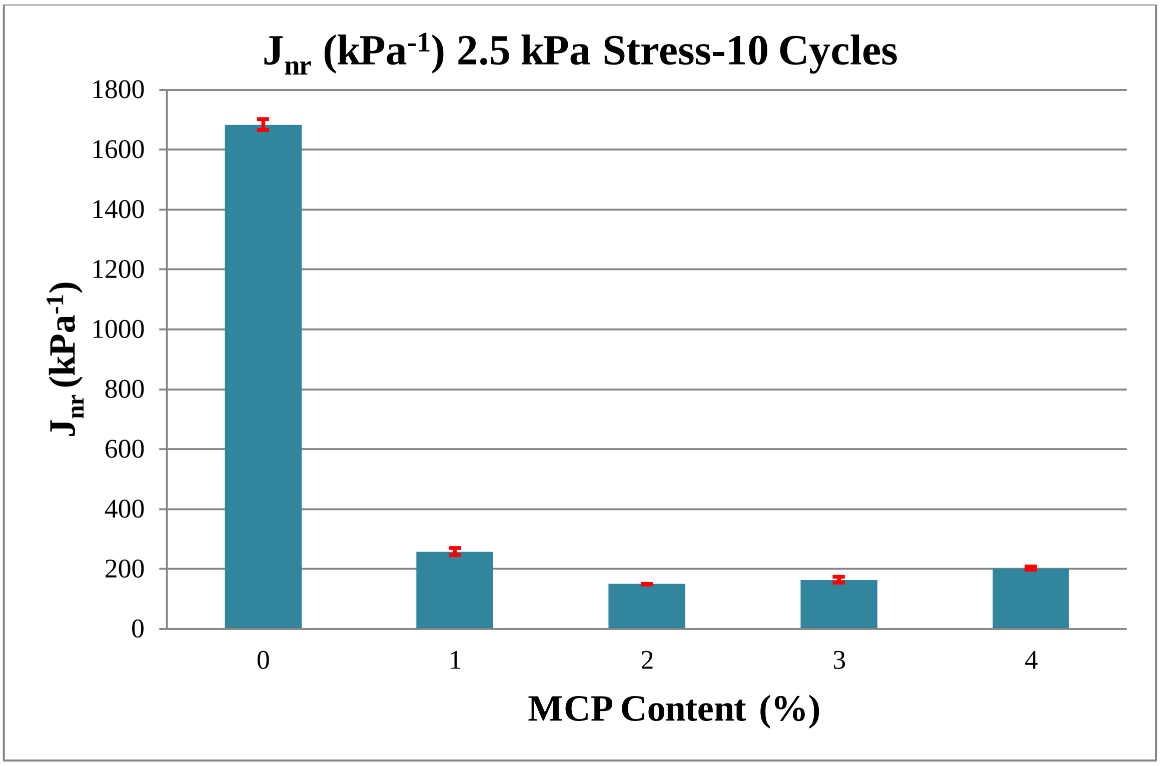
(c)


J_{nr} (kPa^{-1}) 1.5 kPa Stress-10 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0
1
2
3
4
MCP Content (%)

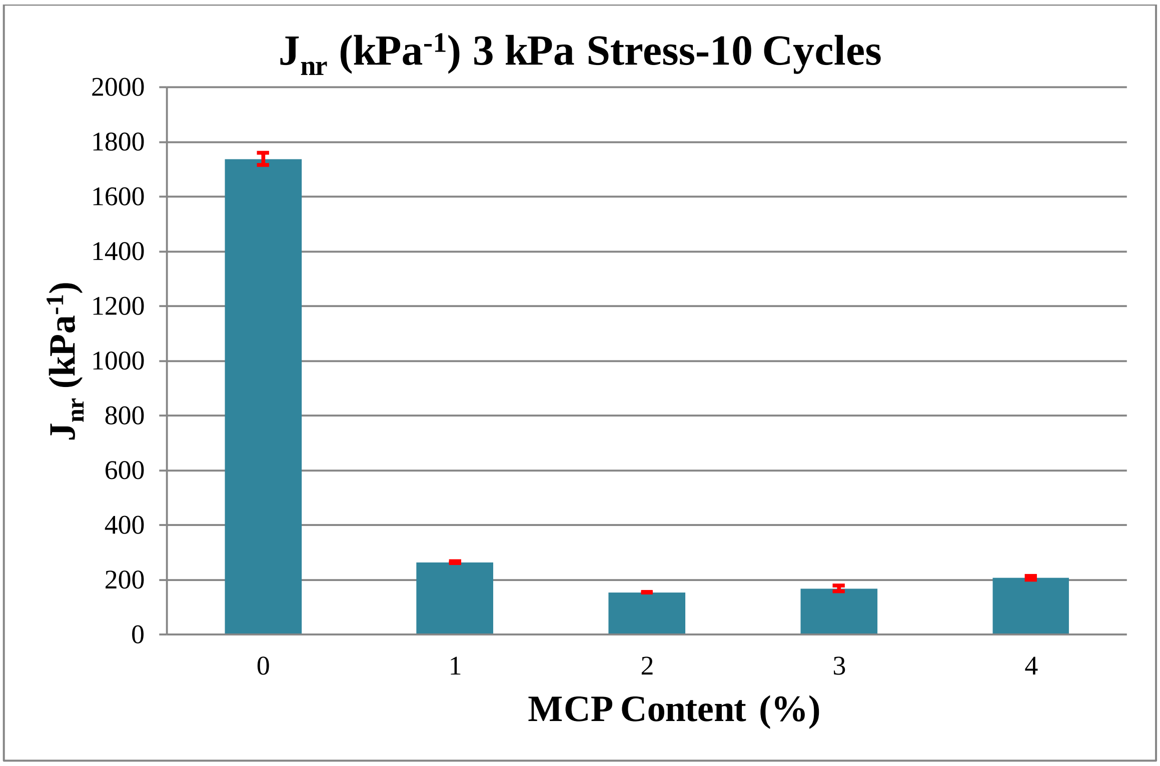
(d)


J_{nr} (kPa^{-1}) 2kPa Stress-10 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0
1
2
3
4
MCP Content (%)

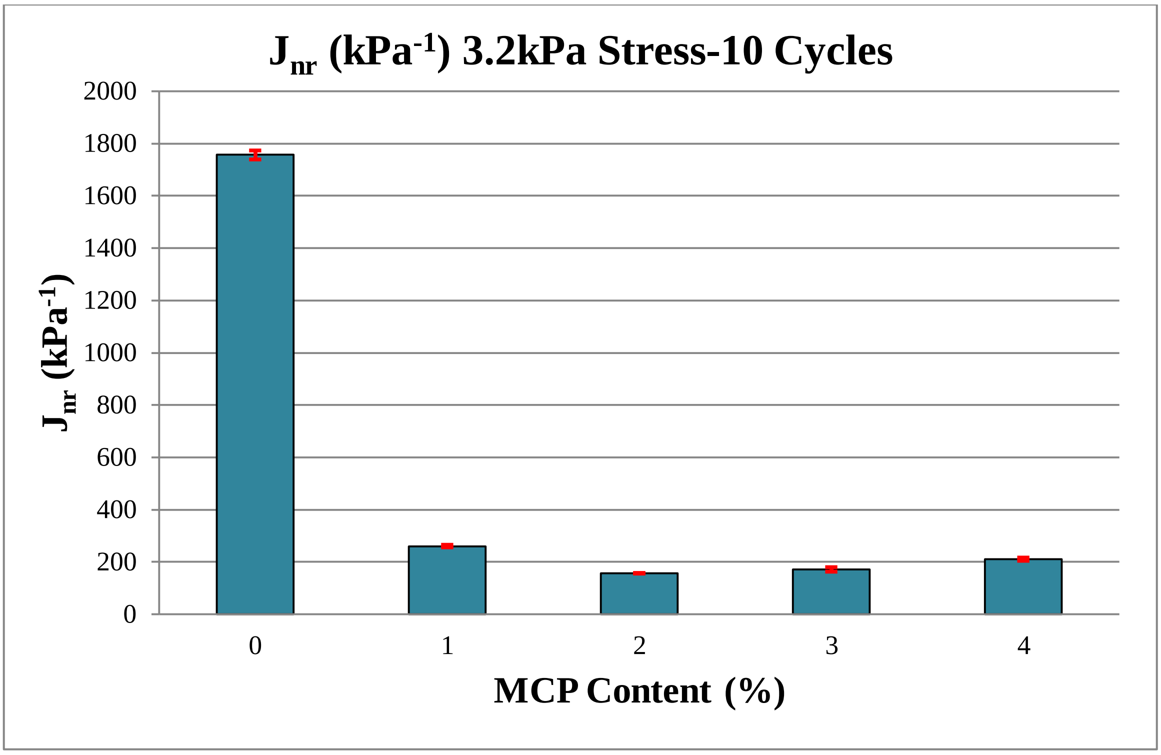
(e)


Jnr (kPa-1) 2.5 kPa Stress-10 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
Jnr (kPa-1)
0
1
2
3
4
MCP Content (%)

(f)


Jnr (kPa-1) 3 kPa Stress-10 Cycles
2000
1800
1600
1400
1200
1000
800
600
400
200
0
Jnr (kPa-1)
0
1
2
3
4
MCP Content (%)

(g)



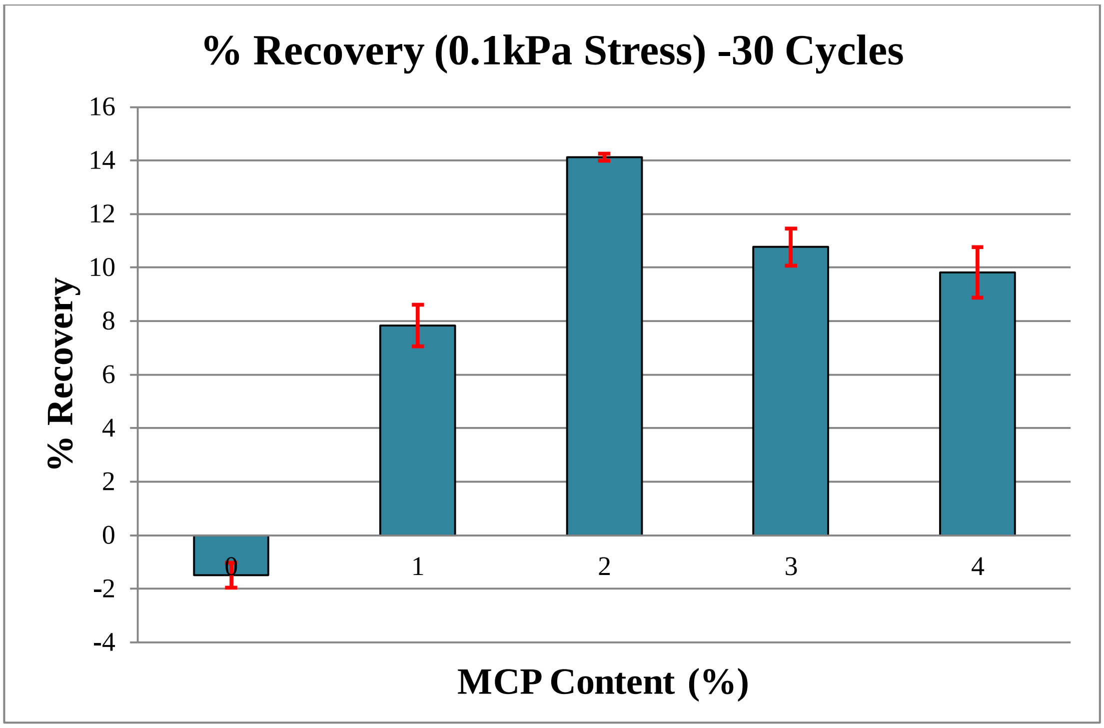
(h)

Figure 4.19 Effects of Creep Stresses on Jnr at Different MCP Dosages under 10 cycles

(a) at 0.1 kPa, (b) 0.5 kPa (c) 1 kPa, (d) 1.5 kPa, (e) 2 kPa, (f) 2.5 kPa,(g) 3kPa and (h) 3.2 kPa Creep Stresses

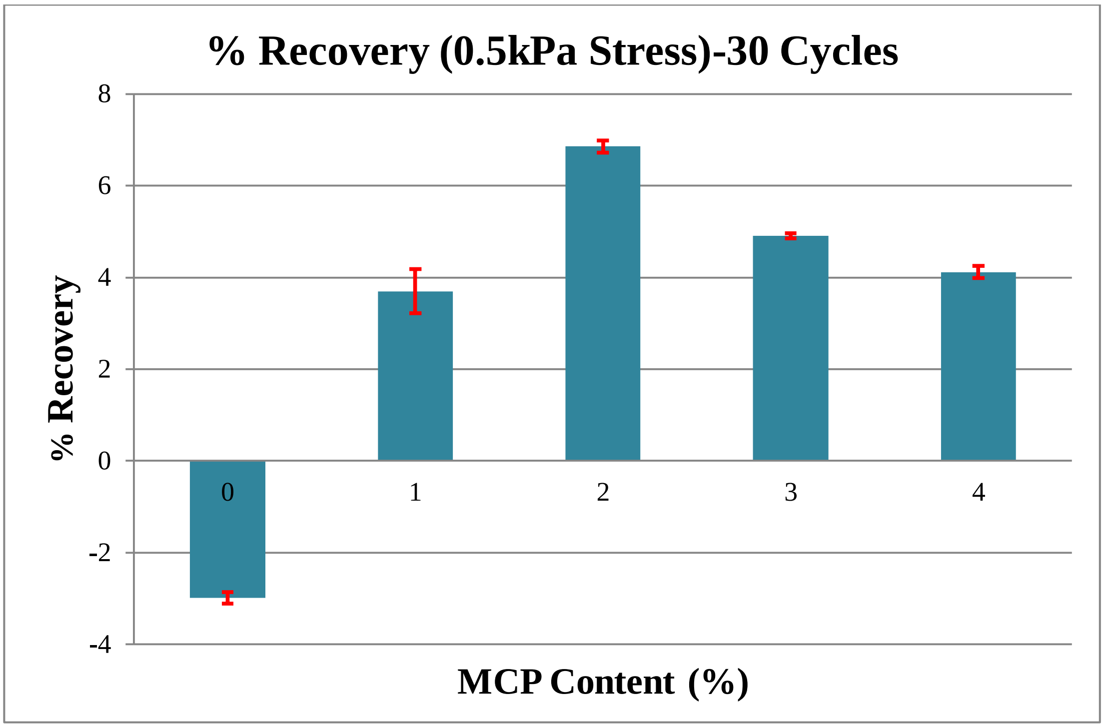
4.2.7.5 Effects of Creep Stresses on %R at Different MCP Dosages under 30 cycles

In the next MSCR test, control and modified binders are subjected to 30 cycles to determine %R and J_{nr} different content of MCP under different values of stress levels. From Figure 4.20 it was conclued that % recovery for 30 cycles was low at every stress levels as comapred to 10 cycles test.

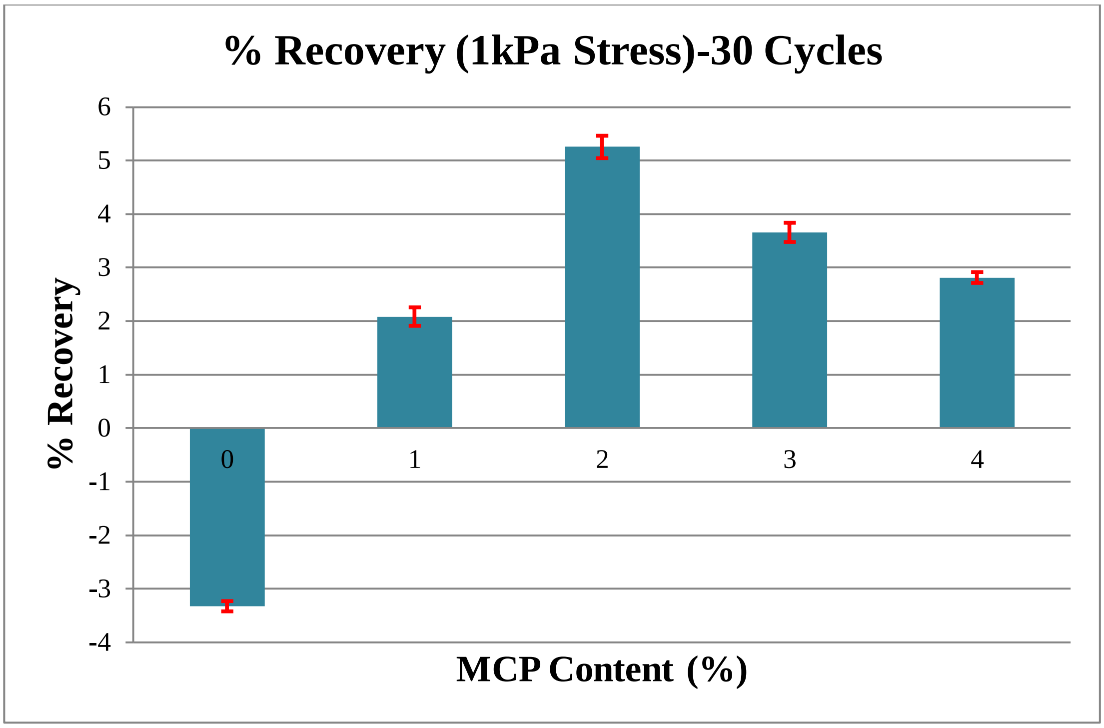


error bar indicates one standard deviation

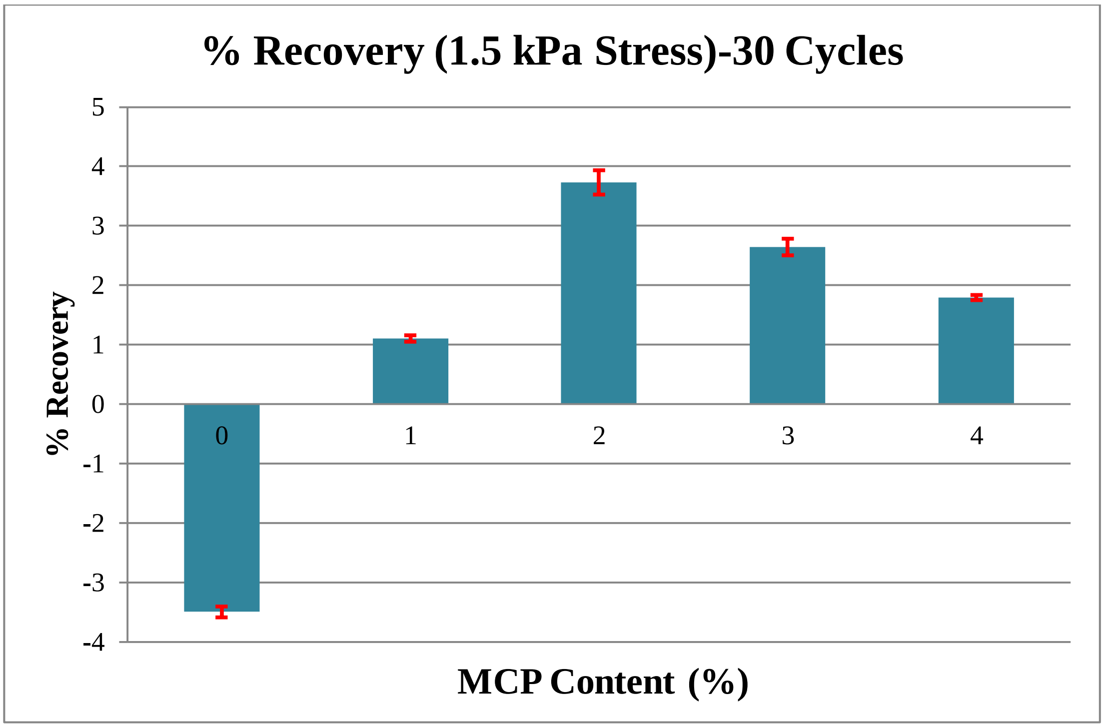
(a)



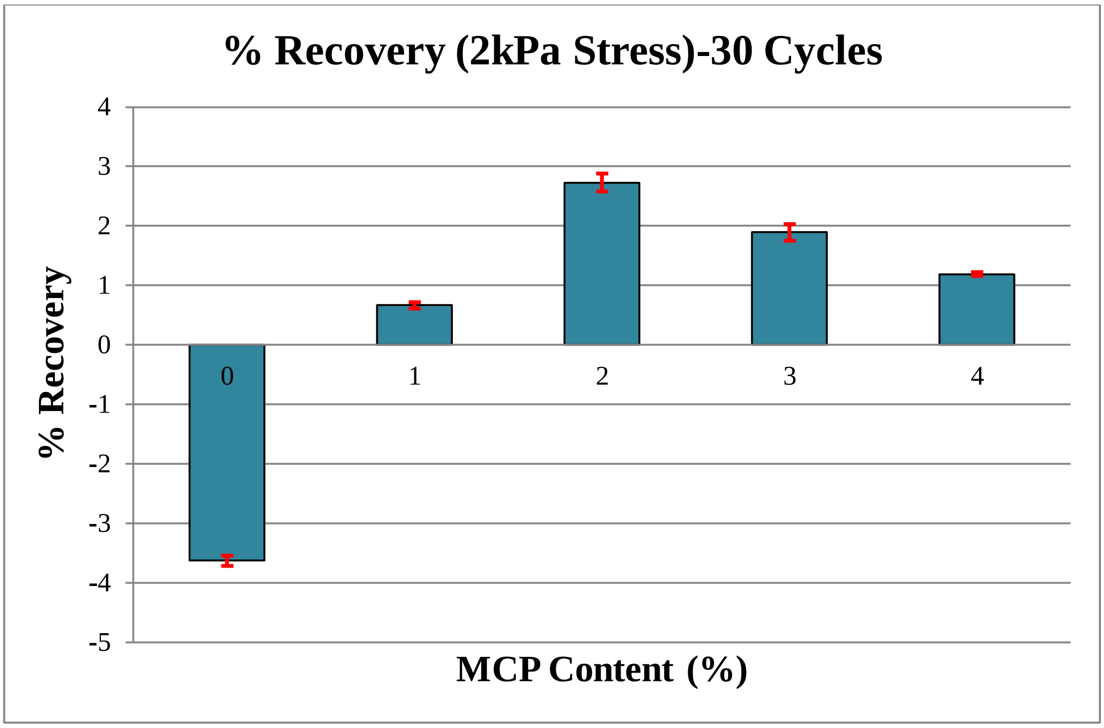
(b)


% Recovery (1kPa Stress)-30 Cycles
6
5
4
3
2
1
0
-1
-2
-3
-4
% Recovery
0
1
2
3
4
MCP Content (%)

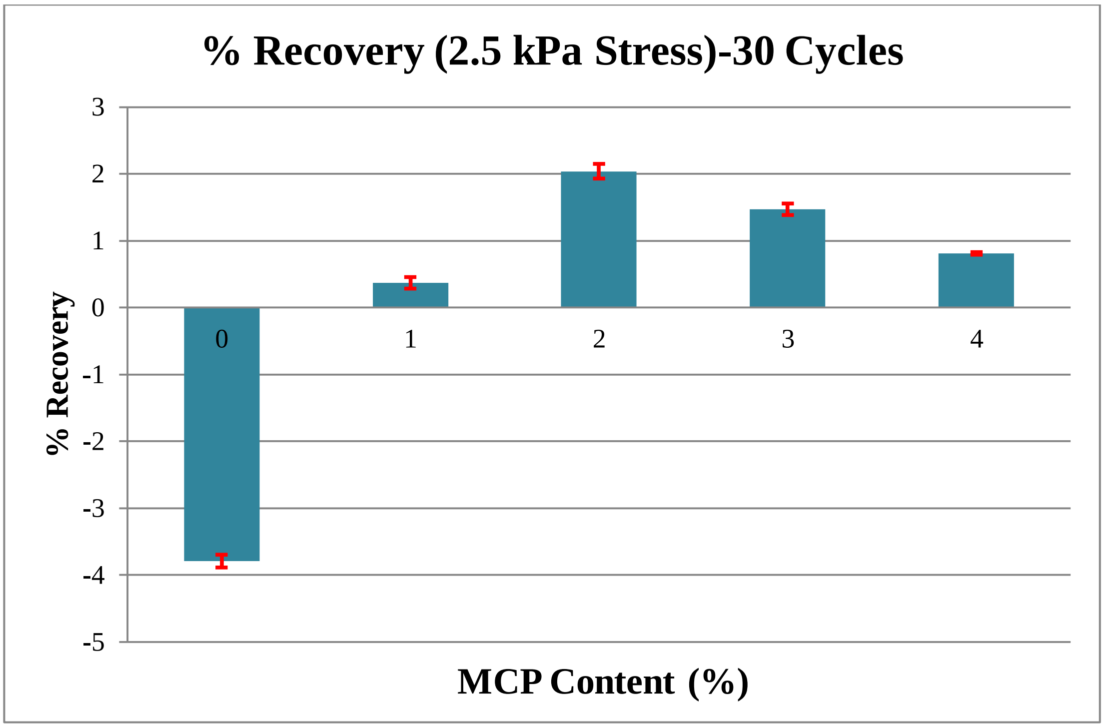
(c)


% Recovery (1.5 kPa Stress)-30 Cycles
5
4
3
2
1
0
-1
-2
-3
-4
% Recovery
0
1
2
3
4
MCP Content (%)

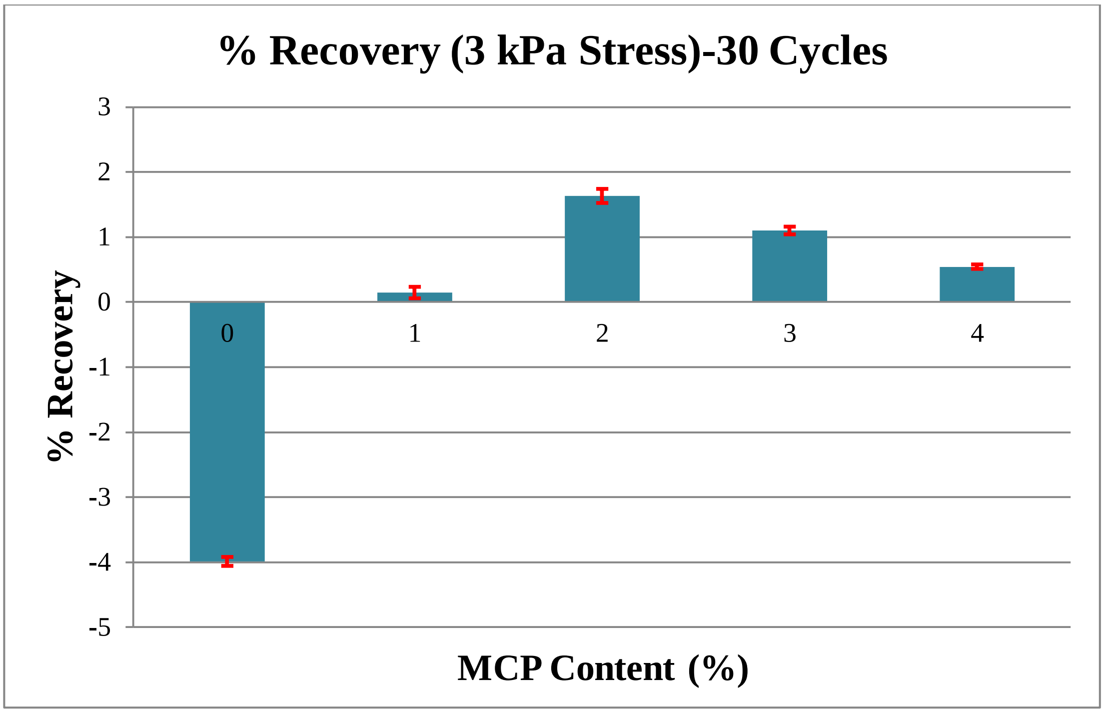
(d)


% Recovery (2kPa Stress)-30 Cycles
4
3
2
1
0
-1
-2
-3
-4
-5
% Recovery
0
1
2
3
4
MCP Content (%)

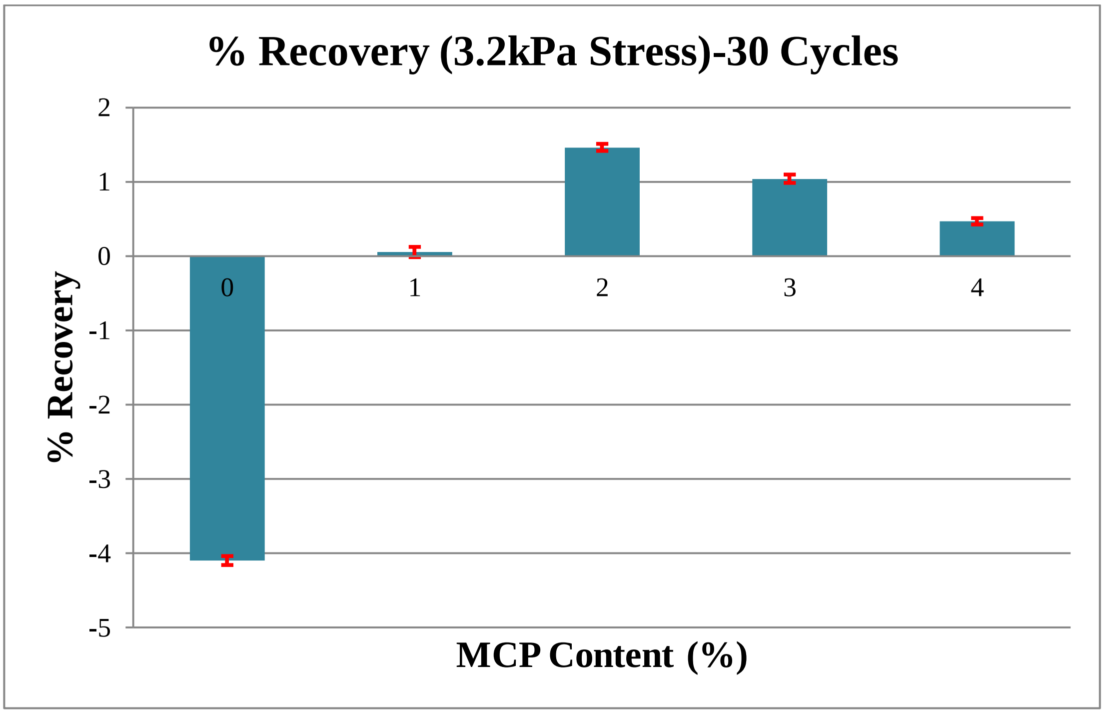
(e)


% Recovery (2.5 kPa Stress)-30 Cycles
3
2
1
0
-1
-2
-3
-4
-5
% Recovery
0
1
2
3
4
MCP Content (%)

(f)



(g)



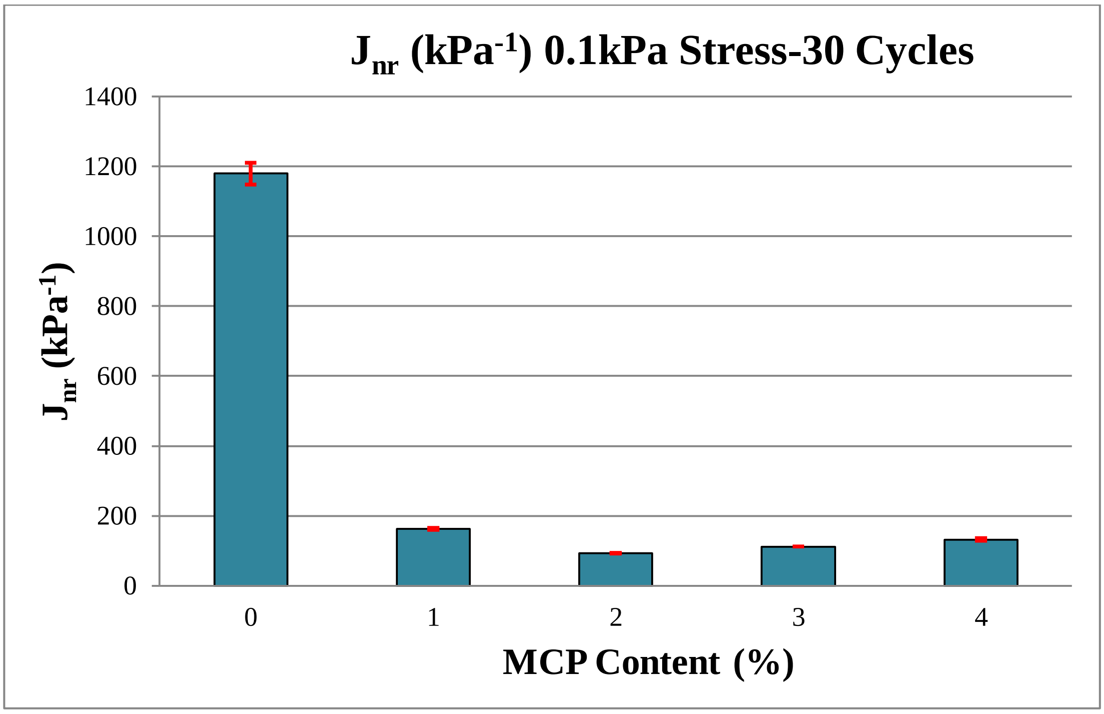
(h)

Figure 4.20 Effects of Creep Stresses on %R at Different MCP Dosages under 30 cycles

(a) at 0.1 kPa, (b) 0.5 kPa (c) 1 kPa, (d) 1.5 kPa, (e) 2 kPa, (f) 2.5 kPa,(g) 3kPa and (h) 3.2 kPa Creep Stresses

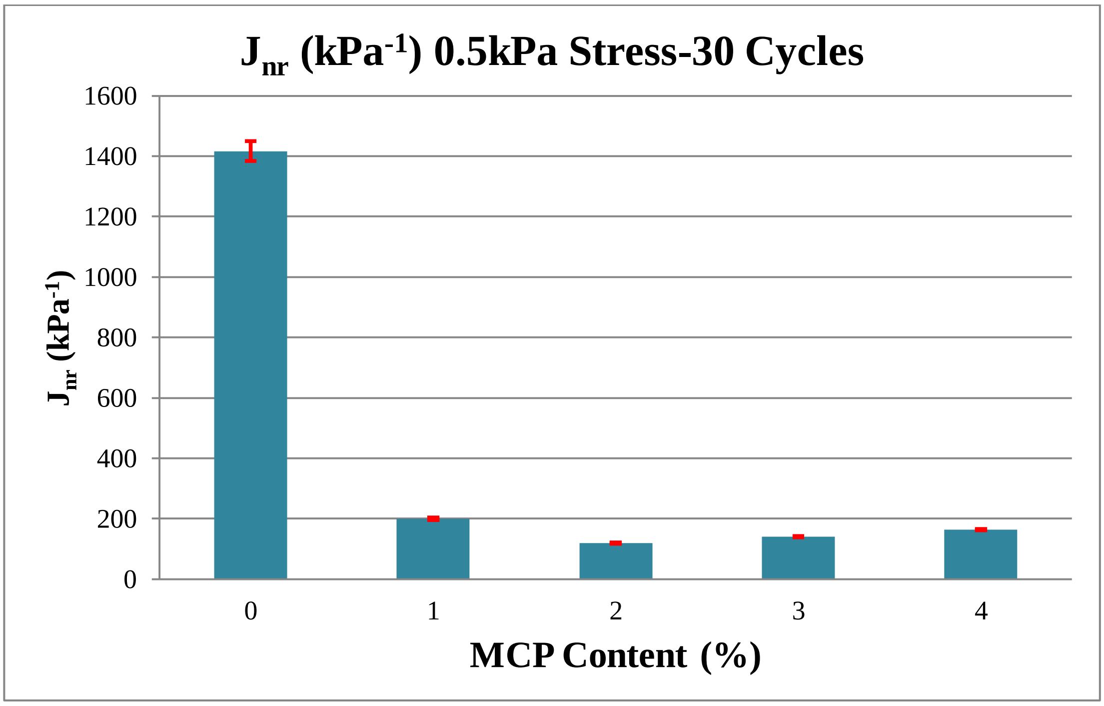
4.2.7.6 Effects of Creep Stresses on Jnr at Different MCP Dosages under 30 cycles

J_{nr} is more as the %R is decreasing but minimum J_{nr} can be seen at C2% addition of MCP as shown in Figure 4.21.

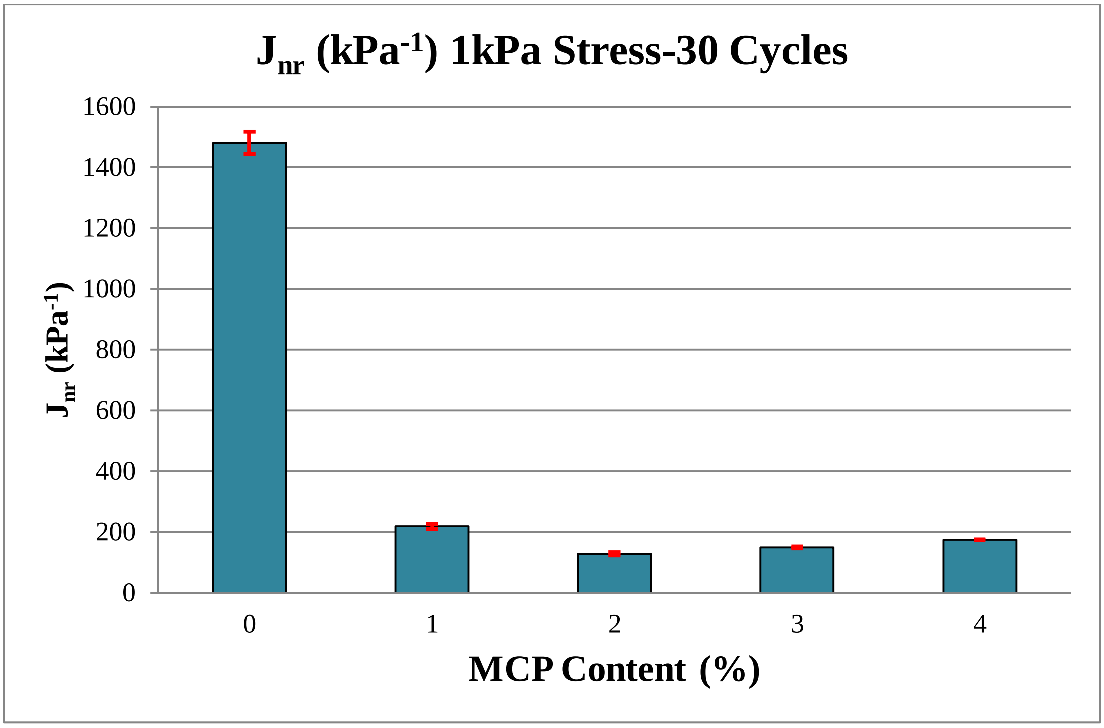


**error bar indicates one standard deviation*

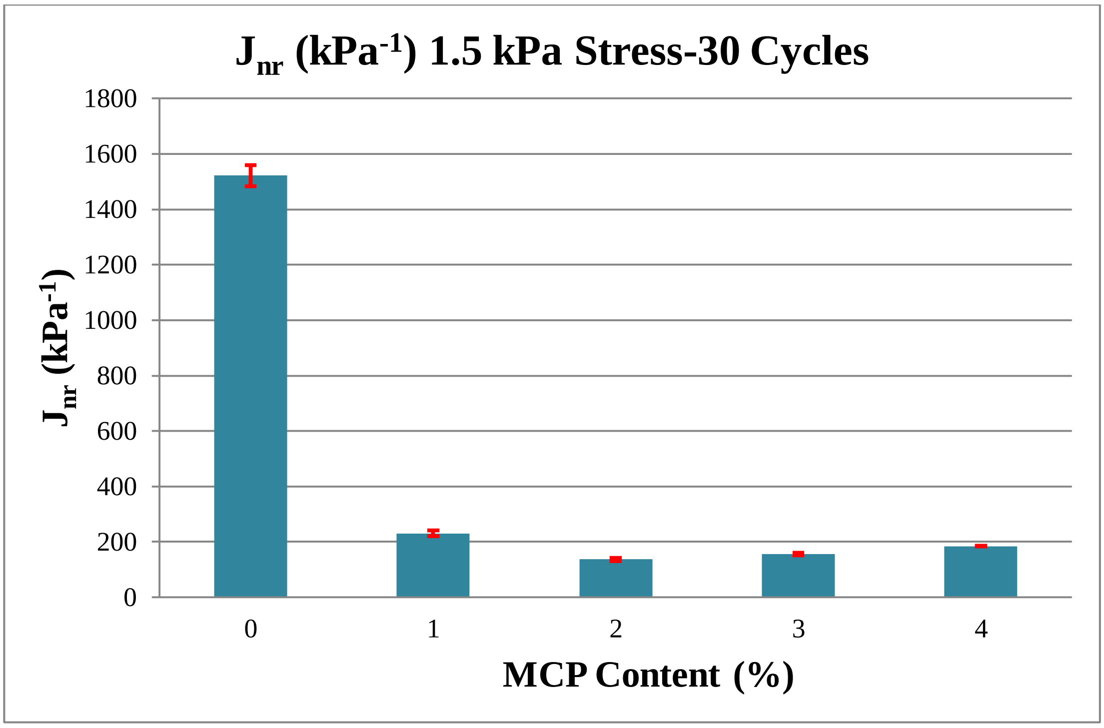
(a)



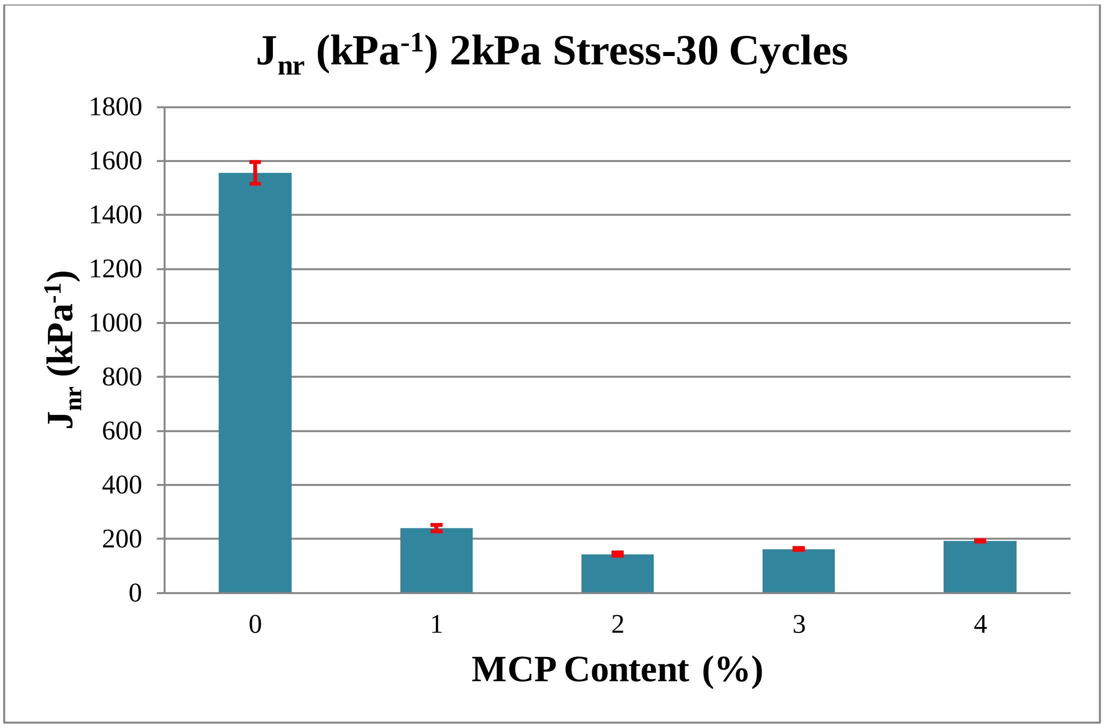
(b)


Jnr (kPa-1) 1kPa Stress-30 Cycles
1600
1400
1200
1000
800
600
400
200
0
Jnr (kPa-1)
0
1
2
3
4
MCP Content (%)

(c)

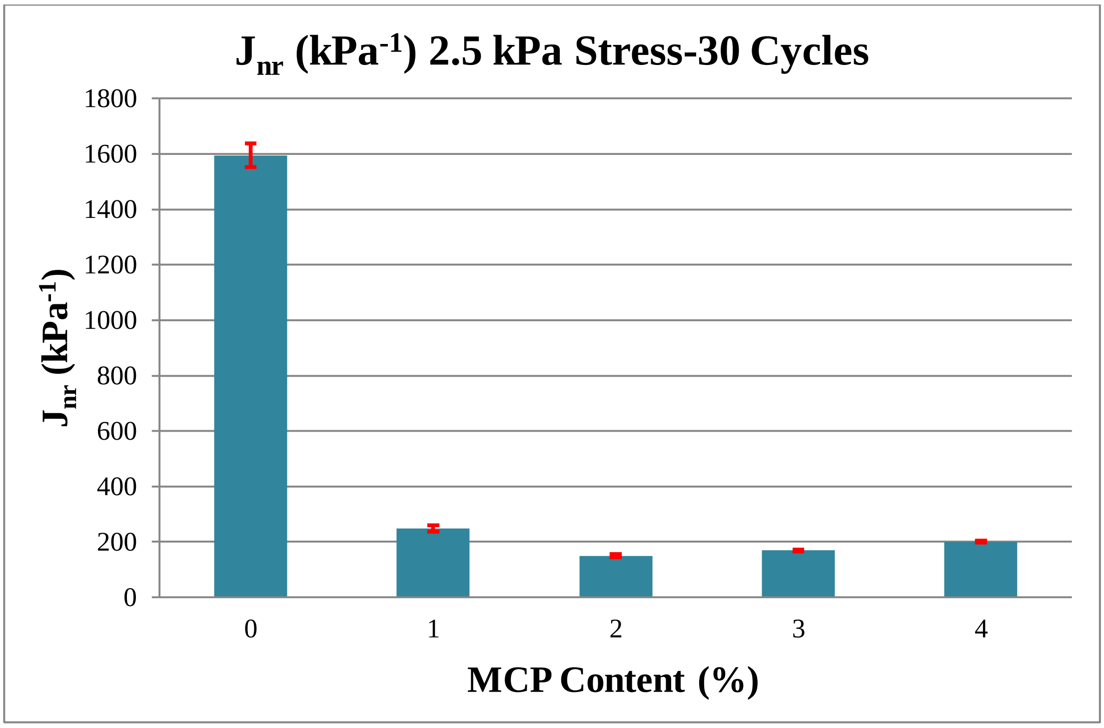

Jnr (kPa-1) 1.5 kPa Stress-30 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
Jnr (kPa-1)
0
1
2
3
4
MCP Content (%)

(d)



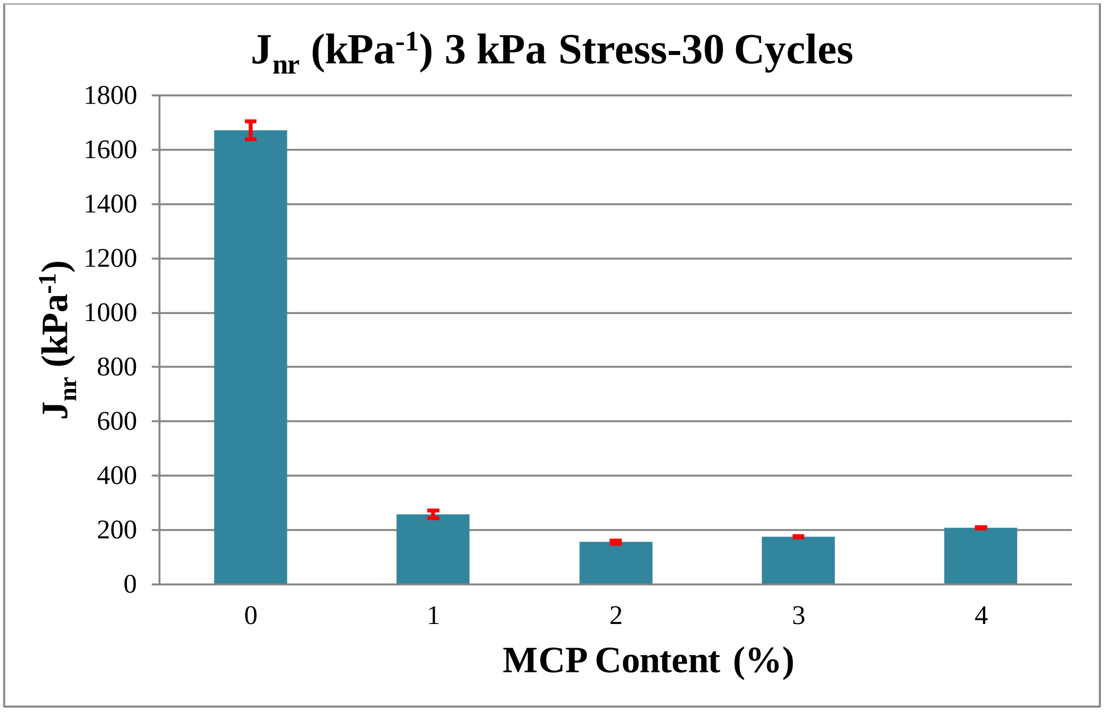
J_{nr} (kPa^{-1}) 2kPa Stress-30 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0
1
2
3
4
MCP Content (%)

(e)

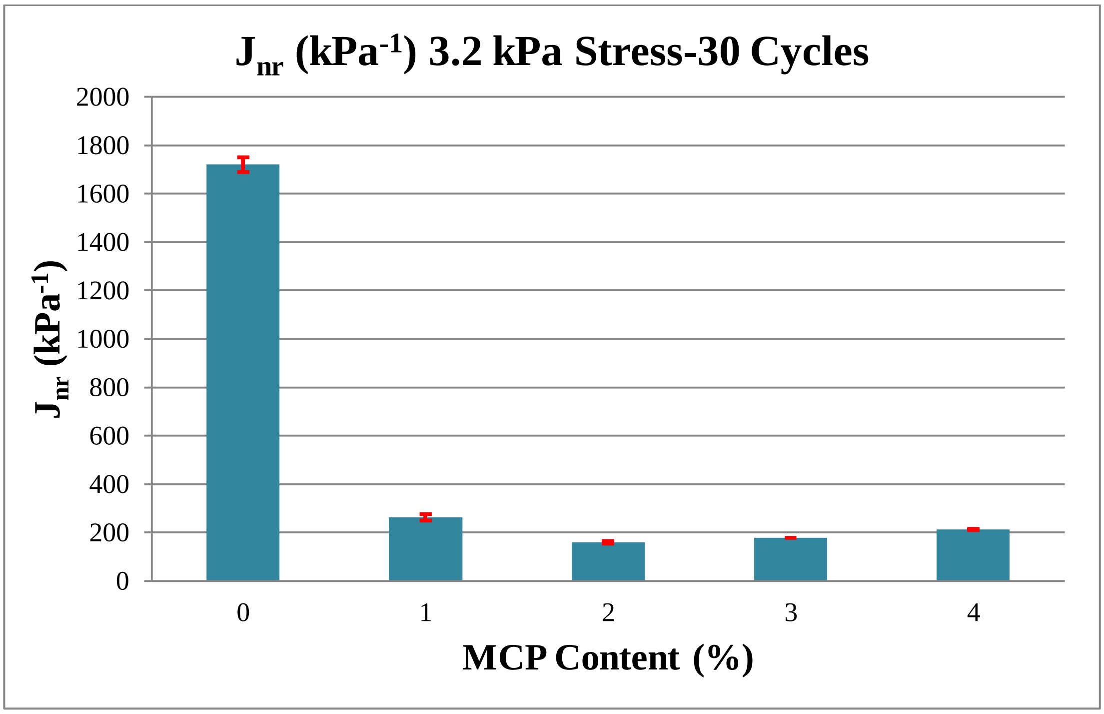


J_{nr} (kPa^{-1}) 2.5 kPa Stress-30 Cycles
1800
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0
1
2
3
4
MCP Content (%)

(f)



(g)



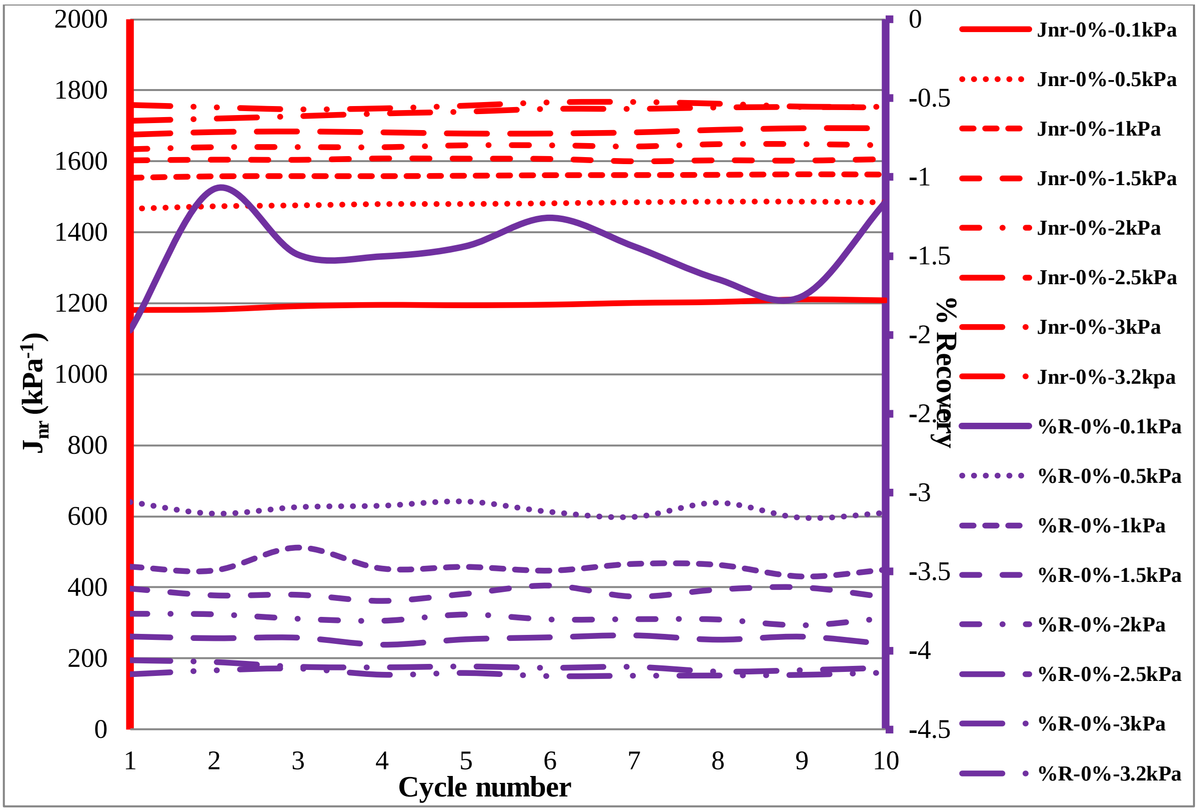
(h)

Figure 4.21 Effects of Creep Stresses on Jnr at Different MCP Dosages under 30 cycles

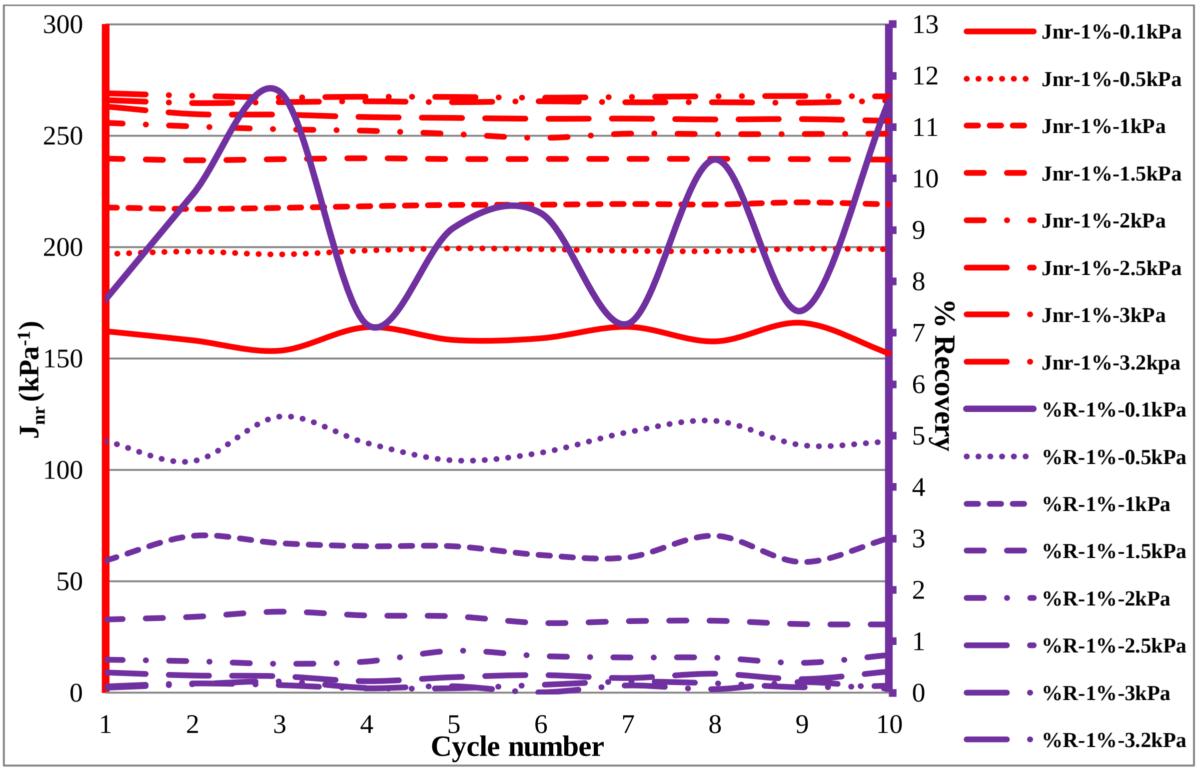
(a) at 0.1 kPa, (b) 0.5 kPa (c) 1 kPa, (d) 1.5 kPa, (e) 2 kPa, (f) 2.5 kPa,(g) 3kPa and (h) 3.2 kPa Creep Stresses

4.2.8 Stress Sensitivity

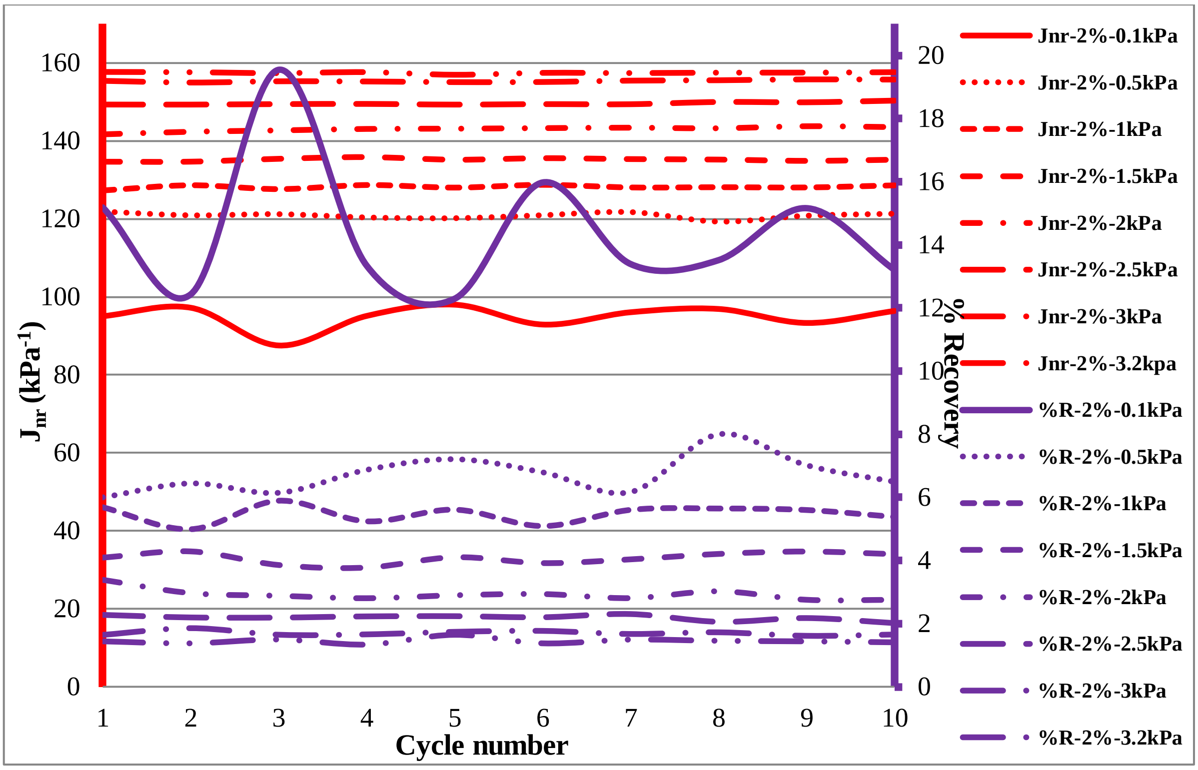
In addition to recovery and J_{nr}, other important parameter is stress sensitivity, which indicates behavior of bitumen for changes in stress levels. This is very important from field considerations as pavements are subjected to different type of loads, which may change bitumen property significantly, if it is stress sensitive. To understand the stress sensitivity, binders were subjected to different stress levels such as 0.1 kPa, 0.5 kPa, 1 kPa, 1.5 kPa, 2 kPa, 2.5 kPa, 3 kPa, 3.2 kPa. Stress sensitivity indicates the behavior of binder when it is subjected to different magnitudes of stresses. A more stress sensitive binder, may not perform intended function. Figure 4.16 and Figure 4.17 shows a plot of J_{nr} and %Recovery for C.B and M.B for 10 and 30 cycles test with 0%, 1%, 2%, 3% and 4% addition of MCP, respectively. For 10 cycles, it is seen that for control binder, J_{nr} increasing drastically, when change creep from 0.1 kPa to 0.5 kPa and keep on increasing as the stresses are increasing as shown in Figure 4.16 (a). But as the addition of 1% of MCP in neat binder the value of J_{nr} decreases as compare to neat binder as shown in Figure 4.16 (b). However, in case of modified binder with 2% MCP, no significant increase in J_{nr} was found when creep stress levels were increased as compare to other modified binder. As the addition of 3% MCP in C.B the value of J_{nr} increases at every stress level. From Figure 4.17 (a-e) it is observed that maximum stress sensitivity is shown by control bonder. This behaviour of binder modified with MCP clearly indicates that it is less stress sensitive and does not yield for changes in stress levels even at high temperatures such as 65^{o}C.


2000
1800
1600
1400
1200
1000
800
600
400
200
0
Jnr (kPa-1)
0
-0.5
-1
-1.5
-2
-2.5
-3
-3.5
-4
-4.5
% Recovery
1
2
3
4
5
6
7
8
9
10
Cycle number
Jnr-0%-0.1kPa
Jnr-0%-0.5kPa
Jnr-0%-1kPa
Jnr-0%-1.5kPa
Jnr-0%-2kPa
Jnr-0%-2.5kPa
Jnr-0%-3kPa
Jnr-0%-3.2kpa
%R-0%-0.1kPa
%R-0%-0.5kPa
%R-0%-1kPa
%R-0%-1.5kPa
%R-0%-2kPa
%R-0%-2.5kPa
%R-0%-3kPa
%R-0%-3.2kPa

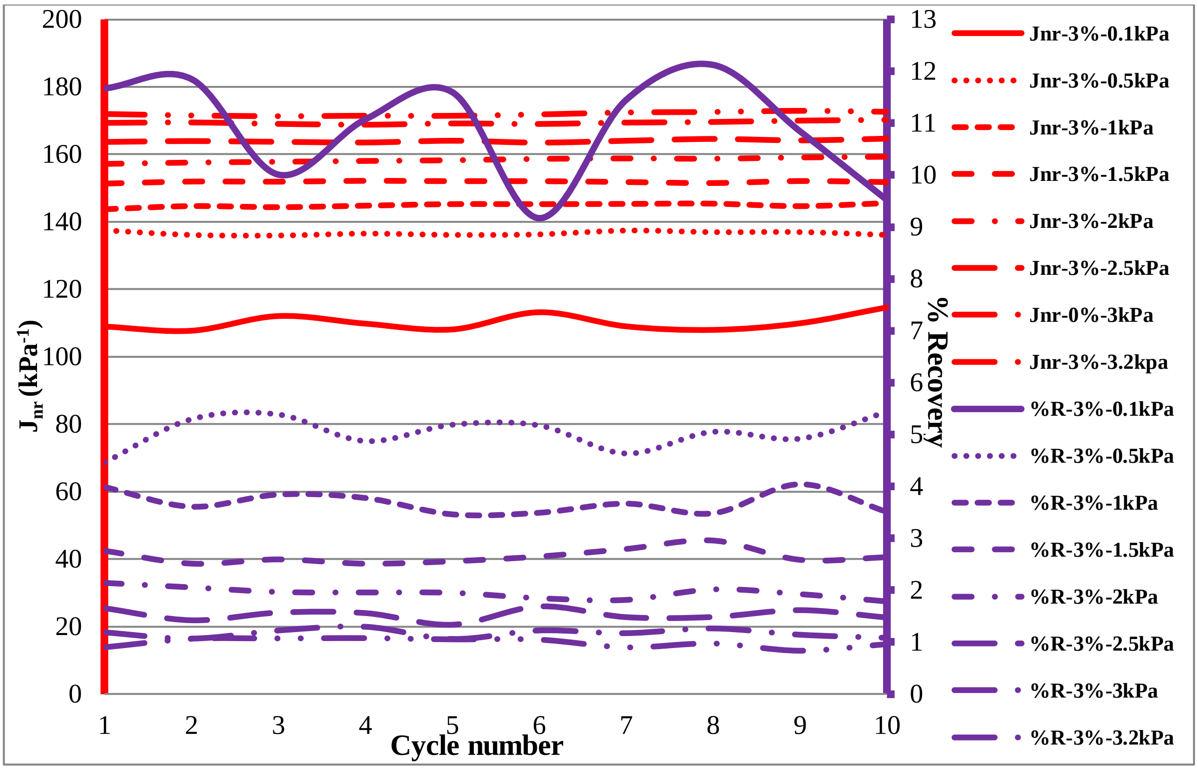
(a)


300
250
200
150
100
50
0
Jnr (kPa-1)
13
12
11
10
9
8
7
6
5
4
3
2
1
0
% Recovery
1
2
3
4
5
6
7
8
9
10
Cycle number
Jnr-1%-0.1kPa
Jnr-1%-0.5kPa
Jnr-1%-1kPa
Jnr-1%-1.5kPa
Jnr-1%-2kPa
Jnr-1%-2.5kPa
Jnr-1%-3kPa
Jnr-1%-3.2kpa
%R-1%-0.1kPa
%R-1%-0.5kPa
%R-1%-1kPa
%R-1%-1.5kPa
%R-1%-2kPa
%R-1%-2.5kPa
%R-1%-3kPa
%R-1%-3.2kPa

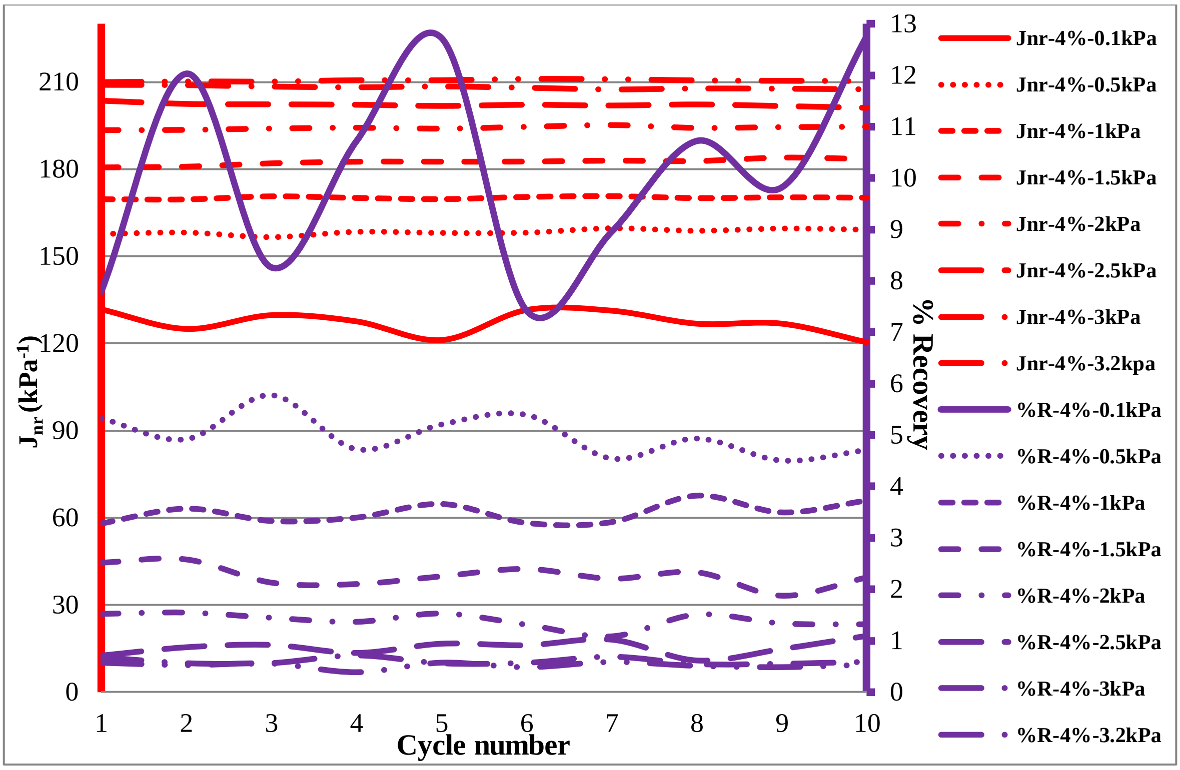
(b)


160
140
120
100
80
60
40
20
0
J_{nr} (kPa^{-1})
20
18
16
14
12
10
8
6
4
2
0
% Recovery
1
2
3
4
5
6
7
8
9
10
Cycle number
Jnr-2%-0.1kPa
Jnr-2%-0.5kPa
Jnr-2%-1kPa
Jnr-2%-1.5kPa
Jnr-2%-2kPa
Jnr-2%-2.5kPa
Jnr-2%-3kPa
Jnr-2%-3.2kpa
%R-2%-0.1kPa
%R-2%-0.5kPa
%R-2%-1kPa
%R-2%-1.5kPa
%R-2%-2kPa
%R-2%-2.5kPa
%R-2%-3kPa
%R-2%-3.2kPa

(c)


200
180
160
140
120
100
80
60
40
20
0
J_{nr} (kPa^{-1})
13
12
11
10
9
8
7
6
5
4
3
2
1
0
% Recovery
1
2
3
4
5
6
7
8
9
10
Cycle number
Jnr-3%-0.1kPa
Jnr-3%-0.5kPa
Jnr-3%-1kPa
Jnr-3%-1.5kPa
Jnr-3%-2kPa
Jnr-3%-2.5kPa
Jnr-0%-3kPa
Jnr-3%-3.2kpa
%R-3%-0.1kPa
%R-3%-0.5kPa
%R-3%-1kPa
%R-3%-1.5kPa
%R-3%-2kPa
%R-3%-2.5kPa
%R-3%-3kPa
%R-3%-3.2kPa

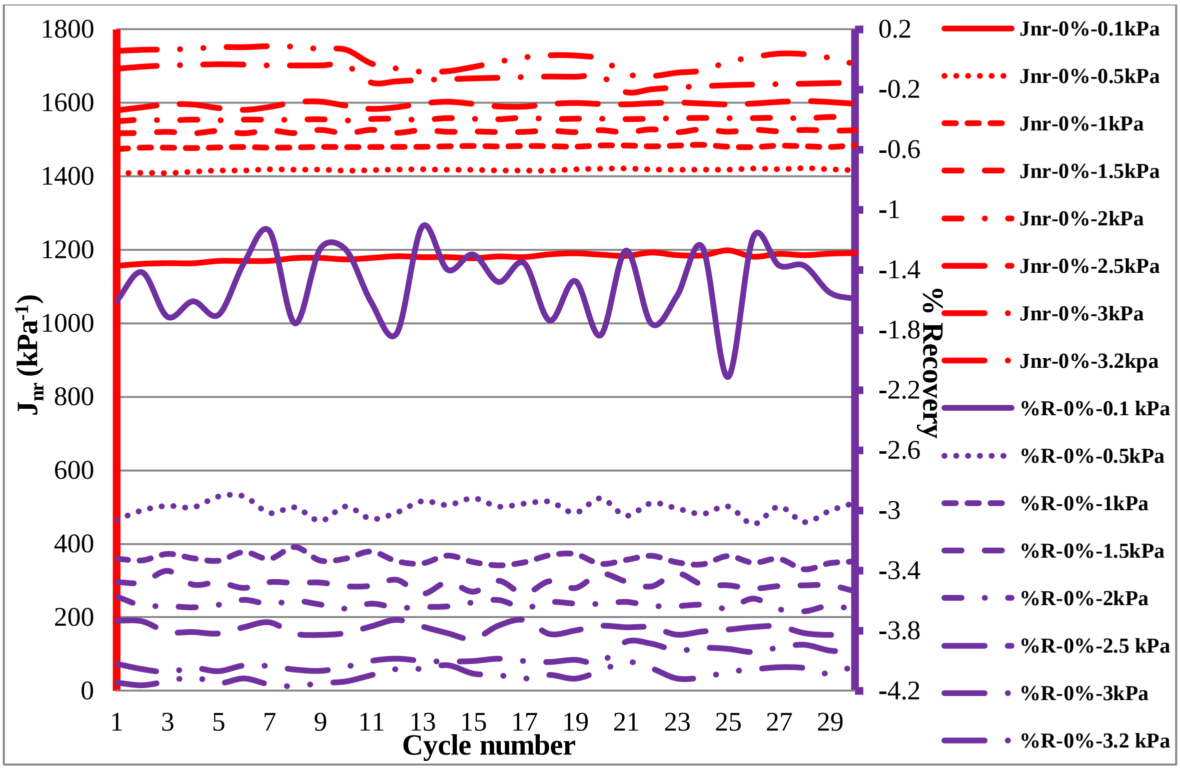
(d)



(e)

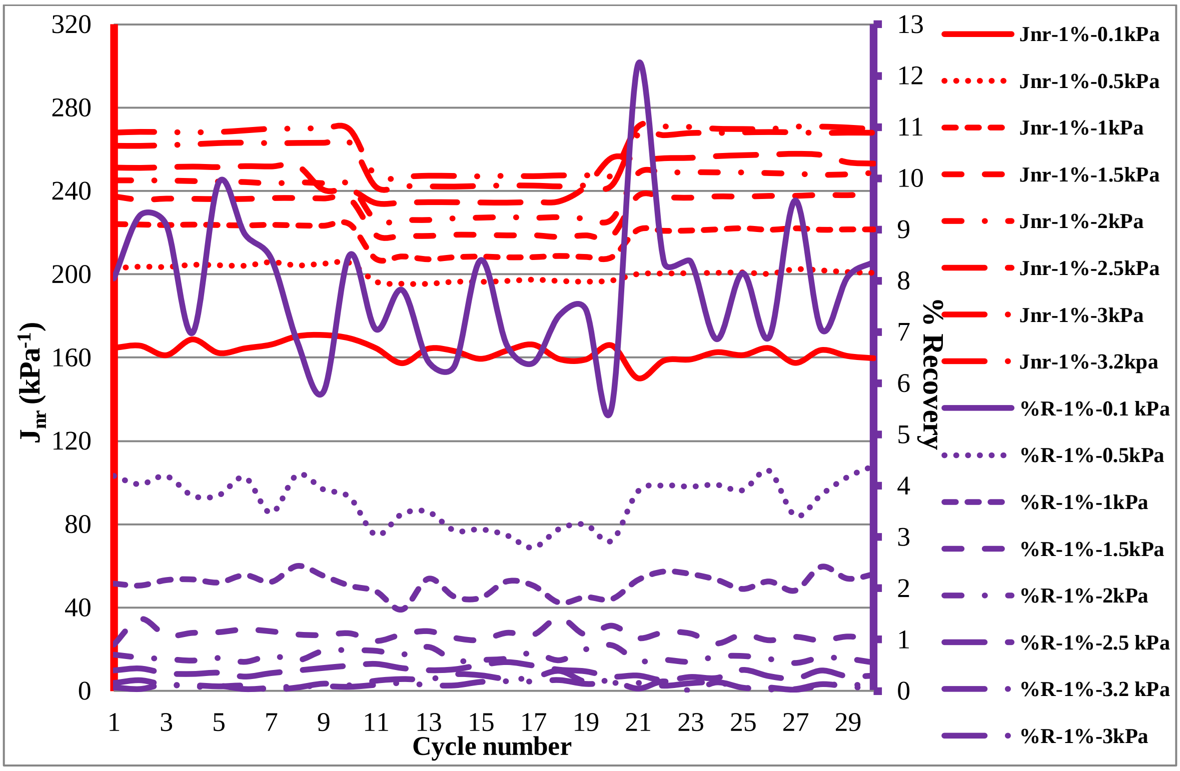
Figure 4.22 Jnr and %R for 10 Creep Recovery Cycles of MSCR Test at Different Stress Levels

(a) C.B, (b) C1% M.B, (c) C2% M.B, (d) C3% M.B and (e) C4% M.B



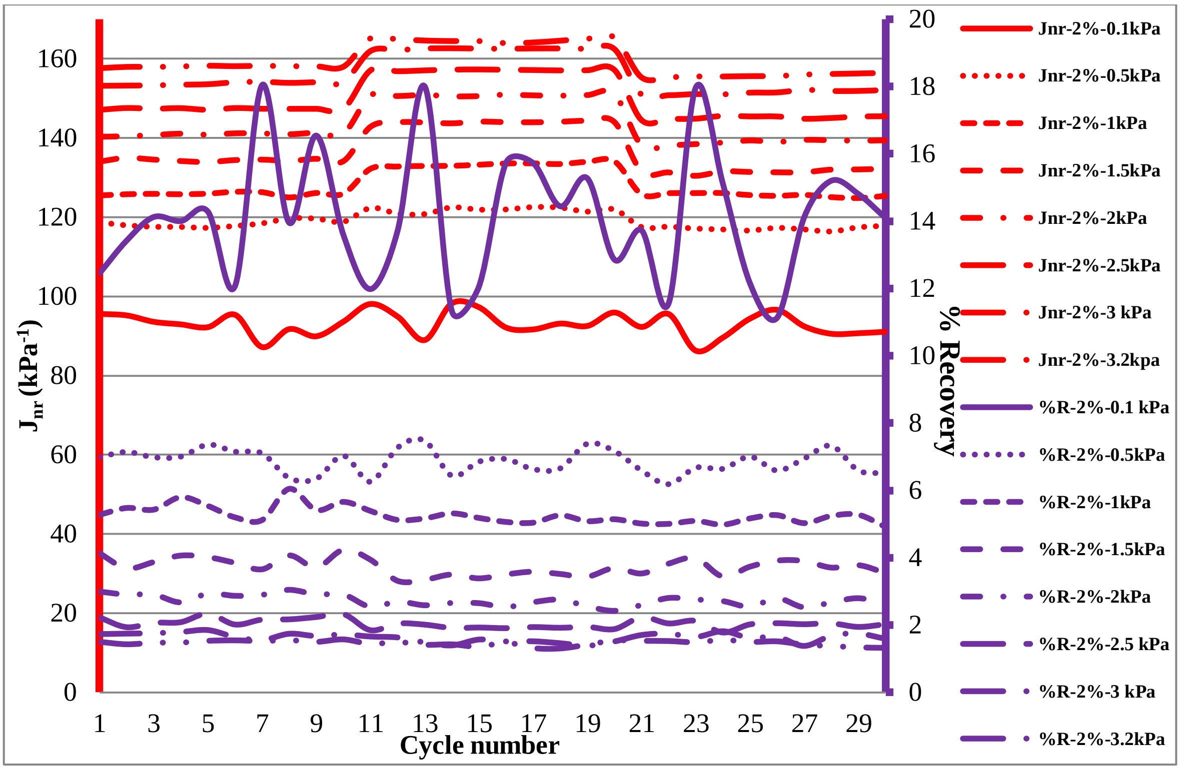
1800
1600
1400
1200
1000
800
600
400
200
0
J_{nr} (kPa^{-1})
0.2
-0.2
-0.6
-1
-1.4
-1.8
-2.2
-2.6
-3
-3.4
-3.8
-4.2
% Recovery
1 3 5 7 9 11 13 15 17 19 21 23 25 27 29
Cycle number
Jnr-0%-0.1kPa
Jnr-0%-0.5kPa
Jnr-0%-1kPa
Jnr-0%-1.5kPa
Jnr-0%-2kPa
Jnr-0%-2.5kPa
Jnr-0%-3kPa
Jnr-0%-3.2kpa
%R-0%-0.1 kPa
%R-0%-0.5kPa
%R-0%-1kPa
%R-0%-1.5kPa
%R-0%-2kPa
%R-0%-2.5 kPa
%R-0%-3kPa
%R-0%-3.2 kPa

(a)

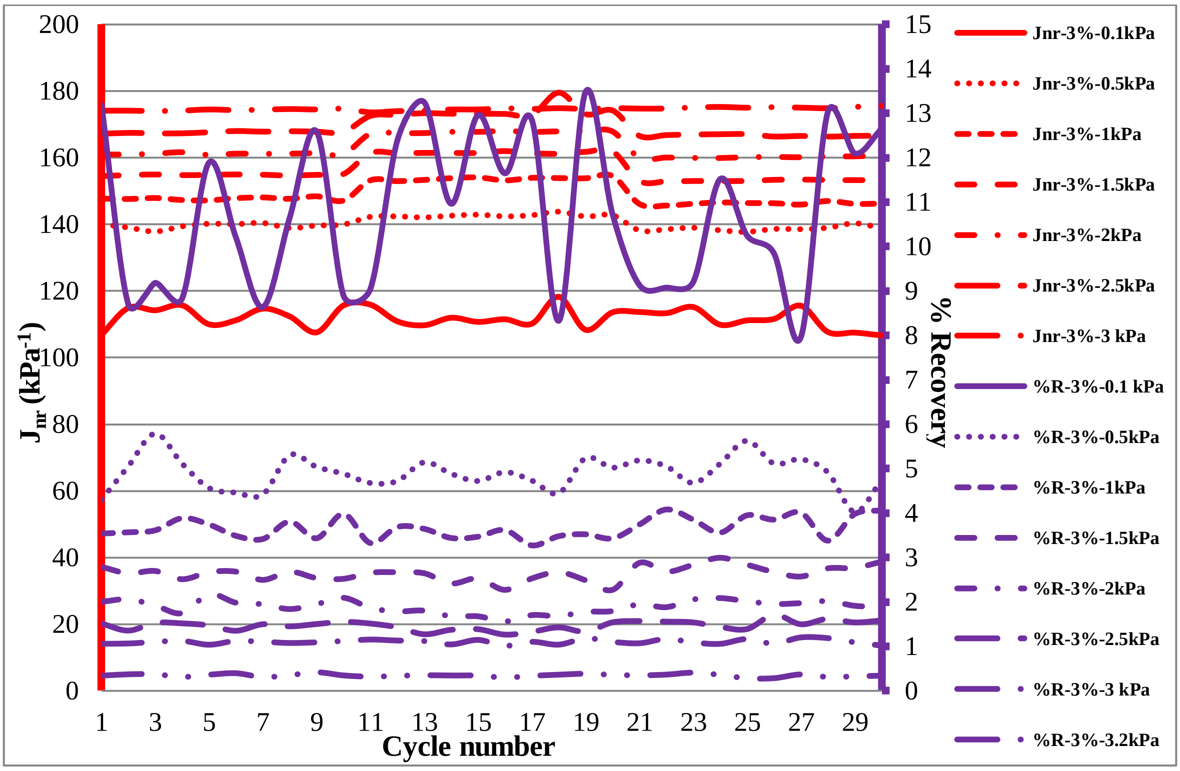


320
280
240
200
160
120
80
40
0
J_{nr} (kPa^{-1})
13
12
11
10
9
8
7
6
5
4
3
2
1
0
% Recovery
1 3 5 7 9 11 13 15 17 19 21 23 25 27 29
Cycle number
Jnr-1%-0.1kPa
Jnr-1%-0.5kPa
Jnr-1%-1kPa
Jnr-1%-1.5kPa
Jnr-1%-2kPa
Jnr-1%-2.5kPa
Jnr-1%-3kPa
Jnr-1%-3.2kpa
%R-1%-0.1 kPa
%R-1%-0.5kPa
%R-1%-1kPa
%R-1%-1.5kPa
%R-1%-2kPa
%R-1%-2.5 kPa
%R-1%-3.2 kPa
%R-1%-3kPa

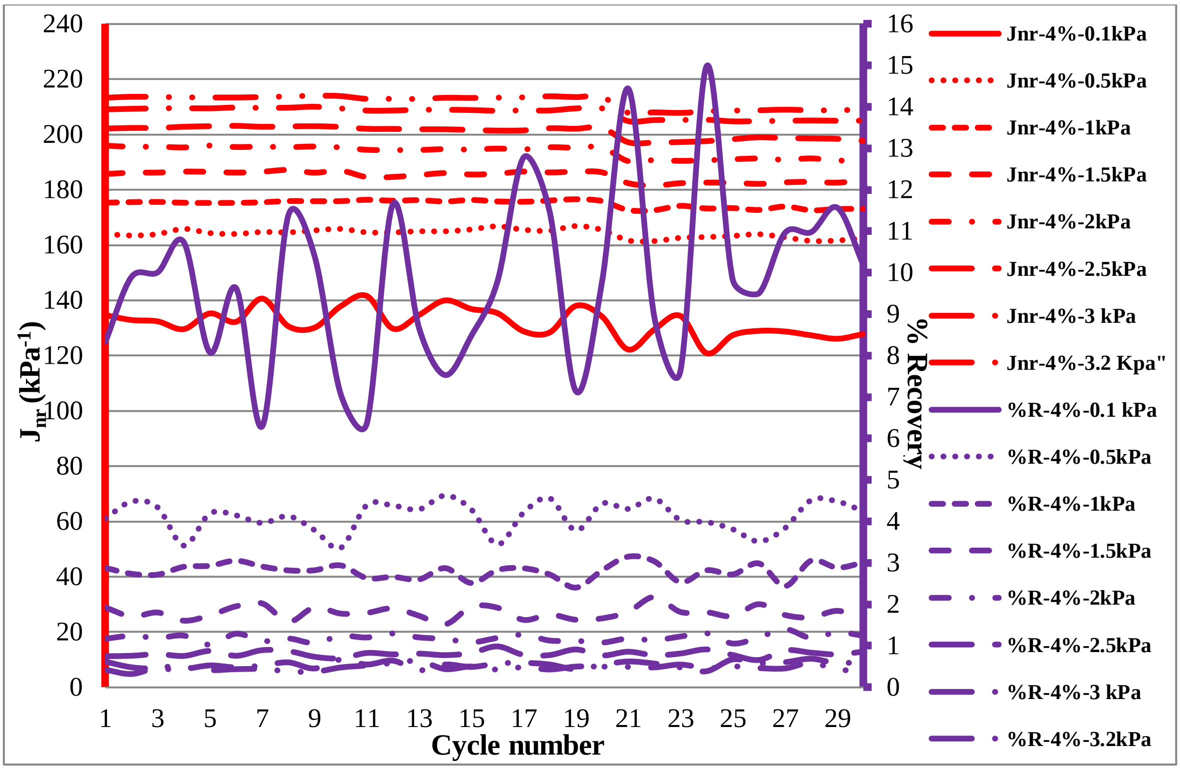
(b)


Jnr (kPa-1)
% Recovery
Cycle number
Jnr-2%-0.1kPa
Jnr-2%-0.5kPa
Jnr-2%-1kPa
Jnr-2%-1.5kPa
Jnr-2%-2kPa
Jnr-2%-2.5kPa
Jnr-2%-3 kPa
Jnr-2%-3.2kpa
%R-2%-0.1 kPa
%R-2%-0.5kPa
%R-2%-1kPa
%R-2%-1.5kPa
%R-2%-2kPa
%R-2%-2.5 kPa
%R-2%-3 kPa
%R-2%-3.2kPa

(c)


Jnr (kPa-1)
% Recovery
Cycle number
Jnr-3%-0.1kPa
Jnr-3%-0.5kPa
Jnr-3%-1kPa
Jnr-3%-1.5kPa
Jnr-3%-2kPa
Jnr-3%-2.5kPa
Jnr-3%-3 kPa
%R-3%-0.1 kPa
%R-3%-0.5kPa
%R-3%-1kPa
%R-3%-1.5kPa
%R-3%-2kPa
%R-3%-2.5kPa
%R-3%-3 kPa
%R-3%-3.2kPa

(d)



(e)

Figure 4.23 Jnr and %R for 10 Creep Recovery Cycles of MSCR Test at Different Stress Levels

(a) C.B, (b) C1% M.B, (c) C2% M.B, (d) C3% M.B and (e) C4% M.B

4.2.9 Rotational viscosity test

Viscosity test is used to determine the resistance of bitumen. Absolute viscosity at 60°C was determined with the help of DSR parallel plate geometry. Viscosity is important parameter during construction of flexible pavements because it analysis the mixing, pumping and compaction temperature of asphalt binder. If the viscosity is low then binder will leads to rutting at higher temperature. At high viscosity cracks will occurs without loading on low temperature. So suitable viscosity is needed according to the climate conditions. Figure4.24 illustrates that the viscosity test was done at different shear rates, to find the particular shear rate for control and modified binder. As the increase of shear rate viscosity is decreasing and at shear rate of 3 (1/s) viscosities was achieved for control binder and at 15 (1/s) shear rate modified binder's viscosity was achieved. But at 2% addition of MCP gives maximum viscosity. This improves the resistance to permanent deformation and increase the stiffness of asphalt binder at high temperature during service period of pavement. The values of viscosity at 15 (1/s) shear rate are 2555.75 P, 3287.8 P, 3421.35 P and 27.15 P for 1%, 2%, 3% and 4% modified binder. Where P is poises.

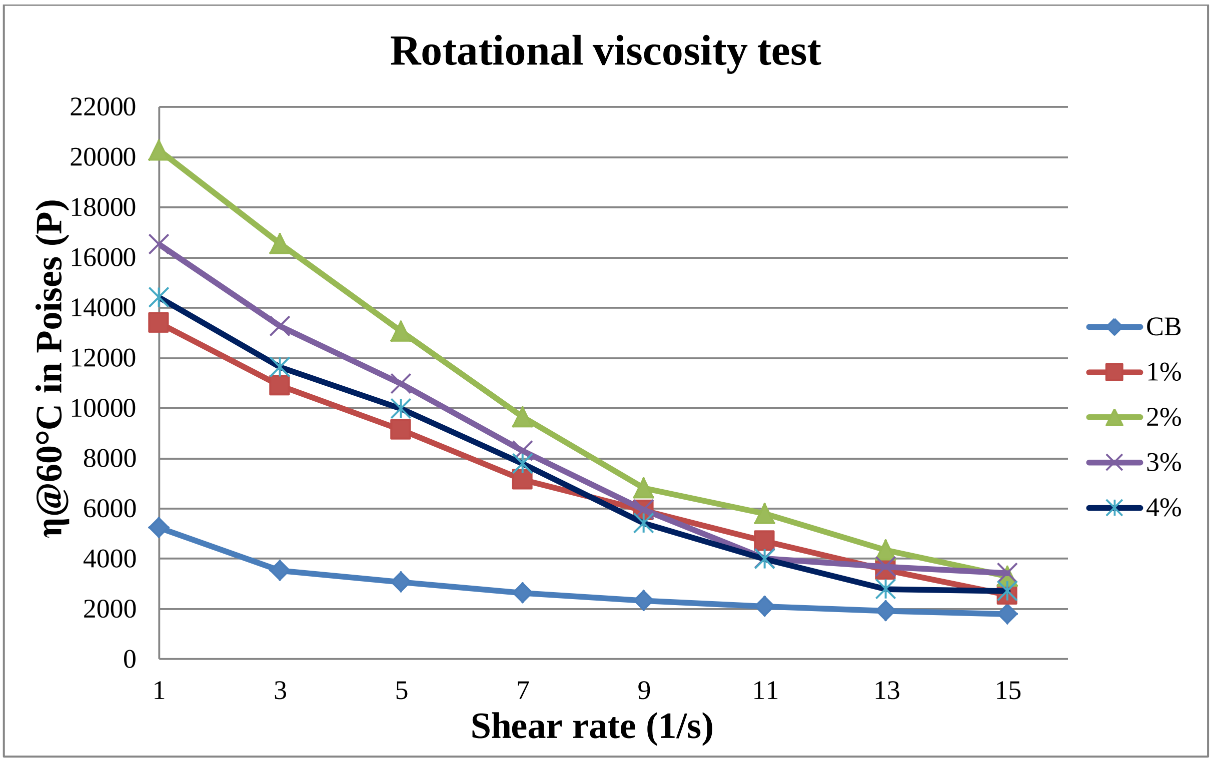
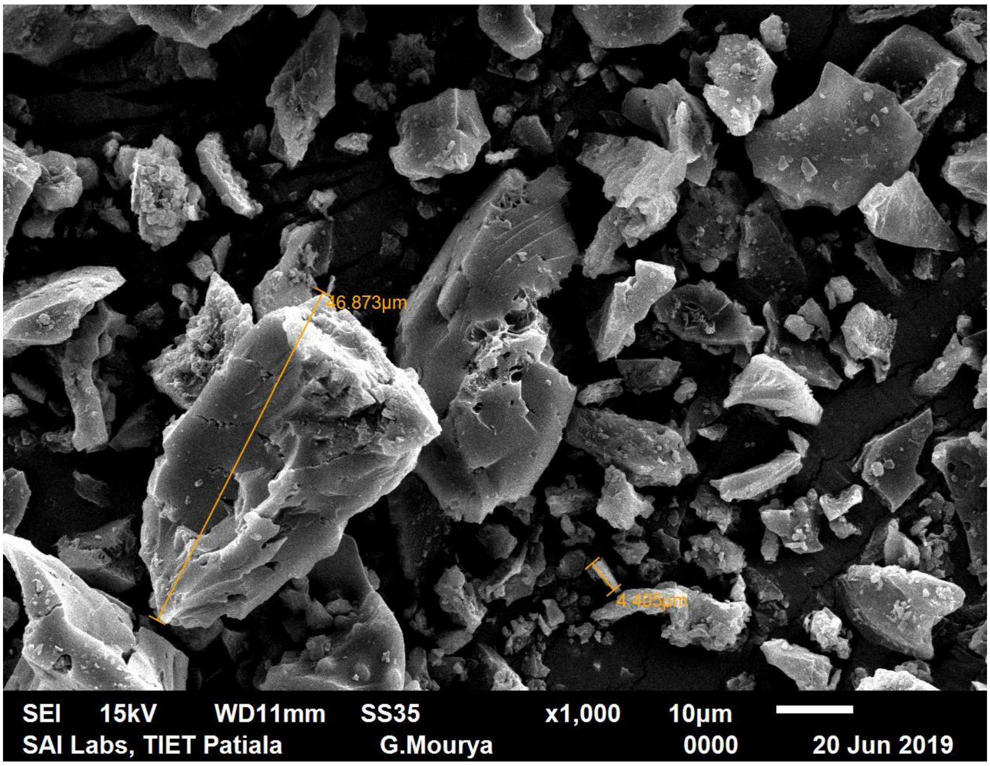


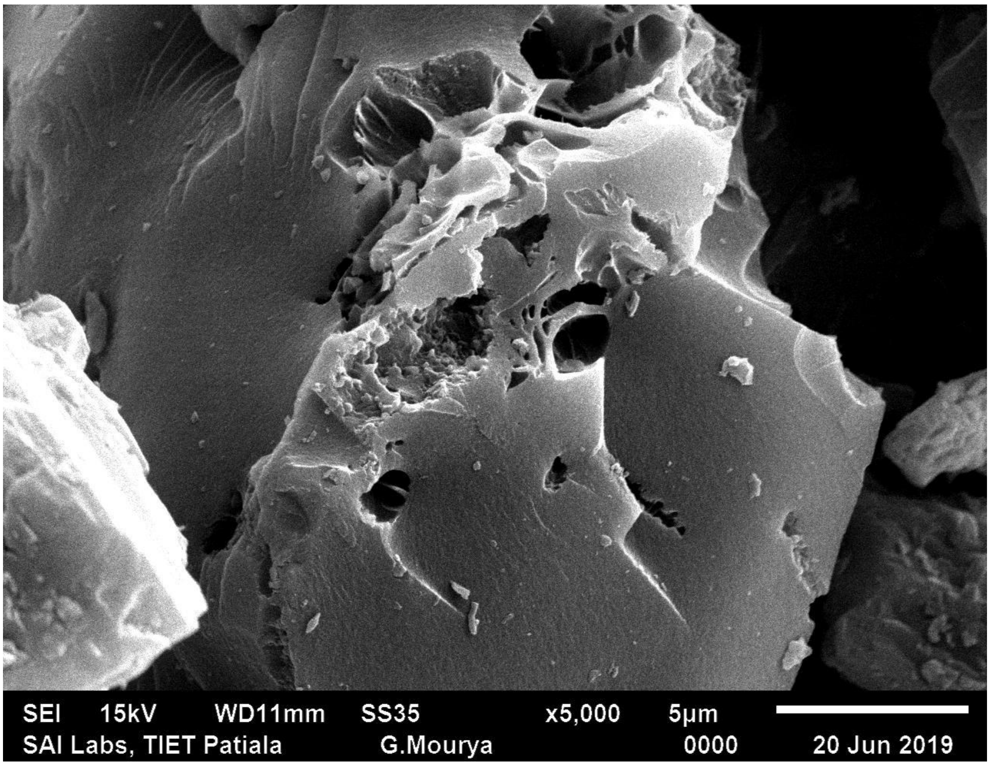
Figure 4.24 Rotational Viscosity Test

4.2.10 Morphology of microcharcoal powder and modified binder (SEM test)

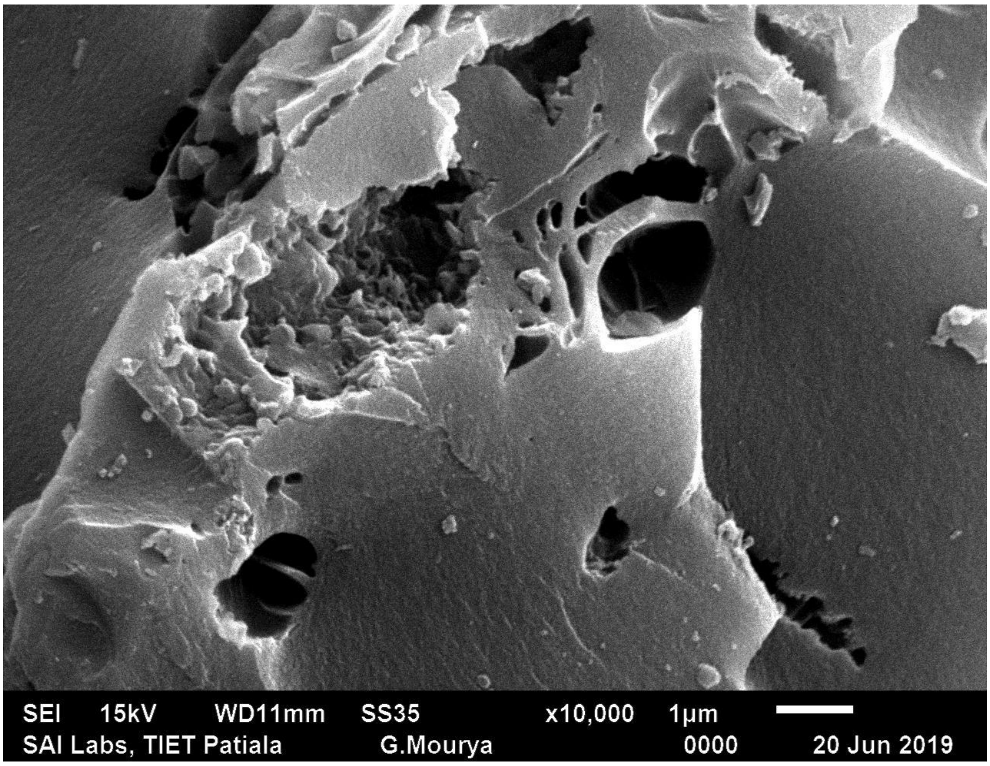
Scanning electron microscope (SEM) test was done for MCP and modified binders to evaluate the shape, size surface structure of MCP and also to see the dispersion of MCP into neat binder. Figure 4.25 (a) shows that the shapes of the MCP is angular and has sharp edges and the maximum and minimum sizes observed as is less than 75 µm i.e. 46.873 µm and 4.405 µm and the surface of MCP is rough have voids on the surface as shown in Figure 4.25 (b) at 5000x magnification. Both the images were taken at 1000 magnification but at different scale, to see the shape and size scale used was 10 µm and for surface analysis 1µm scale was used. Figure 4.25 (c) is the clear image of surface roughness and voids.


36.873µm
4.405µm
SEI 15kV WD11mm SS35 x1,000 10µm
SAI Labs, TIET Patiala G.Mourya 0000 20 Jun 2019

(a)


SEI 15kV WD11mm SS35 x5,000 5µm
SAI Labs, TIET Patiala G.Mourya 0000 20 Jun 2019

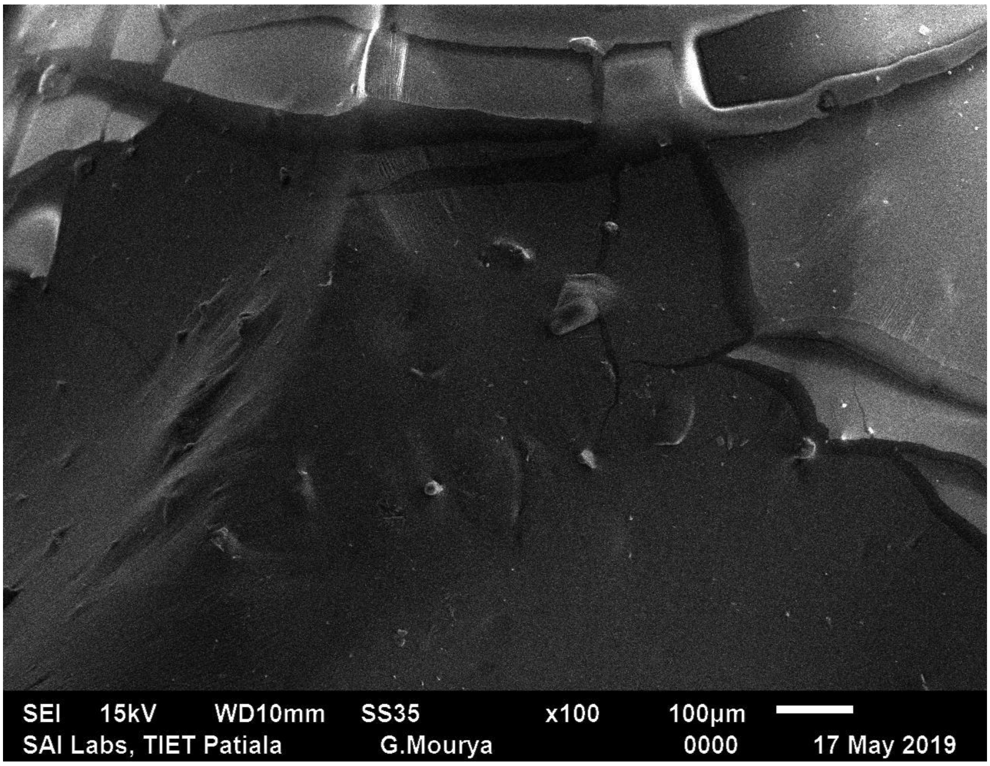
(b)



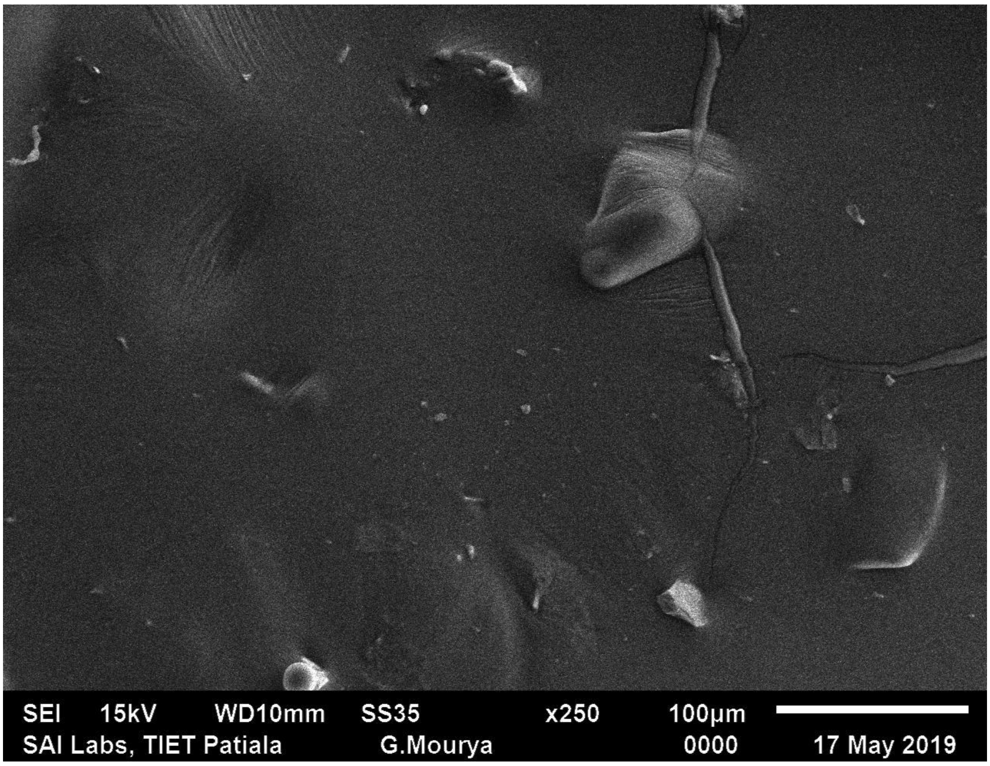
(c)

Figure 4.25 SEM of MCP at Different Magnifications (a) 1000x (b) 5000x and (c) 10,000x

Figure 4.26 (a-b) shows the SEM of C1% MCP modified binder. It can be seen that the addition of MCP at C1% content binder is not mixed properly and clogs were formed which affects the performance of binder during loading periods and at high temperature.



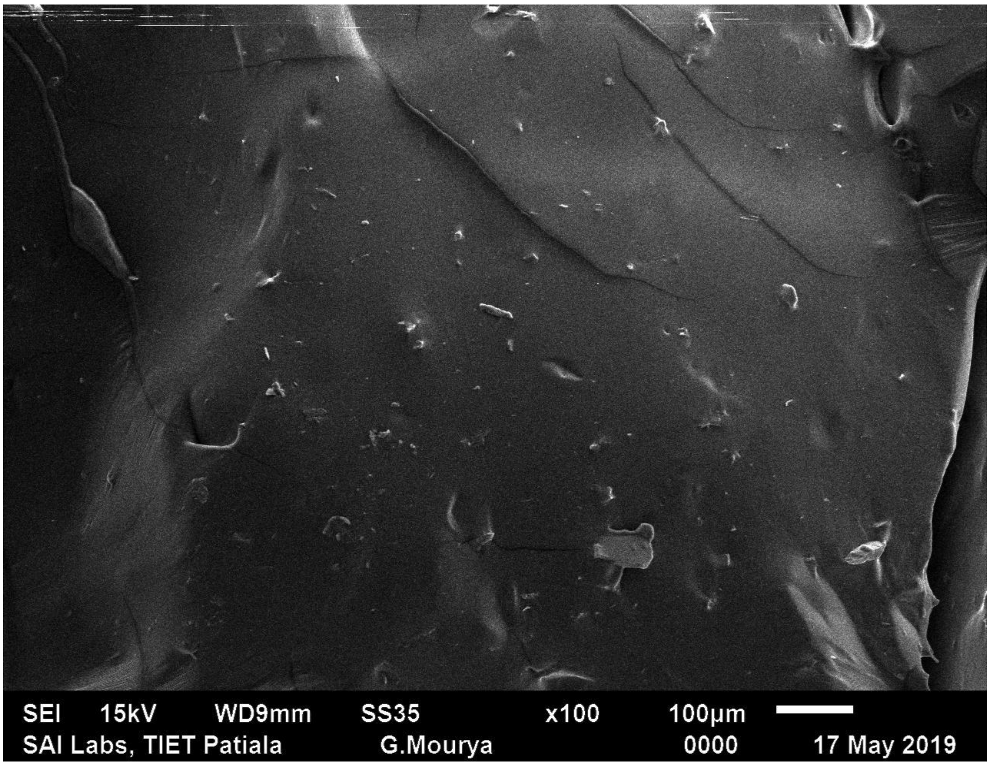
(a)



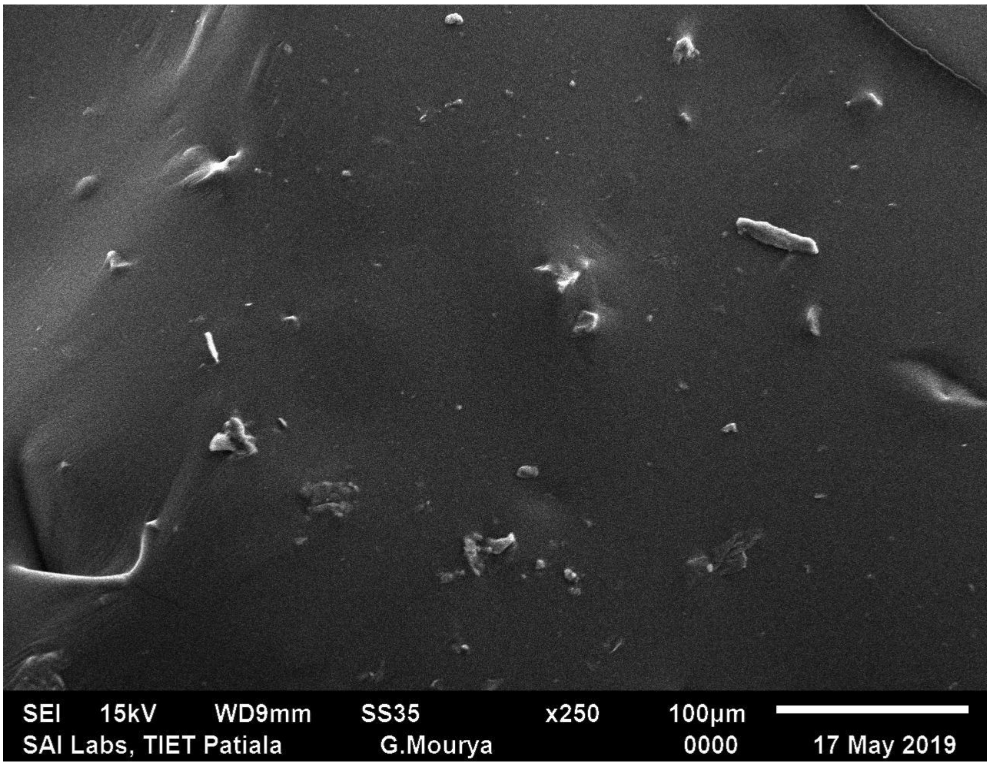
(b)

Figure 4.26 C1% MCP Modified Binder at (a) 100x (b) 250x

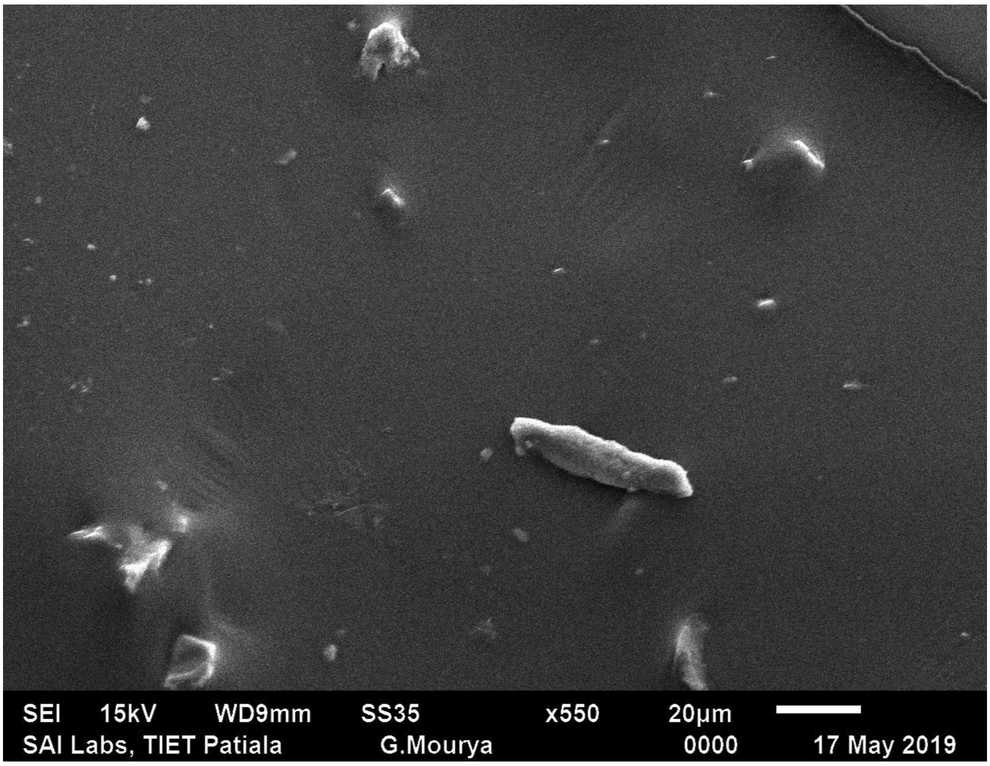
As the increase of MCP content at C2% then MCP is fully dispersed and shows homogeneous mixture which helps to increase the overall performance of asphalt binder as seen in Figure 4.27 (a-c). Because of this reason C2% modified binder gives best results.



(a)



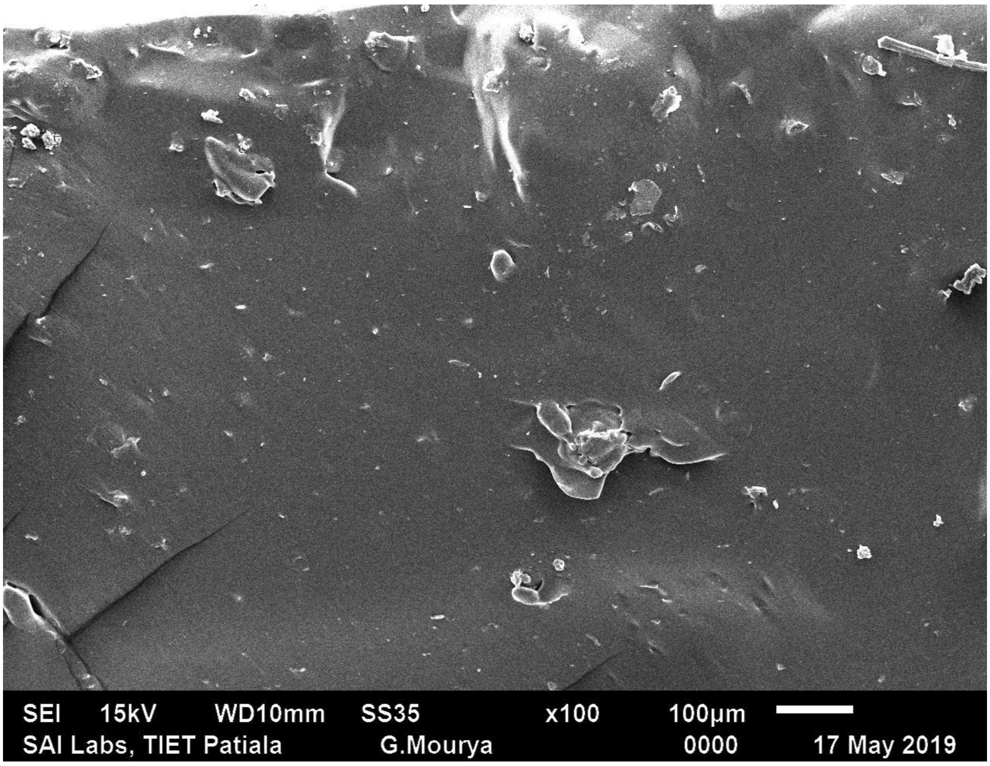
(b)



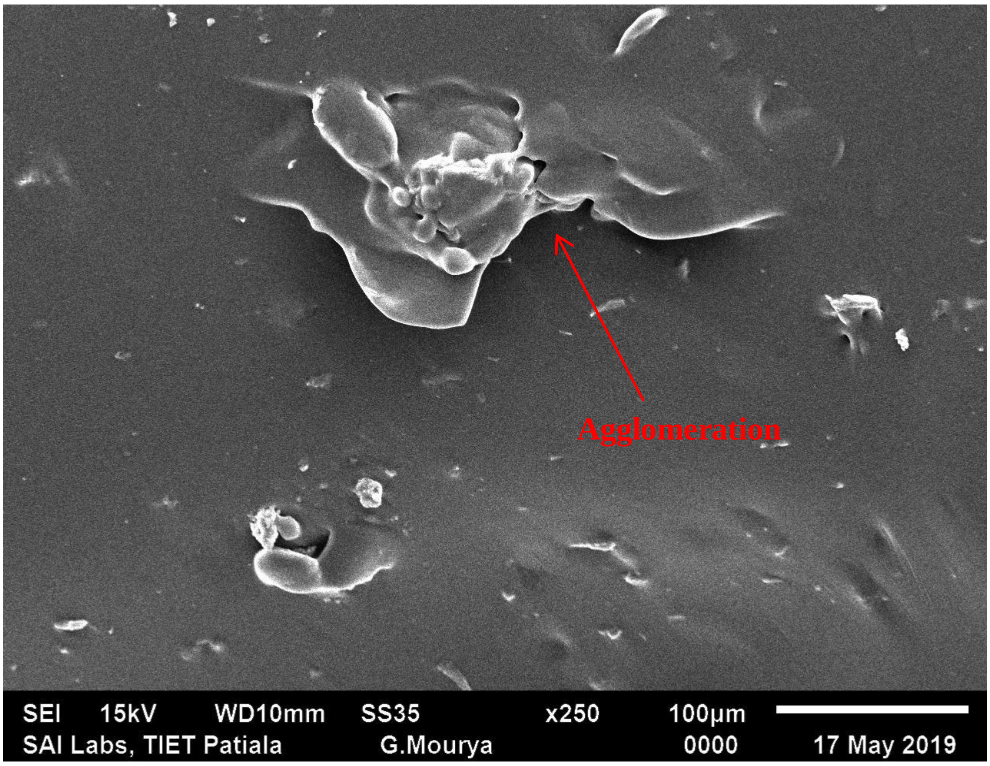
(c)

Figure 4.27 C2% MCP Modified Binder at (a) 100x (b) 250x and (c) 550x

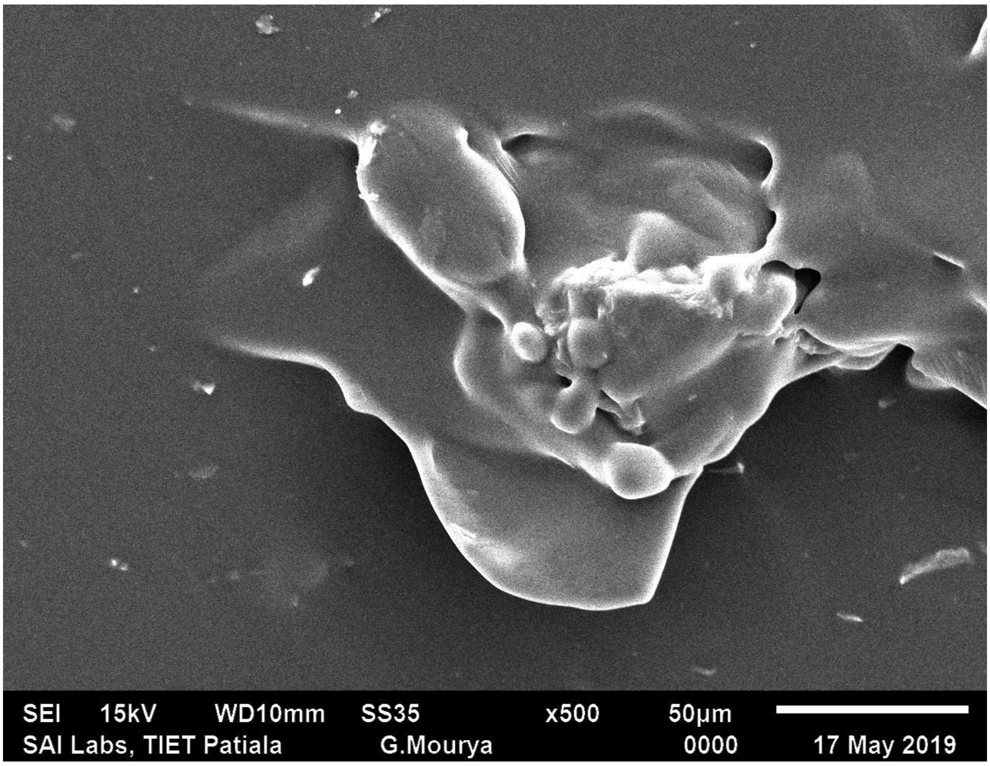
If we add more content of MCP i.e. C3% again it starts to form agglomerations but in this at high rate as shown in Figure 4.28 (c)



(a)



(b)



(c)

Figure 4.28 C3% MCP Modified Binder at (a) 100x (b) 250x and (c) 500x

CHAPTER-5

STATSTICAL ANALYSIS

5.1 GENERAL

This chapter discusses the repeatability of the trials of MSCR test for 10 and 30 cycles at each stress levels with the help of coefficient of variable (CV %) and also done the SPSS for to see the interaction between two independent variables such as proportions of MCP and stress levels on dependent variables such as percentage recovery (%R) and non-recoverable creep compliance (J_{nr}) and compare the mean of groups. In SPSS parametric and non-parametric tests were performed and done the post hoc test to check the interaction between each proportions of MCP and each stress levels.

5.2 ANALYSIS OF REPEATABILITY OF MSCR TEST RESULTS

MSCR test was done for control and modified binders at various proportions of MCP and stress levels to evaluate the percentage recovery (%R) and non-recoverable creep compliance (J_{nr}). For every stress level, three trails were done and run for 10 and 30 cycles. So we get large number of data values. To evaluate the repeatability of the trials of MCSR test coefficient of variable (CV %) method was adopted. CV (%) is the ratio of standard deviation to mean of data sample, which is expressed in percentage. If the value of CV is low, it means data is consistent, homogeneous, stable and has less variation and if CV is high then data is variable. CV% for control binder didn't take because the data of %R are in negative for both the cases i.e. for 10 and 30 cycles.

Table 5.1 shows the CV (%) of %R for 10 cycles at various stress levels of modified binders. It is observed that for C1% modified binder at every stress levels, the CV % is more than 10% (Hilde Soenen, 2013) except for 1.5 kPa; it means variation is more between the trails and data is not consistent. For C2% and 4% modified binders, the value of CV% is less means variations is less between the trails and data is consistent and stable. For C3% modified binder the values are less than 5% as mentioned in box.

Table 5.2 shows the repeatability of J_{nr} for 10 cycles, it can be seen that for all modified binders at every stress levels the values of CV% is less than 10% but for every stress levels and for every modified binder the values are less than 5% or at some places the values are close to 0%, it means there is very less differences between three trail's data and our results are consistent and less variable.

Table 5.1 CV % of %R for 10 Cycles

	Stress Levels							
Modified binder	**0.1 kPa**	**0.5 kPa**	**1 kPa**	**1.5 kPa**	**2 kPa**	**2.5 kPa**	**3 kPa**	**3.2 kPa**
1%	14.53	23.87	21.47	5.58	13.48	39.10	40.01	15.68
2%	9.37	7.65	8.53	8.11	6.23	7.67	7.030	11.12
3%	9.22	4.56	3.27	3.02	3.30	4.42	10.05	12.67
4%	10.50	5.10	8.06	5.09	6.13	7.96	12.70	4.57

Table 5.2 CV % of J_{nr} (1/kPa) For 10 Cycles

	Stress Levels							
Modified binder	**0.1 kPa**	**0.5 kPa**	**1 kPa**	**1.5 kPa**	**2 kPa**	**2.5 kPa**	**3 kPa**	**3.2 kPa**
1%	1.22	0.94	2.55	5.65	6.06	4.85	1.32	1.85
2%	2.36	1.67	1.69	1.25	1.45	1.32	1.14	0.76
3%	4.51	3.38	4.01	4.60	5.02	5.54	5.77	5.45
4%	0.63	1.12	1.74	2.05	2.70	2.65	3.07	3.15

Table 5.3 shows the CV (%) of %R for 30 cycles at different stress levels. It is analysed that the CV% values for 0.5, 2.5, 3 and 3.2 kPa is more than 10% for C1% modified binder. It means trials are highly different to each other and for C2%, C3% and C4% modified binders, values are less than 10% and close to 1% so these trials have consistent data.

Table 5.3 CV % of %R for 30 Cycles

	Stress Levels							
Modified binder	**0.1 kPa**	**0.5 kPa**	**1 kPa**	**1.5 kPa**	**2 kPa**	**2.5 kPa**	**3 kPa**	**3.2 kPa**
1%	9.94	13.02	8.07	4.8	8.32	23.30	62.15	133.45
2%	0.92	1.81	4.00	5.45	5.63	5.48	6.58	3.17
3%	6.43	1.19	4.89	5.25	7.33	6.09	5.39	5.39
4%	9.61	3.06	3.60	2.32	2.48	2.26	5.64	8.83

Table 5.4 showed that for each stress levels and modified binders are values are less than 10% so for every binder the differences in the trials are less it means trials are consistent.

Table 5.4 CV % of J_{nr} (1/kPa) For 30 Cycles

	Stress Levels							
Modified binder	**0.1 kPa**	**0.5 kPa**	**1 kPa**	**1.5 kPa**	**2 kPa**	**2.5 kPa**	**3 kPa**	**3.2 kPa**
1%	2.04	1.97	3.86	4.60	4.82	4.65	5.21	4.93
2%	1.27	2.05	3.40	4.74	4.40	4.25	3.72	2.89
3%	0.51	1.47	2.64	2.82	2.50	2.27	1.83	0.66
4%	3.03	0.95	0.89	1.13	1.32	1.26	1.17	1.34

5.3 STATISTICAL ANALYSIS USING SPSS

Statistical analysis in Statistics Package for Social Science (SPSS) is conducted to check the difference in the mean of groups. The purpose of doing statistical analysis is due to large number of data set produced by MSCR test. This test contains 2 independent variables and 2 dependent variable, now task is to see the interaction between 2 independent variables i.e. proportions of MCP and stress levels on the dependent variables i.e. %R and J_{nr}. To evaluate the interaction two-way ANOVA test is performed.

5.3.1 Analysis of MSCR Test for 10 Cycles

5.3.1.1 Normality and Homogeneity of Variance Assumptions for %R and J_{nr} Based on Proportions

Before performing the two-way ANOVA, first of check the assumptions of two-way ANOVA i.e. normality and homogeneity of variance of data sets, whether data set follows the assumption or not. Table 5.5 shows the results of %R and J_{nr} values based on proportions of MCP. It can be seen that assumptions of ANOVA are not following i.e. neither data values are normally distributed nor homogeneous in the data sets, this is due to significance value (i.e. p- value is 0.00) less than 0.05 value as mentioned in the box below. This shows null hypothesis is rejected. It means there are statistically significantly different between the mean of groups i.e. proportions of MCP effects the %R and J_{nr} values significantly. The assumptions of normality and homogeneity of variance has been violated. In that case we have to do non-parametric tests i.e. Kruskal-Wallis H Test.

Table 5.5 Assumptions for Two Way ANOVA Based on MCP Proportions for 10 Cycles

(a) Normality and (b) Homogeneity of Variance

Tests of Normality							
	Proportion	**Kolmogorov-Smirnov[a]**			**Shapiro-Wilk**		
		Statistic	**df**	**Sig.**	**Statistic**	**df**	**Sig.**
%Recovery	C0%	.215	240	.000	.751	240	.000
	C1%	.221	240	.000	.761	240	.000
	C2%	.192	240	.000	.766	240	.000
	C3%	.194	240	.000	.748	240	.000
	C4%	.204	240	.000	.756	240	.000
J_{nr}	C0%	.164	240	.000	.846	240	.000
	C1%	.135	240	.000	.920	240	.000
	C2%	.117	240	.000	.901	240	.000
	C3%	.103	240	.000	.941	240	.000
	C4%	.124	240	.000	.918	240	.000

(a)

Test of Homogeneity of Variance (Levene's Test of Equality of Error Variances)					
		Levene Statistic	**df1**	**df2**	**Sig.**
%Recovery	Based on Mean	43.262	4	1195	.000
	Based on Median	24.246	4	1195	.000
	Based on Median and with adjusted df	24.246	4	928.985	.000
	Based on trimmed mean	35.070	4	1195	.000
J_{nr}	Based on Mean	206.276	4	1195	.000
	Based on Median	147.438	4	1195	.000
	Based on Median and with adjusted df	147.438	4	275.573	.000
	Based on trimmed mean	186.153	4	1195	.000

(b)

5.3.1.2 Normality and Homogeneity of Variance Assumptions for %R and J_{nr} Based on Stress Levels

Same test is also run for %R and J_{nr} based on stress levels. Table 5.6 shows that there is significance different between the mean of groups because of p- value (0.00) is less than 0.05 value. It means neither data values are normally distributed nor homogeneous in the data sets. So null hypothesis is rejected and stress levels effects the %R and J_{nr} values significantly. The assumptions of normality and homogeneity of variance has been violated. Due to this, performing of two-way ANOVA is inappropriate. So in that case also conducts the non-parametric test i.e. Kruskal-Wallis H Test.

Table 5.6 Assumption for Two Way ANOVA Based Stress levels for 10 Cycles

(a) Normality and (b) Homogeneity of Variance

Tests of Normality							
	Stress Levels	**Kolmogorov-Smirnov**			**Shapiro-Wilk**		
		Statistic	**df**	**Sig.**	**Statistic**	**df**	**Sig.**
%Recovery	0.1 kPa	.149	150	.000	.926	150	.000
	0.5 kPa	.263	150	.000	.761	150	.000
	1 kPa	.262	150	.000	.774	150	.000
	1.5 kPa	.266	150	.000	.791	150	.000
	2 kPa	.257	150	.000	.779	150	.000
	2.5 kPa	.277	150	.000	.757	150	.000
	3 kPa	.319	150	.000	.721	150	.000
	3.2 kPa	.343	150	.000	.700	150	.000
J_{nr}	0.1 kPa	.447	150	.000	.542	150	.000
	0.5 kPa	.458	150	.000	.537	150	.000
	1 kPa	.451	150	.000	.541	150	.000
	1.5 kPa	.440	150	.000	.547	150	.000
	2 kPa	.435	150	.000	.548	150	.000
	2.5 kPa	.436	150	.000	.547	150	.000
	3 kPa	.442	150	.000	.546	150	.000
	3.2 kPa	.441	150	.000	.545	150	.000

(a)

Test of Homogeneity of Variance (Levene's Test of Equality of Error Variances)					
		Levene Statistic (F)	**df1**	**df2**	**Sig.**
%Recovery	Based on Mean	36.557	7	1192	.000
	Based on Median	27.273	7	1192	.000
	Based on Median and with adjusted df	27.273	7	775.043	.000
	Based on trimmed mean	36.227	7	1192	.000
J_{nr}	Based on Mean	3.117	7	1192	.003
	Based on Median	.616	7	1192	.743
	Based on Median and with adjusted df	.616	7	1151.700	.73
	Based on trimmed mean	2.250	7	1192	.028

(b)

5.3.2 Kruskal-Wallis H Test's Results for 10 Cycles MSCR

Kruskal Wallis H test is the non-parametric and rank based test which determines the significant difference between two independent variables to on dependent variables. It is also refers to the one way ANOVA. It tells only whether there is significantly difference between the groups among three or four groups but it doesn't specify which groups are different. To identify the differences follow the post hoc test with the help of Mann-Whitney U test (two independent samples test.

5.3.2.1 Kruskal-Wallis Test Results of %R and J_{nr} Based on MCP Proportions

Table 5.7 (a) showed that mean rank of %R and J_{nr} for each proportions of MCP which can be used to compare the effects of different proportions of MCP. Whether these proportions of MCP have different %R and J_{nr} can be assessed using test statistics as shown in Table 5.7 (b), this shows that the p-value of %R and J_{nr} is 0.00 which is less than 0.05. It means there are statistically significantly differences in the %R and J_{nr} groups due to proportions of MCP and there is 0% chance to find the similarity.

Now to find which proportions of MCP affects the %R and J_{nr} then follow the post hoc test with the help of Mann-Whitney U test (two independent samples test). In this compare the each proportions of MCP and find whether there is significantly difference or not.

Table 5.7 Kruskal-Wallis Test Results of %R and Jnr Based on MCP Proportions for 10 Cycles

(a) Mean Ranks (b) Test Statistics

Ranks			
	Proportion	**N**	**Mean Rank**
%Recovery	C0%	240	120.50
	C1%	240	587.07
	C2%	240	861.79
	C3%	240	753.63
	C4%	240	679.51
	Total	1200	
J_{nr}	C0%	240	1080.50
	C1%	240	790.68
	C2%	240	211.94
	C3%	240	349.23
	C4%	240	570.15
	Total	1200	

(a)

Test Statistics		
	%Recovery	**Jnr**
Chi-Square	656.543	962.412
df	4	4
Asymp. Sig.	.000	.000

(b)

Table 5.8 showed the Mann-Whitney U (Post Hoc) Test for %R and J_{nr} based on proportions of MCP. It can be seen that in post hoc test compare the percentage of proportion of MCP to each other as shown in Table 5.8. In every comparison the p-value is less than 0.05. It means null hypothesis is rejected and there is statistically significantly different between the mean ranks i.e. every proportions of MCP effects the %R and J_{nr} values significantly.

Table 5.8 Mann-Whitney U (Post Hoc) Test Results of %R and Jnr Based on MCP Proportions for 10 Cycles

Test Statistics						
		Stress Levels	**Mann-Whitney U**	**Wilcoxon W**	**Z**	**Asymp. Sig. (2-tailed)**
%R	C0%	C1%	0.00	28920	-18.954	0.00
		C2%	0.00	28920	-18.954	0.00
		C3%	0.00	28920	-18.954	0.00
		C4%	0.00	28920	-18.954	0.00
J_{nr}	C0%	C1%	0.00	28920	-18.954	0.00
		C2%	0.00	28920	-18.954	0.00
		C3%	0.00	28920	-18.954	0.00
		C4%	0.00	28920	-18.954	0.0
%R	C1%	C2%	14274	43194	-9.560	0.00
		C3%	18324	47244	-6.894	0.00
		C4%	21778	50698	-4.621	0.00
J_{nr}	C1%	C2%	530	29450	-18.605	0.00
		C3%	2721	31641	-17163	0.00
		C4%	8706	37626	-13.224	0.00
%R	C2%	C3%	20613	49533	-5.388	0.00
		C4%	17603	46523	-7.369	0.00
J_{nr}	C2%	C3%	15987	44907	-8.433	0.007
		C4%	5428	34348	-15.382	0.00
%R	C3%	C4%	23138	52058	-3.726	0.00
J_{nr}	C3%	C4%	10561	39481	-12.003	0.00

5.3.2.2 Kruskal-Wallis Test Results of %R and J_{nr} Based on Stress Levels

Table 5.9 (a) showed that mean rank of %R and J_{nr} for each stress levels which can be used to compare the effects of different stress levels. Whether these stress levels have different %R and J_{nr} can be assessed using test statistics as shown in Table 5.9 (b), in this case also the p-value of %R and J_{nr} is 0.00 which is less than 0.05. It means there are statistically significantly differences in the %R and J_{nr} groups due to stress levels and there is 0% chance to find the similarity. The whole data is significant and highly effected by stress levels.

Now which stress levels is responsible for affecting the %R and $J_{nr,}$ to see that follow the post hoc test with the help of Mann-Whitney U test (two independent samples test). In this each stress levels are compare the other each stress levels and find whether there is significantly difference or not.

Table 5.9 Kruskal-Wallis Test Results of %R and Jnr Based on Stress levels for 10 Cycles

(a) Mean Ranks and (b) Test Statistics

Ranks			
	Stress levels	**N**	**Mean Rank**
%Recovery	0.1 kPa	150	954.11
	0.5 kPa	150	838.71
	1 kPa	150	743.74
	1.5 kPa	150	625.77
	2 kPa	150	509.70
	2.5 kPa	150	427.83
	3 kPa	150	367.33
	3.2 kPa	150	336.81
	Total	1200	
J_{nr}	0.1 kPa	150	319.09
	0.5 kPa	150	474.79
	1 kPa	150	548.24
	1.5 kPa	150	606.14
	2 kPa	150	653.01
	2.5 kPa	150	697.11
	3 kPa	150	741.50
	3.2 kPa	150	764.12
	Total	1200	

(a)

Test Statistics		
	%Recovery	**J_{nr}**
Chi-Square	455.745	195.461
df	7	7
Asymp. Sig.	.000	.000

(b)

Table 5.10 showed the test statistics for Mann-Whitney U (Post Hoc) Test for %R and J_{nr} Based on stress levels. It can be seen that in post hoc test compare the stress levels to other each stress levels shown in Table 5.10 In every comparison the p-value is less than 0.05. It means null hypothesis is rejected and there is statistically significantly different between the mean ranks i.e. every proportions of MCP effects the %R and J_{nr} values significantly. But this condition is not applied in every case, as mentioned in red box the p-value is more than 0.05 that means it is not significant. For that cases the stress levels are affecting the %R and J_{nr} values and null hypothesis is accepted, it means there is no significant differences.

Table 5.10 Mann-Whitney U (Post Hoc) Test Results of %R and Jnr Based on Stress Levels for 10 Cycles

Test Statistics						
		Stress Levels	**Mann-Whitney U**	**Wilcoxon W**	**Z**	**Asymp. Sig. (2-tailed)**
%R	0.1 kPa	0.5 kPa	4034	15359	-9.605	0.00
		1 kPa	3674	14999	-10.085	0.00
		1.5 kPa	3600	14925	-10.183	0.00
		2 kPa	3600	14925	-10.183	0.00
		2.5 kPa	3600	14925	-10.183	0.00
		3 kPa	3600	14925	-10.183	0.00
		3.2 kPa	3600	14925	-1.183	0.00
J_{nr}	0.1 kPa	0.5 kPa	6807	18132	-5.914	0.00
		1 kPa	5865	17190	-7.168	0.00
		1.5 kPa	5302	16627	-7.917	0.00
		2 kPa	5012	16337	-8.304	0.00
		2.5 kPa	4782	16107	-8.610	0.00
		3 kPa	4462	15787	-9.036	0.00
		3.2 kPa	4309	15634	-9.239	0.00
%R	0.5 kPa	1 kPa	6026	17351	-6.954	0.00
		1.5 kPa	4108	15433	-9.507	0.00
		2 kPa	3619	14944	-10.158	0.00

		2.5 kPa	3600	14925	-10.183	0.00
		3 kPa	3600	14925	-10.183	0.00
		3.2 kPa	3600	14925	-10.183	0.00
J_{nr}	0.5 kPa	1 kPa	9112	20437	-2.846	0.04
		1.5 kPa	8423	19748	-3.763	0.00
		2 kPa	7586	18911	-4.877	0.00
		2.5 kPa	6815	18140	-5.904	0.00
		3 kPa	6287	17612	-6.606	0.00
		3.2 kPa	5977	17302	-7.019	0.00
%R	1 kPa	1.5 kPa	6660	17985	-6.110	0.00
		2 kPa	4331	15656	-9.210	0.00
		2.5 kPa	3746	15071	-9.989	0.00
		3 kPa	3621	14946	-10.155	0.00
		3.2 kPa	3606	14931	-10.175	0.00
J_{nr}	1 kPa	1.5 kPa	9213	20538	-2.711	0.007
		2 kPa	8613	19938	-3.510	0.00
		2.5 kPa	8064	19389	-4.241	0.00
		3 kPa	7607	18932	-4.849	0.00
		3.2 kPa	7391	18716	-5.137	0.00
%R	1.5 kPa	2 kPa	7550	18875	-4.925	0.00
		2.5 kPa	5559	16884	-7.575	0.00
		3 kPa	4602	15927	-8.849	0.00
		3.2 kPa	4117	15442	-9.495	0.00
J_{nr}	1.5 kPa	2 kPa	9511	20836	-2.315	0.00
		2.5 kPa	8905	20230	-3.121	0.00
		3 kPa	8356	19681	-3.852	0.00
		3.2 kPa	8262	19587	-3.977	0.00
%R	2 kPa	2.5 kPa	8520	19845	3.634	0.00
		3 kPa	6840	18165	-5.870	0.00
		3.2 kPa	6110	17435	-6.842	0.00
J_{nr}	2 kPa	2.5 kPa	9632	20957	2.154	0.031
		3 kPa	8930	20255	-3.088	0.002

		3.2 kPa	8787	20112	-3.279	0.001
%R	2.5 kPa	3 kPa	9042	20367	-2.939	0.03
		3.2 kPa	8133	19458	-4.149	0.00
J_{nr}	2.5 kPa	3 kPa	9587	20912	-2.214	0.027
		3.2 kPa	9352	20677	2.526	0.012
%R	3 kPa	3.2 kPa	10031	21356	-1.623	0.105
J_{nr}	3 kPa	3.2 kPa	10129	21454	-1.492	0.136

5.3.3 Analysis of MSCR Test for 30 Cycles

5.3.3.1 Normality and homogeneity of variance Assumptions for %R and J_{nr} based on MCP proportions

In this case also, before conducting the ANOVA test, first step is to check the assumptions of two-way ANOVA. Table 5.11 (a-b) showed that assumptions are not following because p-value for test is less than 0.05 value so null hypothesis is rejected, it means statistically significantly different between the mean of groups i.e. MCP proportions effects the %R and J_{nr} values significantly. In this case perform the non-parametric test i.e. Kruskal-Wallis H Test.

Table 5.11 Assumptions for Two Way ANOVA Based on MCP Proportions for 30 Cycles

(a) Normality and (b) Homogeneity of Variance

Tests of Normality							
	Proportions	**Kolmogorov-Smirnov**			**Shapiro-Wilk**		
		Statistic	**df**	**Sig.**	**Statistic**	**df**	**Sig.**
%Recovery	C0%	.478	720	.000	.040	720	.000
	C1%	.222	719	.000	.739	719	.000
	C2%	.190	720	.000	.764	720	.000
	C3%	.193	660	.000	.756	660	.000
	C4%	.220	720	.000	.703	720	.000
J_{nr}	C0%	.151	720	.000	.578	720	.000
	C1%	.116	719	.000	.929	719	.000
	C2%	.089	720	.000	.928	720	.000
	C3%	.117	660	.000	.902	660	.000
	C4%	.117	720	.000	.904	720	.000

(a)

Test of Homogeneity of Variance (Levene's Test of Equality of Error Variances)					
		Levene Statistic	df1	df2	Sig.
%Recovery	Based on Mean	19.083	4	3534	.000
	Based on Median	13.826	4	3534	.000
	Based on Median and with adjusted df	13.826	4	1290.221	.000
	Based on trimmed mean	16.377	4	3534	.000
J_{nr}	Based on Mean	229.077	4	3534	.000
	Based on Median	227.373	4	3534	.000
	Based on Median and with adjusted df	227.373	4	761.211	.000
	Based on trimmed mean	228.704	4	3534	.000

(b)

5.3.3.2 Normality and homogeneity of variance Assumptions for %R and Jnr based on stress levels

Table 5.12 (a-b) showed that there is significance different between the mean of groups because of p- value (0.00) is less than 0.05 value. So null hypothesis is rejected and stress levels effects the %R and J_{nr} values significantly. The assumptions of normality and homogeneity of variance has been violated. Therefore perform the non-parametric tests i.e. Kruskal-Wallis H Test.

Table 5.12 Assumption for Two Way ANOVA Based Stress levels for 30 Cycles

(a) Normality and (b) Homogeneity of Variance

Tests of Normality							
	Stress_Levels	Kolmogorov-Smirnov			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
%Recovery	0.1 kPa	.127	450	.000	.937	450	.000
	0.5 kPa	.230	450	.000	.811	450	.000
	1 kPa	.236	450	.000	.820	450	.000
	1.5 kPa	.235	450	.000	.810	450	.000
	2 kPa	.274	450	.000	.777	450	.000
	2.5 kPa	.276	450	.000	.743	450	.000
	3 kPa	.323	420	.000	.734	420	.000
	3.2 kPa	.416	419	.000	.083	419	.000
J_{nr}	0.1 kPa	.444	450	.000	.549	450	.000
	0.5 kPa	.452	450	.000	.545	450	.000

	1 kPa	.449	450	.000	.548	450	.000
	1.5 kPa	.447	450	.000	.550	450	.000
	2 kPa	.447	450	.000	.550	450	.000
	2.5 kPa	.447	450	.000	.550	450	.000
	3 kPa	.443	420	.000	.563	420	.000
	3.2 kPa	.439	419	.000	.548	419	.000

(a)

Test of Homogeneity of Variance(Levene's Test of Equality of Error Variances)					
		Levene Statistic	**df1**	**df2**	**Sig.**
%Recovery	Based on Mean	19.250	7	3531	.000
	Based on Median	19.310	7	3531	.000
	Based on Median and with adjusted df	19.310	7	656.917	.000
	Based on trimmed mean	19.395	7	3531	.000
J_{nr}	Based on Mean	12.934	7	3531	.000
	Based on Median	2.506	7	3531	.014
	Based on Median and with adjusted df	2.506	7	3326.221	.014
	Based on trimmed mean	9.485	7	3531	.000

(b)

5.3.4 Kruskal-Wallis H Test's Results for 30 Cycles MSCR

5.3.4.1 Kruskal-Wallis Test Results of %R and J_{nr} Based on MCP Proportions

Table 5.13 (a-b) shows that the p-value of %R and J_{nr} is 0.00 which is less than 0.05. It means there are statistically significantly differences in the %R and J_{nr} groups due to proportions of MCP.

The post hoc test is conducted with the help of Mann-Whitney U test (two independent samples test) to find the MCP proportion's affects the %R and J_{nr}.

Table 5.13 Kruskal-Wallis Test Results of %R and Jnr Based on MCP Proportion for 30 Cycles

(a) Mean Rank (b) Test Statistics

Ranks			
	Proportions	**N**	**Mean Rank**
%Recovery	C0%	720	360.82
	C1%	719	1670.31
	C2%	720	2570.24
	C3%	660	2352.48
	C4%	720	1944.55
	Total	3539	
J_{nr}	C0%	720	3179.50
	C1%	719	2315.27
	C2%	720	597.48
	C3%	660	1017.22
	C4%	720	1678.55
	Total	3539	

(a)

Test Statistics		
	%Recovery	**J_{nr}**
Chi-Square	2053.493	2887.037
df	4	4
Asymp. Sig.	.000	.000

(b)

Table 5.14 showed the Mann-Whitney U (Post Hoc) Test for %R and J_{nr} based on proportions of MCP. It can be seen that in post hoc test compare the percentage of proportion of MCP to each other. In every comparison the p-value is less than 0.05. It means null hypothesis is rejected and there is statistically significantly different between the mean ranks i.e. every proportions of MCP effects the %R and J_{nr} values significantly.

Table 5.14 Mann-Whitney U (Post Hoc) Test Results of %R and Jnr Based on MCP Proportion for 30 Cycles

Test Statistics						
		Stress Levels	**Mann-Whitney U**	**Wilcoxon W**	**Z**	**Asymp. Sig. (2-tailed)**
%R	C0%	C1%	210	259770	-32.814	0.00
		C2%	0.00	259560	-32.852	0.00
		C3%	0.00	259560	-32.129	0.00
		C4%	20	259580	-32.849	0.00
J_{nr}	C0%	C1%	0.00	258840	-32.841	0.00
		C2%	0.00	259560	-32.852	0.00
		C3%	0.00	259560	-32.129	0.00
		C4%	0.00	259560	-32.852	0.0
%R	C1%	C2%	109611	368451	-18.934	0.00
		C3%	121579	380419	-15.661	0.00
		C4%	193452	452292	-8.296	0.00
J_{nr}	C1%	C2%	4673	264233	-32.248	0.00
		C3%	22611	240741	-29.058	0.00
		C4%	76774	336334	-23.100	0.00
%R	C2%	C3%	189473	407603	-6.508	0.00
		C4%	139582	399142	-15.161	0.00
J_{nr}	C2%	C3%	123720	383280	-15.399	0.00
		C4%	42233	301793	-27.499	0.00
%R	C3%	C4%	158327	417887	-10.720	0.00
J_{nr}	C3%	C4%	79142	297272	-21.427	0.00

5.3.4.2 Kruskal-Wallis Test Results of %R and J_{nr} Based on Stress Levels

Table 5.15 also showed that there are not statistically significantly differences in the %R and J_{nr} groups due to stress levels. The p-value of %R and J_{nr} is 0.00 which is less than 0.05.

Table 5.15 Kruskal-Wallis Test Results of %R and Jnr Based on Stress levels for 30 Cycles

(a) Mean Rank (b) Test Statistics

Ranks			
	Stress Levels	**N**	**Mean Rank**
%Recovery	0.1 kPa	450	2804.57
	0.5 kPa	450	2443.23
	1 kPa	450	2158.30
	1.5 kPa	450	1796.52
	2 kPa	450	1510.66
	2.5 kPa	450	1290.28
	3 kPa	420	1065.74
	3.2 kPa	419	990.03
	Total	3539	
J_{nr}	0.1 kPa	450	945.79
	0.5 kPa	450	1417.01
	1 kPa	450	1631.66
	1.5 kPa	450	1780.08
	2 kPa	450	1906.48
	2.5 kPa	450	2039.95
	3 kPa	420	2207.77
	3.2 kPa	419	2296.74
	Total	3539	

(a)

Test Statistics		
	%Recovery	**J_{nr}**
Chi-Square	1293.877	582.700
df	7	7
Asymp. Sig.	.000	.000

(b)

Table 5.16 showed the test statistics for Mann-Whitney U (Post Hoc) Test for %R and J_{nr} Based on stress levels. It can be seen that in post hoc test compare the stress levels to other each stress levels. In every comparison the p-value is less than 0.05. It means null hypothesis is rejected and there is statistically significantly different between the mean ranks i.e. every proportions of MCP effects the %R and J_{nr} values significantl, expect for %R 3 (kPa compare to 3.2 kPa) as mentioned in red box below the p-value

(0.07) is more than 0.05 that means it is non-significant. In this case the stress levels are affecting the %R and J_{nr} values and null hypothesis is accepted, it means there is no significant differences.

Table 5.16 Mann-Whitney U (Post Hoc) Test Results of %R and Jnr Based on Stress Levels for 30 Cycles

Test Statistics						
		Stress Levels	**Mann-Whitney U**	**Wilcoxon W**	**Z**	**Asymp. Sig. (2-tailed)**
%R	0.1 kPa	0.5 kPa	38479	139954	-16.098	0.00
		1 kPa	33748	135223	-17.311	0.00
		1.5 kPa	32543	134018	-17.620	0.00
		2 kPa	32430	133905	-17.649	0.00
		2.5 kPa	33151	134626	-17.465	0.00
		3 kPa	29611	118021	-17.520	0.00
		3.2 kPa	29506	117496	-17.518	0.00
J_{nr}	0.1 kPa	0.5 kPa	61089	162564	-10.3	0.00
		1 kPa	54345	155820	-12.029	0.00
		1.5 kPa	49795	151270	-13.196	0.00
		2 kPa	45456	146931	-14.309	0.00
		2.5 kPa	41614	143089	-15.294	0.00
		3 kPa	36481	137956	-15.665	0.00
		3.2 kPa	35350	136825	-15.937	0.00
%R	0.5 kPa	1 kPa	61708	163183	-10.141	0.00
		1.5 kPa	40133	141608	-15.674	0.00
		2 kPa	33353	134828	-17.413	0.00
		2.5 kPa	33546	135021	-17.363	0.00
		3 kPa	29700	118110	-17.495	0.00
		3.2 kPa	29610	117600	-17.490	0.00
J_{nr}	0.5 kPa	1 kPa	82037	183512	-4.927	0.04
		1.5 kPa	78217	179692	-5.907	0.00
		2 kPa	72528	174003	-7.366	0.00
		2.5 kPa	61101	162576	-10.297	0.00

		3 kPa	51127	152602	-11.710	0.00
		3.2 kPa	49760	151235	-12.040	0.00
%R	1 kPa	1.5 kPa	66004	167479	-9.039	0.00
		2 kPa	46319	147794	-14.087	0.00
		2.5 kPa	37653	139128	-16.310	0.00
		3 kPa	30635	119045	-17.243	0.00
		3.2 kPa	30136	118126	-17.348	0.00
J_{nr}	1 kPa	1.5 kPa	86806	188281	-3.704	0.007
		2 kPa	80132	181607	-5.416	0.00
		2.5 kPa	76395	177870	-6.374	0.00
		3 kPa	63527	165002	-8.362	0.00
		3.2 kPa	57292	158767	-10.003	0.00
%R	1.5 kPa	2 kPa	73586	175061	-7.095	0.00
		2.5 kPa	57406	158881	-11.244	0.00
		3 kPa	43173	131583	-13.858	0.00
		3.2 kPa	40108	128098	-14.651	0.00
J_{nr}	1.5 kPa	2 kPa	88739	190214	-3.209	0.01
		2.5 kPa	81888	183363	-4.966	0.00
		3 kPa	69640	171115	-6.712	0.00
		3.2 kPa	66610	168085	-7.483	0.00
%R	2 kPa	2.5 kPa	78342	179817	-5.875	0.00
		3 kPa	56534	144944	-10.251	0.00
		3.2 kPa	52542	140532	-11.287	0.00
J_{nr}	2 kPa	2.5 kPa	88379	189854	-3.301	0.01
		3 kPa	73148	174623	-5.765	0.00
		3.2 kPa	71768	173243	-6.087	0.00
%R	2.5 kPa	3 kPa	72075	160485	-6.055	0.03
		3.2 kPa	66424	154414	-7.533	0.00
J_{nr}	2.5 kPa	3 kPa	77718	179193	-4.531	0.00
		3.2 kPa	75662	177137	-5.034	0.00
%R	3 kPa	3.2 kPa	78506	166496	-2.702	0.07
J_{nr}	3 kPa	3.2 kPa	76495	164905	-3.275	0.01

CHAPTER-6

CONCLUSIONS

In continuing efforts to enhance the physical and rheological properties of control binder, different types of modifiers were used to increase the performance, to see the behaviour of modified binders and service life of flexible pavements. In this research investigated the influence of coconut shell microcharcoal powder (MCP) on properties of the neat bitumen. To evaluate the rheological measurements of control and bio-modified binders, the dynamic shear rheometer (DSR) was widely used. It helps to measure the viscoelastic response of control and modified bitumen at various loading rates and temperature due to complex nature of bitumen. Many types of tests were conducted with the help of DSR in this study and some of the major findings are listed below:

- Addition of bio-waste has significantly decreased the penetration value of modified binder as compare of control binder. The reduction in penetration values are 19.07%, 24.52%, 17.17%, and 14.99% for C1%, C2%, C3% and C4% respectively. The maximum reduction was 24.52% for C2% addition of MCP. Low penetration value indicates the modified binder is stiff and low susceptibility at high temperature.
- The values of softening point increased for bio-modified binders. The softening point of control binder was 47.5^{o}C whereas the softening point temperature at C2% addition was 58^{o}C indicating a 10^{o}C increase in softening point. After 2%, any further addition did not show significant increase in the softening point temperature. The increase in softening point value shows us that binder can resist some level of high temperature and deformation.
- PI value of control binder is less than -1 indicating that it is highly susceptibility to low temperature. However for modified binders, the value of PI is approaching to +1 and more than control binder indicating modified binder has low susceptibility to high temperature and resist the rutting and cracking in permanent deformation.
- Storage stability test showed that the differences in the upper and lower portion of the specimen are within the prescribed limit i.e. less than 2.2°C. It means added MCP is well homogenously dispersed in bitumen at high temperature.

- The DSR results indicated that the complex shear modulus (G*) at 65°C for control binder is very less even i.e. 0.9118 kPa. It means control binder is highly viscous and couldn't resist the cracking and permanent deformation under loading process. With the increase in content of MCP, G* value is also increases. Addition of C2% addition, value of G* is 6.17 kPa and the percentage increase in modified binder is 577.33%.
- The phase shift angle for control binder was more and indicating highly viscous means less stiffness property but with the addition of MCP, phase shift angle starts decreasing. However the minimum time lag is obtained at C2% of MCP and the percentage decrease in phase angle is 6.58% which helps to retard rutting parameter and shows improved elastic recovery performance and after excessive addition of MCP the phase angle starts increasing again.
- The experimental results showed that the damping factor for control binder was higher. But with the addition of MCP, the loss factor is less and lower loss factor can easily correlate the binder with more rutting resistance performance and improve the elastic properties.
- Moreover frequency and temperature also affects the stiffness and viscoelastic behaviour of binder. It was observed that as the increase in frequency the asphalt binder showed elastic properties because of shorter duration of vehicle loading and the value of rutting parameter and complex modulus also increases which means better will be the performance of binder and vice-versa. The influence of temperature was also significant, bitumen is very sensitive to temperature, so as the temperature increased the performance of rutting, complex shear modulus were decreased and phase shift angle was increased. But for every case C2% of MCP modified binder gives best results.
- The integrated modified binders decreased the accumulated creep strain which helps to increase the rutting resistance by increasing the elastic and stiffness behavior. The accumulated creep strain for 10 cycles was more as compared to 30 cycles creep strains.
- The MSCR parameters such as %R and J_{nr} were found to be more sensitive to MCP dosages and to stress also for control and modified binders. Control binder didn't show any %R but addition of MCP modified binder has given 14.40% recovery for 10 cycles and for 30cycles %R was 14.11% at 0.1 kPa

load stress for C2% addition of MCP as compared to other modified binders and as the stress increased the %R also reduced. Minimum J_{nr} value was observed at highest %R.

- Viscosity determines the resistance of bitumen. Results showed that viscosity was decreased as the increase in shear rate. In this research, it determined that for control binder shear rate will be 3 (1/s) and for modified binder shear rate will be 15 (1/s) at C2% MCP for absolute viscosity.
- Based on the SEM images, results showed that the particle size of MCP was less than 75 µm. The shape of the MCP was angular, sharp edges, rough surface and has voids on the surface. At addition of C2% MCP was fully homogenously dispersed into binder and made the binder rutt resistance which helps to improve the performance of pavements. Agglomeration can be seen expect C2% MCP addition which reduce the performance.
- Statistical analysis has been conducted to see the repeatability between the trains and compared the means of the trails groups with the help of SPSS software. It was concluded that there was significantly differences between the each trials and moreover dosages of MCP and creep stresses have significantly effects on the %R and J_{nr} values.
- In other words there is no doubt to use the environmental waste as modification of conventional binders because it increases the physical and rheological properties significantly.

FUTURE SCOPE OF RESEARCH

- The current study mainly focused on the unaged binder properties. However, during construction of pavement, the bituminous mixture undergoes short term and long term ageing. The effect of short term ageing and long term ageing on the binder needs to be investigated.
- Modified binder can also use in the asphalt mixture to evaluate various tests and performance of modified asphalt mixture.

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